ARCHAEOLOGY AND THE ALCHEMICAL LABORATORY.

Exploring early modern chymical practices at colonial Jamestown (Virginia) and the Old Ashmolean Museum (Oxford)

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I, Umberto Veronesi, confirm that the work presented in this thesis is my own. Where information has been derived from other sources, I confirm that this has been indicated in the thesis.

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ABSTRACT

The present PhD thesis focuses on the study of early modern laboratory apparatus, with the specific aim of adopting a material culture-approach to the history of science and technology. This is achieved through the scientific analysis of two assemblages of crucibles and other reaction vessels, namely that of Jamestown in Virginia (early 17th century) and that of the Ashmolean laboratory in Oxford (late 17th-early 18th century). For each of these case studies the high-temperature activities carried out were reconstructed and contextualised. The analysis of the residues left by the chemical reactions, through optical microscopy and SEM-EDS, allowed to determine what substances were manipulated and what technical processes were followed. While the vast majority of the crucibles from Jamestown were used for testing minerals in search of metals of interest to the settlers, the practitioners at the Ashmolean were found to diversify their work and experiment with technological innovation of the period. As no direct relation exists between the case studies, each of them stands on its own and each brings a novel contribution to its specific historical and archaeological context However, taken together the two case studies illustrate the wider scope of this thesis by indicating the potential of a new methodological approach to the study of laboratory remains, which combines the information of archaeological science with current narratives in the historiography of early modern science. The results are used to build a materials-based network, which tells the story of scientific developments from the bottom up and throws new light on the practical side of doing science. Ultimately, this thesis crosses old disciplinary boundaries and adds new layers of interpretation to both disciplines it engages with.

IMPACT STATEMENT

This thesis started by addressing the very basic question of what it means to do alchemy in early modern Europe, what alchemists did in their laboratories, why they did it and in fact even who alchemists were. Similar research questions lie at the core of an ever-increasing number of works in the history of science, but while historians explore them through the scrutiny of written sources, this thesis proposes a material vantage point to enter the same arena of discussion. Focusing on the remains of two high-profile early modern contexts, namely the metallurgical workshop in colonial Jamestown (Virginia) and Oxford's Ashmolean laboratory, and using scientific analyses of artefacts as its starting point, this thesis threw light on the experiments carried out at the two sites, of which little is known from contemporary documents. It shows how material culture offers an invaluable focus to discuss some of the most influential theories running at the forefront of current historiography and regarding the many overlaps and epistemic exchanges between artisanal and scientific knowledge.

This study and its findings have strong implications for future academic research, both in the field of archaeological science and in Science and Technology Studies (STS). By incorporating research questions and narratives from current historiography, it significantly broadens the scope of at least a portion of archaeological science studies, adding new layers to the interpretation of archaeological remains that relate to past technologies, such as metallurgy and glass making. In this sense, this thesis created a new space in which archaeological scientists can move and that they can further expand. It also brings a relevant contribution to the history of early modern science and technology. Here, scholars are increasingly interested in engaging with the material and the practical side of alchemy, but often fail to recognise the full information potential that artefacts can bring. In this sense, this thesis brought together two disciplines whose significant potential for cross-fertilisation had only been minimally exploited in the past and sketched the contours of such collaboration in the future.

Outside of academia, this thesis represents a significant contribution to the ongoing efforts done by science historians to revise the long-lasting, pejorative view of alchemy that has been dominating both among scholars and the general public. By showing how alchemy involved activities such as testing the yield of potential mines or experimenting with recipes to manufacture coloured glasses, the present work offers a material counterpart to the existing studies that reject the equation alchemy equals magic and irrational behaviour, and provides a new point of view on the complexity and multifaceted nature of this early modern phenomenon.

Finally, the most insightful results achieved in this thesis have already been disseminated in scientific journals and magazines, academic blog posts and at conferences and workshops pertaining to both disciplinary spheres at play, confirming that the methodology brought forward is indeed of interest to a wide variety of specialist.

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It is somewhat strange to find myself here, seven and a half years after first moving to London to join UCL, writing the very last words of a PhD thesis which represents such a big ending. It will mark the end of this doctorate, of course, and that is good because it surely has been an intense, rewarding and extremely challenging process. It will also mean that I will not, after submitting the corrected, final version, be a student any longer, and a student is what I have been all my life. Finally, it will be the end – at least for now - of my own personal story with UCL, a very special place, a place where I have learned to become an academic while London was taking care of shaping me into the person I am today. So, I will begin by thanking London, the city that more than any other has inspired me, the city that has become home, the place I have rejected at times but could not help but love, the place I have struggled to recognise in the last year, when every street I walked down seemed to talk about the past and the present felt suspended.

As I was getting ready to embark on this PhD project, around five years ago, the pleasant feeling of knowing that my life was taking a somewhat defined course, I could only imagine how many things would have changed, rearranged, disappeared, reappeared and simply just happened in the course of this time. And indeed, nearly no one is here that I met when I first arrived. To many of these events I owe something, more or less important inputs that helped with the completion of this piece of research. A lot are trivial and rather anonymous, such as buying the laptop on which I am writing now, on which I have written the whole of the thesis, this laptop I find weirdly pleasing to type on. There are, however, several people and a few institutions to which I owe much more than the silly pleasure of a typing experience, and to these I shall now turn.

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whom goes my gratitude. Dr Stephen Johnston was my very first contact in Oxford, he showed me around the place where the Ashmolean laboratory once stood, what today is the History of Science Museum, of which he is the Head of Research, Teaching and Collections, and he was of great help in my initial steps towards obtaining samples. Sampling proper took place in the conservation department of the Ashmolean Museum and I would like to thank Daniel Bone for his supervision during my visits in February 2017. I am also indebted to Dr Kelly Domoney for organising those visits, helping me sort through the boxes and for her availability and kindness. Finally, Eleanor Standley, Mark Norman and Helen Hovey also played a part in the events that allowed me to obtain the samples and to them I am grateful. The materials from Jamestown had already been sampled by Marcos Martinón-Torres a few years back, but I would like to renew my gratitude to Dr Beverly Straube for initial access and for always being happy to offer her vast knowledge on the subject.

As a PhD student, I have had the opportunity to travel nationally and internationally and to present my work at various conferences, symposia and workshops, engaging both with fellow archaeologists as well as science and art historians. Besides my scholarship and awards from UCL's Institute of Archaeology, part of it has been possible thanks to various institutions and schemes to which goes my deepest gratitude. These include the Society for the History of Alchemy and Chemistry (SHAC); the International Fund (IF) scheme and Princeton Center for Collaborative History; the David A. Gardner '69 Magic Project in the Humanities Council, and the University of Graz.

As a researcher more generally, I owe a great deal to my supervisor, Marcos Martinón-Torres. Besides embodying my UCL imprinting, Marcos is the reason for my interest in the topic of alchemy and for my active involvement in it. He first got me into this project and patiently guided me throughout the process of re-elaborating it my way. As I did my research, I often found myself a little (or a lot) lost, like a mechanical tin toy out of charge and, meeting after meeting, Marcos would put my clockwork back in motion, spreading his enthusiasm and his many ideas.

To my secondary supervisor, Thilo Rehren, I also owe a lot. His deep knowledge of so many aspects of archaeological science has been a vital compass to navigate what turned out to be a kaleidoscope of different technological stories in just a handful of samples. Moreover, Thilo has been an all-rounder mentor, offering me important lessons on such topics as academic writing and how to move across disciplines. It has been a privilege to have him and Marcos as my supervisory team.

Part of what I have become in these past years at UCL is certainly due to professor Ian Freestone, whose office door can always be found ajar. Ian will welcome confused students (including, many times, myself) with a smile and will offer some of his knowledge, experience and a book or two from his vast library on glass, and I would like to thank him for that.

When I decided to engage with the history of science, a discipline that was not my own, I knew I would need a guide for the journey and I found an excellent one in professor Simon Werrett. Simon allowed me to sit in his course and, together with the UCL STS reading group, helped me see the scope of my work as something of interest to historians and archaeologists alike.

A good share of my analytical work would not have been possible without the support and supervision of Tom Gregory, to whom I turned every time the SEM would not behave properly. And because the truth is that it was I who most likely was not behaving properly, blessed be Tom’s patience to get me back on my feet.

Finally, I would like to thank Lisa Daniel, my very first contact at UCL, the person I sent a hesitant email to the day I decided to enrol in the master, an effort that required me to go back to my high school English notes on “how to write a formal letter”. Throughout the years, Lisa has been a reassuring certainty at the IoA, she provided invaluable help with all things administrative as well as a huge amount of early morning kindness.

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As an expat, I have found a piece of home in my fellow nationals, which the IoA never spared. With them, I had the opportunity to articulate in my own language what was in my mind, the new and often complex concepts I was learning and the challenges posed by living in a different country. So, thank you Agnese, your contagious passion and immense heart have been a constant guiding light to me, you showed me what a good teacher should be like, and brought me delicious fried food when I was in troubled waters. I have no doubt you will make a great mother (ciao Amelia!). And thank you Veronica, you have been such an invaluable companion during these past two and a half years, you helped me find my way back every time my mind drifted away and my heart was a little lost. You started your own journey a bit later, but you have always known it all, and from you I learned a lot.

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Like I said, seven and a half years is quite a long time, things get different, lives have to rearrange, sometimes in ways we wouldn't want but need. So, to conclude this list, I would like to thank Tyler. Your big smile that afternoon several Aprils ago, when I first learned about the scholarship, was enough for me to know that I would have got here in the end.

To my uncle Giò

and to change (*il Cambiamento*)

CHAPTER I

INTRODUCTION

Figure 1.1 The Laboratory for the Research on the Structure of Matter at the University of Pennsylvania. According to its mission statement the laboratory "pursues a multidisciplinary approach to solve fundamental materials problems that are likely to underlie future technologies, and thereby, substantially impact the research and educational needs of society".

1.1 THE BACKGROUND: ALCHEMY AND THE HISTORY OF SCIENCE

To virtually any modern-day reader, the term alchemy evokes a very specific host of images and ideas. We tend to associate alchemy to magic and the esoteric rather than to science and rational thinking, and visualise the alchemist as a shadowy old man, quite possibly bearded, quite possibly half-mad, who stirs the content of a crucible surrounded by the smokes and the mess of his laboratory. We can imagine them in pursuit of metallic transmutation or, maybe, the universal medicine that will cure any disease. The language of alchemy is not that of numbers and precise descriptions but rather one of allegorical images, of colourful manuscript pages populated by odd-looking creatures as well as of secretive and seemingly unintelligible symbols. Alchemy is also sometimes perceived as a religious activity, a sort of sacred discipline, whose purpose is spiritual rather than material, in which the adept seeks self-purification and contact with God more than the production of physical effects on whatever it is that he/she is working on. As a consequence, the historiography of early modern science has traditionally accorded little credit to alchemy in the process that, by the end of the 17th century, led to the emergence of values that we relate to the modern scientific method, such as empiricism, collaboration, publication, and the importance of precise quantification and reproducible parameters (**Butterfield 1957; Rossi 1968; 2009**). The narrative of a scientific revolution on which historians have focused for a long time conveys the idea of a fracture between a somewhat obscure and alchemical *before* and a luminous, modern, scientific *after*. Equally, the relationship between alchemy and chemistry has been addressed in terms of opposition, in which the former is a proto or pseudo-science and the latter is the respectable offspring struggling its way out of the murky waters of a magical heritage.

More recent scholarship, however, has come to challenge most of these views. A closer reading of alchemical texts, free from this pejorative bias, has revealed that rather than a monolithic entity alchemy was instead a vital, diverse and multifaceted phenomenon where a varied range of practitioners, aims and ideas coexisted, often in open contrast with one another (**Moran 2005; 2011; Nummedal 2007**). Most historians now reject the grand narrative of a scientific revolution perceived as a complete break with the past, preferring instead to explore lines of continuity with the previous tradition. The very same forefathers of the scientific revolution, figures such as Isaac Newton (1642-1727) and Robert Boyle (1627-1691), were found to be firm believers in alchemical concepts such as metallic transmutation, that in fact these practices occupied a significant part of their time and influenced their discoveries (**Principe 1998; Newman 2010; 2016; 2018**). The perception of a divide between alchemy and chemistry is the product of a persistent presentist bias, one of looking at early modern science through the distorting lenses of a modern-day standpoint (**Principe 2011a**). More contextualised studies of alchemy indicate that no such distinction really existed, at least not in the early modern world, and that alchemy and chemistry were in fact synonyms (**Newman and Principe 1998**). They were employed interchangeably to mean the combination of philosophical theories around natural phenomena and a set of practices involving the manipulation and transformation of natural substances. These were varied and included chemical technologies and various types of crafts. For this reason, the role of artisans and the mechanical arts in the creation and modification of natural knowledge is one major theme addressed by current scholarship, as is that of the many overlaps and epistemic exchanges between artisans and philosophers (**Smith 2004; Dupré 2014**).

In sum, now more than ever before historians are interested in the practical dimension of chymistry, in the experiments carried out in those messy laboratories by a wide array of individuals and for a multitude of reasons (**Roberts and Werrett 2017**). Alongside more theoretical speculations, such active work of manipulation is recognised as the driver of the new experimental sciences of the late 17th century. An immediate consequence of this trend is the potential for contribution from other disciplines, among them the role of archaeology and material culture studies that the present work discusses and argues for. Artefacts have entered the spectrum of sources for the study of science history, alongside vernacular collections of recipes, laboratory notebooks and the re-enactment of alchemical procedures. All the while, it is important to keep in mind that the new and revised understanding of alchemy does not deny that this was often also a deeply religious activity, a way of worshipping and investigating God through his creation, and that secrecy was often advocated by practitioners as a necessary part of their discipline. Equally, metallic transmutation did represent one of the largest branches of alchemical activity, one which led to a number of critical observations on the structure of matter and the nature and behaviour of metals. As visualised in figure 1.1, research around matter, the investigation of what it is and how it changes is still, many centuries later, an active pursuit. This thesis aligns with such current understanding of the alchemy phenomenon in early modern western culture and chapter 2 will delve more deeply into the aspects that most characterise it.

1.2 THE CASE STUDIES

The present work enters this arena from the point of view of materials and recipes, through the analysis of two archaeological assemblages of laboratory equipment. By and large, the artefacts comprise fragments of ceramic crucibles varying in size and shape and employed in high-temperature operations of various nature (cfr chapter 4 and 5). Other types of laboratory tools, also made of ceramic, include flasks, bottles and distillation apparatus. The chemical analysis of the tools themselves as well as of the residues found in them provides a way to reconstruct the details of the experimental processes undergone, of the substances that entered the vessels and the outcomes of the work. In doing so, this thesis offers relevant data on the practical dimension of 17th-century experimental laboratory practice as it happened at two high-profile sites which have attracted the attention of scholars and whose history is therefore quite well known. The first case study concerns the colonial site of Jamestown, Virginia, the first permanent British settlement in North America, founded at the very beginning of the 17th century. The assemblage under investigation here comes from the early fort phases (1607-1610), and links to the metallurgical activities that began soon after the arrival at the New World, with the aim of testing the quality of local minerals. The present work is the most extensive round of analyses performed on the assemblage to date and helps broaden the picture relative to early industrial efforts in the colonial world. The second case study is the basement laboratory of the first Ashmolean Museum in Oxford, opened in the early 1680s and home to the first chair of chemistry at an English university. The apparatus recovered there cannot be dated with precision, but it seems to be at least in part reflecting the experimental programme from the initial decades of the Ashmolean's life (cfr section 5.6.1).

Both case studies hold a great information potential and, as discussed in the next section, they also mirror the two levels on which this thesis operates. On the one hand there is the reconstruction of the single material microhistories, which are initially explored in isolation from one another and that sensibly enrich our understanding of the history and archaeology of two contexts of high profile in historical archaeology. On the other hand, this thesis goes beyond the individual contributions of Jamestown and the Ashmolean laboratory in order to explore broader narratives in the historiography of early modern science and technology. It suggests a methodological approach to the study of early modern alchemical practice through the lenses of its material culture. This last point, the desire to develop a cross-disciplinary methodological framework, is the reason for employing two seemingly unrelated case studies, whose distance is both chronological as well as geographical. Nonetheless, both are historically well-known contexts, whose relevance in early modern times is instrumental to the fundamentally methodological aim at the core of the present project. Moreover, the lack of an immediate connection also helps reinforcing an understanding of alchemy as a multifaceted phenomenon, a broad spectrum made of gradients and where different souls coexisted.

1.3 THE AIMS AND RESEARCH QUESTIONS

As a consequence of the two levels on which this thesis moves, the research questions can also be divided in two broader spheres. The first includes questions specific to the case studies and about the specific contribution that this project makes to our knowledge of the sites and their single microhistories. General questions include:

- What is the range of high-temperature operations carried out at the two sites analysed?
- What tools were employed? Where did they come from?
- What substances, ingredients and reagents, were being manipulated by the laboratory practitioners? Which recipes were followed?
- How does the information thus obtained relate to the information we have from texts and documents?

There are also further and more specific questions that apply to each of the two case studies and that will be exposed and addressed in the relevant sections of the thesis (cfr sections 4.3 and 5.3).

Second, the data obtained on the two separate case studies provides a material point of view on some current research trajectories in the history of early modern science. As historians increasingly widen the scope of what alchemy is in early modern Europe, this thesis equally argues for the varied and multifaceted character of this phenomenon. To investigate such themes, the following questions will be addressed:

- From the point of view of practice, what does it mean to do alchemy? What did alchemists do and who were they?
- What do materials tell us about the many activities, actors and aims driving early modern alchemical practice? Can we see a clear demarcation between the more “philosophical” operations as opposed to more “productive” ones? Did the two levels overlap and interact with one another? And, if yes, in which ways?
- Following from the previous, how does material culture of early modern laboratories reflect shifting values in craft knowledge in this period? Can we see epistemic exchanges between the world of scholars and the world of artisans?

- How does a material approach to the history of early modern science contribute to challenging anachronistic ideas about alchemy and presentists views of its relationship to chemistry and science more generally? In other words, how does material information fit into, or affects and challenges, current historiographic narratives on alchemy?

Finally, this thesis represents an attempt at integrating an archaeological/ scientific approach into the historiography of science, two academic areas that have largely ignored each other despite having a lot in common at their core. This, it is felt, is the most important justification of the present project, which argues that by letting archaeological and historical perspectives interact, chances for new insight will surface that may otherwise be unseen. Thus, the ultimate objectives of the present work are the following:

- To call for a methodological approach to the history of early modern science and technology that takes into account the material culture of laboratories and its scientific analysis alongside more common written and iconographic sources of information.
- Relatedly, to argue that a history of science framework can bring new perspectives to some branches of archaeological science, adding a new layer of interpretation to the existing ones, thereby suggesting new possible avenues for the future of the discipline.

1.4 THE STRUCTURE OF THIS DISSERTATION

This initial chapter introduced the most important themes that lie at the core of this PhD project as well as the case studies under investigation, it outlined the research questions and the reasons why it makes a novel contribution to both archaeological science and the history of science. Chapter 2 expands on the issues presented very briefly in section 1.1, and draws the theoretical framework within which this thesis moves. Importantly, such framework also involves a terminological revision by which the word *alchemy* is substituted with *chymistry* as a way to avoid traditional views of the phenomenon (cfr section 2.1). The chapter discusses the early efforts at crossing disciplinary boundaries and shows the many chances for crossing them again (cfr section 2.3). A description of the kind of materials found in archaeological contexts of relevance follows in chapter 3, together with the details of analytical procedures used to study the assemblages under investigation. Next, chapters 4 and 5 present the two case studies of Jamestown and the Ashmolean laboratory in detail. Both chapters begin with an introduction to the archaeological and historical context of the sites (cfr sections 4.1, 4.2, 5.1 and 5.2), followed by the formulation of site-specific research questions (cfr sections 4.3 and 5.3). Successive sections are devoted to the description of the assemblages analysed (cfr sections 4.4 and 5.4), the results of the analyses and their technical interpretation (cfr sections 4.5 and 5.5), and finally to the discussion of the data within their relevant context (cfr sections 4.6 and 5.6). Starting from material observations that emerge with the case studies, chapter 6 discusses major themes in the history of science and technology (cfr sections 6.1) and proposes a different way of classifying laboratories based on materials and reactions rather than on scientific-artisanal divisions (cfr section 6.2). The chapter also provides future scholars of both sides with possible directions on how to apply the methodological approach discussed and what benefits can be gained (cfr section 6.3). New research questions arising during this work, pointers for future projects and concluding remarks are presented in the final chapter 7.

CHAPTER II

HISTORIOGRAPHY OF ALCHEMY AND CHEMISTRY

The present chapter explores the most relevant theoretical aspects that lie at the core of this PhD project, and prepares the ground for the archaeological approach to the historiography of early modern science presented and discussed in the subsequent chapters. Some of these aspects were introduced in passing in section 1.1, and will find here a more detailed examination. Most of the following theoretical approaches were defined and developed by science historians in the last three decades, oftentimes a re-elaboration of earlier narratives. A notable example is represented by the so-called scholar and craftsman thesis, discussed at length in section 2.2. First developed in the first half of the 20th century, the thesis has been surfacing again in the following decades as historians went back to its core ideas and placed them at the very centre of their agenda. Before exploring these ideas, however, what is important to stress here is that the many lines of enquiry that current scholarship has been following all share the common recognition that the history of science is not just a history of ideas, but also and crucially "a history of the making and using of objects to understand the world" (**Cook et al 2014: 12**). Science was as much a practical endeavour as it was a theoretical one, and historians have paid an ever-increasing attention to the material dimension of knowledge (**Werrett 2014; 2019; Valleriani 2017**). Freed from the almost sacred aura of timelessness that appeared to cover it, science is recognised as "a very human affair, filled with all the complexities and simplicities, errors and insights, pettiness and nobility that customarily attend human activity. And, to be sure, alchemy forms an important part of that story." (**Principe 2011a: 312**). The new material sensitivity which in different ways affects historical research is the reason why, now more than ever before, there is scope for fruitful cooperation between historians of early modern science and specialists of material culture. As discussed in detail in section 2.3, interdisciplinary collaboration and cross-fertilisation are increasingly acknowledged as a major desire for the future of science history (**Martinón-Torres 2011; Nummedal 2011; Rampling 2017; Roberts and Werrett 2017**), and the contribution of figures such as conservators and trained artisans to research projects is becoming more and more common. In this respect, as a discipline looking at past societies through their material culture archaeology can have an important role in the process of redefinition taking place within the historiography of early modern science. The present chapter represents an attempt at entering this very arena. Delving into the most relevant points of contact between archaeology and current historical research, and stressing the potential for more dialogue across the disciplinary divide, it will outline the theoretical framework within which this thesis is going to move.

2.1 ALCHEMY AND CHEMISTRY

One of the most influential outcomes of recent historiography is the radical redefinition of the very understanding of alchemy, and the subsequent reassessment of alchemy's place within the history of early modern science. In the last three decades, some historians have started to oppose the traditional view of alchemy maintained since at least the 19th century.[1] Common traits to the various expressions of such canonical view were the belief that alchemy is a fundamentally ritual discipline, that its motivations are mostly spiritual and symbolic, and that the strongest evidence of that is the secretive language and allegorical images that populate alchemical texts. As a consequence, traditional interpretations have passed down the image of alchemy as a static and monolithic endeavour, myopically obsessed with metallic transmutation and self-purification of the adept (**Boas 1958; Hall 1962; Jung 1968**; **Eliade 1962; Dobbs 1975; 1990**). Moreover, since it belonged to the sphere of religion, of magic and of esoterism, alchemy was thought to have no significance outside of the supramundane realm. Any evidence of actual laboratory practice carried out by alchemists, of their work with real substances was understood as largely symbolic as opposed to the implementation of chemical reactions in order to produce visible effects. Therefore, alchemy was intended as an irrational endeavour devoid of any "scientific" significance. Indeed, when dealing with the relationship between alchemy and the scientific revolution, and more specifically between alchemy and chemistry, the general tendency was to see the former as a developmental and primitive phase of the

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[1] For a summary see Principe and Newman 2001 and Eddy et al 2014

latter. Many historians saw alchemy as still imbued with the obscurity of the dark Middle Ages, and for this reason it was necessarily and radically different from the more respectable chemistry (**Rossi 1968; 2001**). In more recent times, this markedly pejorative view of alchemy has undergone a dramatic change.

Through a more careful scrutiny of historical sources science historians have demonstrated how alchemy was in fact as much a practical endeavour as it was a philosophical one, how alchemical theories were firmly grounded in the observations made during chemical reactions and how alchemists were interested in those reactions and in the production of tangible effects (**Principe and Newman 2001; Principe 2011a**). Together with the realisation that alchemy's practical dimension could not be ignored came the increasing awareness of the width and openness of alchemy's scope. Far from being a static monolith, the alchemical discipline was an incredibly diverse phenomenon, and gold making was only one of the many possible ways to apply the vast body of theoretical and technical knowledge that made up alchemy (**Moran 2005; Nummedal 2007; Beretta 2009; Martelli 2013**). Equally diverse was the profile of individuals who engaged in alchemical work, which spanned from workshop-trained artisanal experts to university-educated natural philosophers, humanists and through to courtly princes. Such diversity of practices and actors inevitably carried with it a host of motivations and aims that fuelled early modern alchemy, some of which we may describe as more philosophical and inquisitive, others more profit-driven, others again more spiritual (**Smith 1994; Principe 1998; 2000; Dupré 2014; Eddy et al 2014; Pastorino 2017**). This dynamism and multifaceted character are central points to the new understanding of alchemy taken forward by recent historians of early modern science. Alchemical activity overlapped with other practical disciplines, sharing with them the underlying theoretical principles as well as the operational knowledge, the equipment employed and the spaces occupied. We can see this continuous exchange in the relationship between alchemy and metallurgy, for instance, where knowledge on metallic generation and transformation maintained by metallurgists was rooted in old philosophical theories developed by alchemists in antiquity and in the Middle Ages (**Haung 2014; Newman 2011; Norris 2009; Principe 2014**). At the same time, a large number of alchemical practitioners engaged in the very same technical operations that metallurgists carried out in their workshops and used the same specialised equipment (**Martinón-Torres and Rehren 2005a**). Marco Beretta has charted a history of alchemy from the perspective of glass making, showing how intimately the development of alchemical theories was intertwined with current technological innovation in the glass industry (**Beretta 2009**). The flow from alchemy to chemical technologies and back, the blurred and fluid boundaries between alchemy and crafts reaffirmed and reinforced the twofold nature of alchemy as a fundamentally theoretical as well as practical endeavour (**Beretta 2004; Moran 2005; Smith 2007; Klein and Spary 2010; Newman 2011; Dupré 2014; Eddy et al 2014; Taape 2014; Beretta 2017; Valleriani 2017**). Where does alchemy end and metallurgy, glass making or distillation, begin? As these studies suggest, rather than looking for a place to draw the line between alchemy and chemical technology, it is more useful - and more historically correct - to explore how knowledge flowed across areas specialisation whose boundaries are characterised by a degree of fluidity in no way comparable to modern-day disciplinary segregation.

In regards of the question "what is alchemy?", a very effective and rather convincing definition is provided by the historian of science William Newman who writes that alchemy is "above all an attempt to understand and manipulate matter" (**Newman 2000: 49**). The historian Tara Nummedal suggests we talk about early modern *alchemies* rather than alchemy and writes that "what practitioners understood to be 'alchemy' included a variety of practices, skills and knowledge bases", and therefore "the only appropriate definition of alchemy is one that encompasses all of these activities and ideas" (**Nummedal 2007: 14**). What both the historians cited ultimately imply is that alchemy can be better understood if we think of it as a multitude of ways of approaching and relating to nature more than as a discipline itself. Only this way can we grasp more effectively the extraordinary breadth of this early modern phenomenon, whose overarching nature makes it "a nexus for intersecting fields of knowledge and techniques of inquiry" (**Moran 2011: 300**). Alchemy was simultaneously an explanatory framework for the natural phenomena and a powerful set of technical information to enquire into nature and to produce things (**Beretta 2004; Principe 2013**).

A journey through alchemy's long and complex history is beyond the scope of this thesis, which has the 17th century as its main focus, and the reader is redirected to the many excellent studies that will be referenced in the following lines. However, it is important to remark that the most fundamental characteristic of alchemy was always the interplay of theory and practice, that such twofold character first emerged in 1st-century AD Graeco-Roman Egypt, out of a substrate composed of Greek philosophical speculation on nature and the sacred technical knowledge of Egyptian priests (**Patai 1982; Beretta 2004; 2009; Martelli 2011; 2014**). The same is true for the Islamic world, where alchemy was re-shaped, incorporating new theories and technical operations, before it made its return to the Latin West again in the later Medieval period (for a history of alchemy see **Principe 2013**). In its long history, never did alchemy cease to be an interaction of philosophical theories and practical knowledge, with the consequence that by looking at it as a solely ritual practice our chances to catch the dynamic reality of alchemy are inevitably low and our understanding of this phenomenon restricted to only one of the innumerable facets that composed it.

Another cornerstone that has traditionally shaped the understanding of alchemy is the perceived opposition with chemistry as rational, respectable and fundamentally modern. This evolutionary narrative often acknowledges that alchemy did contain some seeds of modernity, but it nonetheless presents it as a benchmark of medieval darkness against which chemistry brought scientific light. It is common in such narrative to find claims to the mechanical (and almost secular) character of chemistry, in opposition to which alchemy is often described as an organic and vitalistic phenomenon (**Rossi 2001: 225-228; 2006;** for a discussion on this see **Principe and Newman 2001**). This religious vs secular struggle and the need to sanitising science can be found in the work of several influential scholars of the 20th century. In his *The Forge and the Crucible*, the historian of religion Mircea Eliade draws a parallel between the relationship alchemy-chemistry and the origin of classical theatre in ancient Greece from the sphere of the sacred. "The ritual spectacle", Eliade says, "belonged to the sphere of holy things; it unleashed religious experiences" (**Eliade 1962: 10**). Out of this ritual spectacle originates what he calls the secular drama, which instead "gave rise to experiences of quite a different order; it inspired aesthetic emotions and pursued an ideal of formal perfection quite alien to the values of religious experience” (**Eliade 1962: 10**). In much the same way, chemistry represents a secularised form of alchemy concerned with “exact observations” and “systematic experiments” as opposed to “the passion, the death and the marriage of substances” (**Eliade 1962: 11**). Eliade believes that the allegorical nature of alchemical writings highlights its religious and ritual identity and therefore also its fundamental difference from chemistry. In the lines just cited we can also find the main argument underpinning the grand narrative of the scientific revolution. As a matter of fact, much of the post-World War II historiography of science rests on the assumption that the scientific revolution represented a moment when modern scientific disciplines, ideas and values came to the fore and produced a definitive break with the past (**Butterfield 1957; Koyre 1957; Rossi 1968; 2006; Kuhn 1962; Westfall 1971; Hall 1983**). Even when these histories acknowledged the contributions of the alchemical tradition in the emergence of the new experimental sciences, they invariably do so from a presentist viewpoint through which they pick only what seems to prefigure modernity. For instance, since we know that making artificial gold cannot be achieved, then any attempt at doing so has nothing to contribute to the unfolding of modern science, it rather belongs to the realm of superstition and therefore it is left out of the scope of the history of science.

Mainstream scholarship has since come to reject the idea of a separation between alchemy and chemistry, as this was entirely based on the common tendency of artificially and a-historically superimposing modern knowledge and modern values, thought of as universal, to contexts where they do not apply. The very idea of the scientific revolution has been revisited and a more comprehensive understanding of the individual contexts in which scientific ideas developed has been praised as the attitude historians should embrace (**Osler 2000**). Rather than talking about rupture it is now preferable to explore along the lines of continuity and exchanges between alchemy and chemistry and to look at the blurred boundaries between the two. It was noted that under the veil of allegorical images and of fanciful language that populates alchemical texts one could read real chemical reactions, with relevant ingredients and operational parameters, and that these could be investigated in their own right to better understand alchemical practice (**Karpenko 1992; Principe 1987; Principe 2013: 137-172; Rampling 2014**). This was an enormously important discovery since much of the argument in favour of a supposed irrationality of alchemy revolved around its unintelligible language that would supposedly prove the fantasy of the operations discussed. What was discovered is that alchemists made constant use of *decknamen*, codes and ciphers to conceal information and render it more difficult to access. This could be motivated by the alchemists seeking "prestige" through a specific jargon, by economic reasons (i.e. trade secrets) or instead it may have to do with the need to keep a type of knowledge considered sacred protected from unrestricted dissemination (**Principe 1992; Roos 2014**). In other words, far from being a sign of irrationality and far from only mirroring the ritual dimension of alchemy, secrecy was a way of expressing some aspects of alchemical knowledge using a specific language that had been codified throughout the centuries. In this sense, a useful example is provided by the alchemist and natural philosopher George Starkey (1628-1665), who introduced Robert Boyle to alchemical practice in the 1650s. When writing about his work on metallic transmutation and the philosopher's stone, Starkey made use of both the allegorical and secretive language of traditional alchemy as well as of a more technical one (**Newman and Principe 2002**). Indeed, while the books he published with the pseudonym of Eirenaeus Philalethes are filled with alchemical imagery, his private laboratory notebooks contain detailed reports of his experiments and show the natural philosopher's awareness of issues such as reproducibility and cost efficiency as well as a sound understanding of analytical chemistry (**Newman and Principe 2004**). Not only does this confirm that behind the alchemical imagery were real chemical reactions and natural substances, but Starkey's equally committed involvement on both fronts of the supposed divide alchemy-chemistry shows that no such disjunction really existed in the minds of early modern people, certainly not in the conflictual terms that traditional historiography has maintained. Even when anti-alchemical sentiments were voiced, any criticism was always raised from within and indicates the vital nature of alchemy and the fact that its many facets sometimes clashed. Georg Agricola (1494-1555) and

Andreas Libavius (1555-1616) both fiercely attack alchemy, but what they are against is one side of it, namely the secretive language and the private and uncommunicable nature of the work of some practitioners. However, neither of them denies the possibility of metallic transmutation *per se* or make an explicit distinction with a supposedly more scientific chemistry (**Beretta 1997; Moran 2005: 99-131; Newman 2004; Rossi 2009: 60-78**).

A more attentive reading of the primary literature indicates that most of the figures traditionally considered champions of the scientific revolution and the carriers of modern science firmly believed in metallic transmutation and spent considerable time and resources in pursuit of it (**Newman 2010; 2011**). Notable examples that have been analysed in detail include Robert Boyle (**Principe 1998; Newman 2014a**) and Isaac Newton (**Dobbs 1975; 1991; Debus 1998; Roos 2010; Newman 2010; 2016**). The discovery that such prime figures were committed to gold making and other alchemical quests helped debunk the arguments in favour of alchemy's intrinsic irrationality discussed above. As a matter of fact, most matter theories of the 17th century were coherent with the theoretical possibility of metallic transmutation since all metals were thought to be made of tiny corpuscles whose different shape, arrangement and degree of purity determined the metallic "species". According to this view, lead was different from gold only in terms of its purity, its corpuscles being much grosser than those making up the latter. Theoretically, the practitioner could act at the corpuscular level and transmute a base metal into gold through a process of purifications (**Newman 2006; 2010 Principe 2013**). Consequently, metallic transmutation was an absolutely possible, albeit very hard to achieve, goal, it was consistent with the way people understood nature and the generation of metals at that time. It was not until later, well into the 18th century that people began to differentiate between what we recognise today as separate alchemy and chemistry. This is because by this point background knowledge produced through experimentation increasingly suggested the impossibility of gold making, although there was no consensus over the issue. Not a radical fracture then, but rather an evolving continuity. Chemistry was "an extract, a derivative of alchemy" which at some point in history had had to "shed itself of some very unattractive baggage” (**Moran 2005: 185**), namely the secrecy of the language and the gold making aims which were proving impossible to achieve. It is in this context that the terms alchemy and chemistry slowly started to diverge, with the former becoming increasingly associated with the idea of fraud and delusions and to the sole aim of transmutation of metals, while the latter being used to convey the image of a respectable activity in response to it. It is at this point that “synonyms became non-synonyms” (**Newman and Principe 1998: 41**). Until then, though fraudulent alchemists seeking to make a profit by selling false hopes had always existed (**Nummedal 2007**), a lot of respected alchemy was done in the pursuit of gold making. And even more importantly, it is now largely accepted that the chemical operations done in pursuit of metallic transmutation, despite not being successful in their ultimate outcome, had a significant impact on the understanding of the inner structure of matter (**Martinón-Torres and Rehren 2005a; Newman and Principe 2002; Newman 2004; 2014; Nummedal 2007: 107-109**). Gold making triggered technological developments in the field of metallurgy and weighting which in turn brought an invaluable contribution to corpuscular theories of matter by increasing the awareness that weight and density are important qualities too, besides colour (**Martinón-Torres and Rehren 2005; Rehren and Martinón-Torres 2008**). A rich and varied body of literature has explored the trajectories through which the knowledge - both theoretical and operational – produced by alchemical practitioners has brought a decisive contribution to the emergence of the experimental sciences and the mechanical philosophy in the 17th century (**Clericuzio 1993; Debus 1980; 1990; 1991; 1998; Moran 2005; Newman 1989; 1996; 2006; 2010; Osler 2000; Newman and Principe 2002; Principe 1998**). These histories show how the scientific revolution ultimately rests on this background of laboratory experimentation and on the lively and dynamic discussion of theories of matter and technical information that sprang from them. Thus, to consider alchemical knowledge un-scientific, irrational or to dismiss it as erroneous on the grounds of modern-day scientific facts enormously jeopardises our understanding of the phenomenon. The refusal of most traditional historiography to see alchemical work as a legitimate chemical activity and as integral part of early modern philosophical enquiry is the reflection of an anti-alchemical bias which originated in the 18th century and in a very specific context.

That the division between alchemy and chemistry does not reflect the reality of early modern European culture can also be argued from the fact that at this time the two terms were synonyms. Both were employed interchangeably in primary literature without specific distinction and to refer to many different activities that involved the manipulation and transformation of substances and the philosophical enquiry into nature. Consequently, the historiographic reassessment of alchemy has also passed through a terminological revision that mirrors a current view of alchemy as the dynamic, multifaceted and complex phenomenon discussed in this section. Thus, in order to avoid the risk of anachronistic connotations, it has become commonplace among historians of science to use the archaic term "chymistry" when referring to the period that spans from the reformation to the end of the 17th century (**Newman and Principe 1998**).[2] Like alchemy and chemistry, chymistry is also found in early modern texts as another synonym, but unlike them is not so strongly charged with meaning and prejudice and therefore it offers a very attractive neutral alternative. This way, talking about chymistry rather than alchemy/chemistry helps conveying the idea of original unity between them, the absence of sharp division and modern-day compartmentalisation, as well as the many overlaps and exchanges existing between natural philosophy and various artisanal and artistic practices. Chymistry is that attempt at understanding and manipulating nature mentioned above (**Newman 2000**), and to give the sense of diversification one can adopt other archaic terms that refer to specific branches of chymical activity. One such word is, for instance, the Greek-derived term *chysopoeia* (literally gold making), which identifies the field of metallic transmutation. Another is *iatrochemistry*, or *chymiatria*, encompassing the preparation of remedies through distillation and other chymical methods, and the related knowledge behind it. What is important to keep in mind when making these distinctions is, however, that they were not radically separate activities and that there

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[2] However, not every historian decided to adopt the term chymistry in place of alchemy/chemistry, even among those who adhere to the current understanding of the phenomenon as wide and overarching.

was always mutual exchange between them (**Klein 2008; Nummedal 2011; Newman 2016**). In adhering to the renewed understanding of alchemy/chemistry as an endeavour that is both inquisitive and productive, theoretical and practical, and whose spectrum of practitioners, motivations and sites is overarching, the term chymistry will therefore be employed throughout this thesis with reference to the early modern period. Having discussed how chymistry is understood by current historiography, the following sections focus on how historians of science have analysed this phenomenon and its fundamental role in the process that led, towards the end of the 17th century, to the emergence of that new approach to the natural world also known as scientific revolution. Finally, the last section of this chapter identifies the space where archaeology can enter the arena and contribute to the narratives whose aim is to draw a history of chymistry and chymical practice in the early modern European world.

2.2 THE SCHOLAR AND CRAFTSMAN THESIS AND ITS RECENT DEVELOPMENTS

One of the most prolific perspectives to explore early modern natural philosophy follows the emergence of a renewed attitude towards the material world, where empiricism and practical experience become fundamental in the creation and modification of knowledge. William Newman's definition of alchemy discussed above does introduce the critical issue that to manipulate and to understand are dependent on one another and consequently that artisanal work, with its transformative action exerted upon matter, was an important knowledge-producing mechanism. The skills of the craftsman, their tacit knowledge of natural materials obtained through bodily engagement with substances, are important contributors to the new idea of natural philosophy that develops in the early modern period (**Smith 2004; 2014**). How and in what ways did artisans become producers of philosophical knowledge are questions of prime relevance among current historians of early modern science, but the theme is by no means a new one.

As a matter of fact, the many scholars who now actively engage with the epistemic value of craft knowledge have built and elaborated on an older tradition that emerged in the first half of the 20th century and became known as the scholar and craftsman thesis. Among the first exponents are intellectuals from different backgrounds, such as the philologists Leonardo Olschki (**1919-1927**), the sociologist Robert Merton (**1938**) or the philosopher and physicist Edgar Zilsel (**1941a; 1942**), to cite some of the most renown ones. A detailed examination of the different directions these works took would take this chapter beyond its scope (**see Long 2011: 10-25**), but underpinning them all was the common belief that during the post-medieval period some of the skills, knowledge and values characteristic of the artisanal universe became increasingly embedded into European scientific culture, and decisively shaped the new experimental sciences of the 16th and 17th centuries. As Zilsel wrote, the birth of modern science is to be seen as a socio-economic phenomenon whose single most important drive was "the rise of the methods of the manual workers to the ranks of academically trained scholars" (**Zilsel 1942: 558**). Zilsel refers to the values linked to artisanal practice, such as empirical observation, and experimentation and claims that what triggered the integration of such values in scientific practice was the transition from a feudal system to an early capitalistic economy. The effects were the dramatic increase in the (economic) value of material objects and the parallel rise in importance of those who produced those objects, as well as of the status of manual work itself (**Zilsel 1941a; 1942**). As a consequence, the old social divide that had existed since classical antiquity between intellectual (*episteme*) and manual (*techne*) knowledge slackened, allowing for contact and exchanges between these two worlds (**Newman 1989; 2004; Bensaude-Vincent and Newman 2007; Smith 2007; Smith et al 2014; Dupré 2014**). The birthplace of this phenomenon is the city with its many artisanal-artistic workshops. In 15th-century Florence, for instance, large workshops served major construction efforts and in these, craft activities were carried out alongside the teachings of perspective, mathematics and geometry from the books of Classical authors, where practical and erudite knowledge could meet and influence one another (**Rossi 2009**).

However, the early scholars working on the thesis developed such momentous ideas within the largely presentist framework that more recent historiography has since abandoned (cfr 2.1). In this sense, despite the major role attributed to mechanical arts, early exponents of the scholar and craftsman thesis were primarily interested in highlighting the modernity of the experimental sciences and their new values of openness, cooperation and progression. To them, these values were in sharp contrast with a pre-existing tradition (variably defined as alchemy, magic, astrology, animism, hermeticism and so on) to which little or no epistemic dignity is granted. In investigating the work of Francis Bacon, the historian and philosopher of science Paolo Rossi acknowledges the debt of the natural philosopher towards the magical tradition of his time, but he repeatedly insists on distancing Bacon and the scientific revolution from that tradition (**Rossi 1968; 2006**).[3] Only by breaking with alchemy and mysticism did modern science develop ideas such as publicity of methods and results, desire for clarity, repeatability and controllability of the experiments and a separation from theology. In contrast, alchemy's traits were the usual obscurity, secrecy and a strong religious character that are so often paired with it. Equally common are attempts at sanitising the forefathers of the scientific revolution by downplaying their involvement in alchemical practices, and it was already shown in the previous section how these figures saw no contradiction in alchemy. When talking about the work of the physician and natural philosopher William Gilbert (1544-1603), Edgar Zilsel writes that his scientific method "combines essentially modern with metaphysical, scholastic, and animistic elements" (**Zilsel 1941b: 72**), and continues by remarking that "his animistic metaphysics is nothing but the emotional background of his thinking and does not affect the empirical content of his science" (**Zilsel 1941b: 75**). Like for Mircea Eliade and his parallel with Greek theatre, historians have often referred to the emotional realm when discussing alchemy and its supposed distance from the rationality of modern science. Thus, by insisting on modernity and by imposing modern-day scientific values on the figures of the scientific revolution, the early scholar and craftsman theses

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[3] The very title "Francis Bacon: from Magic to Science" of Rossi's 1968 book sounds like a programmatic statement.

maintain that “tendency to downplay or eliminate any natural philosophical or ‘scientific’ content in alchemy” and show “insufficient interest in the historical and cultural context” that generated ideas (**Principe and Newman 2001: 415-416**). Even though current historians have since moved away from the radical view held by early historiography, a tendency to distinguish between a somewhat more legitimate and also scientifically accurate technical knowledge and the alchemical one still exists. In discussing the work of the alchemist-priest Antonio Neri (1576-1614), author of the first treatise on glassmaking, Maria Grazia Grazzini tells us that “the most widely held image of Neri has been that of the technical expert” whose language was “‘clear’ and ‘distinct’” and whose results were made public “with the aim of being ‘beneficial to the world’” (**Grazzini 2012: 412**). She then contrasts the treatise to another work by Neri, the *Discorso Sopra la Chimica*, which “shows a different side of Antonio Neri, one seemingly at odds with the image of the technician–glassmaker; it reveals the figure of the alchemist deeply committed to Hermetic and Paracelsian doctrine.” Even maintaining that the two sides of knowledge are at odds with each other, Grazzini nevertheless highlights how the example of Neri demonstrates the presence of overlaps between them. To sum up, the early promoters of the scholar and craftsman thesis brought to the table important and innovative themes in the historiography of science. First, they acknowledged that science is a fundamentally social phenomenon and as such it cannot be understood outside of the socio-economic context in which it happened. From this premise derives the idea that artisans and mechanical arts played a crucial role in the development of the experimental sciences and that this role must be analysed. These ideas resonate very strongly in the work of current historians, who brought them back into the scholarly discourse through a process of major revision and elaboration (**Smith 2000; 2004; 2007; Dupré 2014; Roberts et al 2007; Klein and Spary 2010; Long 2001; 2011; 2018; Valleriani 2013; 2017; Pastorino 2009; 2017; Struhal 2017**).

Once the inheritance opposing alchemy and science was put on the side, the relationship and the mutual interactions between the universe of making and that of knowing could become a major research avenue. The reassessment also involved the incorporation of theoretical inputs that had come to the fore in the decades following the first emergence of the thesis. What is perhaps the most relevant example is that material sensitivity that followed the "material turn" of the 1970s and 1980s and that called for the investigation of the practical dimension of science (**Werrett 2014**). For instance, historians have paid more attention to the physical spaces where chymistry was performed, the laboratories and workshops in which scholarly and artisanal cultures met and epistemic exchanges between the two took place (**Shapin and Shaffer 1985; Hannaway 1986; Crosland 2005; Klein 2008; Anderson 2013; Saunders et al 2013; Dupré 2014**). Equally, the study of scientific instruments, laboratory apparatus and other objects from chymistry-related contexts have acquired more and more relevance among historians (**Daston 2000; Taub 2011; Jardine 2018**). One of the tenets of the scholar and craftsman thesis was the role of "superior artisans", individuals who had the necessary education to actually make the encounter happen by incorporating the largely uncodified and non-verbal knowledge of crafts into a natural philosophical system (**Zilsel 1942; Rossi 2009; Olschki 1919-1927**). While the concept is still inspiring current historiographic research delving on the role of such important figures (**Smith 1994; Pastorino 2009; 2017; Valleriani 2013; 2014**), it also triggered important questions on how to access the tacit and embodied knowledge of artisans and artists.

In the influential *The Body of the Artisan* the historian Pamela Smith tackles this topic and outlines the most important shapes that artisanal knowledge took in the early modern period. Smith writes that craft worldviews "saw certainty in nature and in the unmediated experience of nature" (**Smith 2004: 88**) and discussed the contribution of those who theorised the primacy of experiential knowledge and of learning by doing. One central player was the Swiss iatrochemist Paracelsus von Hohenheim (1493-1541) whose work takes forward a whole new philosophical framework or "chemical philosophy" in which chymical procedures become the main tool for a reformation of knowledge that places human artisanal power at its core and advocates hands-on practice as the prime mechanism to investigate the world (**Debus 1977; 1998; Smith 2004; Moran 2005; Principe 2013**). The influence exerted by Paracelsian worldviews on later chymistry is enormous. The concept of *spagyria*, a process of separation based on repeated distillations, became a powerful analytical tool in 17th-century experiments that aimed to reduce substances to its prime components and paved the way for the development of corpuscular theories of matter and the mechanical philosophy (**Principe 1998; Newman 2006; 2010**). One result of Paracelsus' influence on established practice is the gradual inclusion of chymiatria in the sphere of formal medical education, with a growing number of European universities where the consequences of his ideas were discussed and taught (**Debus 1990**).

Besides bringing substantial changes to established university education, Paracelsus' reformation of knowledge and the value of learning by doing was taken up and re-elaborated by influential artist-artisans who wrote books and filled them with similar claims to practical knowledge. In his creations as well as in his writings the French potter Bernard Palissy (1510-1589) actively engaged in and contributed to natural philosophical debates around mineral formation and the generation and the transmutation of matter, and voiced the defence of the power of men to understand nature through processes of imitation (**Smith 2004; Newman 2004**). In one of his most famous passages, Palissy imagines a dialogue between Theory and Practice in which the latter claims the superiority of physical labour over writing and theorising when it comes to natural knowledge. The ideas that begin to be codified by artist-authors of the 16th century were by no means new and were largely shared by an artisanal culture whose education was almost entirely based on practice and imitation of the master, on feeling substances and judging by the eye (**Dupré 2017a**). But what authors such as Palissy or the goldsmith Benvenuto Cellini (1500-1571) brought was a new claim by which artisanal practice was a way of exploring nature on a philosophical level. This vernacular science of matter, as defined by Pamela Smith (especially **2004; 2014**), is based on the artisan's physical manipulation of substances during creative processes, which allows him to listen to nature and capture the intricate web of resonances that connect everything in the natural world. The study of chemical technologies as a knowledge-producing activity has therefore received the interest of science historians.

But besides exploring the ways in which the mechanical arts influenced natural philosophy, recent historiography began to investigate in the opposite direction, asking what natural philosophy had to offer to the world of the mechanical arts, particularly to the field of chemical industry (**Smith 1994; Valleriani 2013; 2014; Dupré 2014; 2019; Klein 2017; Pastorino 2017**). A new wave of studies has concentrated on the contribution of figures who moved halfway between the domains of chemical technologies and that of the intellectual strata, whose university education was often at the service of profit-driven activities. These hybrid experts, as they are sometimes called, need to be understood against the foil of the permeable disciplinary boundaries that characterise early modern chymistry. Their work carried an idea of knowledge that was at once productive and inquisitive and whose aims were to increase profit and explore chymical issues of their time. Their lives, their background and the way they came to acquire chymical knowledge and expertise have been explored in detail. Some, like the German chymists Joachim Becher (1635-1682) and Rudolph Glauber (1604-1670), were empowered by princely patronage and became advisors to rulers (**Smith 1994; Smith 2004: 155-182; Madl 2008**). Others did not depend on patrons and run their own enterprises, using their chymical knowledge to experiment with cutting-edge industrial technologies of their time. One notable example, the chymist-entrepreneur John Dwight (1633-1703) and his work to reproduce Chinese porcelain and other specialised pottery will appear again, and in more detail, later in this thesis (cfr chapter 5) (**MacLeod 1987; Green 1999; Wesley 2014**). This thesis will also discuss the case of another kind of hybrid experts, namely those who worked within the boundaries of formal university education, which in this period begins to open its doors to the teaching of practical chymistry often as a branch of the medical degree (cfr chapter 5) (**Debus 1990; Klein 2016**). Many of these chymical experts were hired by princes at European courts, where they established chemical technology industries depending on what their specialisation was, thereby highlighting the crucial connection between chymistry and issues of politics and governance (**Smith 1994; Beretta 2014; Roberts and Werrett 2017**). Common chymical work included the distillation of medicines, the transmutation of metals, the enhancement of agriculture and the large-scale production of valuable commodities such as glass, porcelain, weapons and gunpowder (**Smith 1994; Sarmiento 1997; Long 2001; 2011; Beretta 2014; Klein 2014**). To many individuals, an expertise in chymical theories and practices provided a way to earn a living and social recognition, and throughout Europe professional chymists began moving within a marketplace that required their skills and productive knowledge. Historical documents give us a sense of how mundane, practical and also important this system was. The detailed contracts of employment, for instance, inform us on the terms of the agreement between patrons and chymists, while trial records bear witness to the consequences of misconduct or failure to meet those terms (**Nummedal 2007; 2011**).

Princely courts and the system of patronage played a significant role in bridging the gap between artisans and scholarly types of knowledge. From the 16th century, incipient forms of a capitalistic and mercantile economy brought an increasing importance of mobile resources over traditionally land-derived wealth, encouraging the European princes to invest large sums of money in potentially profitable industrial enterprises (**Smith 1994; Long 2001; Nummedal 2007; Dupré 2014; Valleriani 2017**). In the numerous mining towns of central Europe, for instance, learned overseers educated in universities operated alongside miners, assayers and refiners, whose chymical expertise derived from workshop training and was ultimately rooted in Medieval alchemy. Metallurgy became a discipline worthy of books written both in Latin and in vernacular and reproduced thanks to the advancing printing technology. The treatises published by Vannoccio Biringuccio (1480-1539) in 1540 (see **Smith and Gnudi 2015**), Georg Agricola in 1556 (see **Hoover and Hoover 1950**) and Lazarus Ercker (1530-1594) in 1574 (see **Sisco and Smith 1951**), as well as the anonymous 16th-century book “Bergwerk- und Probierbüchlein” (see **Sisco and Smith 1949**) contained a wealth of technical information regarding the extraction, working, refining and testing of metals, but also included lengthy sections devoted to the theoretical principles regulating their generation and transformation. The historian Pamela Long has employed the term “trading zones”, which she borrowed from the field of linguistics, to refer to those early modern arenas that promoted contact between individuals from different social strata and contributed to the mutual exchanges between them (**Long 2001; 2011: 94-126**). European courts and mining towns are examples of trading zones, as are the large-scale artistic and engineering projects of early modern cities that were mentioned earlier as the very first place where practical and theoretical expertise worked together (**Rossi 2009; Long 2011; 2018**). Further melting pots for the encounter were offered in this period by the ever-growing number of towns, particularly in the strongly urbanised areas of northern Europe dominated by an early capitalistic mercantile economy (**Braudel 1979**). In places such as the Dutch Republic and the free imperial cities of Germany the class of wealthy merchants was extremely powerful and often ruled the political scene (**Smith 1994; Van Berkel 2010**). The enhanced social mobility of these environments attracted artisans and entrepreneurs who saw more chances of making large profit as well as gaining increased social recognition.

These plural histories challenge the generalisations of grand narratives, where science is seen as unfolding regardless of what happens around it, and re-evaluate the need for understanding the cultural and socio-economic context in which scientific ideas emerged (**Latour 1987; 2005; Law 2010; Osler 2000; Pfaffenberger 1992**). The focus has moved away from the canon of the scientific revolution and the related "heroic" figures that populate it. Instead, attention is now placed on the rich mosaic of people, places and motives that characterise early modern chymistry and to the plurality of ways down which natural knowledge was created and transformed (**Martinón-Torres 2011; Moran 2005**) (**fig. 2.1**). In order to navigate such diverse reality science historians have drawn on the concept of microhistories,[4] where the phenomenon of chymistry is reconstructed from the bottom up, starting from a network of well-contextualised and high-resolution case studies. As the examples mentioned in passing earlier indicate, more and more of such histories scrutinise the work of single figures, often less-studied if not partly (or literally) anonymous characters who moved across the fluid boundaries

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[4] On the concept of microhistory, as a way of entering a historical debate from the perspective of a limited and well-contextualised close-up, the Italian historian Carlo Ginzburg provides us with some of the most renown examples, see for instance his seminal book *The Cheese and the Worms. The Cosmos of a Sixteenth-Century Miller*. For more details on the microhistory method see also Ginzburg 1993.

characterising early modern chymical activity (**Blair 1997; Nummedal 2007; Martinón-Torres 2011; Smith 2012; Dupré 2014**).

Figure 2.1. Illustrations from Antonio Neri's Tesoro del Mondo, 1599. From left to right: The preparation of plants, of animals and of stones.

Through the various narratives presented so far, recent histories of science have shown that chymical practice had the opportunity to play a fundamental role in the process of knowledge reorganisation that affected Western natural philosophy in the early modern period. Because it offered both a sophisticated theoretical framework and a vast body of technical knowledge that fed into those theories, chymistry became a central way of expressing a new relationship with the material world. It provided people with the tools and the language to describe the generation and transformation of matter, it was a focus to negotiate the boundaries of human artisanal power, and offered the necessary knowledge to make profit in a society where values were shifting and technology rapidly progressing (**Moran 2005; Newman 1989; 2004; Smith 1994; Nummedal 2011**). Chymical work contributed to the wealth of individuals and communities, it brought technological advances in the industry and new philosophical theories derived from laboratory experimentation. Chapter 4 will examine the vital contribution of chymistry during the transatlantic explorations, which revealed the existence of previously unknown worlds, facing Europeans with unprecedented challenges to established natural knowledge and world views. Chymical knowledge and practice allowed to test, explore, understand and exploit previously unseen animals, minerals and other resources. As a matter of fact, every expedition included skilled chymists who set up workshops in the European colonies from the Caribbean to the Canadian arctic (**Nöel-Hume 1994; Beaudoin and Auger 2004; Kelso and Straube 2004; Thibodeau et al 2007; Monette 2013; Kelso 2017; Martinón-Torres et al 2018**). Therefore, charting the multifaceted history of chymistry provides a powerful focus on many aspects of the early modern period. As Bruce Moran writes, chymistry "has offered an extremely fertile area for inquiry into various sorts of practice in the history of science, mediating between people, the social world, objects, and texts" (**Moran 2011: 303**).[5]

2.3 NEW SOURCES OF INFORMATION AND THE ARCHAEOLOGY OF CHYMISTRY

The previous sections have discussed the changing understanding of alchemy that has characterised the historiography of early modern science in the past three decades. They also introduced the most relevant directions that such new evaluation has taken, including the introduction and now commonplace use of the term chymistry. In this section, some methodological aspects involved in this revision process are presented, particularly new ways that recent historiography has developed to study the history of chymistry. Indeed, the wide scope of chymistry as a dynamic kaleidoscope composed

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[5] That the history of chymical practice keeps enjoying a growing interest among historians is highlighted by the number of workshops, symposia and conferences that are being devoted to the most different aspects of its study every year. The following is a selection of recent examples that took place in the span of this PhD project: The conference "Ingenuity in the Making: Materials and Techniques in Early Modern Europe" (Centre for Research in the Arts, Social Sciences and Humanities, University of Cambridge, 11-12/05/2017); The Society for the History of Alchemy and Chemistry postgraduate workshop "(Al)Chemical Laboratories: Imagining and Creating Scientific Work-Spaces" (Chemical Heritage Foundation, 01-02/12/2017); the conference 'Learning by the Book: Manuals and Handbooks in the History of Knowledge' (History Department Princeton, 06-10/06/2018); the workshop "Artisans of the Surface in Early Modern Europe, 1450-1750" (King's College London, 20-21/09/2018); the international symposium "Alchemical Laboratories. Practices, texts, material relics" (the University of Vienna, 19-21/02/2020); the virtual conference "Science in the City: 1500-1800 (06-07/04/2020).

of many influences necessarily required a parallel broadening of the range of sources to be employed in telling its history. And indeed, historians have turned to a varied array of less conventional sources of information alongside the more traditional books and manuscripts. Vernacular collections of recipes from workshops and households, for instance, are enjoying an increasing popularity among scholars interested in chymical practice (see particularly **Leong 2008; 2017; 2018** and references therein; **Hagendijk 2019**).[6] Many studies have concentrated on documents such as manuals and how-to texts, laboratory notebooks and work diaries written by both well-known figures as much as by anonymous people (**Newman and Principe 2004; Smith 2012; Hagendijk 2018**).[7] As exemplified by the case of George Starkey mentioned earlier (cfr section 2.1) notebooks can offer significant insights into issues such as, for example, the interplay between openness and secrecy. Inventories and plans of laboratories tell us more about the physical spaces where chymistry was carried out and about the apparatus that furnished them (**Hannaway 1986; De Vos 2007; Newson 2020**). Guild regulations and contracts of various types, between patrons and chymists, remind us of the entrepreneurial dimension of the phenomenon, and the reports of the frequent trials, following misconduct and accusations of fraud, bear witness to how seriously these collaborations were taken (**Nummedal 2007; Roos 2014**).

However, if chymistry was "something that people *did*" (emphasis in original) (**Moran 2005: 10**), it follows that alongside textual sources there exists a different kind of primary evidence that we can draw from when studying it, namely material-based information. Objects of art and technology can be read as embodiments of the craft knowledge of chymical practitioners, a knowledge that is non-verbal and uncodified and the expression of a system of worldviews (**Smith 2007; Dupré 2014; Hanß 2018; 2019**). Building on the example of life-casting as a way to create perfect imitations of small animals and plants, Pamela Smith writes that "The objects that survive from Jamnitzer's busy workshop almost all make reference to the relationship of nature and

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[6] See also the Recipes Project

[7] Another example is represented by the work diaries of Robert Boyle, which have been digitised and can be accessed at http://www.livesandletters.ac.uk/wd/index.html (last accessed July 2020)

art" and that they "manifest the active knowledge embodied in human craft, which can itself comprise a studied reflection of nature at work" (**Smith 2007: 49**). Entering the universe of making and knowing, of activities that are simultaneously investigative and productive, throws light on less accessible mechanisms through which natural knowledge was produced in early modern times (**Dupré 2017a; Shell 2010; Smith 2010; 2017; Valleriani 2013; 2017**). Another way to get information on the vernacular science of artisans is the replication of early modern chymical procedures and recipes (**Moureau and Thomas 2016; Principe 1987; Rampling 2014; Sibum 1995; Smith 2012**). In the last few years, the use of such re-enactments has gained unprecedented momentum thanks to ongoing research projects such as the *Making and Knowing Project* at Columbia University,[8] the *Chymistry of Isaac Newton* at Indiana University[9] and, more recently, *Artechne - Technique in the Arts, 1500-1950*, based at Utrecht University,[10] as well as *Alchemeast* at the university of Bologna.[11] These works have shown how the "experimental" history of science is an invaluable tool to get access to the epistemic categories of the early modern chymists, decipher otherwise obscure passages in texts, or highlight unexpected issues, such as the need of specific skills, materials and knowledge. In light of this dramatic growth in the relevance of materials and material culture of early modern chymistry (**Holmes and Levere 2000; Roberts and Werrett 2017**) and because of its increasing presence within the history of science, opportunities for interaction with material culture-based disciplines are multiplying. The research projects just mentioned have set the example by including a rich mix of diverse expertise in their cohorts.

Nonetheless, it is felt that the contribution of archaeology and archaeological science to the discourse around early modern chymical practice is yet to be fully exploited and developed, though its potential has been pointed out time and again (**Rehren 1996; 1997; 1998; Anderson 2000; Martinón-Torres 2011; Nummedal 2011; Werrett 2014; Rampling 2017**). And a great deal of potential does indeed exist with regard to the

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[8] http://www.makingandknowing.org
[9] http://webapp1.dlib.indiana.edu/newton/
[10] http://artechne.wp.hum.uu.nl
[11] https://alchemeast.eu/

chronological period of interest here, since we have significantly more background information, textual evidence and, importantly, material assemblages available for post-Medieval times than we do for earlier periods. Archaeological science, with its focus on past technologies, significantly overlaps with these shifting historiographic trends and serves well the new focus on early modern chemical technologies adopted by a growing number of science historians. Thus, archaeology offers a different perspective from which to address some of those "surprisingly basic questions" (**Nummedal 2011: 332**) that some historians have become increasingly interested in and that involve "refocusing the history of alchemy on alchemists and their activities, rather than disembodied alchemical ideas", in order to write "the history of alchemical practice" (**Nummedal 2011: 332**). Moreover, such history of chymical practice is often made of interconnecting microhistories, which represents another relevant point of contact with archaeology. Remains of chymistry-related contexts are not always possible to attribute to precise individuals, let alone to the great minds celebrated in the traditional canon of the scientific revolution. The broken pots and rusty tools that we can study are for the most part an open window on anonymous people and everyday situation, and material culture is therefore an exceptionally powerful ally for a history of science that adopts a bottom-up approach.

There are further areas of methodological and theoretical overlap between the two discipline which should encourage more dialogue across the divide. For instance, the use of experimental re-enactments of past processes will be familiar to archaeologists, who have been replicating past processes, technologies and tasks as a means to obtain insights that would have otherwise been impossible.[12] A similar bridge across the disciplinary divide can be found in a methodological framework that is gaining popularity within the history of chymistry, namely that of technical art history. This approach to the history of art and technology "aims at a thorough understanding of the physical object in terms of original intention, choice of materials and techniques, as well as the context in and for which the work was created, its meaning and its

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[12] A detail examination of experimental archaeology falls out of the scope of this thesis. For a general introduction see: **Millson 2011** and references therein.

contemporary perception" (**Hermens 2012: 165**). In its intentions, technical art history is a holistic and interdisciplinary approach in which the many influences making up an object are taken into consideration as equally decisive in contributing to its creation (**Hermens 2012; Dupré 2017a**). This concept, with some differences as to its application, finds a parallel in archaeological theory where the *chaîne opératoire* framework was developed in the field of the anthropology of technology. At its very basic level, the *chaîne opératoire* maintains that any technical activity is determined by a great number of factors and that these have to do with both the material and the social realms (**Lemonnier 1986; 1993; Pfaffenberger 1992; Sillar and Tite 2000; Schlanger 2005**). Ultimately, these frameworks share the aim to acknowledge and explore the complexity of human technological processes and the many aspects influencing them. A last point that is worth raising is that an important component of technical art history is, by definition, the use of scientific analysis to enhance the understanding of works of art. Although this is mainly intended for conservation purposes, the scientific approach and many of the techniques employed are shared with archaeological science and constitute, therefore, another point of contact.

As this thesis will argue, studying the material culture of chymical contexts indeed provides us with an invaluable focus on the crossroads between the spheres of making and knowing in early modern Europe (and beyond) by showing the continuous exchange across the many facets of chymical practice. The material angle exposes a network of interconnected materials, activities and motivations that coexisted in early modern chymistry and reveals how such network was largely characterised by an identity of tools, ingredients as well as of underlying theoretical and operational knowledge. In chapters 4 and 5, presenting the case studies, and with the broader discussion of the results in chapter 6, this archaeology of chymistry will be articulated and discussed in detail. However, it is first necessary to address some important practical and methodological questions that follow the introduction of an archaeological approach to the history of science. Concrete ways to go about exploiting the information potential of material culture need to be laid out and it is necessary to address the question of what this material culture is even composed of, of what the range of materials that we find and study includes. What remains of the present chapter and part of the following present an overview of what we can expect to find in early modern chymical contexts and review the contribution given by the pioneering studies that have entered this arena already.

The types of material traces left behind by chemical technologies and that we can study are many. On the one hand is the rather wide array of instruments that filled the shelves and the working surfaces of the many spaces where chymical activity was performed. Although biased by their own agendas often aimed at creating stereotypes, genre paintings of alchemists at work are a valuable source of information on the material culture of chymical spaces before they became archaeological contexts. In a similar way, useful details can be gained from the descriptive woodcuts of technical treatises and the inventories or the “shopping lists” that are sometimes present in documents of various kinds (**fig. 2.2**). These documents feature furnaces and heating devices of various kinds, crucibles and other vessels used in high-temperature operations, distillation equipment made of glass, ceramic or metal, and then bottles and containers of sorts where chemicals and ingredients were stored. Assemblages of such archaeological materials provide us with a direct link to the rich material landscape of chymical contexts and add a further dimension to the iconographic and text-based evidence on early modern chymical practice.

Figure 2.2. Top: The Alchemist by David Teniers (ca. 1645); bottom: Depiction of an assay laboratory in Agricola's 1556 de Re Metallica (after Hoover and Hoover 1950)

Besides the tools, raw ingredients and the many reagents instrumental to chymical procedures can also be found in material assemblages. Metal oxides, ores and minerals, solvents, fluxes, fuel and so on all fall in this category. The final product of laboratory operations (e.g. refined precious metals, glass-based objects, distilled substances and so on) would be used or sold and their presence is therefore rare in the archaeological record. On the contrary, one of the most represented categories of materials is that of residues that form and end up discarded in the process, such as broken vessels (often with residues stuck within them) and bits of slag. A most informative approach to all these chymistry-related materials is through their scientific analysis. Archaeometric investigation of cultural heritage is carried out through a wide array of analytical techniques and in order to answer a variety of questions involving the identification, provenance, dating, technology of manufacture and material properties of artefacts and other materials. All these questions may be relevant, at varying degrees, to the narratives discussed in this chapter, most notably as ways of reconstructing the material practices of early modern chymistry, the closest we can get to the intentions behind those practices. The chemical characterisation of the materials listed above provide a great deal of technical information regarding which ingredients underwent which operations, following which operational parameters, and aiming to which end (cfr section 3.1). As it will be discussed in more detail in chapter 6, the very same questions are the focus of most archaeological science studies which investigate past technologies, the organisation of production or the provenance of ingredients and finished objects. The overwhelming majority of studies that have analysed assemblages belonging to chymical contexts in the broad sense discussed here, have framed their results within different theoretical narratives from those outlined in this chapter. This is by no means a criticism of these studies per se, whose relevance in the field is out of question. The point that this thesis wants to raise, however, is that the little (if any) exchange existing between archaeologists working on such contexts and science historians indicates that some synergy opportunities may have slipped into the crevices between modern academic disciplines. The case studies of this thesis will provide an example of how the two disciplines can engage in fruitful conversation and of how one's methodology can become instrumental to address some of the other's questions, thereby widening the scope of both. Ideas as to the shapes this widening process can take will also be addressed in chapter 6, which argues how a whole new dimension can be added to archaeological science and to the analysis of production remains of high-temperature activities.

For now, it suffices to say that a smaller number of studies have more explicitly aimed to unite archaeology and history of science by taking a material culture perspective. In these works, the theories around how scientific knowledge was created and modified in early modern Europe were not only acknowledged but also actively engaged with (**Rehren 1996; Martinón-Torres and Rehren 2002; 2005a; 2005b; 2007; 2009; Martinón-Torres et al 2008a; 2008b; 2009; Rehren and Martinón-Torres 2008; Mongiatti et al 2009a; 2009b; Martinón-Torres 2012a**). This archaeology of chymistry takes advantage of the growing available body of material remains of early modern chymical contexts (**Martinón-Torres 2011; 2012b**). The comprehensive analytical investigation of the large assemblage discovered in the metallurgical laboratory of Oberstockstall in Lower Austria, for instance, greatly increased our understanding of the supply of chymical tools and of their use in experimental procedures, that often went beyond contemporary codified knowledge (**Martinón-Torres and Rehren 2005a; Mongiatti et al 2009a; 2009b; Veronesi and Martinón-Torres 2018**). We will come back to the Oberstockstall materials later in the thesis (cfr. section 6.1.2), but what is important to stress here is that these works establish a connection between themes of technology/provenance and the trajectories along which the technical knowledge of experts developed and refined methods for exact quantification, with evident consequences in the emergence of modern analytical chemistry. As it was mentioned above, this is also a period for which we have an unprecedented abundance of textual sources, which boomed after the invention of the printing press. The archaeology of chymistry becomes an opportunity to explore the relation between objects and texts when it comes to chymical practice. In their analysis of theory and practice of brass making Thilo Rehren and Marcos Martinón-Torres point to a significant discrepancy between theoretical speculations found in texts and contemporary material practices as seen through the lenses of material culture (**Rehren and Martinón-Torres 2008**). Such disparities are interesting because they suggest evolving relationships between craftsmen and scholars that otherwise would not be visible and that reinforce some of the central ideas in current historiography outlined in section 2.2.

In the case studies chapter, this thesis will initially shed light on two high profile chymical contexts of which our knowledge is still incomplete. The analytical investigation of the apparatus from Jamestown and the Ashmolean laboratory, respectively, will allow to build two microhistories detailing the main aspects of chymical practice at the sites. The results will then be used to envision a network of interconnected materials and practices that starts from the bottom, from the messy work in the laboratories, and tells us about the communities behind them, their understanding of nature and their hopes and desire to gain knowledge as well as to make profit. While this is left to chapters 4, 5 and 6, the next chapter returns to some of the strictly methodological questions raised above, namely which specific information we can gain from the analysis of chymistry-related assemblages, and what analytical protocol is followed in the present thesis.

CHAPTER III

THE ANALYSIS OF CHYMISTRY-RELATED ARTEFACTS.

ANALYTICAL METHODOLOGY

Figure 3.1. Top view of some ceramic crucibles from the Ashmolean laboratory

After laying the foundation of the theoretical framework within which the present thesis will move, the final section of chapter 2 introduced aspects of the material culture of chymical spaces and highlighted the role this may have in shaping and modifying current historiographic narratives. With a specific focus on the ceramic crucible, which represents the most common type of find in the assemblages analysed in this thesis, the first part of the present chapter discusses the type of information such materials contain and how to go about extracting it through scientific analysis. Drawing from relevant studies in the field of archaeological science, the chapter opens with some methodological notes on the study of crucibles and on the investigation of the high-temperature chymical processes these were used for in the period under investigation (section 3.1). The second part of the chapter provides relevant information regarding the sample selection and laboratory methodology followed in the present project, including instrumental techniques chosen to analyse the samples and details on the analytical protocols followed (section 3.2). Thus, this chapter prepares the way for the two case studies that will follow in chapters 4 and 5 which, alongside the discussion in chapter 6, will allow to make meaningful inferences on some of the wider aspects in the history of early modern science that have been presented in chapter 2.

3.1 THE STUDY OF LABORATORY APPARATUS: SOME TECHNICAL NOTES

One of the most ubiquitous components of the material landscape of early modern chymical contexts and the main focus of the present thesis is the ceramic crucible. Crucibles were by and large the most versatile of chymical instruments, present in virtually every depiction of an alchemist at work in their laboratories (**Principe and De Witt 2002**). What is more, unlike the other category of tools ubiquitous in chymical contexts, namely glass distillation vessels, crucibles tend to survive more easily in the archaeological record due to the much higher resistance of ceramic to breakage and weathering, and its unsuitability to recycling, which makes them among the most common finds in chymistry-related contexts (**Sokup et al 1997; Martinón-Torres and Rehren 2002; 2009; White and Kearns 2010; Veronesi et al 2019**).

Crucibles represent specialised tools in high-temperature activities, and from the beginnings of metallurgy they were produced in a wide variety of shapes and sizes and were essential to several kinds of work. The study of crucibles offers crucial information to the investigation of past technologies, providing us with insights into technical as well as broader cultural aspects shaping them (**Tylecote 1982; Freestone and Tite 1986; Rehren 2003; Bayley and Rehren 2007; Martinón-Torres and Rehren 2014; Rademakers and Rehren 2016; Rademakers et al 2017; 2018a; Liu et al 2019**). Ceramic crucibles were used by glassmakers all over the world to mix the raw ingredients and to melt them into the desired products in furnaces. Equally, mining and metallurgical activities since the early days of extractive technologies heavily relied on the availability of high-quality crucibles where ores could be smelted, metals melted for casting purposes or mixed to produce alloys. Crucibles of smaller scale were key tools in operations that required accurate measurements, such as in metal refining and in fire assay, which involved multiple specialised operations to assess the presence and quantity of the desired metal in a known quantity of ore or in an alloy (**Hoover and Hoover 1950: 219-265; Sisco and Smith 1951**). Small vessels allowed a better control over the experimental conditions and a more accurate outcome (**fig. 3.2**).

Figure 3.2. Assemblages of crucibles from chymistry-related contexts. Left: The conical crucibles from the Tower of London mint (after White and Kearns 2010), right: Small triangular crucibles from the 16th-

century laboratory of Oberstockstall (Austria). In both cases the vessels were used in fire assay activities to test the quality of ores or coinage.

Crucibles belong to the broad category of finds known in the archaeological literature as technical ceramics, which also includes such tools as distilling vessels, moulds for casting, furnaces, kilns, hearths and ovens, and auxiliary elements such as kiln furniture and tuyères, long hollow tubes employed for controlling air access into the furnace. What distinguishes technical ceramics from common pottery is that the former was purpose-made to ensure better performances under extreme conditions. Indeed, crucibles were often manufactured to maximise their refractory properties, that is the ability to withstand extreme temperatures and chemical attack without failing (i.e. melting or breaking). One common way of doing it was by selecting specific kaolinitic clays especially rich in alumina (Al_2O_3) and relatively poor in oxides that promote melting, such as alkali and alkali earth oxides (Na_2O, MgO, K_2O, CaO). The renown triangular crucibles from Hesse in Germany, which will be thoroughly discussed in the following chapters, have exceptionally high levels of alumina, typically exceeding 35 wt% Al_2O_3, while the figure is normally between 15 and 30 wt% Al_2O_3 in most other crucibles of the post-Medieval period (**Dungworth 2006; Martinón-Torres and Rehren 2009; White and Kearns 2010; Monette 2013**).

After the choice of clays, a second step was to add specific tempering agents to the paste, which would further improve physical and thermal resistance and promote chemical inertness, thereby ensuring better performances. As the examples from the case studies analysed will show, the most common additives were sand, graphite flakes and grog (i.e. used crucibles crushed to a powder). Another known way to make crucibles stronger, again seen in German productions, is to pre-fire them at temperatures of over 1200C, which caused the ceramic to vitrify and promoted the formation of mullite, a highly refractory mineral phase, in the ceramic matrix (cfr section 4.5.1) (**Martinón-Torres et al 2006; 2008a**). However, in spite of their generally high resistance, crucibles suffered inevitable damages and contaminations during their use, and many operations required breaking the vessel after completion to retrieve its content. Therefore, crucibles were often used only once, and the care and the effort that went into producing them resulted in what effectively were disposable objects, as used crucibles were generally thrown out or reused to make grog (**Hoover and Hoover 1950: 230; Dungworth 2006; White and Kearns 2010**).

Technical ceramics, and crucibles in particular, represent a major branch of investigation in technological studies with special reference to the field of archaeometallurgy. Although a detailed methodological enquiry into how crucibles are analysed in archaeological science is outside the scope of this project, some general aspects need to be discussed here before outlining the results of the analyses in the following chapters. For a more exhaustive treatment of technical aspects of crucible analysis the reader is redirected to the recent excellent works by Frederik Rademakers and collaborators (**Rademakers and Rehren 2016; Rademakers et al 2017; 2018a**), as well as to other relevant technological studies by Tite, Freestone, Tylecote, Rehren, Bayley, Dungworth and Martinón-Torres cited so far, that laid the methodological foundations of the field. The scientific analysis of crucibles gives us insight into the aspects of crucible manufacture detailed above, namely what type of clays were employed, what tempering strategies and any other material information on the vessels themselves. These are stimulating avenues of research as they allow us to make inferences on issues such as the technical properties of the vessels, whether they were imported or locally made and on underlying supply strategies of chymical laboratories and what drove them (**Martinón-Torres and Rehren 2009**). It was already mentioned how crucibles of superior quality tend to have a very high content of alumina in the ceramic matrix, while the fact that a handful of German manufacturers supplied a vast network of chymical laboratories all over the world will be discussed later on.

The analysis of crucibles, however, and in particular of the residues preserved in them, is also an effective way to reconstruct the operations that were carried out within them and both these kinds of information will be addressed in the case studies presented in this thesis. Because a multitude of chymical activities made use of crucibles, it follows that the substances that at any point may have entered the vessels to undergo high-temperature treatment were many and very diverse. These substances included the main ingredients (e.g. a mineral to be assayed) as well as the reagents necessary to achieve the required conditions or to promote chemical reactions (e.g. fluxes to break down the minerals, or charcoal, when reducing conditions were required). The combination of ingredients and reagents is referred to as crucible charge and the term will be used throughout this thesis. The interaction, at high temperature, between the charge and the crucible's ceramic produced the residues that can often be seen adhering to the inner (and sometimes outer) surface of the reaction vessels (**fig. 3.3**). Other terms employed for these residues in the chapters that follow include interaction layer, vitrified layer, slag/slagged/slaggy layer and glassy residue, all referring to the same feature in the analysed vessels. The multiple names reflect the different looks that these residues can have, which depending on the nature of the reagents may display a more or less glassy look, may include metallic droplets or may show colours that indicate the presence of specific ingredients. Examples of very different residues will be presented and discussed in chapters 4 and 5. Because the residues contain the chemical signature of the charge and of its interaction with the ceramic, they provide us with the details of the operations the vessels had been used for. First, important observations can be derived from the composition of the glassy matrix of the residue. The matrix is made of all the elements in the charge that melted to a more or less uniform glass, and may contain other phases - if present, either residual from the ingredients or newly-formed during the reactions.

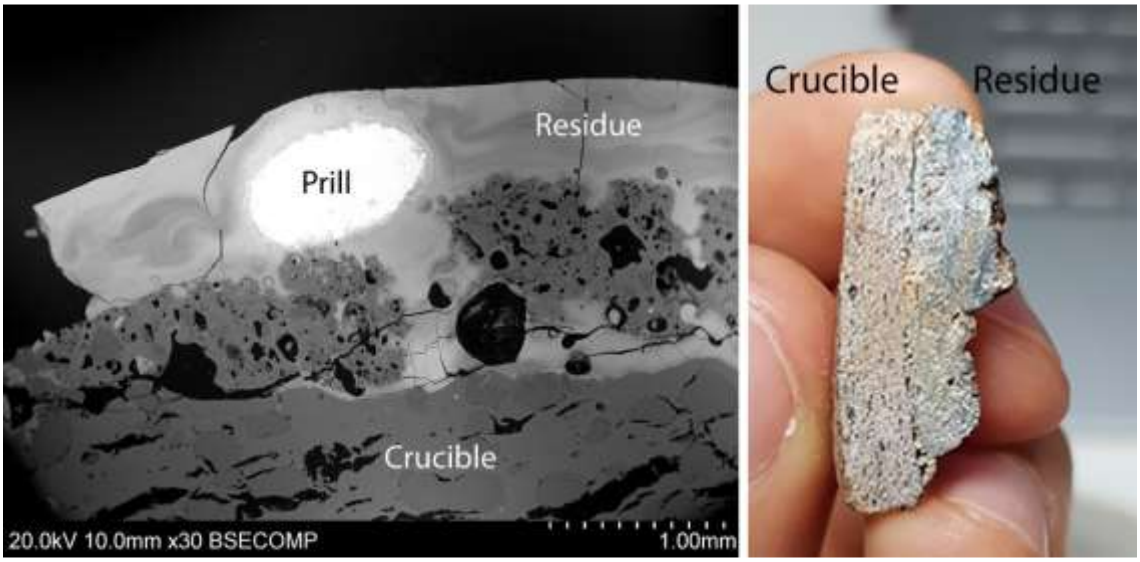


Figure 3.3. Examples of crucibles residues. Left: SEM backscattered micrograph from Jamestown sample JR 1339C, showing a glassy-looking residue adhering to the crucible wall. Note that the residue contains some molten ceramic as well as a large metallic prill. Right: Section through sample JR2718W from Jamestown showing the sharp boundary between the crucible and the metal-rich residue stuck to it.

By comparing the composition of the matrix with that of the crucible's ceramic one can assess the relative contribution of ceramic and charge to the matrix. For instance, a strong enrichment in alumina in the residue's matrix is likely caused by ceramic absorption, since highly refractory crucibles like the ones discussed in the present thesis are especially rich in this oxide, while it is less likely to be introduced by the charge itself. For this reason, the ratio of alumina to other elements can be employed as a sort of benchmark for assessing the presence of significant enrichments in the residue matrix and their origin. The contribution of charge components, for example, will produce notable enrichments in other elements, such as heavy ones coming from ores, or alkali and alkali earth oxides coming from charcoal and other reagents, when compared to the ceramic of the crucible (**Rademakers et al 2018a**). As shown in **figure 3**, discrete phases within the residue matrix can be seen under high magnification. These may represent unreacted or partially-reacted ingredients, such as sand grains in glassmaking crucibles (cfr section 5.5.2) or remnants of minerals when the vessels had been used to smelt ores (cfr section 4.5). Chapter 4 discusses the metallurgical crucibles from the colonial site of Jamestown (Virginia), mostly employed in operations of fire assay. The analysis of mineral relics discovered in the crucibles' residue layer makes it possible to identify the range of minerals tested and link the content of the vessels to the geology of Virginia (cfr sections 4.6). Thus, targeting these specific features in the residue is crucial to reconstruct aspects of practical chymistry in the contexts under investigation. Newly formed phases resulting from chemical reactions happening during the high-temperature processes may also be observed. One example of such phases are the metallic globules trapped in the residues of metallurgy-related crucibles (**fig. 3.3**). Their chemical composition may tell us what alloy the chymists were working with or what metal they were attempting to smelt from the ore. Thus, alongside the chemical composition of the residue's matrix, the correct identification of the different phases, through visual and chemical analysis, is a way to reconstruct the initial components of the charge, assess thermodynamic parameters such as temperature and redox conditions, and make inferences on the desired final product (**Mongiatti et al 2009a; Rehren and Nixon 2014; Martinón-Torres et al 2018**).

In general, similar information can be obtained from the analysis of other chymistry-related artefacts that preserve a residue on the surface. This is the case for other pieces of apparatus investigated in this thesis, such as distillation vessels and containers of different kinds. Like technical ceramics, distillation equipment is a specialised class of tools employed for specific chymical activities. Retorts, alembics and other vessels with articulated shapes are by and large the most iconic component of any chymical laboratory, commonly found in early modern genre paintings as well as in woodcuts and illustrations of all periods (**fig. 3.4**) (**Principe and De Witt 2002**). The retort is a spherical vessel with a narrow and elongated neck that could be made in glass, ceramic or metal (**Moorehouse 1972; Booth 2016**). The liquid to be distilled was placed in the vessel and a gentle or moderate heat was applied. The rising vapour would condense at the top of the retort and the liquid was channelled along the neck into another vessel called receiver. Similar in shape to the retort, but open at the bottom, is the still head. It was the upper part of the so-called alembic (**fig. 3.4**) and was placed on top of a closed-profile vessel called cucurbit (or aludel), where the substances to be distilled were placed.

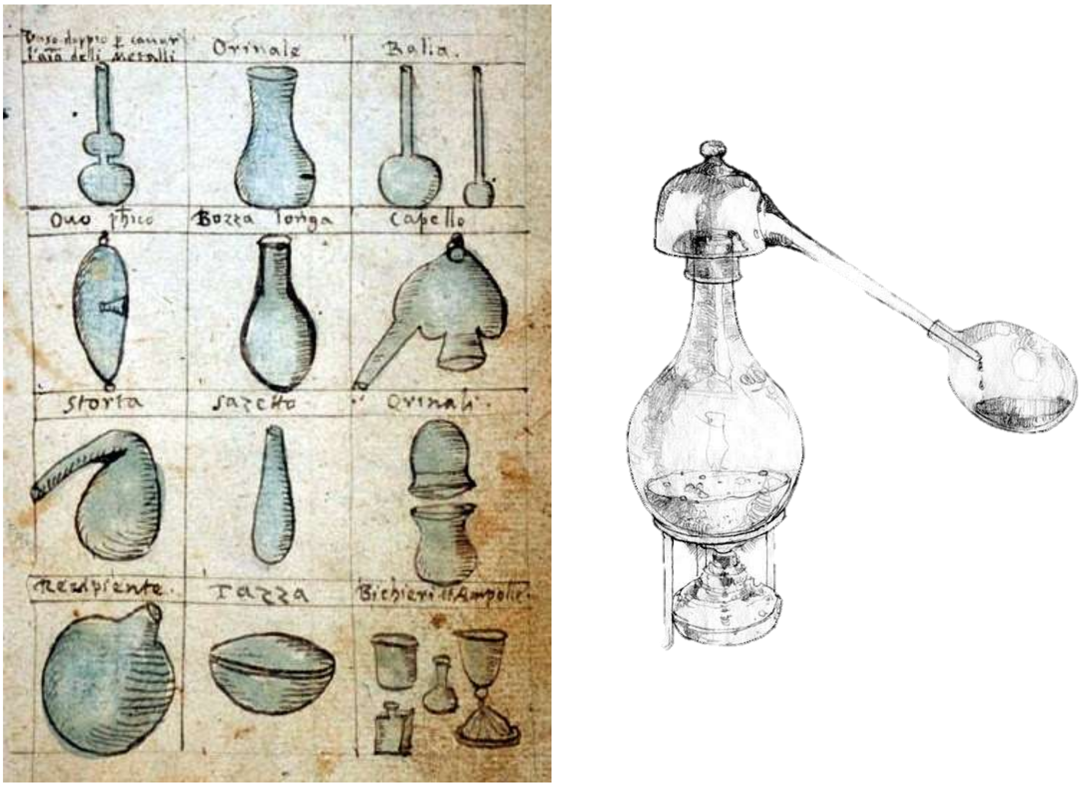


Figure 3.4. Left: Examples of glass distillation vessels from Antonio Neri's Tesoro del Mondo (1599); right: The alembic with its various compinents, namely the roubded cucurbit where the liquid is heated, the still head on placed on top of it and the receiver where the distilled liquid is collected.

In early modern times, distillation was employed to produce medicines, perfumes, alcoholic beverages and strong mineral acids by allowing a wide array of substances both organic and inorganic to evaporate and condense again. But distillation was also in incredibly powerful way of decomposing matter into its components and observing the effects, which indeed led to important new arguments in fields such as the corpuscular view of nature (**Newman and Principe 2002: 77; Newman 2006**). Glass constituted the preferred material for distillation apparatus since it allowed the chymists to literally keep an eye on the progress of the chemical reactions going on inside of the vessels. However, although the investigation of glass and other vitreous materials constitutes a major branch in the archaeometric literature, the specific study of chymistry-related glass apparatus has received virtually no attention. As noted before, this is in large part due to the lower number of findings in the archaeological record, partly because glass does not survive burial as well as pottery does, and partly because glass was normally recycled once it broke. In addition, distillation entailed using a much gentler heat than that needed in crucible operations, way below the point when glass would begin to melt and interact with the charge. As a consequence, glass distillation equipment does not generally contain a residue layer comparable to that in crucibles, even though the substances being distilled sometimes leave a deposit on the surface of the vessel (see, for instance **Moorehouse 1972: 95, 120**) (**fig. 3.5**). But other important questions can be successfully addressed through the chemical analysis of glass vessels, questions involving who produced them and using which ingredients, and how chymists sourced their vessels. Some initial steps in this direction will be presented in section 6.1.2, with the analysis of a small case study of glass apparatus that may hopefully lay the ground for future work (**Veronesi and Martinón-Torres 2018**).

On the other hand, distillation equipment made of ceramic was much more likely than glass to absorb the vapours produced during distillation and allow some deposit to adhere to the walls of the vessel (**fig. 3.5**). In some cases, the temperature and conditions in the vessels were enough to also produce an interaction layer between charge and ceramic akin to that found in crucibles (cfr section 5.5.3). And just like with crucibles, the analysis of similar residues and deposits in distillation vessels can yield precious information on what was being worked in the apparatus.

Non-specialised containers such as phials bottles and jars, again made of different materials, were employed to store ingredients and chemicals in various chymical contexts (**Hull 2003; Castillo Cardena 2014; Mass and Russow 2015**) (**fig. 3.6**). They were not normally subjected to high temperatures as crucibles and, to a lesser extent, distillation vessels were. Nonetheless, the substances they were holding were sometimes highly reactive, and information on their content may be obtained from these vessels too, either in the form of residues on the walls or as traces absorbed by the ceramic.

Figure 3.5. Residues left on chymical apparatus after distillation. Left: A reddish deposit can be seen on some of the glass vessels from the 16th-century laboratory of Oberstockstall (Austria); right: one of the tall crucibles from the Ashmolean laboratory, showing a white deposit rich in zinc.

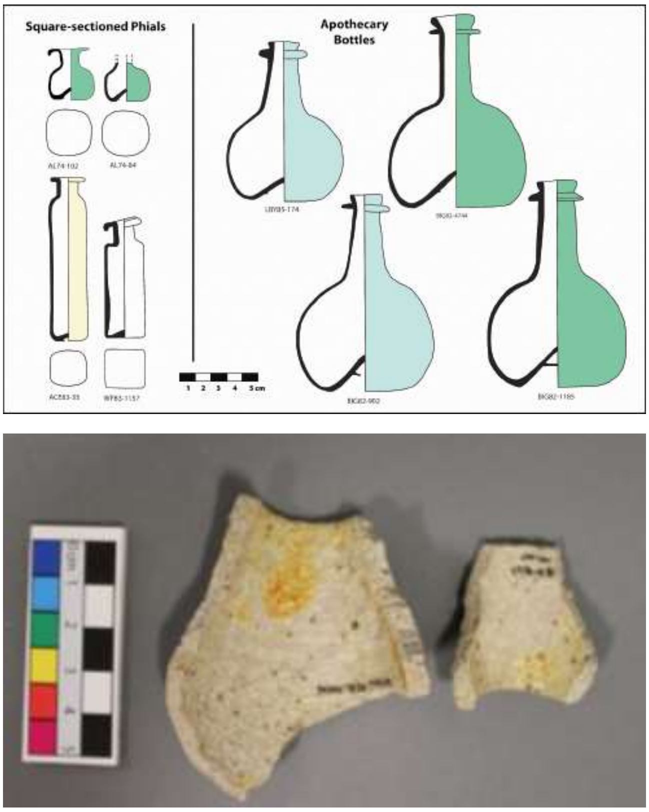


Figure 3.6. Left: Phials and apothecary bottles from 17th and 18th century London (After Castillo Cardena 2014); right: Fragment of jar from the Ashmolean laboratory showing an orange spot on its internal wall.

Another crucial tool of early modern chymical practice is the so-called cupel. Despite not as ubiquitous as crucibles and distillation vessels, cupels were essential in post-Medieval times during fire assay and refining activities and are usually found in specific chymical contexts such as mints and mining sites. In fire assay, the main function of cupels was to separate, by means of an oxidising reaction, the precious metals from the lead bullion into which they had previously been concentrated while processing the ore in the crucible. Like crucibles, cupels were specialised tools whose engineering was tuned for their purpose. Their porous body, made mostly of compacted bone ash, was meant to absorb the lead oxide (PbO) forming during the process without reacting with it, thereby leaving behind the unreacted noble metal/s as a small button on the surface (**Rehren and Klappauf 1995; Rehren 1996, 1998; Martinón-Torres et al 2008b**) (**fig. 3.7**). This way, by knowing how much ore had initially entered the crucible and by weighing the desired metal after cupellation, chymists could calculate the richness of a mineral vein, or assess the quality of coinage or other metal.

Like what was seen with crucibles, not all cupels were of the same quality. While the best ones were made of pure bone ash only, some contained additives such as wood ashes and clay and this is reflected in both contemporary recipes (**Hoover and Hoover 1950: 228-231; Sisco and Smith 1951: 27-30; Smith and Gnudi 2015: 137** and from analytical investigation of archaeological samples (**Rehren and Klappauf 1995; Martinón-Torres et al 2008b; 2009; White 2010; Ulseth et al 2015; Hsu and Martinón-Torres 2019**). Additives would inevitably bring impurities with them and that would increase the chances of unwanted reactions with the lead oxide. Silica (SiO_2), for instance, is especially detrimental in cupels because it readily reacts with lead oxide to form a lead-rich glaze (**fig. 3.7**). This would have prevented some of the lead oxide from being absorbed by the thick body of the cupel, and potentially hindered the precision of the process. The chemical composition of cupels tells us what the vessel was made of, what it was used for and indicates the presence of any such impurities. Because of the extremely perishable nature of bone ash, very few examples of unused cupels have been discovered in the archaeological record (but see **Rehren 1998**), while those that we can analyse are heavily contaminated by lead oxide and any other impurities. Thus, pure bone ash cupels are expected to contain almost only lime (CaO) and phosphate (P_2O_5), plus the lead oxide absorbed from the bullion. Small quantities of other oxidised base metals, associated with the source of lead, gold or silver, can also be found, such as copper (CuO), antimony (Sb_2O_3) or bismuth (Bi_2O_3); any other elements that are detected are likely to come from additives to the initial cupel body. Thus, by subtracting the lead oxide and re-normalising the data one can assess the initial components of a cupel and its degree of technical quality. Furthermore, small quantities of precious metals often end up in the cupel matrix and remain lost at the end of the process, with higher amounts indicating a less successful operation in which some of the gold or silver ended up non accounted for in the final bullion.

Figure 3.7. Left: A set of bone ash cupels from the Tower of London (after White 2010); right: Experimental replica of a cupel showing the silver button in the centre. Note the heavy interaction between the lead oxide and the cupel which produced the green lead silicate glaze (after Moureau and Thomas 2016).

3.2 SAMPLING OF THE ARTEFACTS AND ANALYTICAL METHODOLOGY

When selecting archaeological materials for laboratory analysis a multitude of factors may drive and constrain the choice of samples to be taken. The sampling strategy followed here aimed at obtaining what are deemed the most informative samples while minimising invasiveness, destructiveness and general disturbance within the assemblage. A technique is usually considered invasive if it requires a sample to be taken from the objects, and destructive it this sample is consumed during analysis, e.g. through dissolution in acid etc. Invasive sampling has the benefit that a sample taken from the object is retained and can be re-analysed more than once. This is the case of microscopy and microanalytical techniques such as those employed here (cfr section 3.2.3). There are also non-destructive and non-invasive techniques where the objects (or fragment) do not need to undergo further sampling and where the impact of the analysis is so minimal that it is considered virtually non-existent. One example discussed in this thesis is that of portable XRF, for which other constraints apply (cfr section 3.2.1). Another technique-related constraint (for destructive or invasive techniques) involves the kind of sample one needs. Indeed, some instruments require fine powders that may be compressed into pellets or further treated with reagents, while for other analytical techniques, such as most microscopy-based ones, a cross-section of the object is needed.

Other factors affecting the sampling strategy can be linked back to either the need of minimising disturbance to the object or assemblage and that of obtaining the most information out of the materials analysed. The former has to do with issues of object integrity, and invasive sampling need to consider this (**Rehren 2002**). It follows that in some cases only very small samples can be taken, or only from specific areas of the object, or only from those objects that are already fragmentary as opposed to the whole ones. Sampling is heavily dependent upon the research questions and for this reason the analyst will try and target specific parts of an object that may more effectively help answer those questions. In the case of the present thesis, since the attention was directed towards the contents of the chymical apparatus more than at the apparatus itself the sampling specifically targeted the residue layers present inside of the crucibles and other vessels. When this is a possibility, sampling of large assemblages may be guided by an initial screening by means of a portable XRF instrument, as it was done in the case of the Ashmolean Museum (cfr section 5.4). Chapters 4 and 5 will address the relevant sampling constraints and strategies adopted in the two case studies, detailing which factors had the most influence and how samples were taken. The remainder of this chapter outlines the analytical techniques used in the present thesis and details regarding sample preparation and protocols followed during analyses.

3.2.1 pXRF

A Portable X-Ray Fluorescence spectrometer (pXRF) is an analytical instrument “about the size, weight, and shape as a cordless drill” (**Frahm 2013a: 1080**). It is not to be confused with the bench-top XRF, which is portable only in the sense that it can be dismounted and carried around, but still needs to be operated on a support. Benchtop machines are also used widely in archaeology, particular in museum collections (**Craig et al 2007**). PXRF enables mass-sampling of large assemblages quickly and at a much lower cost compared to other instruments. It allows overcoming legal issues concerning the moving of objects from museums or deposits and the export of samples to foreign countries for analysis, and permits the analysis in situ in a complete non-destructive way. It requires no sample preparation (but samples may be treated before analysis if desired), and it can (and should) be brought directly to the field where it holds the potential to guide surveys and excavation in real-time (**Frahm et al. 2014**). These characteristics make pXRF a very useful tool to be used in fieldwork to rapidly assess finds or soil, as well as a very convenient sorting tool for sample selection. However, and despite the fast developments, this technique also has important limitations when it comes to the data produced. The lower voltage of a pXRF is not enough to generate the amount of energy needed to excite and displace the inner electrons of elements with high atomic number (**Shackley 2011: 17**) while some lighter elements (generally up to aluminium) whose x-rays are absorbed in the air path are also impossible to detect or have rather high detection limits. Even though these limitations can be at least partially overcome using newer instruments and under the right operational conditions (**Goren et al 2011; Forster and Grave 2012; Kennedy et al 2013**), pXRF is generally more common and more effective in the analysis of materials such as metals and obsidian (**Martinón-Torres et al 2012; Kellett et al 2013; Frahm 2014a; 2014b**) over others (e.g. ceramics and glass), where light elements make up too much of the composition. Other debated issues involve whether or not pXRF can be considered a fully quantitative method, especially when its accuracy and comparability with other established techniques is taken into account. In general, the need for a methodological protocol for the calibration of the machine is the main concern of some scholars as well as sources of scepticism towards many pXRF studies (**Shackley 2011; 2012; Speakman and Shackley 2013**). Similar concerns do not apply in the present case, where pXRF was only employed for guiding the sample selection and presence vs absence of certain elements was enough information.

Figure 3.8. The pXRF used for the present work during the initial screening at the Ashmolean Museum (cfr section 5.4).

All pXRF analyses were carried out on site in the conservation department of the Ashmolean Museum over two consecutive days (**fig. 3.8**). The machine used was an Olympus Innov-X Delta Handheld XRF analyser employing the so-called “Soils” factory-set calibration, which operates a Compton-normalised algorithm optimised for the detection of minor and trace elements in silica-based matrices. The “Soils” mode operates with three different consecutive beam settings, for a total of sixty seconds live time. Beams 1 and 2 work at 40kV accelerating voltage, better for measuring heavy elements. Beam 1 targets Sr, Zr, Mo, Ag, Cd, Sn, Sb as primary elements, as well as Ti, Ba, Cr, Mn, Fe, Co, Ni, Cu, Zn, Hg, As, Se, Pb, Rb and LE as secondary elements. Beam 2 targets Fe, Co, Ni, Cu, Zn, Hg, As, Se, Pb, Rb as primary and Ti, Ba, Cr, Mn, Sr, Zr, Mo, LE, Ag, Cd, Sn and Sb as secondary elements. Finally, the accelerating voltage of beam 3 is set at 15kV, which allows to detect lighter elements, and the beam targets P, S, Cl, K, Ca, Ti, Ba, Cr, Mn as primary elements and then Fe and LE as secondary elements.

Different areas of each artefact were targeted with no surface preparation, usually pointing at any visible residue as well as a cleaner spot on the ceramic for comparison.

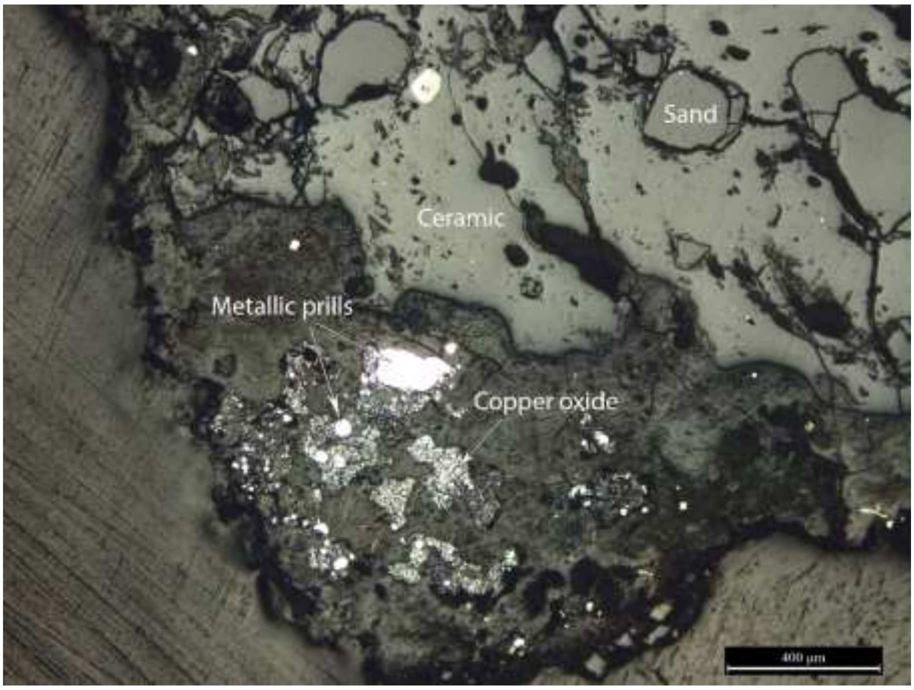
Where possible, flat areas were analysed, but in several circumstances the instrument had to be pointed at concave areas as well, as in the case of the interior of most crucibles. No certified reference materials were analysed to monitor data quality and no further calibration was performed during the analytical sessions except an internal calibration of the machine through a cobalt standard. Thus, the results are to be considered qualitative only and mainly aimed at confirming the presence or the absence of certain elements of interests in the objects that were analysed. As mentioned before, the pXRF screening was performed in order to inform the selection of samples that would undergo further chemical analysis as well as to provide some rough guidance during the successive electron microscopy investigation. Once the samples were taken and appropriately recorded through photos and measurements they were prepared for further analytical investigation.

3.2.2 Macroscopic assessment, sample preparation and Optical Microscopy

Some important preliminary information regarding the samples chosen for microanalysis can be obtained through the visual examination of macroscopic features. When dealing with ceramic crucibles and containers, as in the case of most samples here, size is one such feature. Specifically, vessels of small size are indicative of small-scale operations such as quantification and refining of precious metals where the need for closely controlled parameters is of high importance. As it will be shown in the relevant chapters, small vessels of this type are abundant at the sites of Jamestown and the Old Ashmolean Museum. In a similar fashion, vessels with a closed profile point to chymical operations that required specific conditions in order to be successful, a markedly reducing atmosphere for instance, or a closed environment which would not let gases escape (cfr section 5.5.3). A macroscopic assessment of the samples is also apt to provide us with some preliminary information regarding some of the ingredients and reagents that made up the crucible’s charge, as well as details on the kind of chemical reactions these underwent during the high-temperature processes. The macroscopically visible effects caused by these processes include, for instance, a particularly intense distortion or melting of the ceramic body, an indicator of extreme temperatures and/or of highly reactive and aggressive chemicals being worked in the crucibles (**fig. 3.9**). Equally, slaggy layers adhering to the vessels' walls may vary in thickness, texture and colour and their aspect tell us something about the components responsible for their formation. Surface discolouration is perhaps the most immediate clue as to charge ingredients, as in the case of the characteristic green corrosion colour usually visible in crucibles and other technical ceramics that have been used to melt, smelt or alloy copper (**fig. 3.9**). Chapters 4 and 5 abound in instances where initial interpretations are formulated on the basis of macroscopic assessment, and the reader is redirected there for further examples.

Figure 3.9. Crucible fragments with evidence of interaction between ceramic and charge. Left: A fragment from the Ashmolean laboratory (AN1999.205.31) showing intense distortion of the ceramic and a greenish discolouration due to the corrosive attack of alkalis. Right: Fragment from Jamestown (JR 2361D) with green spots on its internal surface corresponding to corroded brass prills.

Cross-sections of the selected ceramic bodies and residue layers were cut, mounted in epoxy resin blocks which were successively ground and polished to a flat, mirror-like surface down to 1µm following standard procedures. These were now ready for reflected light optical microscopy (OM). The main aim of this technique, which allows to look at samples under relatively high magnification, was to obtain an initial understanding of the most relevant microstructural features of each sample. The advantage of an optical microscope is its speed, which allows to process a large number of blocks in a relatively short amount of time, and to distinguish quickly between metallic, sulfidic, oxidic or silicate components based on reflectivity, colour and opacity (**fig. 3.10**). For instance, in cross-polarised mode, OM allows to see the colours of the phases, thus helping to distinguish, for instance, between different phases which may appear similar in plain reflected light, such as copper sulfide and copper oxide. This way, any relevant anomalies or areas of particular interest could be noted down and recorded, creating informed expectations for the successive microanalysis. The instrument used was a Leica DM4500 Led microscope with a Leica DFC290 HD camera attached.



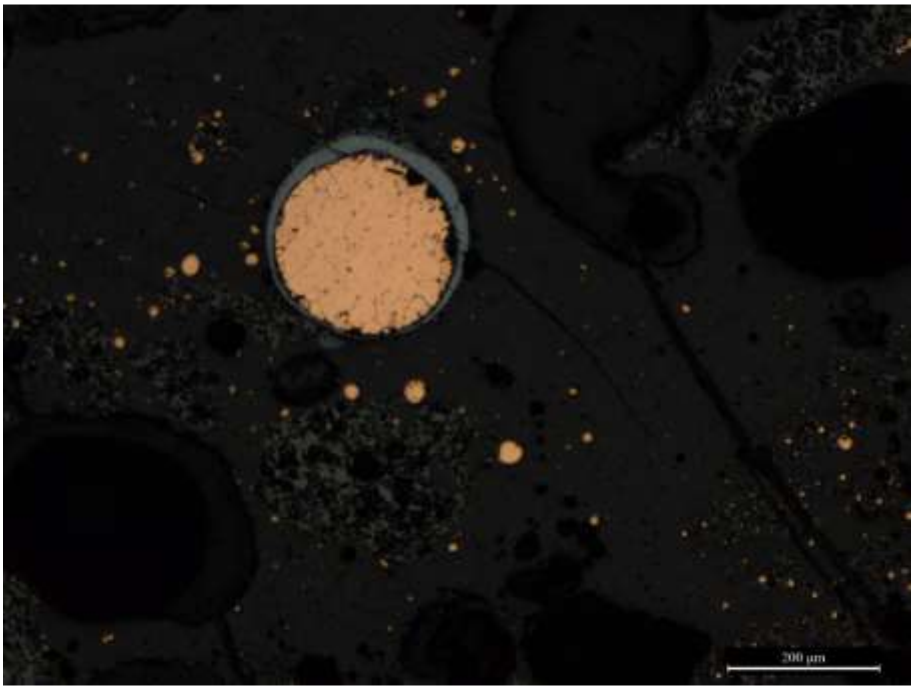


Figure 3.10. Copper-rich prills under the optical microscope. In plain reflected light (top, sample JR1400B II) the metal appears bright white, while the area of copper oxide is less bright. Different shades of grey characterise other oxidic or silicate phases such as the ceramic and the sand grains. In the cross-polarised light mode (bottom, sample JR965B), prills show the typical orange colour of copper, while the copper sulfide shell surrounding one of the prills appears light grey.

3.2.3 SEM-EDS

After optical microscopy analysis the blocks were carbon-coated prior to analysis with scanning electron microscopy (SEM) with energy dispersive spectrometry (EDS) operated under high-vacuum conditions. This instrument has the advantage of combining the investigation into the bulk chemistry of the sample with the analysis of discrete microstructural phases thanks to high-magnification imaging and point or small-area analysis, therefore making it possible to analyse, in the case of crucibles and other laboratory tools, the ceramic and the residues separately, and even individual particles or inclusions within them (**fig. 3.3**).

All analyses were performed in the Wolfson Archaeological Science Laboratories at the UCL Institute of Archaeology using a Hitachi S-3400 with an Oxford Instruments X-sight energy dispersive spectrometer. 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 µm across, avoiding large inclusions and metallic areas. For the ceramic, the matrix was analysed at high magnification, also avoiding larger inclusions such as quartz or grog temper (**Freestone and Tite 1986; Martinón-Torres and Rehren 2009**). Spot analysis of discrete phases was performed at higher magnification on smaller areas of variable size. Images were obtained in backscattered electron (BSE) mode, that allows to detect compositional differences across the samples indicated by different shades of grey. Results were processed using INCA Oxford Instruments software and are reported in wt% of oxides for ceramic and glassy matrices and for mineral phases, with the exception of chlorine which is always presented as elemental wt%, as are metallic phases. Bulk areas with comparable microstructure were analysed three to five times and the average calculated, whereas discrete phases were normally analysed once (full results in **Appendices B-C**). In order to account for corrosion, porosity and to facilitate comparability, data is shown normalised to 100 wt%. Internal quantification calibration was optimised through the repeated analysis of a cobalt standard at intervals of around thirty minutes, while certified reference standards were routinely analysed during the course of the analyses in order to monitor the quality of the analytical data. Selected standards include USGS BHVO-2, a basalt standard useful for assessing the accuracy for major and minor elements of ceramic materials, Corning glass standards A, C and D covering a wide spectrum of glass compositions, and alloys CURM 42.23.02, AgA2 and AgA3 for assessing the accuracy of elements such as silver, tin, copper and zinc. Data shows that quantification was normally within 10% error relative to the recommended value for most major and minor elements in the case of basalt and glass standards. Data is less reliable for the elements lead and tin in low concentrations (less than 2 wt% and less than 1 wt% respectively) as analysed in silver standard AgA3, and generally for heavier elements in low concentrations. Full results on certified standards are presented in **Appendix A**.

Finally, besides simple bivariate plots, the SEM data was used to generate the cluster dendrogram of crucible compositions shown in figure 4.9, by means of the statistical analysis software R. The groups were initially separated through hierarchical cluster analysis in combination with principal component analysis. The dendrogram was obtained using Ward's method as clustering algorithm and the oxides selected were restricted to the seven main ones, detected in all samples analysed, namely Na_2O, MgO, Al_2O_3, SiO_2, K_2O, CaO, TiO_2, FeO.

CHAPTER IV

JAMESTOWN

Figure 4.1. The island and the river James

4.1 INTRODUCTION: A LAND OF HOPE

This chapter explores the first of the two case studies covered in the present thesis, namely Jamestown in Virginia, the first permanent English settlement in the new world. Its foundation in 1607 followed decades of unsuccessful attempts at establishing colonies in the northern part of the American continent. These include Martin Frobisher's (1535-1594) 1570s expeditions in the Canadian arctic in search of the northwest passage (**Hogarth 1999; Beaudoin and Auger 2004**), and, in the following decade, the infamous lost colony of Roanoke, in modern-day North Carolina (**Nöel-Hume 1994; Luccketti 1997**). The chosen place for the new settlement was a small island up the river James, not far from its mouth and the Chesapeake Bay (**fig. 4.1**). Until the foundation of Williamsburg in 1632, Jamestown remained the main British settlement in the new world (**Kelso 2017**). The colonial expedition had been sponsored by the Virginia Company of London, established shortly before for the purpose. The Company had been granted a royal charter for the exploitation of the resources to be found in Virginia, and its shareholders had invested their capital in the hope of profiting from the venture. Indeed, the colony prospected the promise of great wealth which would derive from the new industrial and trade opportunities and particularly from the discovery of mineral resources. Gold and silver were understandably at the top of the list, but other desired resources included zinc and tin minerals to be employed in copper metallurgy, where several of the Company's members had economic interests (**Hudgins et al 2009; Straube 2013**). Besides metallurgical aspirations the shareholders also expected to set up new trade routes towards the Pacific Ocean and the markets of East Asia through the discovery of the northwest passage. The very same ambitions had been the driving forces behind earlier colonial attempts made by the Europeans in north America which, despite the ultimately unsuccessful outcome, had nonetheless set up a model for the subsequent English colonisation of Virginia (**Heuvel 2007**). Besides the previously noted English exploration voyages in Canada and North Carolina, other 16th-century colonisation efforts include the first French settlements in Quebec (**Cote 2009; Fiset and Samson**

2009). In both instances, the settlers had brought along a host of specialised apparatus and trained professionals who would perform tests on the mineral resources of the area, and target potential mining sites where gold and silver could be found. Of these activities we know relatively little. For Jamestown, written sources are for the most part chronicles of the events surrounding the early phase of the fort, as exemplified by the writings of captain John Smith, the first man in charge of the colony (**Smith 1907**). Captain Smith's pages very well reflect the initial sense of excitement and the great hope underpinning the colonisation efforts:

> The mildnesse of the ayre, the fertilitie of the soyle, and situation of the rivers are so propitious to the nature and use of man, as no place is more convenient for pleasure, profit, and mans sustenance, under that latitude or climat. (**Smith 1907: 60**)

Virginia must have looked like a promised land with seemingly endless resources, and Smith's list of the many goods England was forced to import from other European countries reveals the desire to become more independent on the market:

> Muscovia and Polonia doe yearely receive many thousands, for pitch, tarre, sope-ashes, Rosen, Flax, Cordage, Sturgeon, Masts, Yards, Wainscot, Firres, Glasse, and such like; also Swethland for Iron and Copper. France in like manner, for Wine, Canvas, and Salt. Spaine as much for Iron, Steele, Figges, Reasons and Sackes. Italy with Silkes and Velvets consumes our chiefe Commodities, Holland maintaines it selfe by fishing and trading at our owne doores. (**Smith 1907 61**)

From captain Smith we also know the name and profession of many of the settlers who arrived at Jamestown in the spring of 1607, as well as of those who came with the first supply ships the following year. Smith mentions specialised workmen practicing various professions such as carpenters, masons, tailors, apothecaries, blacksmiths, goldsmiths, assayers, refiners and glassmakers (**Smith 1907: 90-91**). The list indicates that mineral prospection and the quest for noble metals was not the only industrial activity to be carried out at the fort. In fact, the Virginia Company hoped to start a large-scale production of glass, a much-desired commodity at the time, and another which England had to import heavily from the continent. The new land of Virginia and its pristine woodlands offered the rare possibility of producing glass without the restrictive forestry legislation that at the time were in place in England (**Crossley 1972; 1998**). German glassmakers had been sent for the purpose in 1608 (**Harrington 1972**) and in a matter of weeks had produced a first trials of "Pitch, Tarre, Glasse, Frankincense, Sope Ashes" (**Smith 1907: 147**). Nothing is known about the fate of this locally-produced glass as we have no in-depth account of the chymical operations of the fort's early years. However, we do know that little was produced and that the settlers eventually failed in starting a large-scale glass and mining industry, and this has led to the widespread negative opinion of the colonists as lazy and incompetent men (**Morgan 1975**). One of the aspects that written accounts report in greater detail are the many hardships the colony had to sustain throughout the early years, when the settlers were decimated by the harsh climate, diseases, the hostility of the native populations and by the "starving time" of the years 1609-1610. George Percy, one of the original members of the Jamestown council and the author of another chronicle of these early months, provides a long list of men who died during the first summer (**Tyler 1907:20-22**). Ultimately, documents give us a clear sense of how to the explorers who first arrived in the Chesapeake Bay, Virginia had appeared as a land that promised a bright future and great industrial expansion and of how such optimistic expectations soon had to confront the reality of an unknown and often overwhelming new world, and of the enormous difficulties encountered as a consequence. In 1610, just when the few survivors were preparing to abandon the colony more supply ships arrived to Jamestown and the fort was re-established. Nonetheless, this second phase did not differ much from the early one, with frequent Indian attacks throughout the 1620s and a second "starving time" in 1622-23. This eventually led to the Virginia Company losing the royal charter and to Jamestown becoming a colony of the crown in 1624.

4.2 ARCHAEOLOGICAL BACKGROUND

While the documents provide us with a good amount of information on the timeline of events surrounding the creation and the life of Jamestown in its initial years, very little can be found about practical aspects of the artisanal activities carried out at the fort. This is hardly surprising if we consider how rapidly the situation must have escalated into trouble and consequently how the amount of time and energies devoted to such workshop activities must have been constantly diminishing. An example in this sense is offered by the story of the German glassmakers who, not long after their arrival and their initial work, decided it was more desirable to smuggle weapons for the Indians in exchange for food since “there was no time and little incentive to make glass” (**Kelso and Straube 2000: 66**).

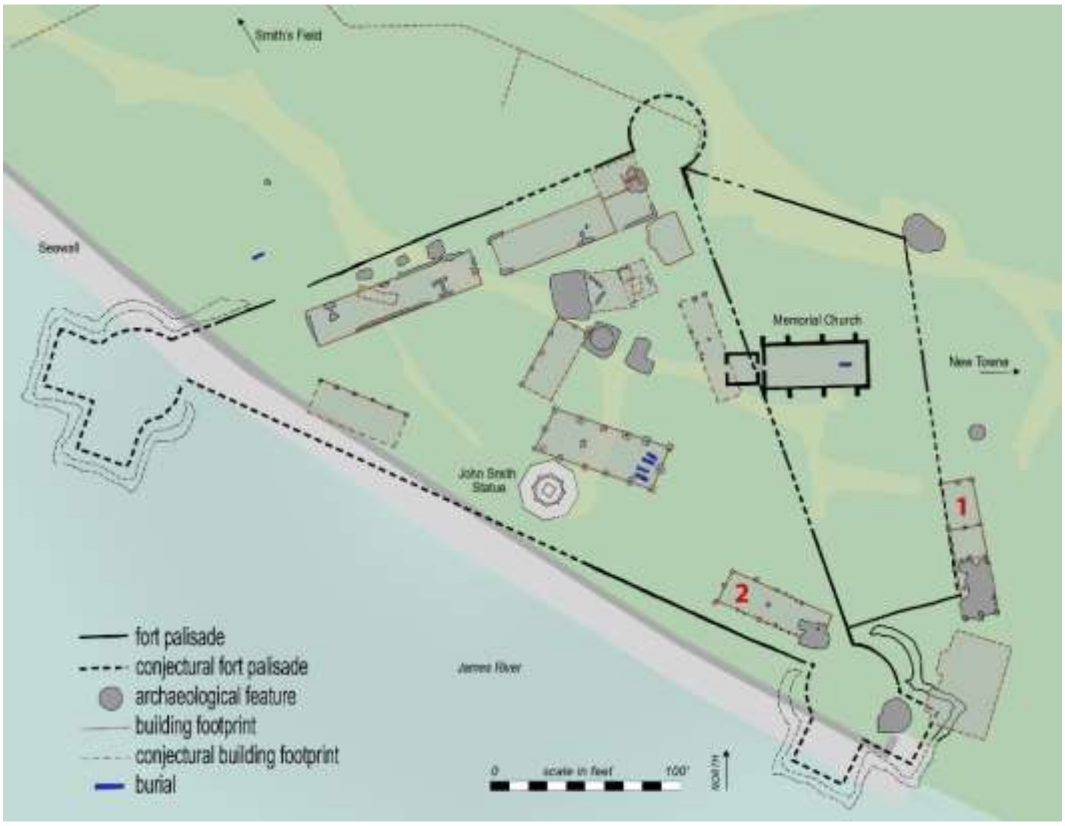


Figure 4.2. Map of the excavated fort (after Jamestown Rediscovery). Number 1 indicates the factory outside of the fort walls, number 2 indicates the barracks.

However, since 1994 the archaeologists of the Jamestown Rediscovery Project have been excavating the site of the early James Fort, and the ongoing archaeological work is enormously enriching our knowledge of the colony's life. The exact location of the fort had been a debated issue for a long time, and until its discovery it was largely believed that it must have disappeared with changes in water levels. However, many of the structures belonging to the original triangular fort and to later additions have been brought to light by the archaeologists. These include part of the walls and defensive constructions, the church, the barracks, the storage facilities and various other buildings (**Kelso 2017: 46-117**) (**fig. 4.2**). Thousands of artefacts have been unearthed during the over twenty-five years of digging, providing us with a comprehensive view of many aspects of colonial life and with the stories of the people who inhabited it. Among the materials recovered are abundant traces that can be linked to the metallurgical and, more generally, to the artisanal efforts of the colony. Ceramic crucibles of various sizes, mostly triangular but also of the beaker-type, fragments of bone ash cupels and other specialised ceramic vessels, pieces of distillation apparatus made of glass and pottery and bits of ores and glass cullet are examples of such finds (**fig. 4.3**) (**Kelso and Straube 2000: 33-66; Kelso and Straube 2004: 157-161; Hudgins 2005; Martinón-Torres and Rehren 2007; Hudgins et al 2009; Owen et al 2014**). They bear us witness to the work of metallurgists, assayers, refiners, glassmakers and apothecaries operating in the fort during the early phases of occupation, and represent an invaluable source of information on the great variety of individuals and activities that must have characterised the colonial venture (**Kelso 2017: 143-164**). The vast majority of the vessels display slagged walls and the melting/discolouration of the ceramic surfaces, which indicate that they were used in high-temperatures operations and that chemical reactions went on in them. Many of the chymistry-related artefacts were found within the context of a large dump layer inside of what archaeologists have labelled "the factory", a long building situated at the perimeter of the fort (**Kelso 2017: 92-101**).[13] The location of the factory just outside of the fort walls and the types of

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[13] Further information on the factory, as well as on other contexts, can be found at: https://historicjamestowne.org/archaeology/map-of-discoveries/the-factory/ (last accessed May 2020)

artefacts found in it, including glass beads and pieces of scrap copper used to commerce with the Indians, point to its use as trading centre. The crucibles, alembic, distilling flasks and distilling dishes suggest that the building was also the site of metallurgical operations of testing and precious metals refining carried out in the brick hearths also discovered in one of the rooms.

Figure 4.3. The assemblage of chymistry-related materials from Jamestown. Top row: ceramic crucibles of the Hessian (left) and non-Hessian (right) types; mid row: two cupels (left) and distillation equipment (right); bottom row: glass cullet (left) and bits of stibnite (Sb2S3) (right) (photos by Marcos Martinón-Torres).

More triangular crucibles and the assemblage of glass cullet were recovered from a pit/trash layer pertaining to the "barracks", another long building to be found in close proximity to the east bulwark (**fig. 4.2**) (**Kelso 2017: 80-89**). Finally, archaeologists also recovered thousands of small copper offcuts and sheets, most likely representing a reservoir of scrap metal to be employed as reagent in workshop operations as well as for the everyday needs of the settlement (**Hudgins 2005**). Some of the materials mentioned here have been the subject of archaeometric analyses in the past years. In 2007, Marcos Martinón-Torres and Thilo Rehren have carried out a pilot study on a small selection of triangular crucible fragments. They studied the samples using a scanning electron microscope and obtained some insight into the metallurgical processes that the crucibles had been used for (**Martinón-Torres and Rehren 2007**). The vessels were found to be German imports from the region of Hesse, which in this period produced the most technically-advanced ceramic laboratory equipment in the world (cfr 4.5.1) (**Cotter 1992; Martinón-Torres and Rehren 2009**). The analysis of the slaggy residues revealed the presence of copper, zinc, iron and tin, plus a multitude of other reagents, and indicate that settlers were probably testing minerals with an eye to noble metals or to copper alloys. Two more papers have focused on remains related to copper metallurgy by way of analysing some of the pieces of scrap copper that were mentioned before (**Hudgins 2005; Hudgins et al 2009**). Analyses performed using inductively coupled-plasma atomic emission spectrometry (ICP-AES) allowed to detect the pattern of trace elements characterising the metal and to infer its European origin, thereby confirming that the copper had been brought along to be used at the fort. More recently, some broken pieces of glass cullet, fragments of glass objects and suspected primary glassmaking waste, including glass-on-crucibles, were investigated using laser ablation inductively coupled-plasma mass spectrometry (LA-ICP-MS) (**Owen et al 2014**). While most of the glass appears to be of European manufacture, it was hypothesised that the crucibles contain residues of the 1608 glassmaking trials made by the German experts using local ingredients. The history of glass production in Jamestown at this very early stage is still a rather unclear one (**Harrington 1972**).

Section 4.5.4.1 will address this in more detail and in light of the new discoveries presented here. Building on previous literature, the rest of this chapter will present new analytical data on the Jamestown assemblage deriving from the investigation of a larger set of crucibles used in chymical operations during the early years of Jamestown.

4.3 SITE-SPECIFIC RESEARCH QUESTIONS

The analysis of the two case studies in this and in the next chapter aim to address the research questions that were outlined in section 1.3. There are, however, other relevant lines of enquiry besides the broader themes that those questions raise, contributions that the analysis of a comprehensive assemblage can make to a better understanding of the history of the sites under investigation. Site-specific issues of this kind revolve around the history of chymical practice during the very early days of the Jamestown colony, as well as the place of the colony itself within the phenomenon of early modern transatlantic explorations. The following questions will therefore be investigated in this chapter:

1. What is the range of chymical operations that the settlers pursued in the initial period (1607-1610)?
2. What tools, ingredients and reagents were involved in Jamestown's chymistry?
3. What types of minerals were tested? What did the settlers hope to find? Most of the vessels appear to be related to metallurgical operations, and we know that metallurgists were sent to expeditions to find and assess the quality of minerals of the area.
4. We know from documentary evidence that glassmaking was an important activity during this period. Is this reflected in the assemblage? Are there remains that may be linked to the local production of glass? If so, what ingredients did the German glassmakers use in their workshop?

5. And with regard to both metallurgy and glassmaking, did chymists working at Jamestown appear to follow established European practices or did they instead deviate from codified knowledge of the period?

4.4 SAMPLE SELECTION AND MATERIALS ANALYSED

The initial selection of the artefacts to undergo invasive sampling and microanalysis was carried out in the storage room of the Jamestown Rediscovery Project by Marcos Martinón-Torres (**fig. 4.4**). As mentioned in section 4.2, the objects come from contexts relative to dumping events and they all pertain to the early phase of the fort, datable to the period 1607-1610.

Figure 4.4. The storage room of the Jamestown rediscovery Project (photo by Marcos Martinón-Torres)

The sampling targeted those materials that could be related more closely to industrial activities and focused on finds that were in a fragmentary state, so as to minimise any further damage to the assemblage. A few of the chosen samples became the object of the 2007 pilot study by Marcos Martinón-Torres and Thilo Rehren that was noted earlier (**Martinón-Torres and Rehren 2007**), while the majority were not investigated at the time and have been picked up for the present work. The assemblage comprises twenty-one fragments (**table 4.1**), whose individual description can be found in **Appendix B**. Seventeen samples belong to ceramic crucibles, but they are usually too small and undiagnostic to offer significant indication as to the original shape and size of the vessels. Some observations resulting from the visual inspection of the surface and section, and from SEM-EDS characterisation (cfr section 4.5.1) allow to establish that fifteen fragments come from Hessian crucibles. Only in one instance (A-630) does the fragment clearly indicates a different crucible type with circular body, tapering walls and a small bulge above the base. One very small fragment (JR741A) is of more unsure attribution, given the high degree of contamination between the ceramic and the content of the crucible. The base is present in four specimens only (A-630, 152-JR, JR1400B and JR1416C), with one (JR1400B) also preserving the spout, while the remaining samples are all undiagnostic wall fragments. Where bases do survive, the limited diameter that can be reconstructed indicates that they belong to small vessels, with a height that in all likelihood did not exceed 10 cm. The thickness of the walls is limited too and this also seems to point to small crucibles. The tendency towards small scale is further confirmed by the recovery of several entire or nearly entire triangular vessels, which were not sampled for the present project. Nonetheless, crucibles of larger dimensions were discovered too, but again none was analysed in neither previous studies nor in the present work. All fragments but one (J-crucibles) come from crucibles used in high-temperature furnace operations as suggested by the presence of slaggy layers stuck to the walls. To finish, there are four samples that don't belong to crucibles. Of these, one is a rounded lump of greenish slag (JR1115B), one is the fragment of a bone ash cupel (JR1957A II), another belongs to a piece of green flat glass (JR2G) and the last one is a small sample of a frothy glassy residue (JR2718N).

Sample ID	**Type**	**Part**
JR1957A I	Triangular crucible	Wall
JR392A	Triangular crucible	Wall
JR1339C	Triangular crucible	Wall
JR2436B	Triangular crucible	Wall
JR1545C	Triangular crucible	Wall
JR1400B I	Triangular crucible	Wall + base
JR1400B II	Triangular crucible	Wall + spout
JR1416C	Triangular crucible	Wall + base
JR965B	Triangular crucible	Wall
JR2361D	Triangular crucible	Wall
JR2718J	Triangular crucible	Wall
JR2718M	Triangular crucible	Wall
JR2718W	Triangular crucible	Wall
A-630	Non-triangular crucible	Wall + base
152-JR	Triangular crucible	Wall + base
JR741A	Crucible	Wall
J-crucibles	Triangular crucible	Wall
JR1957A II	Cupel	
JR2G	Glass cullet	
JR1115B	Slag	
JR2718N	Frothy residue	

Table 4.1. List of the samples analysed. Further details can be found in Appendix B.

4.5 RESULTS AND TECHNICAL INTERPRETATION

The twenty-one samples chosen were analysed with SEM-EDS in order to explore the chemical composition and microstructure of both the crucible and the residue layer. The analyses followed the analytical protocol presented in section 3.2.3. The results are in general agreement with the available information coming from the written accounts (cfr section 4.1), as metallurgy is by and large the most represented chymical activity. The data is also consistent with what was reported in the previous analytical campaigns mentioned above, and substantially expand on them by offering new insight into the range of processes carried out by Jamestown's specialists. In order to structure the complex emerging scenario of high-temperature chymical practice at Jamestown, the present section will report and discuss the results in relevant sub-sections according to the type of operations mirrored in the samples. The first aspect to be addressed is the data on the ceramic crucibles themselves, disregarding the residues within them (4.5.1). These are instead the focus of the following sub-sections, where the various enrichments and microstructural features detected are reported with reference to the metallurgy of noble metals (4.5.2) and that of copper (4.5.3) and copper alloys (4.5.3.1). A group of samples with less clear links to metallurgy is then explored (4.5.4) and their potential interpretation within the glassmaking efforts discussed (4.5.4.1). The last sub-section includes samples that, despite sharing some traits with the previous categories, could not be securely attributed to any (4.5.5). Due to the heterogeneity of the crucible residues and to the marked diversity encountered, each of these sections includes, besides the technical interpretation of the data, also a brief discussion grounded in the relevant technological context the results touch upon. A broader discussion follows in section 4.6, contextualising the emerging technological features within the larger picture of the archaeology and the chymistry of the colonial world.

4.5.1 THE CERAMIC CRUCIBLES

As briefly noted in passing, macroscopic and SEM-EDS characterisation of the crucibles confirm that out of the seventeen fragments under investigation, fifteen are consistent with the Hessian type. Where the walls are sufficiently free from distortion effects caused by the use it is possible to see the characteristic grainy look caused by the heavy sand tempering (**fig. 4.5**) (**Martinón-Torres and Rehren 2009**). In cross-section, the sand particles can be seen more clearly, together with the grey-white and strongly vitrified appearance of the ceramic, another typical indication of the Hessian type (**fig. 4.5**). The microstructure of these crucibles shows a fine-grained clay, elongated voids running parallel to the surface and tempering inclusions almost entirely made of round quartz particles varying in size from few tens up to 500 µm across (**fig. 4.5**). As observed macroscopically, the ceramic appears highly vitrified and homogeneous with a sharp crucible-residue boundary and very little interaction between the two (**fig. 4.5**).

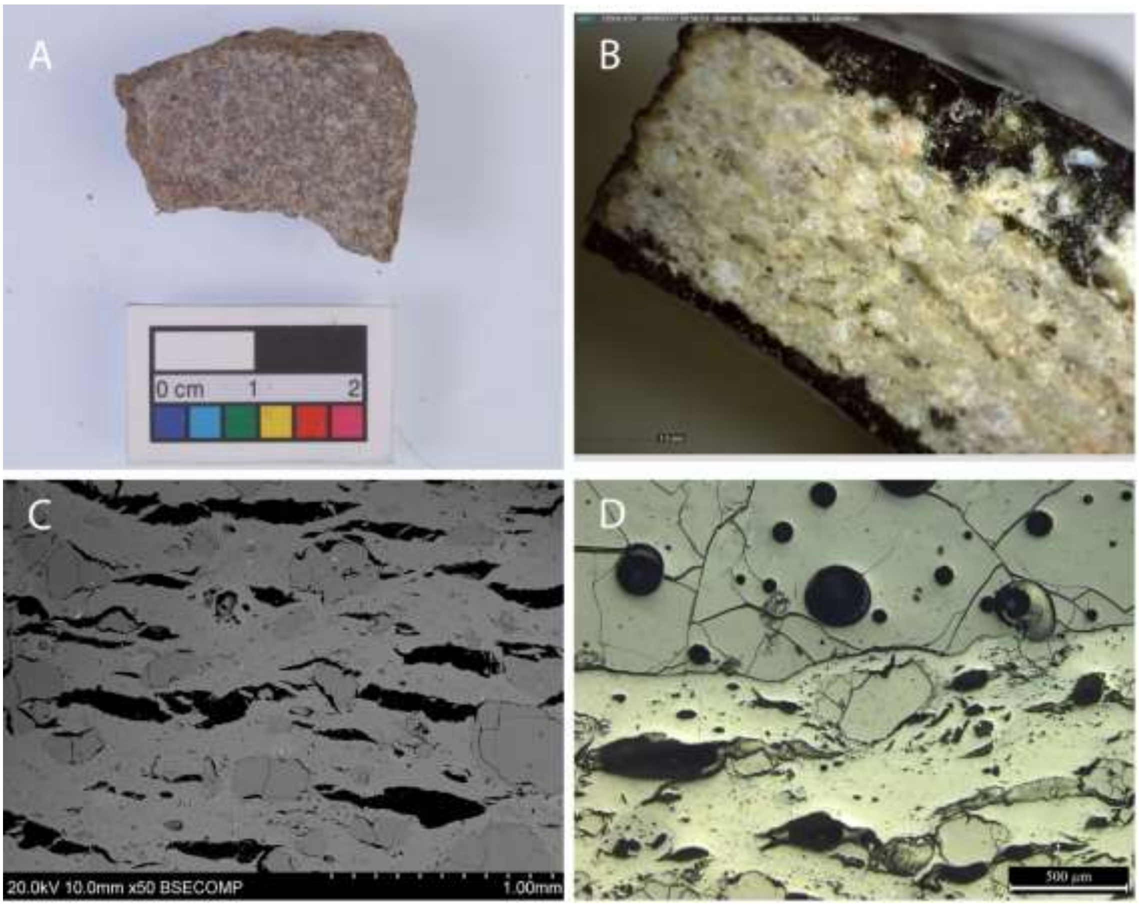


Figure 4.5. Hessian crucibles from Jamestown. The typical sandy texture of the ceramic can be seen both on the surface (A, sample JR2718M) and in section (B, sample JR1339C). Quartz inclusions and elongated voids can be seen in the microstructure at high magnification (C, sample J-crucibles, one of the outliers in figure 4.8), while the sharp boundary between the ceramic and the residue is shown in the optical microscopy image D (sample JR1957A I, one of the outliers in figure 4.8).

The chemical composition of the clay matrices (**table 4.2**) shows levels of alumina generally exceeding 35 wt% Al_2O_3, low total alkali and alkali earth oxides (generally below 4wt%) and iron oxide mostly between 1 and 2 wt%.

Sample	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	FeO
JR1957A I	0.7	0.5	39.3	54.2	1.5	0.3	1.9	1.4
JR392A	0.2	0.6	39.2	54.9	1.5	0.2	1.9	1.6
JR1339C	0.3	0.6	38.3	55.5	1.4	bdl	1.7	2.2
JR2436B	1.2	0.5	37.8	55.2	2.1	0.2	1.7	1.1
JR1545C	0.3	0.6	37.6	56.3	1.1	0.4	1.9	1.7
JR1400B I	0.3	0.6	39.3	55.0	1.2	0.3	1.8	1.6
JR1400B II	0.3	0.6	38.8	55.6	1.1	0.3	1.8	1.6
JR1416C	0.2	0.7	36.4	57.7	1.2	0.3	1.9	1.7
JR965B	0.3	0.6	36.8	57.6	1.1	0.4	1.8	1.6
JR2361D	bdl	0.6	38.3	55.7	1.5	0.2	1.8	1.9
JR2718J	0.2	0.6	38.9	55.1	1.4	0.3	1.8	1.8
JR2718M	bdl	0.6	38.9	55.8	1.1	0.3	1.8	1.6
JR2718W	0.2	0.6	37.2	56.7	1.6	0.4	1.9	1.5
A-630	0.2	0.5	23.1	70.6	1.8	0.4	1.9	1.6
152-JR	1.6	0.4	31.5	62.7	0.3	bdl	1.8	1.8
JR741A	0.5	1.0	25.4	64.6	3.2	0.2	1.2	4.0
J-crucibles	bdl	0.6	37.1	57.7	1.1	bdl	1.8	1.8

Table 4.2. Average chemical composition by SEM-EDS of the ceramic matrices of the fragments analysed.

These compositions are comparable with the data reported by Martinón-Torres and Rehren for the three crucibles analysed in the preliminary study (**Martinón-Torres and Rehren 2007**), and in every respect consistent with German crucibles discovered at several sites all over Europe and in the colonial world (**Martinón-Torres and Rehren 2009**). Four "Hessian" crucibles appear to deviate slightly from the main cluster in figure 4.8. The most likely reason is contamination from components of the charge during use, especially from aggressive alkali-rich reagents (cfr. section 5.5.4). The shifting Al_2O_3/SiO_2 ratios can also be explained as a result of ceramic distortion. That these samples come from German crucibles seems confirmed by their microstructure, with features such as the elongated voids parallel to the surface and the high degree of vitrification of the ceramic clearly comparable to that of Hessian vessels (**fig.4.5 and 4.6**).

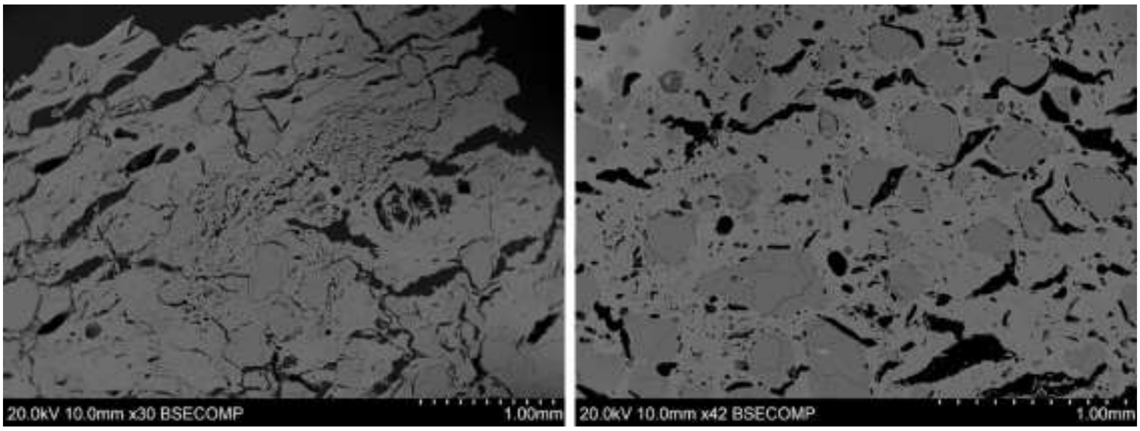


Figure 4.6. Hessian outliers 152-JR (left) and JR2436B (right) showing ceramic distortion due to use and causing minor differences in chemical composition when compared to the other Hessian samples. Note, however, the voids parallel to the surface of the vessel and the high degree of vitrification which, together with sand tempering, are typical of Hessian productions.

As briefly introduced in chapter 3, these crucibles owed their outstanding thermal refractoriness and resistance to chemical attack to the special kaolinitic clay they were made with, to the tempering technology and to the high-temperature pre-firing they underwent (**Martinón-Torres et al 2006; 2008a**). Kaolinitic clays are generally high in alumina and low in fluxing elements such as potash and iron oxide, and therefore ideal to produce heat resistant pots (**Martinón-Torres and Rehren 2014**). Tempering through the addition of sand further strengthened the crucibles as the expanding and shrinking of quartz during heating and cooling resulted in the formation of elongated voids that acted as a barrier or “firebreak” against the propagation of further and larger cracks. Finally, these crucibles were subjected to pre-firing at temperatures of over 1300C, which determined the formation of mullite, an aluminosilicate with excellent thermal and mechanical-shock resistance properties (**Martinón-Torres et al 2006; 2008a**).

In contrast, sample A-630 from the conical crucible described in section 4.4 shows significant differences with the Hessian types in both its macro and microscopic appearance, as well as in the chemical composition. First, the ceramic appears much more distorted externally, suggesting a lower level of refractoriness of the clay employed (**fig. 4.7**). As visible from the section, the vessel was tempered with sand (**fig. 4.7**), and high magnification images show that the quartz grains are heavily shattered due to the expansion and shrinkage of the body provoked by the intense heat (**fig. 4.7**). The microstructure also confirms the distortion of the ceramic matrix, showing a state of continuous melting and the development of circular bloating pores throughout.

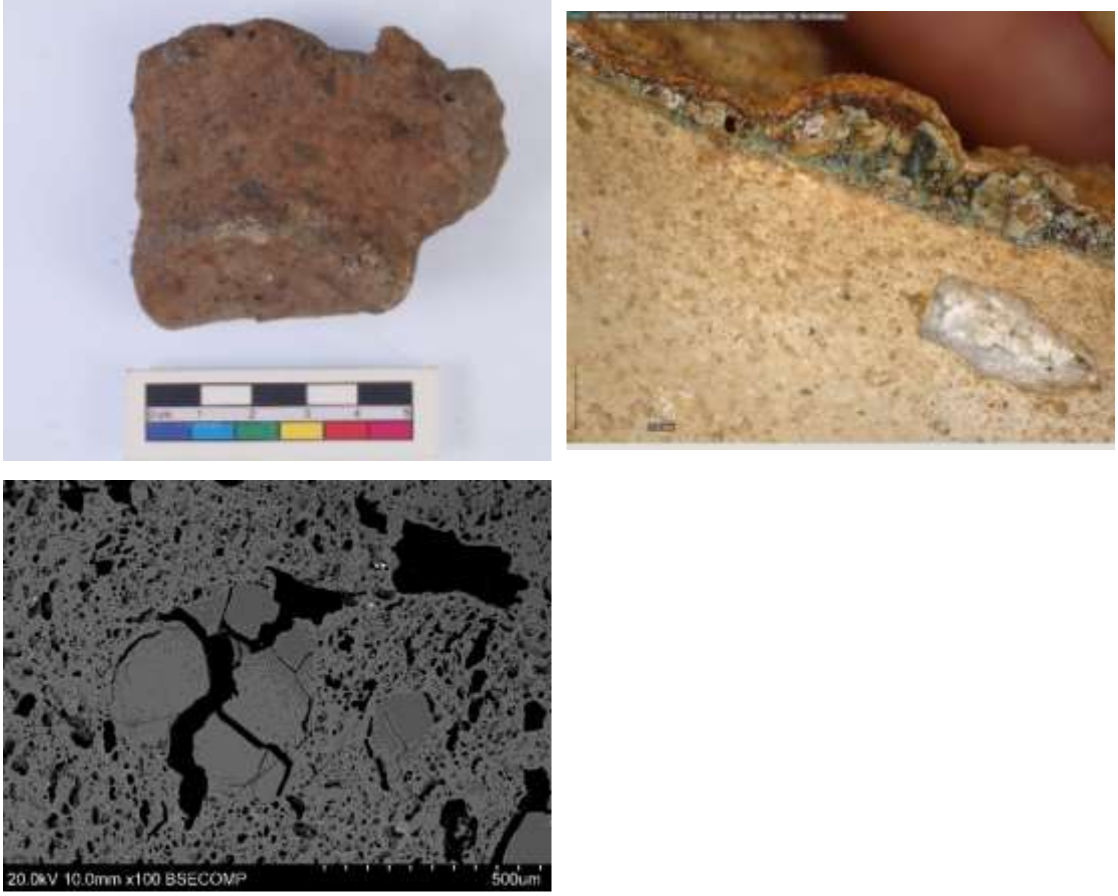


Figure 4.7. Sample A-630 from a non-Hessian crucible, showing molten external ceramic and sand grains in section (top). Note the sharp boundary between the ceramic and the slaggy residue indicating high refractoriness. At high magnification, shattered quartz and bloating pores can be seen in the ceramic matrix (bottom).

Figure 4.8. Round crucible from Cartier-Roberval, Quebec (after Monette 2013)

All of these features are in line with a lower degree of refractoriness when compared to the ones previously discussed. This is further confirmed by the chemical makeup, and particularly by the considerably lower levels of alumina of sample A-630 (23.1 wt% Al_2O_3). Nonetheless, the low amounts of fluxing elements (i.e. alkali and alkali earth oxides and iron oxides mainly) and the generally sharp boundary between crucible and slaggy residue are indicators of a rather refractory clay (**fig. 4.7**). Non-Hessian crucibles are relatively uncommon in Jamestown, but they represent the norm at other sites, like the 16th-century French colony of Cartier-Roberval, in Canada, where the vast majority of the crucibles discovered show a conical shape, a tapering profile and an externally bulging foot that closely resemble sample A-630 (**fig. 4.8**) (**Cote 2009; Martinón-Torres 2010**). These vessels were found to be of French manufacture, the analyses indicating that they most likely come from the pottery workshop of Louviers, a village not far from Rouen (**Martinón-Torres 2010; Monette 2013**). In England, non-triangular crucibles of various kinds were found at several post-Medieval sites related to chymical activities, such as glass houses (refs **Crossley 1967; Ashrurst 1970; 1987; Vose 1994; Welch 1997; Jackson et al 2005; Dungworth 2006; 2007; Blakelock 2007; Dungworth et al 2013**) metallurgical workshops (**White and Kearns 2010**) and at the laboratory of the Ashmolean Museum in Oxford (cfr chapter 5; **Martinón-Torres 2012a**). The documents do not provide relevant clues as to where A-630 and the other non-Hessian crucibles used in Jamestown may come from. Equally, a comparison with the chemical composition of the other assemblages is not particularly illuminating either, since there don't seem to be well-defined geographical groups as far as major elements are concerned (**fig. 4.9**). Sample A-630 (dark red dot in **fig. 4.9**) appears to sit in an area which is in between the French compositions from Quebec and the Louviers and the English crucibles from various post-Medieval sites. The same scenario emerges from taking into account all major and minor elements. In the cluster dendrogram of **figure 4.10** samples are arranged in branches according to compositional proximity, the closer two samples the more similar their chemical signature. As we can see, none of the samples around A-630 form tight clusters but instead several smaller branches are present, indicating similar but not identical compositions. In this less homogenous area, A-630 can be found in proximity to both French ("CR" IDs) and English ("LM" and site name IDs) crucibles.

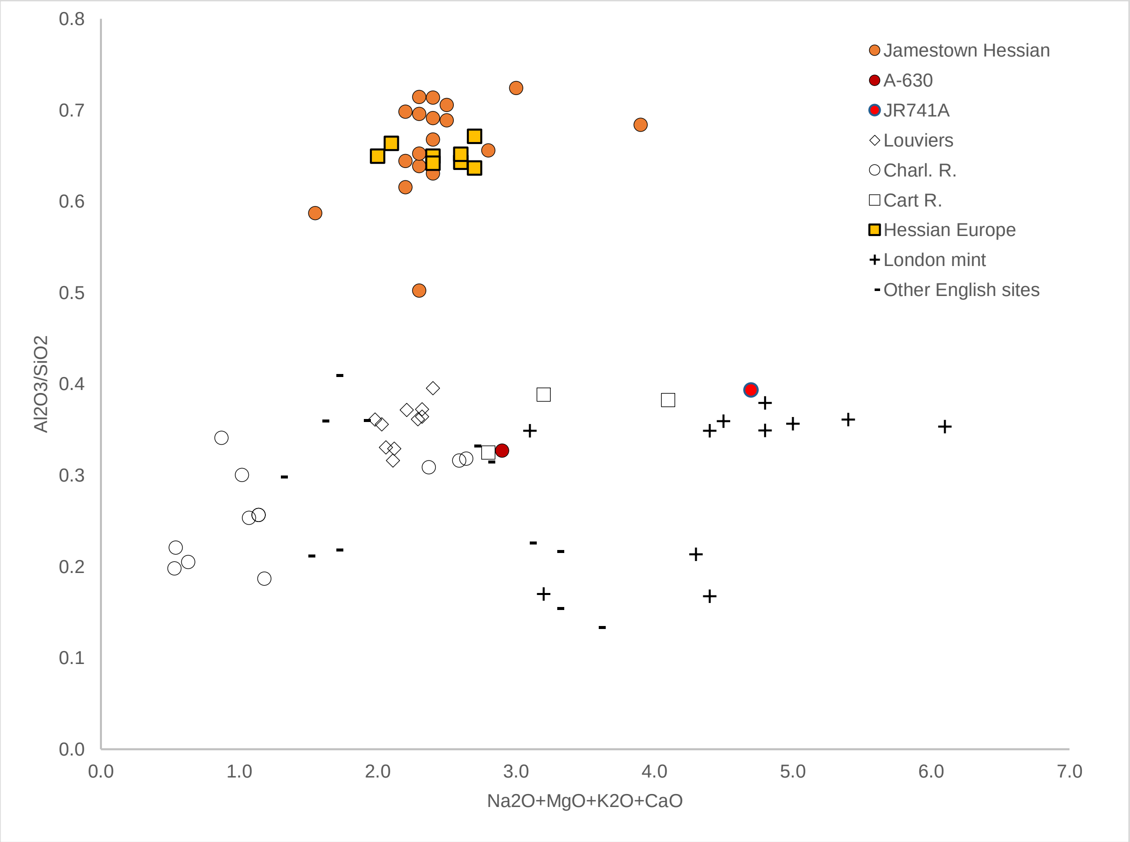


Figure 4.9. Alumina to silica ratio vs total alkalis in the Jamestown crucibles and in other European post-Medieval crucibles. Data on Hessian crucibles is from Martinón-Torres and Rehren 2009; on Louviers and Charlesbourg-Royal from Monette 2013; on Cartier-Roberval from Martinón-Torres 2010; on London mint from White and Kearns 2010 and on other English sites from Dungworth 2006.

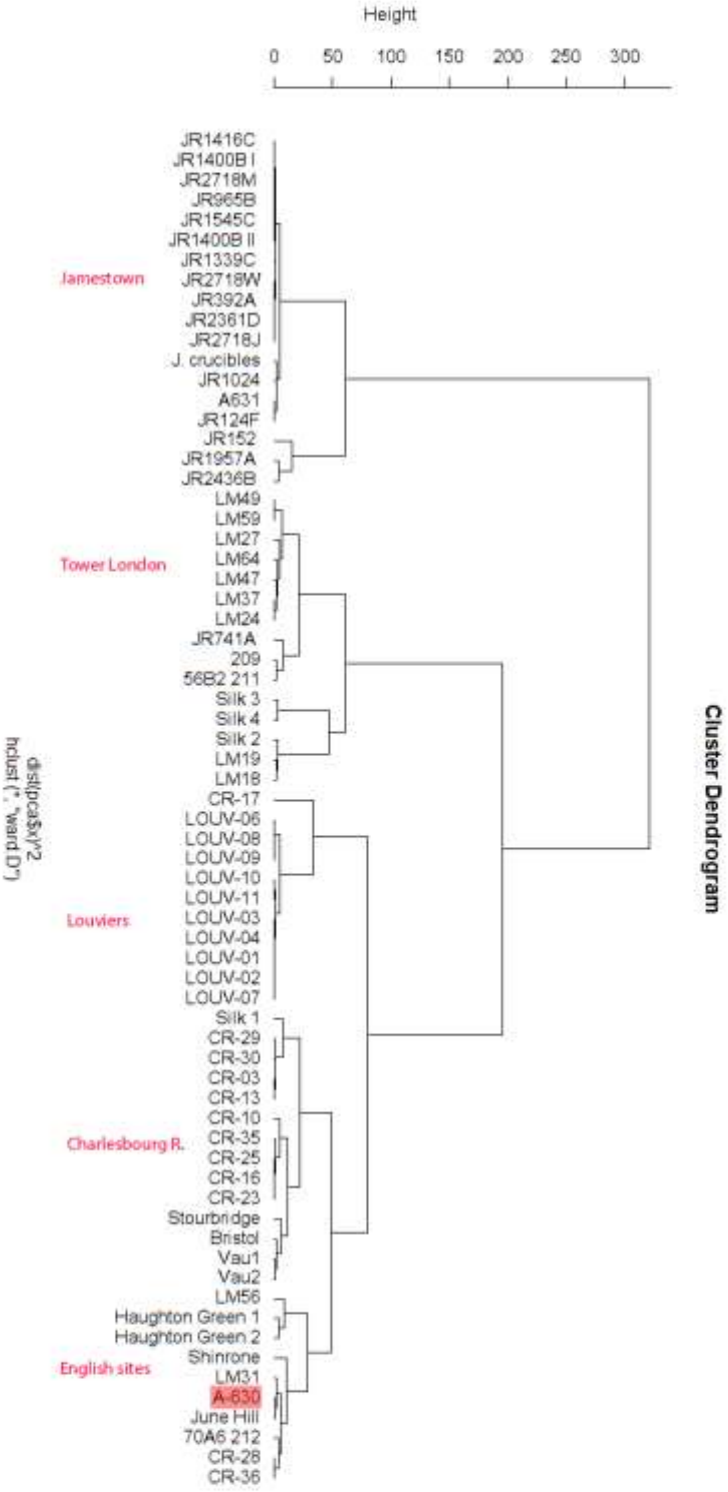


Figure 4.10. Cluster dendrogram with data from post-Medieval European crucibles. Sources are the same as in previous figure. Note sample A-630 sitting on the opposite end from the rest of Jamestown crucibles and in proximity with vessels from the French site of Cartier-Roberval and English contexts.

A decisive interpretation of the Jamestown outlier as to its origin is not possible without 1) the analysis of further non-Hessian examples and 2) the pattern of trace elements that SEM-EDS cannot detect and that may be more significant in distinguishing between clays. In the meantime, typological considerations such as the conical shape, tapering profile and bulging foot already noted seem to push A-630 somewhat closer to the French vessels that to their English counterparts, which in this respect differ quite substantially (see, for instance, the crucibles from the Tower of London in **White and Kearns 2010**). At any rate, both the typological as well as the compositional profile of sample A-630 challenges the observation, made during the analysis of the Canadian assemblage, that "Jamestown collection does not contain any circular crucibles similar to those found at Cap Rouge" (**Cote 2009: 80-81**). Sample JR741A is an outlier too, and compared to the rest of the assemblage it shows high alkalis (4.8 wt% total) and alumina levels similar to those of sample A-630 (25.4 wt% Al_2O_3). A highly vitrified body and the presence of bloating pores characterise the matrix of this sample (**fig. 4.11**) and are due to extreme temperature and an intense interaction between the crucible and the charge. Such interaction is most likely what has caused the elevated levels of potash and iron oxide detected (3.1 wt% and 4 wt% respectively), responsible for the extended melting of the ceramic (cfr section 4.5.5). Lastly, despite showing the typical microstructure of Hessian crucibles (**fig. 4.6**), sample 152-JR has lower alumina and higher silica and soda than the other triangular vessels (31.5 wt%, 62.7 wt% and 1.6 wt% respectively). This too may be due to contamination from the charge, which could also be the reason for the higher degree of distortion and more abundant bloating pores this sample displays when compared to the rest of the Hessian crucibles.

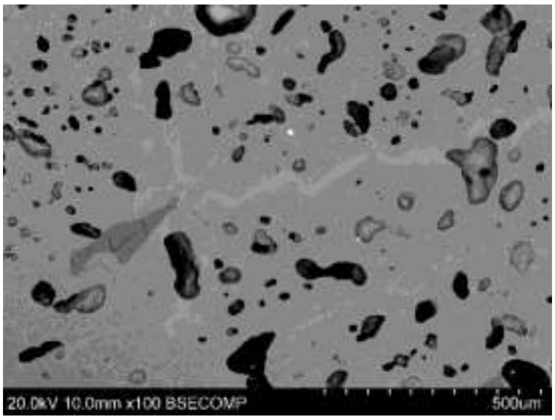


Figure 4.11. SEM backscattered micrograph of the ceramic matrix of sample JR741A, showing heavy vitrification with frequent bloating pores.

4.5.2 NOBLE METALS

Five of the crucible fragments (JR1339C, JR1416C, JR 1400B I and II and A-630) under investigation as well as the one sample of bone ash cupel (JR1957A II) can be related, either directly or indirectly, to metallurgical activities involving noble metals and particularly silver. Two of the samples (JR 1400B I and II) are fragments from the spout and the base of the same vessel (**fig. 4.12**), while the rest are wall fragments. The residues adhering to the inner and often also to the outer walls of the crucibles can vary very much in appearance and range from a well-defined black and glossy layer to simple patches of dark burned areas or orange-reddish spots sometimes showing shiny grains (**Appendix B**). All of them lack evident signs of green discolouration associated with copper corrosion products and instead visible in other samples of the assemblage (cfr section 4.5.3).

Figure 4.12. The fragments of crucible base and spout from which samples JR1400B I and II were obtained (photo by Marcos Martinón-Torres).

As discussed in section 3.1, the analysis of the slaggy layer on the crucibles focused on characterising 1) the bulk composition, 2) the various oxide phases and 3) any metallic area present within the residue matrix. The results of the SEM-EDS analysis indicate a marked microstructural heterogeneity and compositional variability within the assemblage. However, neither in this sub-section on noble metals nor in the next ones will the samples be considered one by one as this would result in excessive fragmentation and repetition and would render it more complicated to look at broader trends in the data. Instead, more general aspects emerging from the analysis of the different phases encountered will be reported and discussed. The glassy background (**table 4.3**) of all of the samples investigated here (except for the cupel) is typically rich in silica (average ranging from 27.7 to 56.5 wt% SiO_2), with variable amounts of oxides of aluminium (5.6-15.5 wt% Al_2O_3), calcium (2.8-9.7 wt% CaO) and titanium (0.4-1.3 wt% TiO_2). Lead is also very frequent, but not as ubiquitous as the elements just mentioned (0.7-11.5 wt% PbO). There is then a host of oxides that tend to recur less frequently and include soda (0.6-4.7 wt% Na2O), magnesia (0.3-3 wt% MgO), potash (0.2-5.1 wt% K_2O) and the oxides of phosphorus (0.4-2 wt% P_2O_5), sulfur (0.1-1 wt% SO_3) and manganese (0.1-0.7 wt% MnO). The residue of sample A-630 is by far the one with the highest levels of iron oxide (25 wt% FeO), whereas in the rest of the assemblage this varies between 3.6 and 11.6 wt%. Finally, some of the crucibles' residues are also variably enriched in heavier elements more specifically linked to the metallurgical activities carried out. These include copper oxide (1-29.6 wt% CuO), tin oxide (1.6-3.9 wt% SnO_2) and zinc oxide (1.2-8 wt% ZnO) detected in JR 1400BI, II and JR1416C, the oxides of arsenic (0.8-4.4 As_2O_3) and nickel (0.2 wt% NiO) in sample JR1339C and that of barium (1.7-34 wt% BaO) detected in the residue of samples JR 1339C and JR 1416C. Worth noting here are two such phases. The first comprises clusters of tin-rich crystals that can be seen in sample JR 1416C (**fig. 4.13**). Besides tin oxide (ca. 62-73 wt% SnO_2), their composition includes elements most likely associated with the mineral assayed, such as the oxides of copper (ca. 1wt% CuO), iron (2-3 wt% FeO), zinc (0.5-1 wt% ZnO), barium (ca. 1wt% BaO), lead (1.5-2.5 wt% PbO) and lime (ca. 1-2 wt% CaO), as well as alumina (ca. 3.8-5.3 wt Al2O3) and silica (ca. 14-22 wt% SiO2), which may at least partly result from contamination with the ceramic.. The second, from the fragment of crucible spout JR1400B II, are the extended areas of cuprite visible as the light grey phase in **figure 4.13**. Besides the predominant copper oxide (> 85 wt% CuO), the phase also contains some minor contamination from the surrounding glassy matrix as well as more substantial contamination from charge components such as the oxides of silver and lead (ca. 8 and 2 wt% respectively). The presence of silver, in particular, is due to the many metallic prills that can be seen scattered across the residue (**fig. 4.14**).

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO	FeO	NiO	CuO	ZnO	As_2O_3	SrO	AgO	SnO_2	BaO	PbO
JR1339C internal	3.9	bdl	9.3	27.7	bdl	1.0	0.5	2.1	7.9	0.4	bdl	8.0	bdl	bdl	bdl	4.4	bdl	bdl	bdl	34.0	0.8
JR1339C external	4.7	3.0	13.5	45.6	0.4	bdl	0.2	5.1	8.9	0.8	0.3	6.6	0.2	bdl	bdl	0.8	bdl	bdl	bdl	9.4	0.7
JR1416C	bdl	0.3	12.9	56.5	1.5	0.8	bdl	bdl	5.6	0.7	bdl	6.1	bdl	3.4	1.4	bdl	bdl	0.3	3.9	1.7	5.0
JR1400B I base	bdl	bdl	15.5	55.0	2.0	0.1	bdl	0.2	4.6	1.3	0.1	11.6	bdl	1.0	8.0	bdl	bdl	0.6	bdl	bdl	bdl
JR1400B II spout	bdl	1.2	5.6	43.3	bdl	bdl	bdl	bdl	2.8	0.7	bdl	3.6	bdl	29.6	1.2	bdl	bdl	0.3	1.6	bdl	11.5
A-630	0.6	2.6	7.2	48.3	1.0	bdl	bdl	4.3	9.7	0.7	0.7	25.0	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl	bdl
JR1957A II cupel	0.3	0.3	0.2	1.4	15.7	bdl	1.0	bdl	18.7	bdl	bdl	0.4	bdl	bdl	bdl	bdl	0.7	bdl	bdl	bdl	61.4

Table 4.3. Average bulk composition by SEM-EDS of the residue layers in sample from silver-related samples. Except when mentioned, the data refers to internal residues.

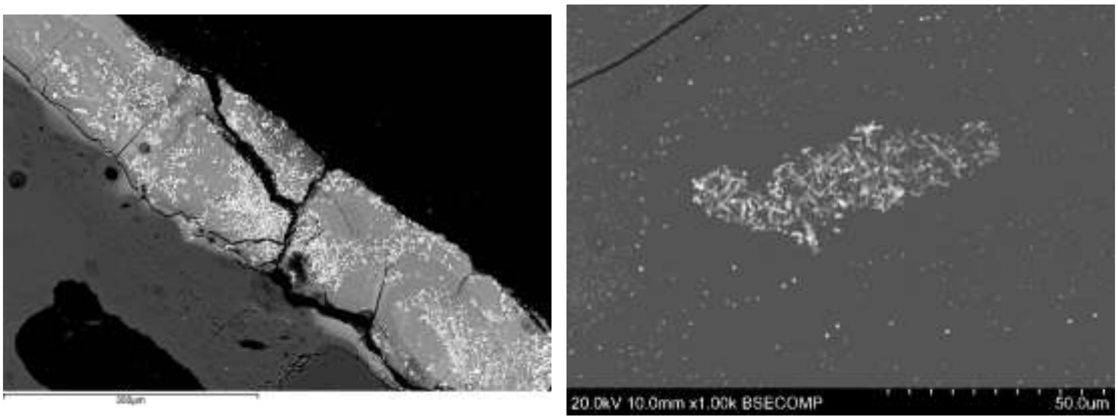


Figure 4.13. SEM-EDS micrographs showing the scatter of tin-rich crystals (bright white) in the residue from sample JR1416C representing remnants of the mineral tested in the crucible.

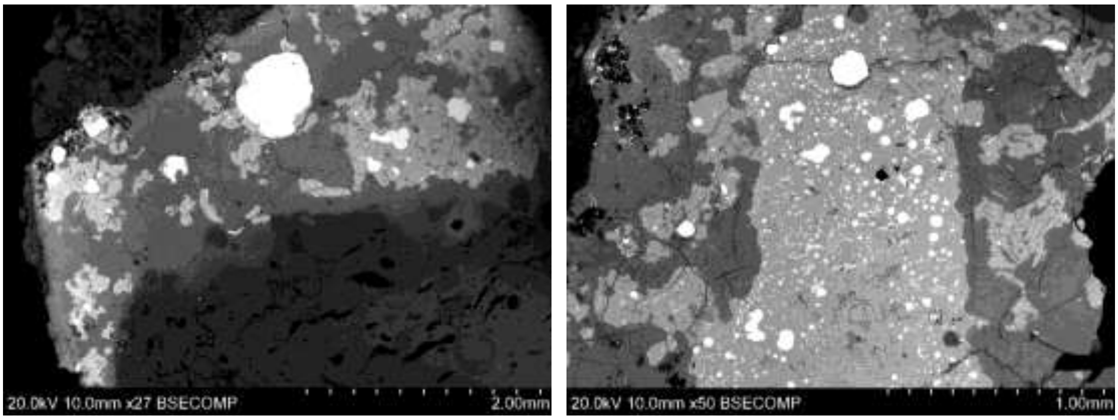


Figure 4.14. SEM-EDS micrographs showing the cuprite (light-grey) areas developing around the metallic prills (bright) in the residue from sample JR1400B II.

Prills of various dimensions are formed during metallurgical operations when the oxides reduce into metallic form. In an ideal scenario, all of the metal that forms is collected after smelting, although often small pools of metal get trapped in the viscous slaggy layer and are not retrieved. These metallic phases are a precious source of information as they can reveal details of the metallurgical processes that produced them. Indeed, the presence of silver-rich droplets was the most important indicator connecting the crucibles discussed here to the metallurgy of noble metals. Except for sample A-630 silver is present in all of the prills analysed and is generally the main component, with copper being the most common associated element (**table 4.4**). The purest droplets, with levels of silver ranging between around 91 and 99 wt% and the balance being copper, were detected in sample JR1400B II, which also has the highest number of metallic phases left in the slaggy layer (**fig. 4.14**). Very pure silver prills were found in sample JR1416C too, though these appear more heavily corroded with resulting presence of elements such as oxygen, chlorine and bromine. In sample JR1339C metallic prills were discovered in both the internal and external residue layer. In the former (**fig. 4.15**), the main components of the prill are lead (42.2 wt% Pb), silver (34.6 wt% Ag) and sulfur (8.2 wt% S), with the balance being corrosion elements. At high magnification it is possible to observe small, brighter segregation areas of pure lead sulfide within the prill. Their composition is around 85 wt% lead and 15 wt% sulfur. The prill in the external residue layer (**fig. 4.15**) is much larger and shows a remarkably complex composition, with only around 2 wt% silver and the rest being nickel (54.9 wt% Ni), arsenic (31.3 wt% As), sulfur (3.9 wt% S), lead (3.5 wt% Pb), antimony (0.9 wt% Sb) and cobalt (0.6 wt% Co). The microstructure shows round areas of segregation with a similar elemental makeup but much more enriched in silver compared to the bulk composition. Sample A-630 doesn't provide immediate connections to silver metallurgy in the same way the other samples do. The elevated presence of iron reported for the glassy residue layer can also be seen in the metallic prills where this is the main component (92.5-99 wt% Fe). Copper was found at low levels in every prill (0.5-5.4 wt% Cu), while in a few of them phosphorus (0.3-1.7 wt% P), nickel (0.7 wt% Ni) and tin (0.5-1.1 wt% Sn) were also detected.

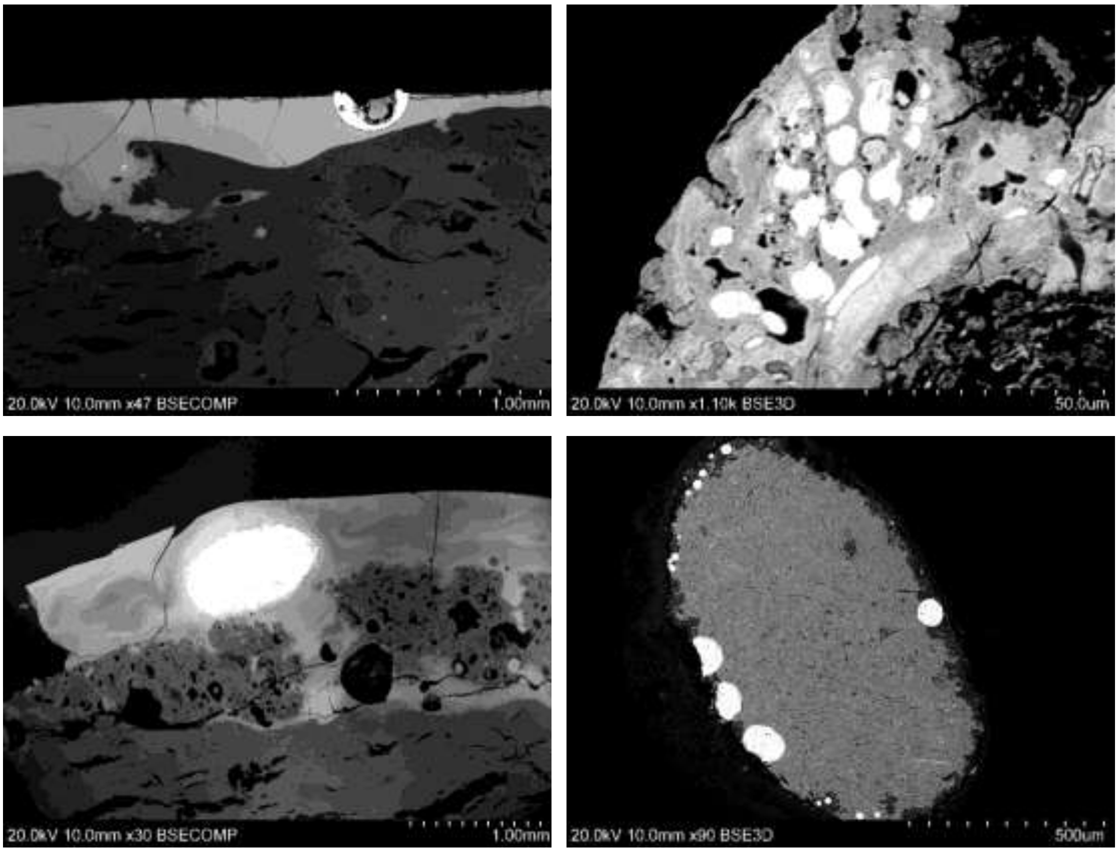


Figure 4.15. SEM-EDS micrographs showing metallic prills in internal (top) and external (bottom) residue from sample JR1339C. Note the denser segregation areas (right side) within the two droplets.

Sample	O	P	S	Cl	Fe	Co	Ni	Cu	As	Br	Ag	Sn	Sb	Pb
1400BII spout	1.9	bdl	bdl	0.1	bdl	bdl	bdl	6.7	bdl	bdl	91.3	bdl	bdl	bdl
1400BII spout	bdl	bdl	bdl	bdl	bdl	bdl	bdl	6.1	bdl	bdl	93.9	bdl	bdl	bdl
1400BII spout	2.5	bdl	bdl	bdl	bdl	bdl	bdl	1.7	bdl	bdl	95.8	bdl	bdl	bdl
1400BII spout	0.3	bdl	bdl	bdl	bdl	bdl	bdl	0.6	bdl	bdl	99.2	bdl	bdl	bdl
1400BII spout	1.9	bdl	bdl	bdl	bdl	bdl	bdl	3.3	bdl	bdl	94.8	bdl	bdl	bdl
1400BII spout	bdl	bdl	bdl	bdl	bdl	bdl	bdl	1.1	bdl	bdl	98.9	bdl	bdl	bdl
1416C	6.5	bdl	bdl	3.9	bdl	bdl	bdl	2.8	bdl	2.2	84.6	bdl	bdl	bdl
1416C	8.1	bdl	0.1	0.3	bdl	bdl	bdl	4.9	bdl	bdl	86.6	bdl	bdl	bdl
1339C int.	14.8	bdl	8.2	0.2	bdl	bdl	bdl	bdl	bdl	bdl	34.6	bdl	bdl	42.2
1339C ext.	3.0	bdl	3.9	bdl	bdl	0.6	54.9	bdl	31.3	bdl	1.9	bdl	0.9	3.5
A-630	bdl	0.6	bdl	bdl	96.0	bdl	bdl	2.9	bdl	bdl	bdl	0.5	bdl	bdl
A-630	bdl	0.3	bdl	bdl	92.5	bdl	0.7	5.4	bdl	bdl	bdl	1.1	bdl	bdl
A-630	bdl	bdl	bdl	bdl	97.1	bdl	bdl	2.9	bdl	bdl	bdl	bdl	bdl	bdl

A-630	bdl	1.7	bdl	bdl	97.0	bdl	bdl	1.3	bdl	bdl	bdl	bdl	bdl	bdl
A-630	bdl	0.7	bdl	bdl	98.8	bdl	bdl	0.5	bdl	bdl	bdl	bdl	bdl	bdl
A-630	bdl	0.4	bdl	bdl	99.0	bdl	bdl	0.7	bdl	bdl	bdl	bdl	bdl	bdl

Table 4.4. Chemical composition by SEM-EDS of metallic prills found in silver-related samples

The various and variable components of the slag layers outlined above are a strong indication that a mixture of unrefined metallic ores and of different reagents made up the charge of the crucibles analysed. Simpler operations such as casting and melting of metals would have produced a much “cleaner” residue, with most of the enriched elements being those from molten ceramic and with only faint traces of the molten metal. One such example, sample JR 2718J, will be discussed in the next section. Here, the results rather point to operations of assay aimed to assess the presence and the quantity of precious metals in local mineral resources. The enrichments in the bulk composition of the residue layers are the product of the interaction between charge components (i.e. minerals, reagents, fuel ashes, cfr section 3.1) and crucible’s ceramic. If we assume that most of the alumina comes from the latter, and if we compare other enrichments against it, it is possible to highlight what, besides molten ceramic, has contributed to the formation of the residues (cfr section 3.1). Here, all of the slag layers appear to contain extra lime, iron oxide and silica when compared to the ceramic composition and some show an extra source of alkalis too (**fig. 4.16-17**). Possible sources for the excess lime and silica are mineral gangue from the ores tested (i.e. calcite and quartz) or crushed glass possibly employed as a flux to promote the formation of slag (**Mongiatti et al 2009a**). Alkali and alkali earth oxides are easily incorporated from fuel ash, but crucibles in this period were generally heated from the outside and great care was taken to avoid precisely such contamination, all the more during high-precision operations like fire assay. For this reason, the generalised presence of lime and magnesia is more likely a result of the addition of charcoal to the charge, as a reducing agent (**Rademakers et al 2018a: 1656**). Similarly, the presence of potash and soda most likely mirrors other reagents, fluxes such as saltpetre (KNO_3) or common salt (NaCl), the latter suggested by the recurring chlorine in association with sodium (**Hoover and Hoover 1950: 238; Sisco and Smith 1951: 108-110; Smith and

Gnudi 2015: 143, 333). Finally, contemporary sources often mention iron filings as yet another possible flux (**Hoover and Hoover 1950: 397-398; Sisco and Smith 1951: 114**) and this could have caused the iron oxide enrichment of some of the samples. However, iron was a very common component of the ore bodies that Jamestown chymists were assaying, and its presence in the slag layers therefore needs to be - at least in part - explained this way.

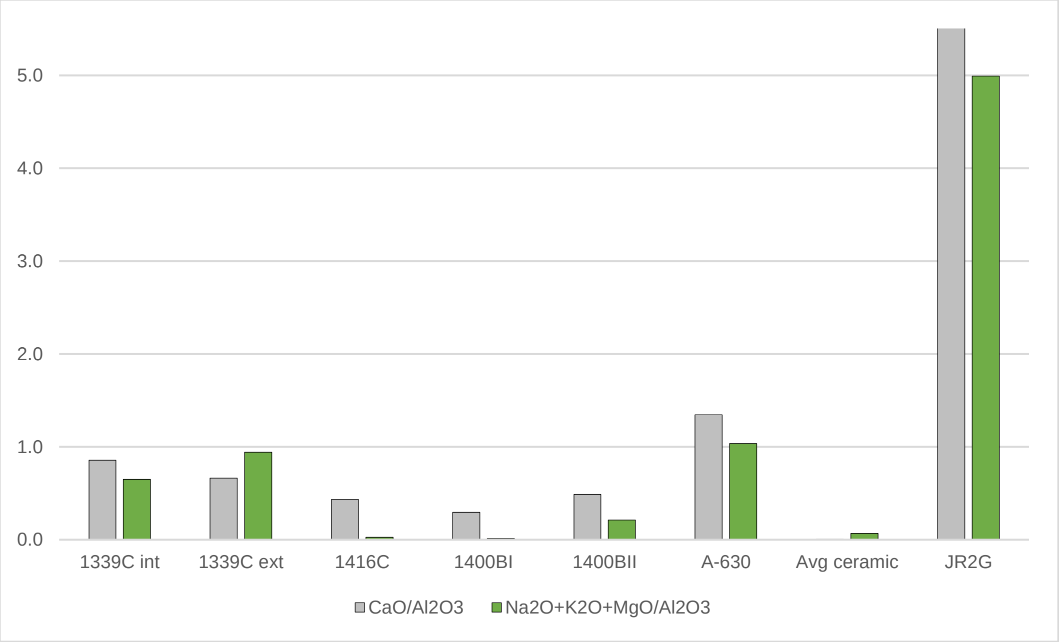


Figure 4.16. Diagram showing the CaO/Al_2O_3 (grey) and Na_2O+K_2O+MgO/Al_2O_3 (green) ratios in the residue layers of silver-related samples. They are compared to the average composition of the ceramic fabric from the crucibles analysed and the glass cullet sample JR2G (CaO/Al_2O_3 value 13.9). The bars show an extra source of lime, and alkalis compared to the ceramic which derive from additives employed during fire assay.

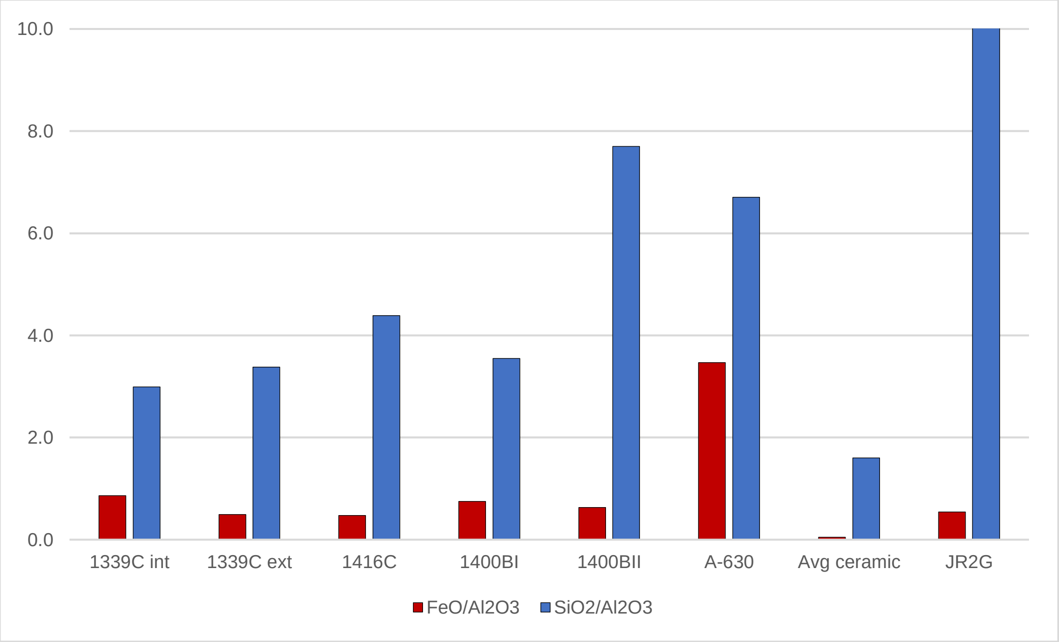


Figure 4.17. Diagram showing the FeO/Al_2O_3 (red) and SiO_2/Al_2O_3 (blue) ratios in the residue layers of silver-related samples. They are compared to the average composition of the ceramic fabric from the crucibles analysed and the glass cullet sample JR2G (SiO_2/Al_2O_3 value 39). The bars show an extra source of iron oxide and silica compared to the ceramic which derive from additives employed during fire assay.

The results on bulk slag composition, oxide phases and metallic prills reveal suits of heavy elements that tend to recur in association. One of these comprises tin, copper, zinc, lead, iron, sulfur and barium and is found in samples JR1400B I and II and JR1416C. A similar association had also been reported by Martinón-Torres and Rehren (**2007: 88**). The presence of sulfur, in particular, points to silver-bearing sulfide lenses of complex polymetallic nature. Indeed, there are numerous bodies of galena (PbS), chalcopyrite ($CuFeS_2$), sphalerite ((Zn,Fe)S) and pyrrhotite (FeS) in Virginia from which silver could be mined (**Sweet 1976**). Such associations occur in different parts of Virginia (**Wright et al 1948; Sweet et al 1989**) and these bodies often contain tin minerals too (**Koschmann et al 1942: 284;**), while barium is present as barite gangue (**Edmunson 1938: 9**) (**fig. 4.18**). It is likely that the selection of different fractions of these ore deposit may have caused the compositional differences witnessed here between the samples, such as the prevalence of tin enrichments in JR1416C or of copper in JR1400B II. A second notable suite of associated elements differs markedly from the previous and is characterised by nickel, arsenic, cobalt and antimony, plus the ubiquitous lead, barium and iron. Samples of this type include JR 1339C from this section and JR 392A, which will be discussed in section 4.5.4. The occurrence of such associated minerals in Virginia is reported in the south-west of the state, in the counties of Floyd and Carroll and can further include pyrites and pyrrhotites, galena and arsenopyrite (FeAsS) (**Watson 1907: 578**) (**fig. 4.19**).

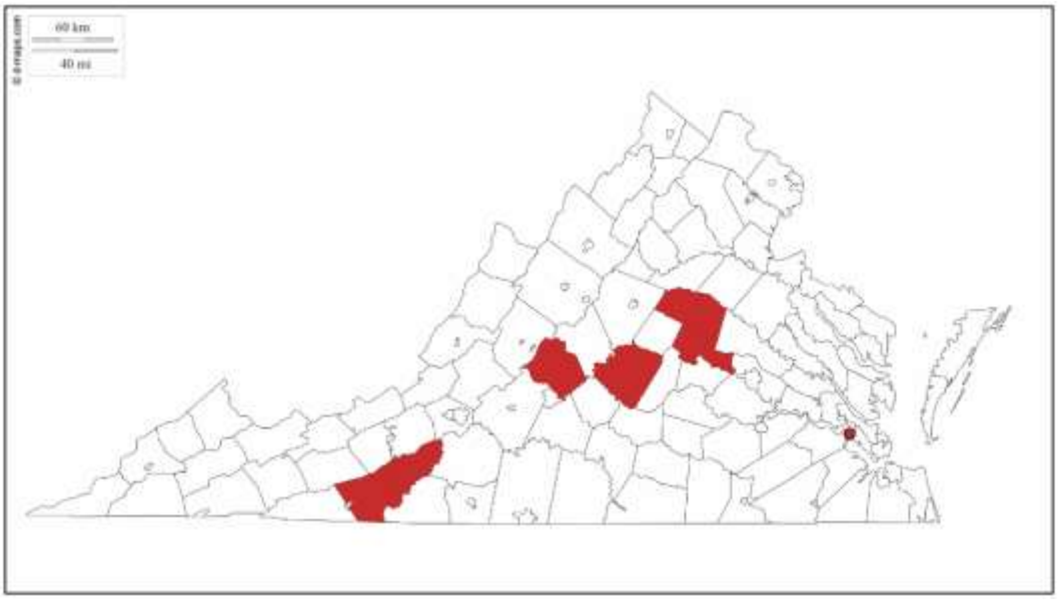


Figure 4.18. Map of Virginia with location of major bodies of mixed zinc-copper-iron-lead sulfides (dot is Jamestown).

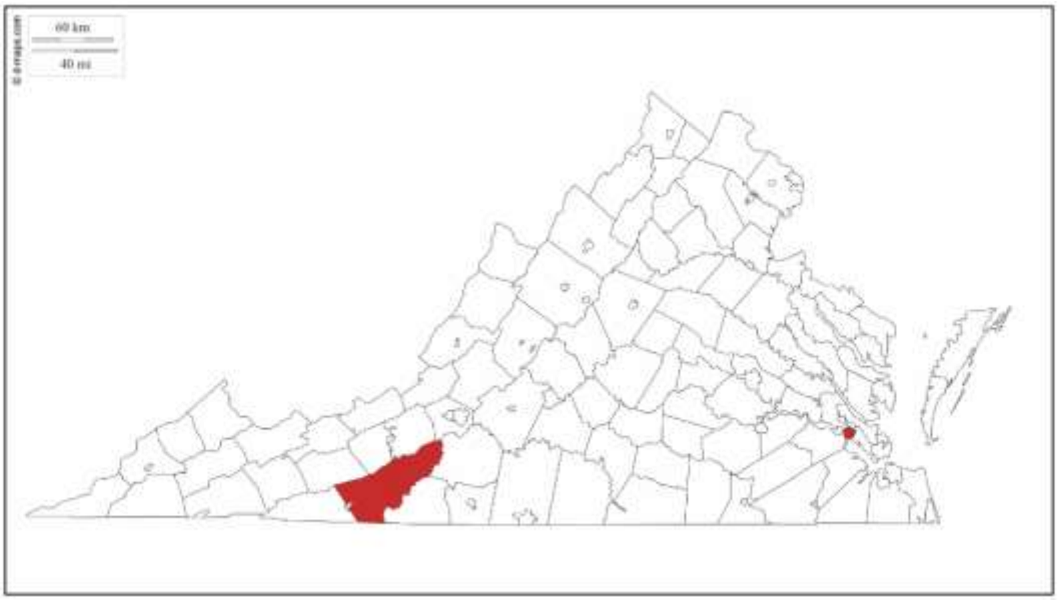


Figure 4.19. Map of Virginia with location of major bodies of mixed nickel-arsenic-cobalt-iron-lead (dot is Jamestown).

The last type of mineral which appears to have been assayed by Jamestown metallurgists is pyrite, represented by sample A-630 and most likely tested in the hope of finding gold in it. Despite sometimes containing small quantities of the precious metal, pyrites were often mistaken for much richer gold ores due to their shiny appearance and for this reason they were known as 'fool's gold'. Incidents with pyrite being mistaken for gold seem to be a constant feature of early European colonisations in north America. In the latter part of the 16th century loads of worthless pyrite had been shipped back to France during the exploration of Canada to be assayed (**Hogarth 1999; Monette 2013: 91**), and a similar issue had probably characterised the Jamestown colony too, when mineral specimens thought to be rich in gold were discovered to be nothing but sterile rocks (**Barbour 1969: 108**). Whether or not this is the case here, belts of gold deposits in Virginia are known along the James river basin where they occur in veins that contain most of the sulfide minerals cited so far and also large quantities of pyrite (**fig. 4.20**) (**Taber 1913**). Settlers may therefore have collected samples of pyrite during one of the expeditions upriver mentioned in captain Smith's chronicles (**Smith 1907: 115- 145**) and later tested them. However, the absence of any trace of gold (or, in fact, of silver) in sample A-630 does not allow to link this vessel to either of the noble metals. The presence of copper and some tin in the prills of A-630 is again consistent with the other polymetallic ores being tested, while nickel was also found in a comparable iron-rich sample analysed in 2007 (**Martinón-Torres and Rehren 2007: 91-94**).

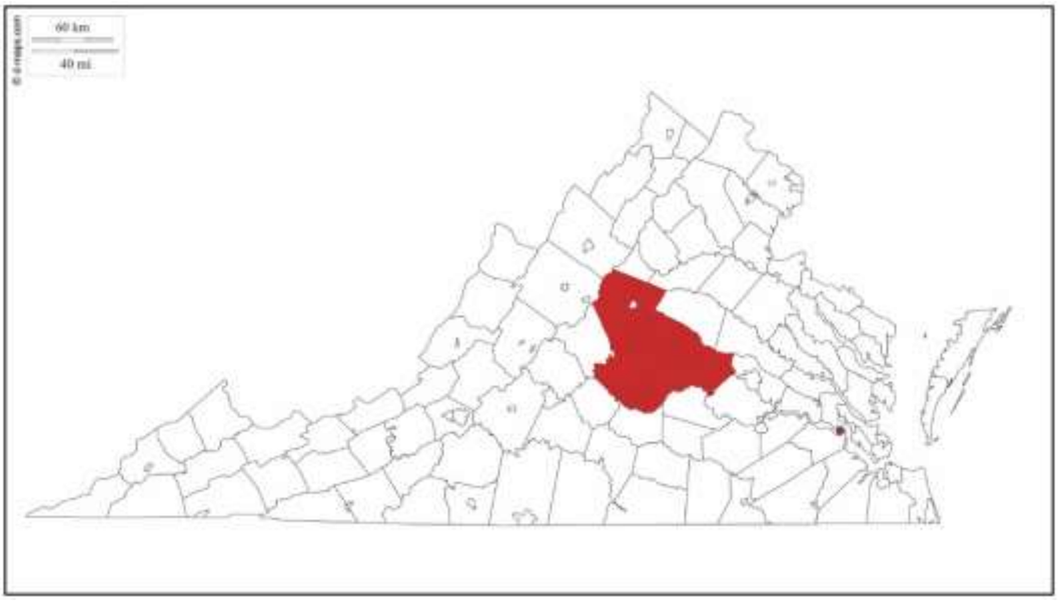


Figure 4.20. Map of Virginia with location of major gold deposits (dot is Jamestown).

During fire assay procedures such as those conducted at Jamestown, the first step was the extraction of the metal of interest through a strongly reducing process carried out in a crucible. As gold or silver (or both) reduced out of the ore it sank towards the bottom of the vessel where it got absorbed by the pool of lead especially added as collector (**Hoover and Hoover 1950: 239; Sisco and Smith 1951: 45-46; Smith and Gnudi 1990: 139**). An aspect worth noting in all of these slag layers is the relatively low amounts of lead oxide in most samples (**table 4.3**). This can be explained by the fact that lead would have been preferentially enriched in the metal phase collecting at the bottom of the crucible, rather than in the oxidised slag layer forming on top (**Mongiatti et al 2009a; Liu et al 2015**). The lead bullion was now ready to undergo cupellation in order to isolate and retrieve the noble metal. This second step, visible here in sample JR 1957A II happened in the cupel and entailed an oxidising reaction (cfr section 3.1). The bulk composition of the cupel body is made of the oxides of phosphorus and calcium, which add up to 34.4 wt% of the total, with the balance being almost entirely the absorbed lead oxide (61.4 wt% PbO). Small quantities of alumina (0.2 wt% Al2O3) magnesia (0.3 wt% MgO), soda (0.3 wt% Na_2O), iron (0.4 wt% FeO) and silica (1.4 wt% SiO_2) and around 0.7 wt% strontium oxide complete the composition of the cupel. Microscopic examination of the cupel's body displays a heterogeneous microstructure dominated by discrete grains with the fine cellular structure typical of bone (**fig. 4.21**), which suggest that the main ingredient of the cupel was bone ash (cfr **Girbal 2011; Ulseth et al 2015; Hsu and Martinón-Torres 2019**). However, the ratio between phosphate and lime is higher than the 1:1 expected if bone ash was the sole source of the two elements and points to an extra source of phosphate (**Martinón-Torres et al 2009**). This could be represented by pulverised fish bones, an ingredient documented in various treatises of the time (**Hoover and Hoover 1950: 229-231; Sisco and Smith 1949:97-98; 1951: 31**). Fish bone would also explain the presence of strontium oxide, silica, soda, magnesia and alumina in the cupel's matrix (**Naga et al 2014**). An alternative explanation for the relatively high content of strontium oxide in the cupel's body is contamination from the gangue of the silver-rich mineral assayed in the previous step, as this element was found in one sample (JR1416C, cfr section 4.5.2).

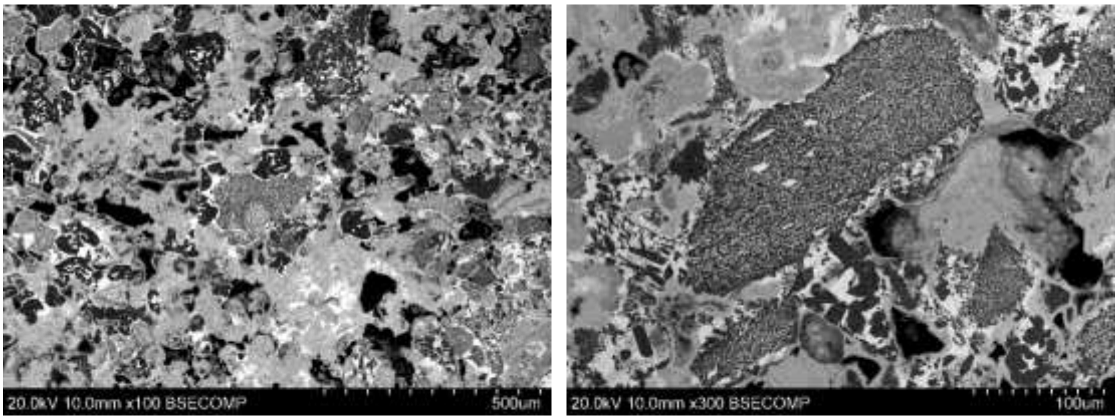


Figure 4.21. SEM-EDS backscattered micrographs showing the microstructure of cupel JR1957A II, with frequent fragments of bone visible as discrete areas of fine cellular structure (dark grey).

4.5.3 COPPER-BASED METALLURGY

In four of the samples analysed as part of this thesis the focus of metallurgical operations appears to be the metallurgy of copper. Of these, three are undiagnostic fragments of crucible walls (JR965B, JR2361D and JR2718J) with a residue layer adhering to the inner surface. The fourth sample (JR1115B) is a lump of melted slag whose peculiar round shape narrowing down towards one end is indicative of it having formed inside of a triangular crucible. This same sample is mentioned in a 2009 study of scrap copper from the site, but was not analysed at the time (**Hudgins et al 2009: 170** and figure 8.4 therein). The immediate link to copper metallurgy is suggested by the characteristic green discolouration and confirmed, upon cutting through, by the presence of extended areas of fresh metal in its section (**fig. 4.22**). Alongside JR1115B, only one other sample can be readily linked to chymical operations involving copper from a macroscopic assessment, namely crucible fragment JR2361D, which also displays green patches on its surface as well as rounded metallic prills embedded in the slaggy residue (**fig. 4.22**). For the other two samples discussed in this section the connection to copper metallurgy is shown by SEM-EDS characterisation.

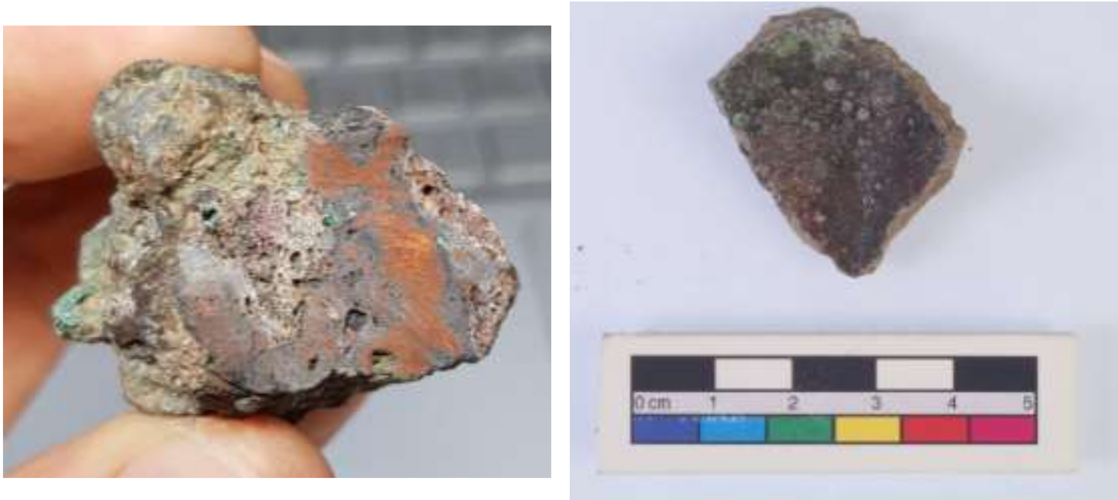


Figure 4.22. Copper-related samples showing macroscopic evidence of copper metallurgy. Left: slag sample JR1115B with extended areas of metallic bronze visible in cross-section; right: crucible JR2361D with green round droplets embedded in the residue layer.

Indeed, the bulk chemical composition of the slag lump and of the crucible slag matrices (**table 4.5**) shows that besides the generalised high levels of silica (42.2-62 wt% SiO_2), an expected feature that was also observed in the previous section, copper oxide was notably detected in all of the samples (0.3-13.1 wt% CuO). In addition, the residues also have common enrichments from interaction with the crucible's ceramic, such as those in alumina (3.7-15.5 wt% Al_2O_3) and titania (0.6-1.4 wt% TiO_2), while other recurring elements are lime (0.2-17.9 wt% CaO), iron oxide (0.8-22.5 wt% FeO) and alkalis (0.7-9 wt% Na_2O, 3-17.3 wt% K_2O and 0.6-3.7 wt% MgO). There are then less ubiquitous enrichments which do not appear in every sample analysed. These include the oxides of phosphorus (0.2-1.6 wt% P_2O_5) and manganese (0.2-0.4 wt% MnO), and heavy elements such as tin oxide (2 wt% SnO_2 in sample JR1115B), zinc oxide (1 wt% ZnO in JR2361D) and finally lead oxide (0.8-1.4 wt% PbO in samples JR1115B and JR965B).

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO	FeO	CuO	SnO_2	ZnO	PbO
JR965B	9.0	1.0	3.7	42.2	1.6	bdl	0.9	6.2	4.3	0.6	0.2	22.5	6.4	bdl	bdl	1.4
JR2718J	1.7	1.3	6.6	62.0	0.2	bdl	bdl	17.3	7.2	1.0	0.3	2.2	0.3	bdl	bdl	bdl
JR2361D	2.7	3.7	7.0	53.1	1.0	0.5	0.2	10.5	17.9	0.8	0.4	0.8	0.4	bdl	1.0	bdl
JR1115B	0.7	0.6	15.5	59.7	bdl	bdl	bdl	3.0	0.2	1.4	bdl	3.1	13.1	2.0	bdl	0.8

Table 4.5. Average bulk composition by SEM-EDS of the residue layers in samples from copper-related samples.

Sample	O	P	S	Cl	Fe	Ni	Cu	Zn	As	Sn	Sb	Pb
JR965B	1.1	bdl	bdl	bdl	0.3	bdl	98.5	bdl	bdl	bdl	bdl	bdl
JR965B	1.0	bdl	0.4	bdl	bdl	bdl	98.2	bdl	bdl	bdl	0.4	bdl
JR965B	0.8	bdl	bdl	bdl	1.9	bdl	97.2	bdl	bdl	bdl	bdl	bdl
JR965B	0.9	bdl	bdl	bdl	0.5	bdl	98.6	bdl	bdl	bdl	bdl	bdl
JR965B	0.9	bdl	bdl	bdl	2.3	bdl	96.8	bdl	bdl	bdl	bdl	bdl
JR965B	1.8	bdl	bdl	bdl	3.8	bdl	94.4	bdl	bdl	bdl	bdl	bdl
JR965B	1.1	bdl	bdl	bdl	3.5	bdl	95.4	bdl	bdl	bdl	bdl	bdl
JR965B	0.8	bdl	bdl	bdl	3.6	bdl	95.7	bdl	bdl	bdl	bdl	bdl
JR965B	1.4	bdl	bdl	bdl	4.5	bdl	94.1	bdl	bdl	bdl	bdl	bdl
JR2718J	9.1	bdl	bdl	bdl	0.5	bdl	90.4	bdl	bdl	bdl	bdl	bdl

Table 4.6. Chemical composition by SEM-EDS of metallic prills found in silver-related samples

Again, the wide array of enrichments reported here can be tentatively linked to single components of the crucible charge. As noted in the previous section, the presence of charcoal is chiefly indicated by enrichments in lime and magnesia, while soda and potash are suggestive of the addition of alkali-based fluxes. In particular, the recurrence of chlorine and soda in association in sample JR965B strongly suggests the use of common salt. Aside from molten ceramic, detected enrichments such as those in silica and alumina could in part derive from the gangue associated with minerals being worked, and specifically to the quartz and feldspar fraction of ore bodies. Finally, heavy elements are diagnostic of the type of metals and ores that entered the crucible as charge. Sample JR2718J only shows faint traces of metallurgical enrichments, it is mostly free from heavy elements except for low concentrations of copper oxide (0.3 wt% CuO). When compared to average ceramic composition, the matrix indicates an excess source of silica, lime, alkalis, and a little iron oxide (**fig. 4.23-24**). These must represent reagents that were added to the charge to promote melting, perhaps fuel ashes or crushed glass of the high-lime type discovered in abundance at the site (cfr 4.5.5). Further relevant impurities are absent. This indicates that the original vessel was probably used in casting copper for internal needs of the colony, rather than for the testing of minerals. The same interpretation was given for one very similar fragment analysed in the 2007 pilot study (**Martinón-Torres and Rehren 2007: 86-87**), with the alternative hypothesis that the vessels could have also been used to test a calcium-rich mineral, possibly in the hope of finding a source of zinc (**Hudgins et al 2009: 171-172**).

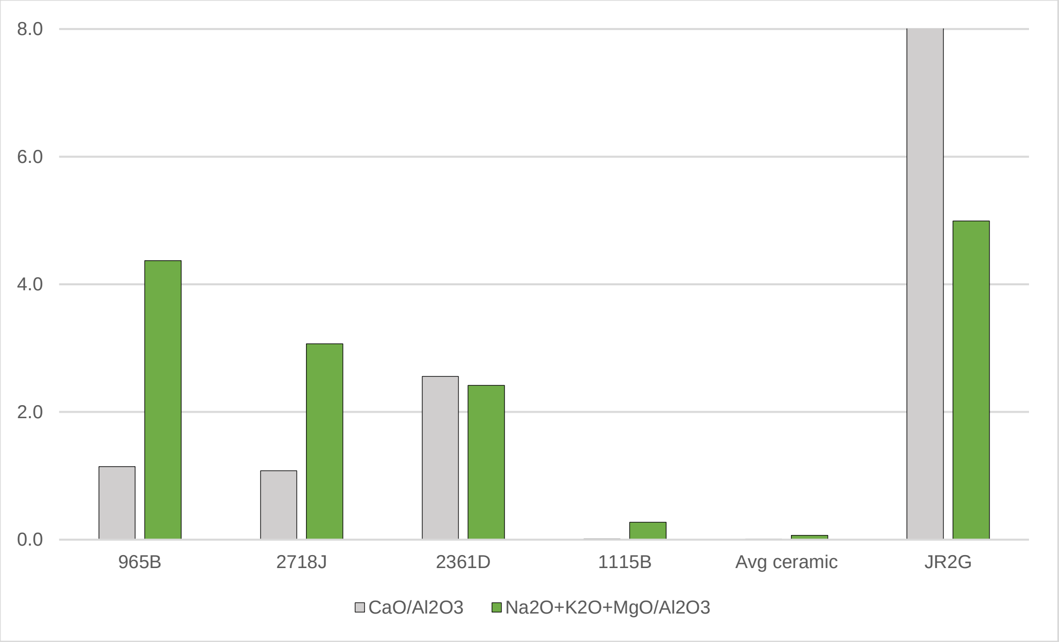


Figure 4.23. Diagram showing the CaO/Al_2O_3 (grey) and Na_2O+K_2O+MgO/Al_2O_3 (green) ratios in the residue layers of copper-related samples. They are compared to the average composition of the ceramic fabric from the crucibles analysed and the glass cullet sample JR2G (CaO/Al_2O_3 value 13.9). The bars show an extra source of lime, and alkalis compared to the ceramic which derive from additives employed during fire assay.

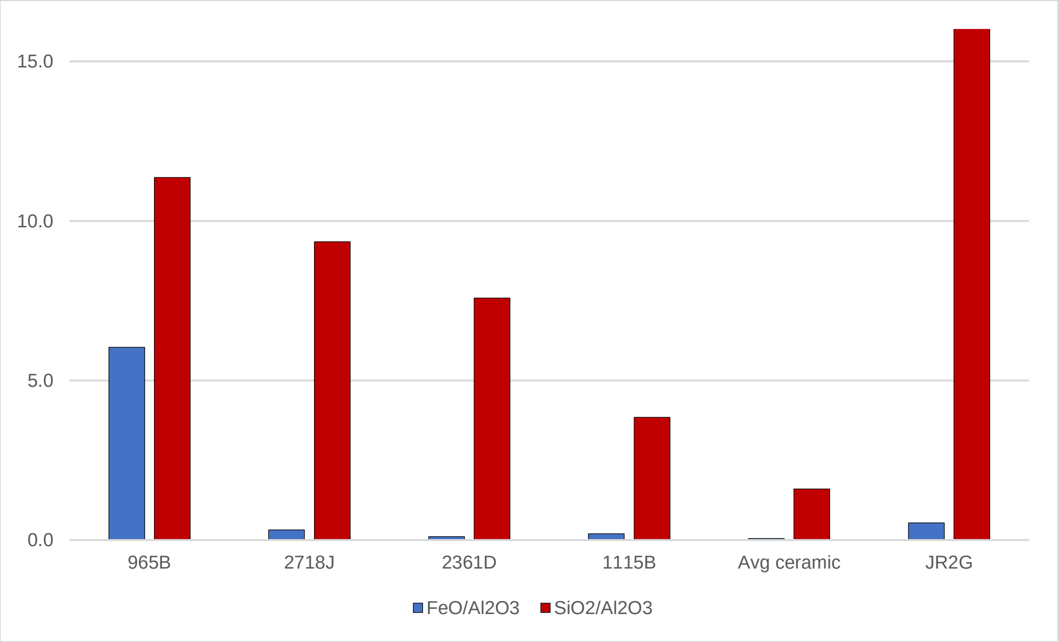


Figure 4.24. Diagram showing the FeO/Al_2O_3 (red) and SiO_2/Al_2O_3 (blue) ratios in the residue layers of copper-related samples. They are compared to the average composition of the ceramic fabric from the crucibles analysed and the glass cullet sample JR2G (SiO_2/Al_2O_3 value 39). The bars show an extra source of iron oxide and silica compared to the ceramic which derive from additives employed during fire assay.

Sample JR965B is characterised by a very different situation. The residue matrix was found to contain especially high iron oxide levels, suggesting an excess source of this element is at play (**fig. 4.24**). At high magnification it is possible to see that iron comes as clusters of residual magnetite (Fe_3O_4) scattered across the matrix, variably enriched in the oxides of copper (up to 5 wt% CuO), nickel (ca. 1.5 wt% NiO) and lead (up to 2.5 wt% PbO) (**fig. 4.25**). There is also a large number of bright metallic droplets that in many cases appear to reduce out of the magnetite areas (**fig. 4.25**). They are made of almost pure copper (ca. 94-99 wt% Cu) with the balance being iron and oxygen from corrosion (**table 4.6**). One larger prill also contains little sulfur (0.4 wt% S) and antimony (0.4 wt% Sb) and shows a grey copper sulfide shell surrounding it (**fig. 4.25**). In light of these results, the sample is believed to be the remain of assay operations roughly comparable to the ones discussed in the previous section. In this case however, the lack of appreciable quantities of silver in either the prills or other phases analysed probably means that an iron-rich sulfide mineral was being tested for its copper content. It was noted that pyrite, pyrrhotite and chalcopyrite are very common features in the geology of Virginia and that these may have been deemed worth for their possible noble metal content. One cannot exclude, therefore, that this sample could also have been assayed for testing the presence of silver or gold. The need for reducing conditions is confirmed by the enrichment in alkali earth oxides, brought in by charcoal, while soda and potash allow to hypothesise the addition of an alkali-based flux.

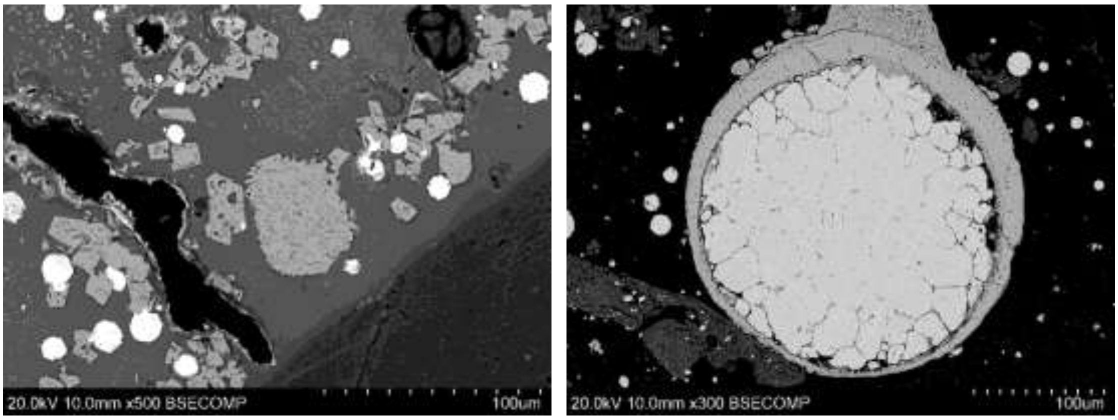


Figure 4.25. SEM-EDS micrographs showing the microstructure of sample JR965B. Left: clusters of residual magnetite (mid-grey) and round copper-rich prills (bright white) in the glassy matrix (dark grey); right: copper-rich prill with copper sulfide shell around it.

4.5.3.1 Copper alloys

Sample JR2361D and the slag lump JR1115B need to be addressed separately as data shows their coming from metallurgical operations aimed at searching and testing local sources of alloying metals. Sample JR2361D contains two large metallic prills (ca. 0.5-1.5 mm across) where a high degree of corrosion has left only limited areas of fresh metal (**fig. 4.26 A-D**). SEM-EDS revealed that the fresh metal is brass and contains approximately 11 wt% zinc, plus variable quantities of iron (0.8-9.1 wt% Fe), lead (1.2-1.3 wt% Pb) nickel (0.3-0.4 wt% Ni), sulfur (up to 0.5 wt% S) and arsenic (up to 1.5 wt% As) (**table 4.7**). The relatively low zinc content of this brass is most likely to be linked to the preferential oxidation of zinc to copper and its subsequent loss to corrosion. Some very small segregation areas are also present, mostly made of lead, with little copper, zinc, arsenic, phosphorus, sulfur and chlorine. Moreover, one of the large prills is surrounded by a layer of copper sulfide (**fig. 4.26D**) comparable to the one discovered in sample JR965B and discussed earlier in section 4.5.3. Tiny rounded prills were also discovered all over the glassy matrix (**fig. 4.26E**) and on top of the elements reported so far also contain little amounts of antimony (0.8 wt% Sb) (**table 4.7**). During excavation several thousand pieces of scrap copper offcuts were discovered. These were found to be of European origin and it is likely that – at least in part - they had been brought along from England to be employed as ingredients in the testing of local minerals for alloying purposes through cementation of local sources of zinc (**Hudgins 2005; Hudgins et al 2009**). The strong enrichment of the residue layer in lime and magnesia (17.9 wt% CaO and 3.7 wt% MgO) (**table 4.5**) is an indication that charcoal was an important part of the crucible's charge and supports the use of this crucible for cementation, which indeed required strongly reducing conditions. During the process, layers of metallic copper were combined with calcined zinc ore and charcoal in a sealed vessel. The vessel was then heated until zinc reduced in the form of vapour at around 907C and became absorbed by the copper (**Bayley 1998; Rehren 1999a; Martinón-Torres and Rehren 2002; Craddock and Eckstein 2003; Bayley and Rehren 2007**). That the residue in sample JR2361D does not reflect simple melting of brass is indicated by the impurities detected in the prills, including the sulfur mentioned above. The impurity profile of these metallic phases is consistent with the complex polymetallic sulfides already abundantly discussed in previous sections, with elements such as iron, lead, copper and sulfur often associated with sphalerites (**Wright and Raman 1948; Sweet et al 1989**). Some sheets of scrap brass have also been discovered by archaeologists, but these have a different impurity pattern and were most likely brough from Europe for everyday metallurgical purposes at the fort (**Hudgins 2005; Hudgins et al 2009**). Thus, while the evidence so far available seemed to point to a lack of brass cementation activities at the site, the data from sample JR2361D may tell a different story. The results presented in this thesis appear to indicate that the crucible had been employed to test a local ore of zinc by cementing it with copper in an attempt to produce brass.

On the other hand, the lump of crucible slag JR1115B has metallic phases that range quite dramatically in size and shape from small rounded prills to large amorphous pools of metal (**fig. 4.27A-B**). They are made of bronze, with a tin content that was found to vary greatly, oscillating between just a few percent to exceptionally high levels nearing 50 wt% (**table 4.7**). A scatter of elongated tin oxide crystals can be observed all around the large metallic phases (**fig. 4.27A, D**). Their angular shape is indicative of the fact that they were caused by the preferential oxidation of tin burning out of the bronze melt (**Dungworth 2000; Rademakers and Farci 2018; Rademakers et al 2018a: 1663**).

The fact that the molten slag is not the result of simple re-melting of bronze is indicated by the remarkable variability in the tin content of the metallic phases, a characteristic feature encountered in residues of active alloying of copper with a fresh source of tin where any composition between pure copper and pure tin can be found (**Renzi and Rovira 2016; Rademakers and Rehren 2016; Rademakers et al 2018a; 2018b**). Corrosion could also be a factor in the changing levels of tin, but the negligible amounts of oxygen detected in the prills allows to discard this possibility. At any rate, a more complicated issue is to establish whether tin was introduced in metallic or in mineral form as ground cassiterite. While the first merely involved melting both metals together, the second case would have required a cementation process akin to what discussed above, in which tin ore was mixed with copper sheets and heated until the former would reduce out of the ore and combine with the latter. The resulting bronze bullion would have sunk towards the bottom of the crucible and on top of it floated a layer of slag containing the gangue from cassiterite and molten ceramic, plus any metal that might have been trapped in it. The occurrence of mineral relics is believed to be the most reliable clue in favour of cementation vs the alloying of copper and tin in metallic form (**Rademakers and Farci 2018**). In the case of sample JR1115B, although the tin oxide crystals sometimes seem to cluster in what could represent cassiterite pseudomorphs (**fig. 4.27D**), their presence is not straightforward to establish (see also **Rademakers et al 2018a: 1665**). A further potential indication of cementation in the sample discussed here is the presence of around 0.5 wt% tantalum oxide (Ta_2O_5) in many of the tin oxide crystals analysed, a common element associated with tin deposits (**Farci et al 2017**). In Virginia, columbite-tantalite is often associated with cassiterite in Amelia and Powhatan counties (**Penick 1987**). The hypothesis of the cementation of cassiterite and English copper is also largely consistent with the scenario reconstructed so far, of tests and experiments with local minerals, and with the inquisitive attitude that characterises all experimental work at the site. Finally, Hudgins and collaborators analysed similar masses of waste copper and came to the same interpretation (**Hudgins et al 2009: 174-175**).

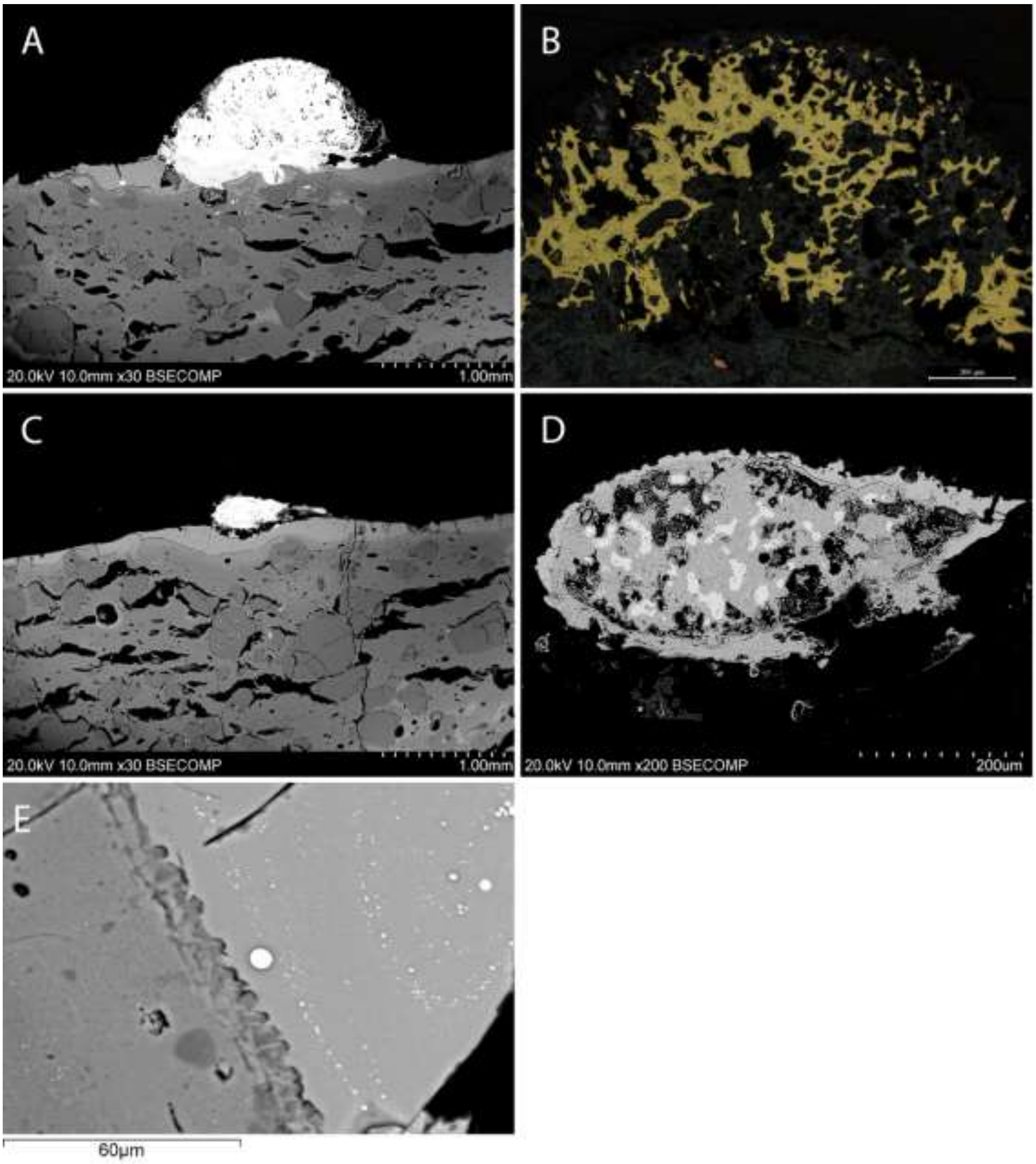


Figure 4.26. Overview of brass prills in sample JR2361D. A-B: larger prill seen under SEM-EDS (left) and OM (right) and showing a high degree of corrosion; C-D: smaller prill, also very corroded. Note the thin copper sulfide layer around it (grey in D); E: tiny brass prills floating in the glassy residue.

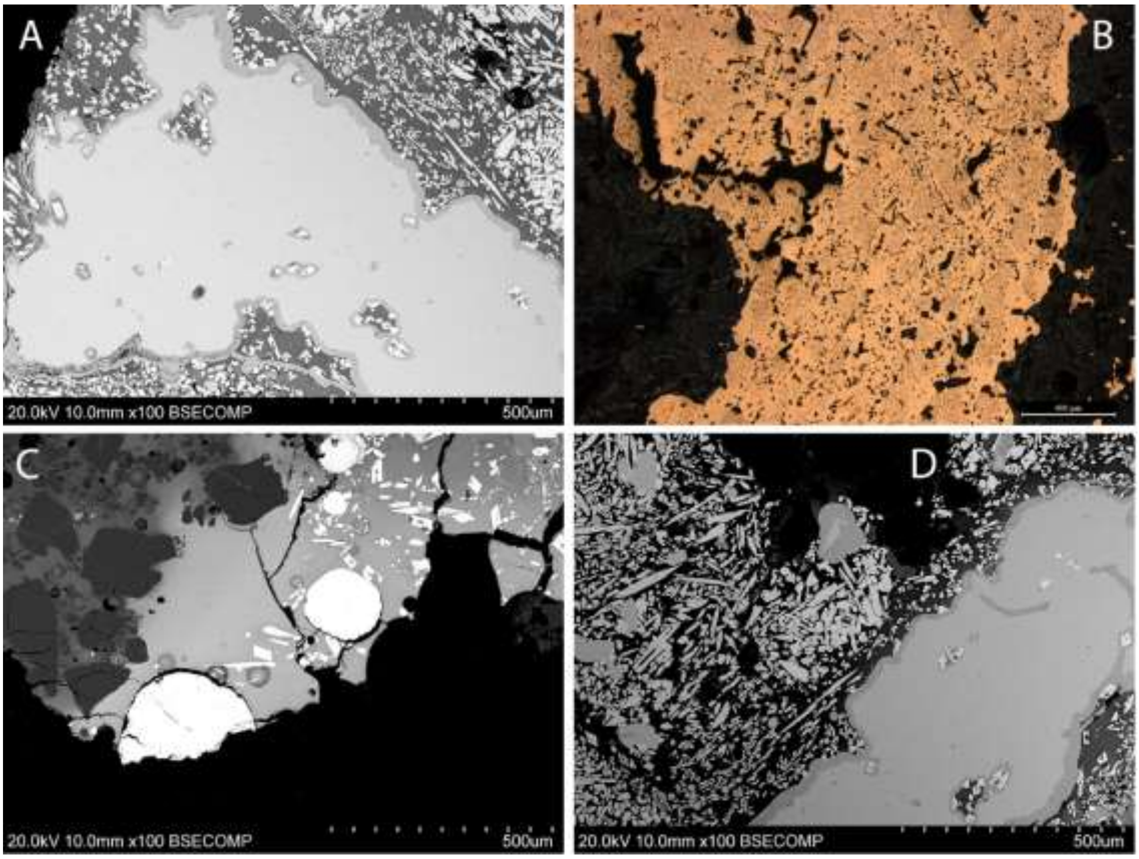


Figure 4.27. Overview of bronze areas in sample JR1115B. A-B: SEM-EDS backscattered micrograph (left) and OM image (right) of large pools of bronze surrounded by a scatter of elongated tin oxide crystals (bright white). C: SEM-EDS backscattered micrograph showing rounded prills in the glassy matrix as well as residues of the ceramic from the crucible (dark grey); D: SEM-EDS backscattered micrograph with possible cassiterite pseudomorph left of the bronze area.

Sample	O	P	S	Cl	Fe	Ni	Cu	Zn	As	Sn	Sb	Pb
JR2361D	1.3	bdl	0.5	bdl	0.8	0.4	83.6	12.3	bdl	bdl	bdl	1.2
JR2361D	1.9	bdl	0.5	0.2	0.4	0.4	83.4	11.9	bdl	bdl	bdl	1.3
JR2361D	1.3	bdl	bdl	bdl	0.5	0.3	86.2	11.6	bdl	bdl	bdl	bdl
JR2361D	1.2	bdl	bdl	bdl	0.2	0.3	86.8	10.9	0.5	bdl	bdl	bdl
JR2361D	1.7	1.4	bdl	bdl	9.1	bdl	73.6	10.6	1.5	bdl	0.8	1.2
JR1115B	1.9	bdl	bdl	bdl	bdl	bdl	98.1	bdl	bdl	bdl	bdl	bdl
JR1115B	1.3	bdl	bdl	bdl	bdl	bdl	85.3	bdl	bdl	13.5	bdl	bdl
JR1115B	bdl	bdl	bdl	bdl	bdl	bdl	86.4	bdl	bdl	13.6	bdl	bdl
JR1115B	bdl	bdl	bdl	bdl	bdl	bdl	56.3	bdl	bdl	43.7	bdl	bdl
JR1115B	3.9	bdl	bdl	0.2	bdl	bdl	48.3	bdl	bdl	47.6	bdl	bdl
JR1115B	1.0	bdl	0.2	bdl	bdl	bdl	97.6	bdl	bdl	1.2	bdl	bdl

Table 4.7. Chemical composition by SEM-EDS of metallic phases in copper alloys.

4.5.4 FELDSPAR-RICH INCLUSIONS

Three of the crucible fragments investigated (JR392A, 1957A and 1545C) cannot be readily linked to metallurgical activities. Indeed, the residues adhering to them don't show clear signs of metallic corrosion as those observed in other samples. Instead, the residues appear black and glassy, sometimes quite smooth and translucent and other times grainy and heterogeneous (**fig. 4.28**). Both the microstructure and the chemical makeup of these three residue layers confirm the impressions deriving from the macroscopic assessment, namely 1) that they form a separate group and 2) that their connection with metallurgy is more dubious than that of the samples discussed so far. As **table 4.8** shows, no traces of either silver or copper were detected in the bulk composition of the residues, which appear closely comparable and are mainly composed of silica (52.1-60.4 wt% SiO_2), with strong enrichments in both soda and potash (5.3-8.7 wt% Na_2O and 13.1-17.9 wt% K_2O) and alumina (8.7-12.2 wt% Al_2O_3). Other notable enrichments include lime and magnesia (0.9-4.6 and 0.6-2.4 wt% respectively) and iron oxide (2.5-4.5 wt% FeO), while the oxides of lead (0.5-1.3 wt% PbO), arsenic (0.4 wt% As_2O_3) and sulfur (0.2-0.7 wt% SO_3) are not common to all three samples.

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	FeO	As_2O_3	PbO
JR1957A I	8.6	1.8	8.7	57.8	bdl	bdl	0.8	14.0	2.6	0.7	4.5	bdl	0.5
JR1545C	8.7	0.6	12.2	60.4	bdl	0.2	0.6	13.1	0.9	0.8	2.4	bdl	bdl
JR392A	5.3	2.4	11.2	52.1	0.4	0.7	0.5	17.9	4.6	0.9	2.5	0.4	1.3

Table 4.8. Average bulk composition by SEM-EDS of the residue layers in samples with feldspar-rich residues.

Figure 4.28. Fragments with black and glassy feldspar-rich residues. Note the grainy texture due to the presence of unreacted feldspars in the matrix of some samples. Left: JR1957A I, right: JR1445C (photos Marcos Martinón-Torres).

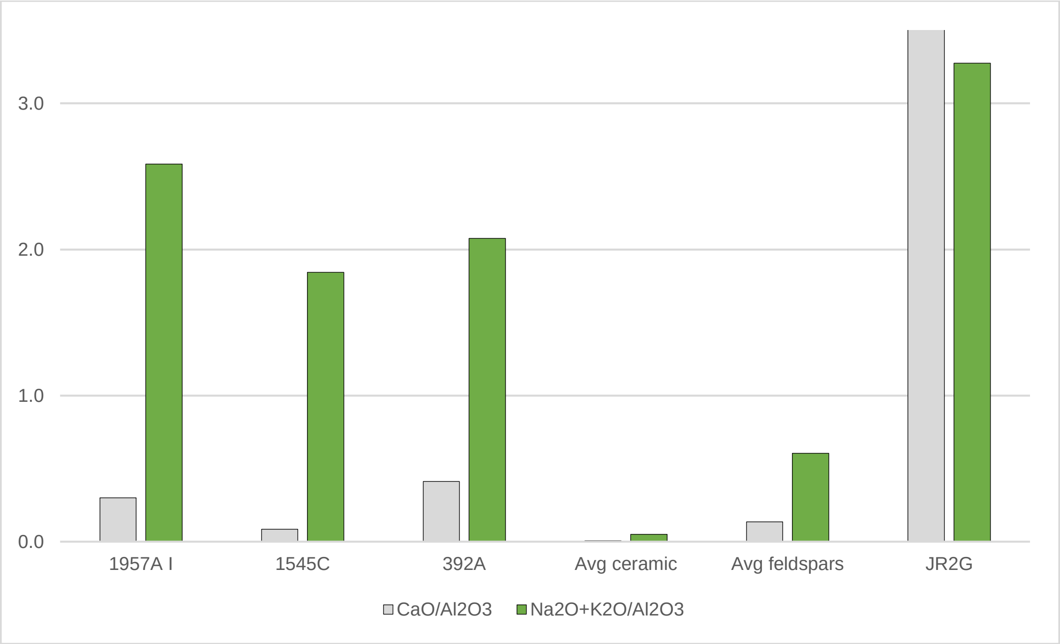


Figure 4.29. Diagram showing the CaO/Al_2O_3 (grey) and Na_2O+K_2O/Al_2O_3 (green) ratios in the residue layers of feldspar-rich samples. They are compared to the average composition of the ceramic fabric from the crucibles analysed, the glass cullet sample JR2G (CaO/Al_2O_3 value 13.9), and the average of the feldspar inclusions analysed (from table B.3 in Appendix B). The bars show an extra source of lime, alumina and alkalis compared to the ceramic which derive from the feldspars as well as from additives employed during fire assay.

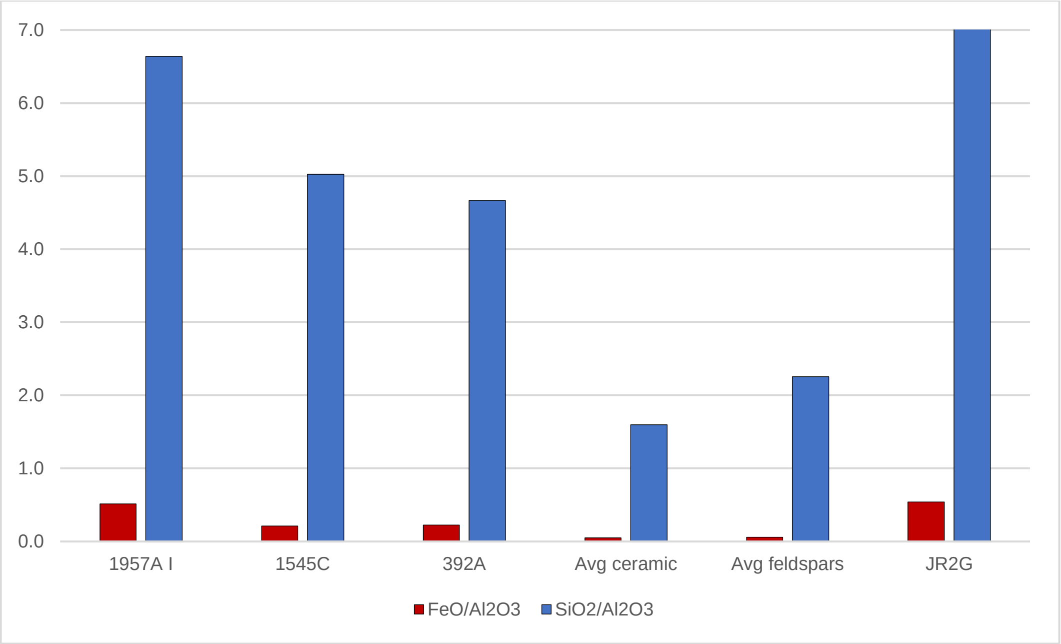


Figure 4.30. Diagram showing the FeO/Al_2O_3 *(red) and* SiO_2/Al_2O_3 *(blue) ratios in the residue layers of feldspar-rich samples. They are compared to the average composition of the ceramic fabric from the crucibles analysed, the glass cullet sample JR2G (*SiO_2/Al_2O_3 *value 39), and the average of the feldspar inclusions analysed (from table B.3 in Appendix B). The bars show an extra source of iron oxide and silica compared to the ceramic which derive from the feldspars as well as from additives employed during fire assay.*

Not unlike what was observed in the previous sections, lime and magnesia suggest that charcoal was a component of the charge, and impressions left by charcoal fragments can indeed be seen on the surface of sample JR392A. However, the most interesting enrichments detected in these samples are those in alumina and in both alkalis (**fig. 4.29**). It was already noted that the most likely contribution of alumina in the bulk composition of a slag layer derives from molten crucible matter (cfr section 3.1), though this does not seem to be the case here. Instead, a look at the microstructure of the residue reveals the presence of numerous grains of partially melted alumina-rich inclusions (**fig. 4.31-32**), which can also be seen macroscopically, embedded in the glassy matrix (**fig. 4.28**). They are responsible for the high alumina in the residue's bulk, and in large part also for the high levels of potash and soda (**Table 4.9**). Element ratios in **figures 4.29 and 4.30** indicate that a certain amount of extra alkalis, as well as silica, must have entered the charge with other substances too. This is in line with the rest of the samples analysed and points to fluxes such as salt and crushed glass or to initial ingredients like mineral gangue. While a number of the inclusions resemble plagioclase feldspars, with roughly 1:1 soda to alumina molar ratio, others have a more complex composition. This is especially the case in sample JR392A, whose inclusions have too low silica and too high alumina to alkali ratios for them to be pure feldspars. Moreover, SEM-EDS shows tiny prills of galena (PbS) either intergrown with the mineral grains or scattered around them (**fig. 4.32**), whose presence explains the associated enrichment in lead oxide of the residue's bulk noted before. The inclusions therefore represent fragments of partially-reacted ore which may have contained some feldspars, but also other alkali-bearing phases such as micaceous material or slate impregnated with sulfide inclusions. The microstructure of some of the inclusions from **figure 4.32** shows parallel foliation in the microstructure that appears to confirm this interpretation.

Sample JR 392A is especially informative for reconstructing what must have been going on in these crucibles. The discovery of galena is important because it offers the strongest clue to the use of these crucibles in metallurgical operations, while the chemical composition of the metal droplets allows to place these samples within the same geological horizon of the ones discussed in the previous sections. The many tiny prills contain iron and sulfur as main impurities and are not in themselves especially telling. However, one much larger metallic globule can be seen trapped just below the border between the residue and the crucible (**fig. 4.32**), its chemical composition comprising, besides sulfur and iron, also relevant quantities of nickel and antimony (**table 4.10**), an association which mirrors quite closely that of one of the silver-related samples from section 4.5.2. And indeed, the large formations of Virginia's feldspar-rich pegmatites often contain those polymetallic sulfides that Jamestown chymists were testing for noble metals, tin and zinc (**Pegau 1937**). In sum, the data presented in this section is interpreted as evidence of the assay of galena-rich minerals, perhaps with an eye to their potential noble metal content. On a final note, the same interpretation of JR392A can be extended to other too as well, even though no lead-rich feldspars were found in them. However, the three appear closely comparable in their bulk chemical composition and in their macroscopic features too, and especially in the black and glassy look of their residue. It is exactly this glass-like appearance that requires, in the next section, further discussion as to a potential interpretation as glassmaking residues.

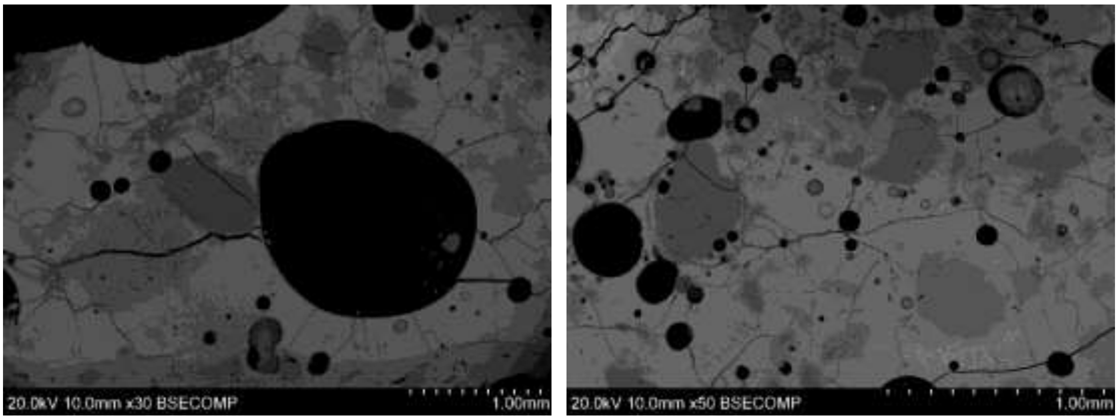


Figure 4.31. SEM backscattered micrographs showing unreacted feldspars (dark-grey) in sample JR1957A I.

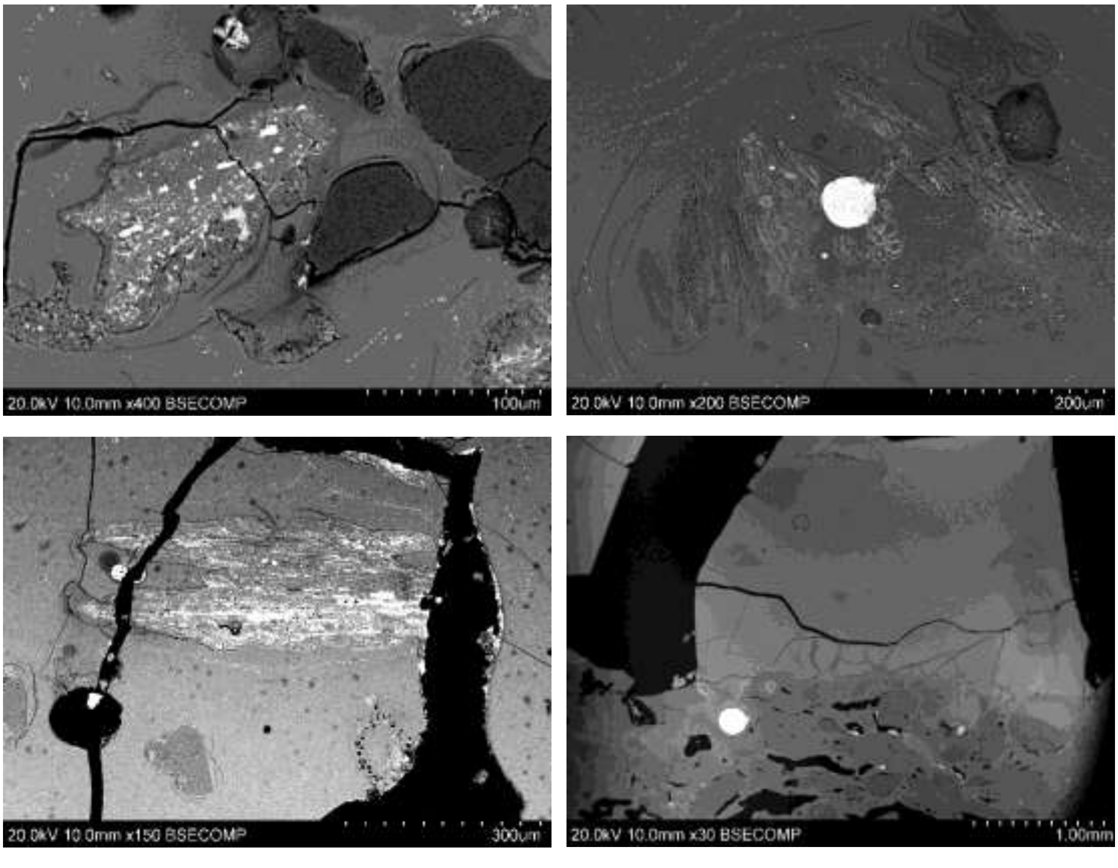


Figure 4.32. SEM backscattered micrographs showing lead-bearing feldspar inclusions in sample JR392A. Note the lead sulfide prills within the mineral or scattered in the glassy matrix.

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_20	CaO	TiO_2	MnO	FeO	As_2O_3	PbO
JR 392A	0.5	0.6	21.5	44.7	1.1	0.2	bdl	17.3	1.9	0.6	bdl	3.5	bdl	8.1
JR 392A	1.1	2.9	18.0	46.8	1.1	0.1	bdl	17.2	bdl	2.8	bdl	2.7	bdl	7.4
JR 392A	1.1	bdl	25.9	57.9	bdl	bdl	bdl	10.0	5.0	bdl	bdl	bdl	bdl	bdl
JR 392A	1.9	bdl	24.7	57.5	bdl	bdl	bdl	8.2	6.4	bdl	bdl	0.5	bdl	0.8
JR 392A	bdl	bdl	23.8	55.4	bdl	bdl	bdl	20.8	bdl	bdl	bdl	bdl	bdl	bdl
JR 392A	2.3	bdl	24.7	54.9	bdl	bdl	bdl	14.4	1.7	bdl	bdl	bdl	0.1	1.8
JR 392A	3.8	bdl	25.2	58.8	bdl	bdl	bdl	6.3	6.0	bdl	bdl	bdl	bdl	bdl
JR 392A	bdl	bdl	23.3	56.2	bdl	bdl	bdl	19.3	1.2	bdl	bdl	bdl	bdl	bdl
JR 392A	1.8	bdl	20.1	59.2	bdl	bdl	0.1	16.2	0.9	bdl	bdl	0.5	bdl	1.3
JR 392A	0.6	0.3	22.8	55.7	bdl	bdl	bdl	20.1	bdl	bdl	bdl	0.5	bdl	bdl
JR 392A	6.8	bdl	26.4	59.2	bdl	bdl	bdl	0.1	7.4	bdl	bdl	bdl	bdl	bdl
JR 392A	0.7	0.9	19.4	48.2	0.3	1.0	bdl	16.7	bdl	0.6	bdl	2.7	bdl	9.6
JR 392A	0.2	0.5	22.7	48.8	0.3	bdl	bdl	19.8	bdl	0.8	bdl	1.2	bdl	5.6
JR 392A	0.5	0.9	23.5	47.7	0.3	0.4	bdl	21.2	bdl	0.8	bdl	1.3	bdl	3.5
JR 392A	0.4	bdl	23.3	56.1	bdl	bdl	bdl	19.8	bdl	bdl	bdl	0.4	bdl	bdl
JR 392A	0.7	1.9	22.6	39.7	0.5	2.7	bdl	17.8	bdl	1.6	bdl	6.6	0.1	5.9
JR 392A	2.1	bdl	23.1	56.9	bdl	bdl	bdl	15.3	1.1	bdl	bdl	bdl	bdl	1.6
JR 392A	0.7	0.8	22.8	40.2	0.6	2.3	bdl	19.7	bdl	0.7	bdl	2.3	bdl	10.1
JR 392A	1.4	bdl	23.3	56.2	bdl	bdl	bdl	17.4	1.0	bdl	bdl	bdl	bdl	0.7
JR 392A	0.6	0.7	25.2	36.7	bdl	1.4	bdl	21.2	bdl	0.7	bdl	2.0	bdl	11.4
JR1957A I	8.0	bdl	25.0	61.1	0.2	bdl	bdl	0.2	5.7	bdl	bdl	bdl	bdl	bdl
JR1957A I	5.3	1.0	22.2	40.0	bdl	bdl	bdl	18.2	2.0	bdl	0.7	10.5	bdl	bdl
JR1957A I	5.3	1.1	21.4	39.7	0.3	bdl	bdl	17.7	2.0	bdl	0.6	11.2	bdl	0.6
JR1957A I	7.7	bdl	25.1	61.1	bdl	bdl	bdl	0.2	5.9	bdl	bdl	bdl	bdl	bdl
JR1957A I	7.0	bdl	26.1	59.7	bdl	bdl	bdl	0.4	6.8	bdl	bdl	bdl	bdl	bdl
JR1957A I	7.9	bdl	24.9	61.3	bdl	bdl	bdl	0.3	5.6	bdl	bdl	bdl	bdl	bdl
JR1957A I	7.5	bdl	25.2	60.9	bdl	bdl	bdl	0.4	6.0	bdl	bdl	bdl	bdl	bdl
JR1957A I	7.2	bdl	25.9	59.9	bdl	bdl	bdl	0.2	6.9	bdl	bdl	bdl	bdl	bdl

JR1957A I	6.9	bdl	26.5	59.3	bdl	bdl	bdl	0.2	7.1	bdl	bdl	bdl	bdl	bdl
JR1957A I	6.7	bdl	25.0	59.5	1.4	bdl	bdl	0.5	6.3	bdl	bdl	0.5	bdl	0.3
JR1957A I	6.5	bdl	27.3	57.7	0.2	bdl	bdl	0.1	8.2	bdl	bdl	bdl	bdl	bdl
JR1957A I	7.4	bdl	25.7	60.1	bdl	bdl	bdl	0.5	6.4	bdl	bdl	bdl	bdl	bdl
JR1957A I	7.6	bdl	25.3	60.6	bdl	bdl	bdl	0.3	6.2	bdl	bdl	bdl	bdl	bdl
JR 2718M	5.5	1.2	12.4	51.7	bdl	bdl	0.6	23.1	2.4	0.5	bdl	2.6	bdl	bdl
JR 2718M	3.0	bdl	22.0	55.0	bdl	bdl	0.1	18.7	1.2	bdl	bdl	bdl	bdl	bdl
JR 2718M	0.4	1.3	24.9	48.1	bdl	bdl	bdl	20.9	1.4	0.5	bdl	2.4	bdl	bdl
JR 2718M	3.1	bdl	20.9	53.8	bdl	bdl	0.2	21.7	bdl	bdl	bdl	0.4	bdl	bdl
JR 2718N	13.9	1.9	14.8	43.9	bdl	bdl	1.4	9.2	bdl	bdl	bdl	14.9	bdl	bdl

Table 4.9. Average bulk composition of mineral inclusions. All results in wt% and normalised to 100%

JR392A	O	S	Fe	Ni	Sb	Pb
Prill (large)	8.5	3.3	0.3	5.1	0.5	82.2
Prill	10.6	11.8	0.6	0.6	bdl	76.5
Prill	35.7	0.6	10.4	bdl	bdl	53.3
Prill	8.3	12.3	bdl	bdl	bdl	79.4

Table 4.10. Chemical composition by SEM-EDS of metallic prills found in sample JR392A

4.5.4.1 GLASSMAKING EVIDENCE?

In a recent study, a number of crucibles fragments from the same early fort context and showing the same dark glassy residue were analysed for their chemical composition (**Owen et al 2014**). The results are in general accordance with the data presented here, with a closely compatible bulk chemistry of the residues. It is safe to assume, therefore, that the two sets of samples are the product of the same chymical operations. But rather than to metallurgy, Owen and collaborators connect the crucibles to the glassmaking efforts that in 1608 brough to the production of the already mentioned trial of glass by German glassmakers (cfr section 4.1) (**Smith 1907: 147; Harrington 1972**). A comparable sample had already been reported, with the same interpretation as glassmaking waste, by Robert Brill in his *Chemical Analyses of Early Glasses* (**Brill 1999: 443**). On the basis of the chemical composition of the glassy residues, Owen and collaborators hypothesise that the ingredients used for making the glass were sand, beech ashes from the nearby woodlands, kelp ashes from the shoreline, pulverised oyster shells as a source of lime and (potentially) broken glass cullet of the type imported from Europe. However, as argued in a recent article based on the new data produced in this thesis, there seem to be several aspects which cast serious doubts on this interpretation (**Rehren et al 2019**). First, it is sensible to expect that any glass made in Jamestown would have followed contemporary European practice, using either wood ash or the ashes of other alkali-rich plants like kelp as fluxing agent. The former would have produced a composition rich in potash, lime and phosphate (**Jackson and Smedley 2004; Jackson et al 2005; Wedepohl and Simon 2010; Dungworth et al 2013**) while in the latter soda would have been predominant (**De Raedt et al 1999; Dungworth and Degryse 2009**). But this is clearly not the case here, as the samples analysed are sensibly enriched in both alkalis but have very low levels of phosphate and lime. It was shown how, for the most part, the high alkalis derive from feldspar grains, and only minimally (if at all) from other potential fluxing substances. The practice of employing feldspars as glassmaking ingredients/reagents is not unknown at the time, but its occurrence is extremely rare and geographically restricted to West Africa (**Lankton et al 2006; Freestone 2006; Babalola et al 2018**). A second observation is that Owen and collaborators report some notable enrichments in the glass matrices, namely the oxides of antimony (ca. 6-7 wt% Sb_2O_5) and boron (ca. 2wt% B_2O_3), but could not find a satisfactory explanation as to their source (**Owen et al 2014: 77**). Indeed, while antimony was a common decolourising reagent added to glass in antiquity, the levels of antimony oxide in glasses decolourised this way are usually below one percent (**Jackson 2005; Silvestri et al 2008; Foster and Jackson 2010**). Moreover, in post-Medieval Europe it is manganese and not antimony the common reagent employed to decolour glass (**Sayre 1962; Verità 2013**). A much more sensible explanation for the antimony enrichment is that it was part of the assayed ore, not unlike what seen in other samples analysed here. The same is true for boron. Again, high-boron glasses obtained by using borate evaporites as flux have been recently recognised at late antique to Medieval Pergamon, in Turkey (**Schibille 2011; Rehren et al 2015**) and in 16th-century Iznik glazes (**Tite et al 2016**). However, there is no evidence suggesting that this technology was known to European glassmakers of the 17th century or even still in use at this time. More likely, boron entered the batch as contamination in the mineral source, probably in the form of boron-rich tourmaline minerals which are known to occur in pegmatite bodies of Virginia (**fig. 4.33**) (**Pegau 1937; Brown 1962; Sweet 1989: 27**).

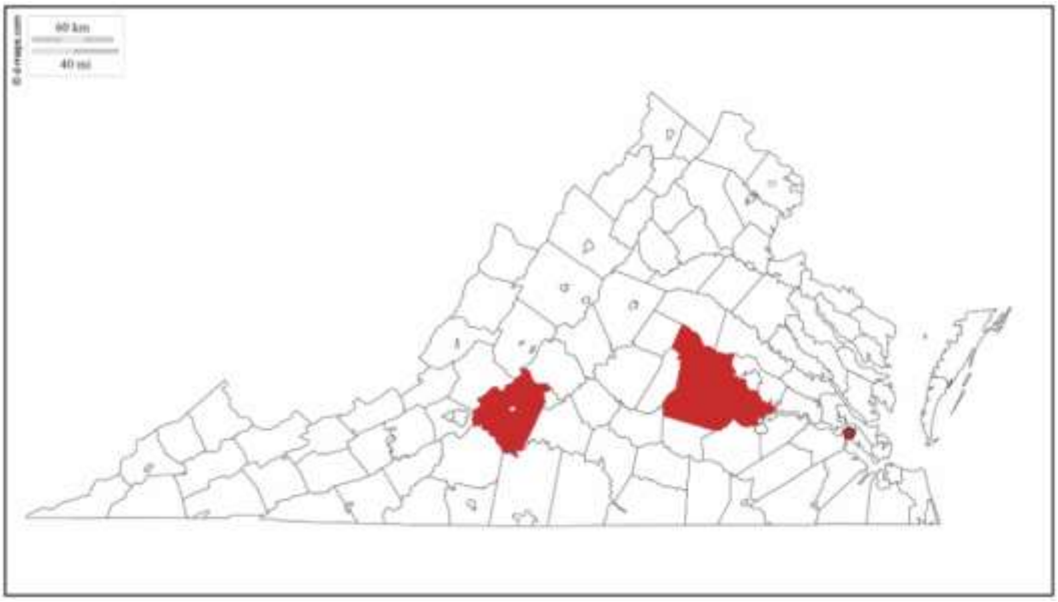


Figure 4.33. Map of Virginia with location of main pegmatite bodies (dot is Jamestown).

Third, a close observation of the glassy residue stuck to the crucible analysed by Owen and collaborators further points to its origin during metallurgical activities. The form and distribution of the slag displays what appears to be the negative impression left by the bath of liquid metal that solidified underneath the slag, leaving a smooth surface where the two were in contact (**fig. 4.34A**). Moreover, the "fin" protruding from the inner side wall of the crucible, visible in both the sample analysed in 2014 and in this thesis, indicates that the slag was floating on top of the pool of denser metal (**fig. 4.34B-C**) (see Alipour et al 2021). Finally, the crucible to which one of the samples analysed here belongs clearly shows that the slag is only preserved towards the rim, while the rest of the surface, protected by the liquid metal, is not coated (**fig. 4.34D**) (**Rehren et al 2019**).

Figure 4.34 A-B: Sample J14 from Owen et al 2014. Note the smooth surface underneath the residue and corresponding to the contact area with the pool of molten metal underneath (A) and the "fin" protruding from the internal side of the vessel (B). C: Sample JR92A also showing a similar protruding feature. D: crucible fragments from the same context of JR1945C. Note the clean internal wall of the vessel, protected by the molten metal and the residue only forming towards the rim (Photos: Marcos Martinón-Torres and Preservation Virginia's Jamestown Rediscovery Foundation).

Analysis of glassmaking crucibles from the literature shows that the glass settles at the bottom of the vessel and that no glassy slag is normally found on the side walls (**Rehren and Pusch 2005**, particularly figures 2 and 4). In light of these observations, some of which were made possible by the SEM-EDS characterisation of the microstructure not available so far, the crucible fragments investigated here and those reported by Owen and collaborators are more consistent with the testing of a feldspar-rich local mineral rather than with primary glassmaking. Nonetheless, there is no reason to doubt that some glass was indeed produced at Jamestown in 1608. In this respect, two large triangular crucibles with a thick porous off-white residue in them are probably more appropriate candidates as glass-related (**fig. 4.35**). None has been thoroughly analysed yet. However, one very small sample which seem to be coming from one of them (JR741A) is discussed in the next section, although its high corrosion does not allow to make more informed hypotheses.

Figure 4.35. Large triangular crucibles with white frothy residue cakes, likely used for glass making (photos by Marcos Martinón-Torres)

4.5.5 Others

There are seven samples which need to be considered separately as they cannot be confidently ascribed to the categories discussed so far. However, we shall see that for most of them a connection with the same chymical operations of mineral testing can be hypothesised. The first is sample JR2G, a fragment of translucent green window glass with a crusty brownish corrosion film on the surface. It is one of the thousands of pieces of scrap glass, or cullet, recovered during excavation (**fig. 4.3**). The bulk chemical composition (**table 4.11**) resulted particularly enriched in lime (22.6 wt% CaO), with other notable enrichments being those in magnesia (2.8 wt% MgO) and phosphate (1.9 wt% P_2O_5) that indicate the glass was made using plant ashes as opposed to a mineral or synthetic form of alkali flux (**Jackson et al 2005**). However, the low levels of soda (0.8 wt% Na_2O) and potash (4.5 wt% K_2O) distinguishes this composition from that of contemporary wood ash glasses (**Jackson and Smedley 2004; Wedepohl and Simon 2010**), and rather indicates the so-called high lime low alkali type (HLLA) of post-Medieval northern Europe (**Dungworth 2011; 2012; Dungworth et al 2013**). Some fragments of HLLA glass of comparable composition are reported in the 2014 paper by Owen and collaborators, together with fragments with higher alkalis and lower lime, indicating that both types were present at Jamestown in the form of cullet (**Owen et al 2014**). Sample JR2G must therefore be seen as part of the fort's reservoir of scrap glass, which the settlers had brought from Europe presumably to be employed for everyday needs like glazing buildings and as a potential reagent in furnace operations (cfr sections 4.1). The rest of the samples to be considered here belong to crucibles. Two of these come from small crucibles and can be connected to similar furnace operations. The first, sample JR2718W, displays a thick, grainy and rusty-looking residue, indicating the presence of metal and suggestive of extended corrosion (**fig. 4.35A**). This is confirmed by the bulk chemical composition of the residue matrix, which is made of lead and chlorine (ca 75 wt% Pb and 14 wt% Cl). Scattered throughout the matrix are also frequent metallic droplets (**fig. 4.36B**), whose composition (ca. 85 wt% Pb and 15 wt% S) is consistent with galena (PbS). This offers a connection with the typology of minerals assayed in search of noble metals and discussed in earlier in the chapter as galena often contains variable quantities of silver. Since the fragment comes from the lower section of the original crucible it is likely to represent lead bullion, now very corroded, which originally would have collected any gold or silver after the ore reducing process. However, no gold or silver was detected in appreciable quantities here.

In a similar way to what just observed, sample 152-JR is the fragment of a very small triangular crucible and it displays a uniform black discolouration on both its inside and outside surface (**fig. 4.36C**). No fully formed residue can be seen adhering to the vessel like in most of the samples analysed here, and the inner surface appears particularly clean. At high magnification the cross-section doesn't contain slagged layers, metallic droplets or any other phase worth noting, except for one very thin and heterogeneous deposit showing various small phases (**fig. 4.36D**). The bright areas are rich in silver, lead, phosphorus and iron, while the surrounding light grey matrix also contains sulfur and barium. The enrichment in lime and phosphate detected in the deposit points to the usual presence of charcoal to attain the necessary reducing atmosphere, while the association of silver, lead and iron and the presence of sulfur and barium is reminiscent of the silver-bearing galena minerals with barite gangue discussed in section 4.5.2. In this sense, sample 152-JR too is likely to be a product of the very same activities of mineral prospection mirrored in the vast majority of the assemblage investigated here. Presumably, the pool of metal sitting at the bottom of the vessel is the reason why the inner surface looks free of a slaggy layer (cfr section 4.5.4.1). But unlike what seen in sample JR2718W, no trace is left of this metallic bullion. That these two crucibles were probably employed in testing and refining activities is further hinted by their small size, offering better control and more careful and precise quantification of the components.

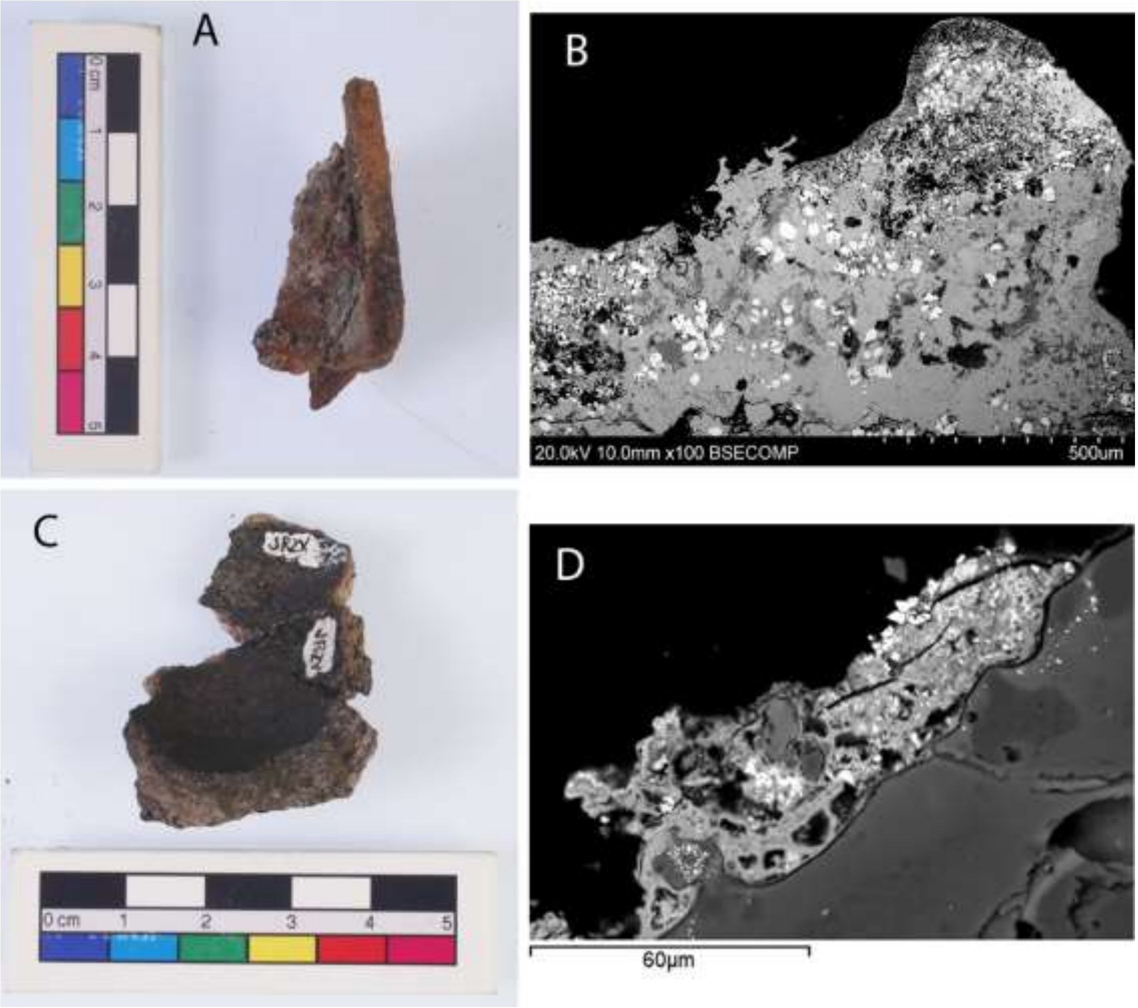


Figure 4.36. A-B: Sample JR2718W with corroded lead-rich residue stuck to the internal surface. The microstructure of the residue shows bright areas of lead sulfide in a matrix of corroded metal. C-D: Sample 152-JR with clean internal surface and a very thin and heterogenous deposit enriched in lead and silver.

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO	FeO	AgO	BaO	PbO
JR2G	0.8	2.8	1.6	63.5	1.9	0.4	bdl	4.5	22.6	0.3	0.7	0.9	bdl	bdl	bdl
152-JR	bdl	0.3	1.5	bdl	14.5	4.3	0.3	bdl	5.6	bdl	bdl	36.2	19.8	0.9	16.5

Table 4.11. Average bulk composition by SEM-EDS of the residue layers in samples JR2G and 152-JR.

The next three samples to be considered here can also be tentatively linked to similar crucible operations. The first is JR2718M, with a dark and grainy residue that visually resembles very closely the feldspar-rich slaggy layers discussed earlier (**fig. 4.37**). The resemblance also applies to compositional and microstructural features. Indeed, under the SEM the residue looks glassy and rather homogeneous, with light grey amorphous phases corresponding to partially melted potassium-rich feldspars (**fig. 4.37**). The bulk composition of the residue (**table 4.12**) is very rich in silica (67.0 wt% SiO_2) and in alumina (14.8 wt% Al_2O_3), while the alkalis expected from the feldspar inclusions appear to have partially leached away through weathering (4.4 wt% K_2O and 1.8 wt% Na_2O). Other enrichments include iron oxide (3.9 wt% FeO), as well as charcoal-related elements lime (5.2 wt% CaO) and magnesia (1.6 wt% MgO). SEM-EDS didn't detect any traces of heavy elements potentially associated with a metallurgical use of this crucible. However, the strong similarities with the samples discussed in section 4.5.4 align this fragment with the same prospecting activities, most likely in search of noble metals through the reducing fusion of feldspar-rich minerals.

The second related sample is JR2718N, a lump of frothy and grainy residue taken from the inside of a crucible (**fig. 4.38**). Reading this sample is complicated by the degree of corrosion and porosity, which has caused a leaching of soluble components like alkalis and an increase in silica (**table 4.12**). What can be said is that the microstructure has a cracked glassy appearance that is remarkably similar to that of JR2718M, just discussed. Some of the enrichments detected such as those in alumina, iron oxide and alkalis derive from light grey phases representing remnants of iron-bearing feldspar inclusions (**fig. 4.38**), while the high lime might derive from charcoal or mineral gangue components in the original charge. Overall, it is rather difficult to provide a satisfactory explanation regarding the process that resulted in this residue, but the similarities with JR2718M allow to hypothesise a relation to similar assay activities.

The third sample, JR2436B, again displays significant visual similarities to the feldspar-rich class of fragments as well as to JR2718M. It is a small crucible fragment with a dark grainy residue stuck to it and showing orange rusty spots on the surface (**fig. 4.39A**). Under high magnification, the first thing to note is the high degree of interaction between the glassy residue and the ceramic body, with quartz grains coming from the latter which can be seen floating in the former (**fig. 4.39B**). The contribution of crucible matter in the residue's matrix takes the form of a substantial enrichment in alumina (14.9 wt% Al_2O_3) and titania (0.9 wt% TiO_2). Conversely, the relatively high levels of lime (20.2 wt% CaO), magnesia (4.0 wt% MgO) and phosphate (1.3 wt% P_2O_5) suggest that the main component of the charge was charcoal. Such enrichments may also have been caused by the addition of crushed glass of the high-lime type as reagent. However, a remnant of charcoal with characteristic cellular structure can be seen in the residue's matrix (**fig. 4.39C**) confirming the presence of this reagent in the charge. That the chemical reactions undergone in the crucible must have been strongly reducing is further indicated by the scatter of tiny iron phosphate prills that formed around the piece of charcoal (**fig. 4.39D**). Thus, the overall picture for sample JR2436B appears compatible with the processing of iron-rich minerals under particularly reducing conditions. As discussed before, pyrites and similar minerals are abundant in Virginia and Jamestown chymists seem to have been tested them possibly in search of gold or silver.

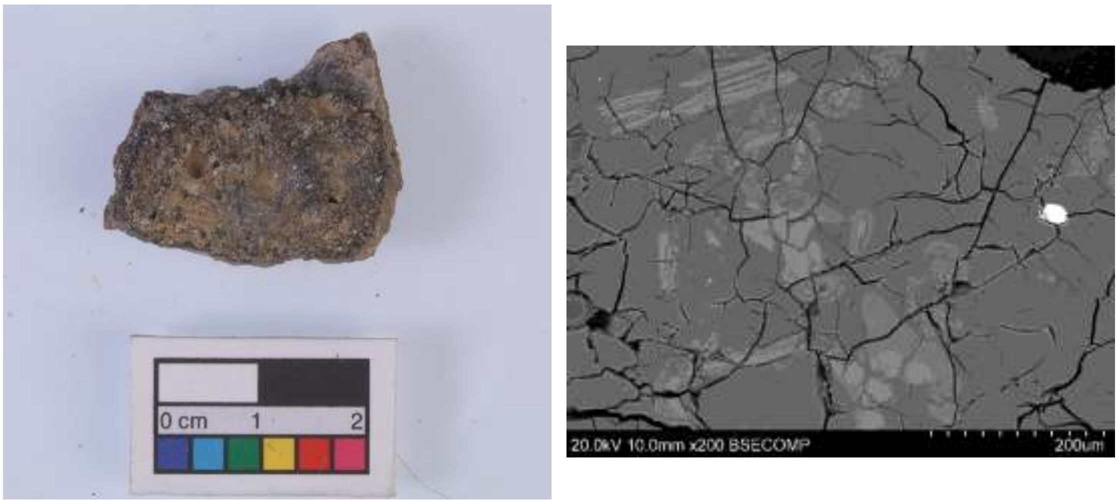


Figure 4.37. Overview of sample JR2718M. Note the light grey phases indicating remnants of feldspar inclusions (right).

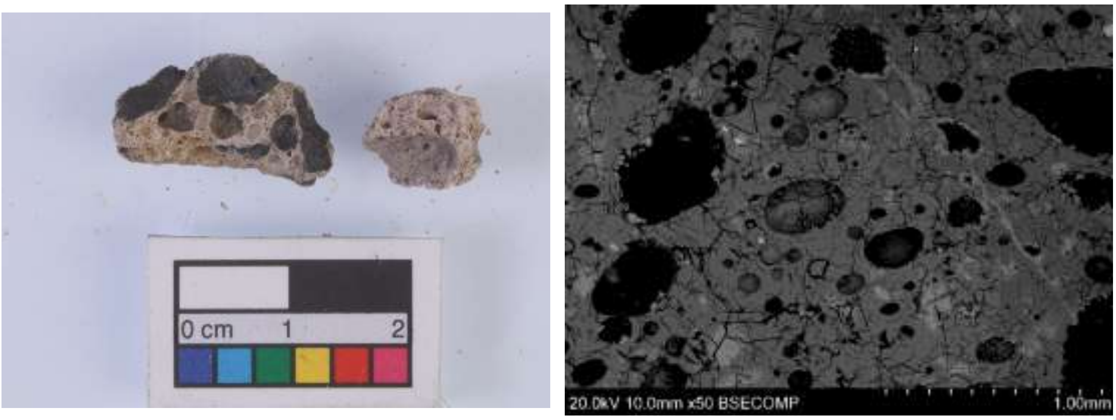


Figure 4.38. Overview of sample JR2718N, with corroded cracked microstructure and also showing light grey areas representing remnants of feldspar inclusions (right).

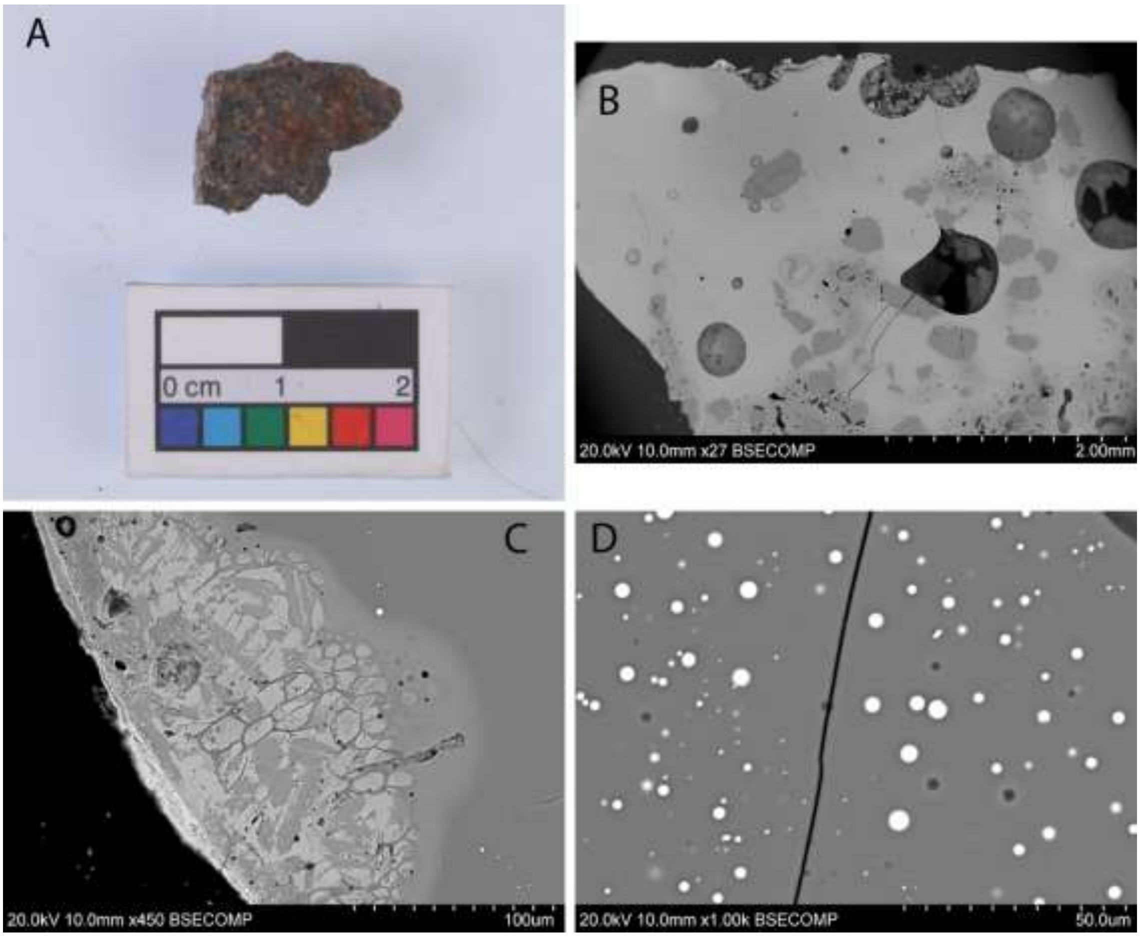


Figure 4.39. Overview of sample JR2436B. The residue appears glassy and the boundary with the ceramic is diffused with a high degree of interaction (B). Note the charcoal imprint towards the edge of the residue (C) as well as the scatter of tiny iron prills due to very reducing conditions around it (D)

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO	FeO	AgO	BaO	PbO
JR2718M	1.8	1.6	14.8	67.0	0.1	0.3	0.3	4.4	5.2	0.6	bdl	3.9	bdl	bdl	bdl
JR2718N	1.0	0.6	6.3	83.0	bdl	bdl	0.2	2.5	3.2	0.8	bdl	2.6	bdl	bdl	bdl
JR2436B	1.2	4.0	14.9	50.3	1.3	bdl	bdl	3.2	20.2	0.9	0.6	3.5	bdl	bdl	bdl

Table 4.12. Average bulk composition by SEM-EDS of the residue layers in samples JR2718M, JR2718N and JR2436B

The final sample to be discussed, JR741A, has already been mentioned at the end of the previous section in potential relationship to glassmaking activities. It is a very small fragment which appears to come from one of the large triangular crucibles with a frothy off-white residue shown in **figure 4.35**. The sample shows a vitrified intermediate layer sitting between the ceramic and a thin whitish residue (**fig. 4.40A-B**). At high magnification the latter appears fully corroded (**fig. 4.40C**), with a chemical composition dominated by wood ash elements such as magnesia (7.6 wt% MgO), phosphate (1.9 wt% P_2O_5), lime (12 wt% CaO), manganese oxide (1.2 wt% MnO) and enriched in ceramic components such as alumina (7.9 wt% Al2O3), iron oxide (2.6 wt% FeO) and titania (1.4 wt% TiO_2.) (**table 4.13**). The intermediate vitrified layer gradually diffuses into the ceramic phase without clear demarcation (**fig. 4.40D**). Its chemical composition is comparable to European potash-lime-silica glasses of the time (**Jackson and Smedley 2004; Wedepohl and Simon 2010**), but it is also unsurprisingly enriched in components from the molten ceramic such as alumina (18 wt% Al_2O_3), titania and iron oxide (both at 1 wt%), and represents a mixture between glass batch and crucible elements. Finally, the ceramic is highly vitrified and severely affected by the heat, with the frequent presence of rounded bloating pores throughout the matrix (**fig. 4.40D**). The chemical makeup of the ceramic fabric shows that the transfer of elements has also occurred in the opposite direction, with contamination from the glass batch visible in levels of silica (64.6 wt% SiO_2), potash (3.2 wt% K_2O), magnesia (1 wt% MgO) and iron oxide (4 wt% FeO) that are higher than in the other crucibles, and lower levels of alumina (25.4 wt% Al_2O_3) and titania (1.2 wt% TiO_2).

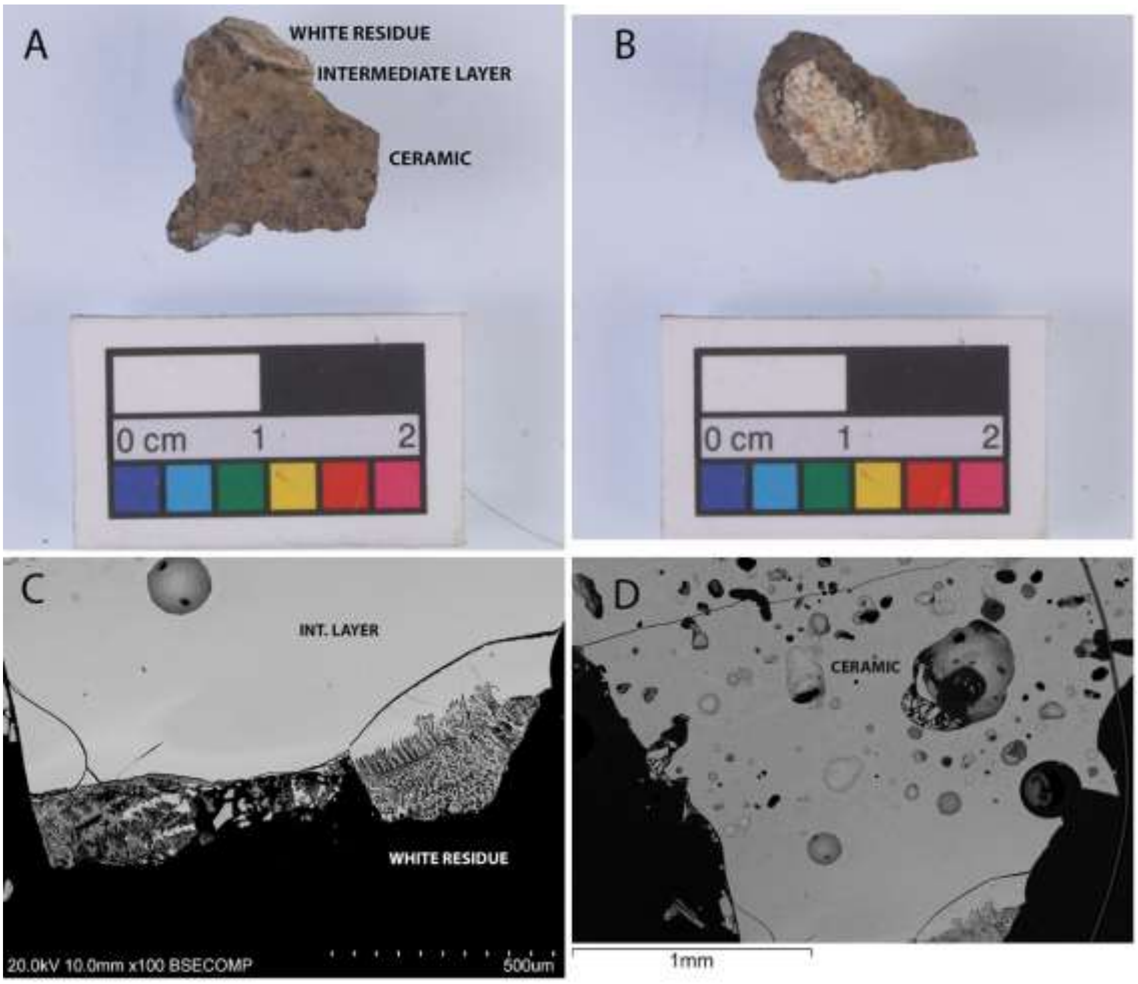


Figure 4.40. A-B: Side and top view of sample JR741A showing the white residue, the intermediate vitrified layer and the ceramic underneath. C-D: SEM backscattered micrographs showing the three layers at high magnification. Note the extreme corrosion characterising the white residue and the high degree of interaction between the glassy intermediate layer and the crucible ceramic.

JR741A	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	Cl	K_2O	CaO	TiO_2	MnO	FeO
White residue	1.2	7.6	7.9	59.0	1.9	0.2	5.0	12.0	1.4	1.2	2.6
Intermediate layer	2.1	0.9	18.0	60.6	bdl	0.1	8.2	8.2	1.0	bdl	1.0
Ceramic	0.5	1.0	25.4	64.6	bdl	bdl	3.2	0.2	1.2	bdl	4.0

Table 4.13. Average bulk composition by SEM-EDS of the three layers in samples JR741A

4.6 DISCUSSION: TESTING THE NEW WORLD

The preceding sections provide a wealth of new information on high-temperature chymical practices at the site of early Jamestown. The data helps drawing the contours of the complex and rich scenario surrounding the early English attempts to establish an industrial enterprise in the new world. Prospecting the mineral resources of Virginia is by and large the most represented type of activity surfacing from the analysis of the crucibles, and the remarkable compositional variability encountered within the residues bears witness to the wide spectrum of ingredients and reagents that were involved in the assaying operations. Among the several minerals tested, the quest for noble metals seems to play a major role in the assemblage, hardly surprising since they constituted one of the biggest driving forces behind the colonisation of the American continent. Deposits of gold in Virginia could have been accessed by navigating the river James upward (**Taber 1913; Sweet et al 1989: 2-3**), which we know the settlers did in more than one occasion, and gold features in some of the written accounts at our disposal (cfr section 4.1). However, the assemblage investigated only offers indirect evidence of gold prospecting. Silver, on the other hand, is well represented in the crucibles, partly as an enrichment in the slag layers and in oxide phases found therein, but mostly as metallic prills incorporated in the residue matrices. The chemical characterisation of the residues suggests that the minerals assayed by Jamestown metallurgists were complex silver-bearing polymetallic sulfides (**Sweet 1976**). These were first subjected to reducible fusion in the crucible so to concentrate the silver in a lead bullion, with cupellation of the bullion following and resulting in the isolation of the precious metal. The ores tested are strongly reminiscent of fahlores that we know were assayed and exploited in post-Medieval central Europe (**Mongiatti et al 2009a**), and described in metallurgical texts like Agricola's *De Re Metallica* (**Hoover and Hoover 1950: 111-114**). Agricola even mentions silver sources that contain cobalt, antimony, and arsenic (**Hoover and Hoover 1950: 114**), an association that we have seen in one of the samples analysed here as well (cfr section 4.5.2 and see **Watson 1907:**

578). During one upriver exploration trip in 1608, soon after the coming of the supply ships, John Smith informs us that rock samples of potential interest were collected:

> "And in our returnes searched many places we supposed mines, about which we spent some time refyning, having one William Callicut, a refyner fitted for that purpose. From that crust of earth we digged, he perswaded us to beleeve he extracted some small quantitie of silver; and (not unlikely) better stuffe might be had for the digging." (**Smith 1907: 143**)

From the passage we known that assay operations – what Smith calls *refyning* – were carried out on the spot, presumably using small crucibles of the kind analysed here. The men then most likely dug some of the presumed silver ore and brought it back to the fort to undergo further and more thorough testing in the metallurgical workshop. The same or related ore bodies were also prospected for zinc and tin sources which may be used in the production of copper alloys. Copper industry was indeed a field where England had been lagging behind due to the scarcity of suitable mineral resources within its territory, with the effect that large quantities of tin and zinc had to be imported. In the 16th century, during the reign of Queen Elizabeth, the Society of Mines Royal and the Society of Mineral and Battery Works were created and large-scale prospection and metalworking had become a priority (**Day 1998; Hudgins 2005**). The colonisation of Virginia was a great opportunity to further implement this strategy of independence. Moreover, establishing a production of brass and bronze would certainly have been a very inviting prospect to the Virginia Company, many of whose shareholders were themselves members in the English copper monopolies (**Hudgins et al 2009**).

However, when it comes to written documents we know even less about copper than what little we do know about noble metals prospection. The most interesting pieces of evidence is a letter from 1607 in which the Spanish ambassador in London Don Pedro de Zuniga informs his king that among the resources discovered by the English in Jamestown is a "soil from which it seems to them they may obtain bronse" (**Hudgins**

2005: 61). It has been suggested that *bronse* is indeed to be read as brass (**Hudgins et al 2009**), which makes the ambassador's letter the only direct evidence explicitly mentioning this metal (and, by extension, zinc) and its quest in relation to the colonial venture. But in spite of the high expectations, the early life of the colony was a period of enormous difficulties for the settlers whose number got quickly decimated by diseases, lack of food and the frequent conflicts with the indigenous populations. No gold, silver or copper alloys were ever exploited on the scale everyone expected, even though the results presented in this thesis show that some prospecting did happen and that sources of silver as well as of zinc and tin were indeed discovered and tested by the metallurgists. Equally, the plan to establish a glassmaking industry in Jamestown and begin a production of glass on a scale that could satisfy the English market became impossible, and as far as we know only the initial test batch was produced and shipped back home (**Harrington 1972**). However, nothing more is known regarding this trial of glass and so far, only a few crucibles discovered at the site seem to contain residues that can be tentatively linked to glass production. The tiny fragment analysed in this thesis appears so heavily corroded that virtually no informed inference can be made at this stage regarding what local glass was made with. Some compositional clues seem to suggest a substantial similarity to European wood ash glass of the period, but a satisfactory answer will have to wait for more data. The other glassy slag layers found on a number of crucibles previously interpreted as glassmaking remains (**Owen et al 2014**), were re-assessed here and their link to metallurgical operations of mineral prospection seems now more likely (**Rehren et al 2019**).

Thus, if Jamestown was to be judged against the benchmark of the initial expectations it can be regarded as a failed colonial and industrial endeavour, the settlers uncapable of fulfilling the commands of the Virginia Company investors who had ventured their money in it. Indeed, if we take a look at virtually all early European transatlantic colonies, that of failure appears to be a recurring narrative. Common to all these ventures were the same gilded aspirations and dreams of wealth, and all witnessed metallurgical activities which were quickly abandoned or produced disappointing outcomes. At La Isabela, the first European town in the new world, founded in 1496 by

Christopher Columbus in today's Haiti, archaeologists have discovered triangular crucibles and other remains of metallurgical activity (**Deagan and Cruxent 2002; Thibodeau et al 2007;**). But here too European hopes clashed with the need to survive in a hostile environment and with the failure to find any viable sources of precious metals. A similar fate was met by the short-lived French settlement of Cap Rouge in Quebec, only active between 1541 and 1543, where archaeological discoveries bear us witness to the attempts at locating precious metals, which we know were unsuccessful too (**Cote 2009; Martinón-Torres 2010; Monette 2013**). A few decades later it was the English who set out to America with the same desires. In the 1570 Martin Frobisher guided an expedition to the Canadian arctic in search of the northwest passage and potential gold sources, setting up assay operations which resulted in shiploads of worthless rock mistaken for gold (but containing none) being sent to England (**Beaudoin and Auger 2004**). Evidence of metallurgical chymistry was also discovered at the site of one of the North Carolina settlements founded in the 1580s by Walter Raleigh (1552-1618), where experimental work was directed by the natural philosopher Thomas Hariot (1560-1621) (**Nöel-Hume 1994**). Attempts at establishing a permanent colony failed here too and ended with the notorious mystery of Roanoke's lost colony.

In this sense, the story of Jamestown overlaps with that of many other early European colonies where precious metals were never found in substantial amounts and the events were soon to take a turbulent turn. But the material landscape emerging from the study of the chymistry-related contexts shows how the similarities between early colonial efforts are not restricted to the aims that fuelled them and the initial disappointing outcomes of the initial years. The results outlined in the previous sections have shown that material culture exposes a whole network made of shared chymical practices and supply of laboratory equipment, allowing us to explore similarities that revolve around day-to-day histories. The example of the non-Hessian crucible from Jamestown and its potential link with French colonial activities is of course a drop in the ocean, but it indicates that material evidence holds the potential for new and fresh points of view which may help writing a more comprehensive history of European colonisation of the new world. In this respect, the traditional view that blamed the failure on the incompetence and even the laziness of Jamestown's colonists (**Morgan 1975**) appears an oversimplification. On the contrary, the archaeological record of Jamestown as well as that of the other contexts mentioned above, has shown us that some of the most technologically advanced pieces of apparatus were employed in search of mineral wealth, like the triangular German crucibles recovered from all the sites excluding Cap Rouge where metallurgists employed high-quality French vessels instead (**Monette 2013**). The activity of skilled professionals is further suggested by the fact that on number one in the long list of 'sorts of men which are to be passed in this voyage', written by Richard Hakluyt in view of the 1580s expeditions to America, we read 'men skilful in all mineral causes' (**Wright 1965:34**), and that the list of men who arrived in Jamestown with the supply ships in 1608 contain several metallurgical specialists as well as glassmaker and practitioners of other professions. In some cases, mining experts were especially hired from Germany as Germans were renowned for their metallurgical skills. The chymist Joachim Gans was in charge of prospecting at Roanoke, while another German, Jonas Schutz, commanded the assay operations during the Frobisher enterprise of 1577-1578 (**Beaudoin and Auger 2004**). The activity of Germans is also known at Jamestown where they had been brought along as glassmaking experts (**Harrington 1972; Owen et al 2014; Kelso 2017**). Later in the 1620s, another attempt was made at producing glass once again by hiring foreign experts, this time Italians.

Where analytical studies have been carried out like in the present case, the reconstruction of the laboratory activities clearly indicates that, albeit short-lived, the chymical efforts were well-rooted in contemporaneous European mining and metallurgical knowledge, with the metallurgists targeting the typology of ores from which experience told them they could obtain silver and other metals. It is reasonable to assume that the same happened in regard to glassmaking and that technological traditions and artisanal recipes of the time were being followed in making glass with local ingredients. Nonetheless, the wholly new environment of Virginia is also likely to have triggered at least some departure from the established artisanal tradition of Europe. Indeed, it is reasonable to assume that the chymists would have encountered types of minerals and of other natural resources which they had not seen before. One possible example comes from the residues discussed in section 4.5.4, reflecting the assay of feldspar-rich bodies associated with silver-bearing minerals. The analysis of the inclusions found in the glassy residue shows that these are for the most part lead-rich potassium feldspars (**Appendix B**). These may be relics of the mineral amazonite, a blue-green variety of microcline ($KAlSi_3O_8$) considered a gemstone for its attractive look, that is abundant in pegmatite deposits in Virginia (**Pegau 1937: 111; Brown 1962: 49, 60; Sweet 1989: 27**) (**fig. 4.41**). The presence of lead impurities is believed to be the sources of amazonite's peculiar colour (see **Pivec et al 1981; Hofmeister and Rossman 1985; Julg 1998**) and there are relatively few known sources of the mineral in the world, virtually none of them in Europe. If this was the case then one could imagine how such beautiful green rock would have stimulated the curiosity of the chymists who may have thought of it as malachite ($Cu_2CO_3(OH)_2$) and consequently tested it for copper, or instead they may simply have wanted to know more about this somewhat mysterious substance. Whether or not shifting artisanal practices can be seen in the crucibles investigated here, the discovery voyages remain a moment of deep fracture with the past, a moment where early modern Europeans experienced a dramatic rearrangement of traditional worldviews. Existing philosophical schemes had no longer the power to explain the sheer amount of novelty that colonists were faced with and it was time for a new framework (**Principe 2011b; Rossi 2009**). It is in this sense that the transatlantic expeditions and discoveries of the 16th and 17th century played a key part in creating a new space for chymistry and chymical knowledge as a set of theoretical and practical spheres of knowledge, a most powerful tool that through

Figure 4.41. Specimen of amazonite from Virginia.

active manipulation allowed to explore, understand and, crucially, also to exploit nature. In many respects, chymistry was an overarching system capable of directing the shifting worldview that characterised early modern Europe. It was a key player in the narrative of progress that accompanies innovation in fields related to the discovery voyages, such as cartography, navigation and instrument making. In one of Johannes Stradanus's notorious series of engravings *Nova Reperta*, (**fig. 4.42**), from around the period of the Jamestown foundation, a female figure representing the new world can be seen entering from the left, while the man opposite, the old world, is ushered out. While the author had a clear agenda of exalting chymistry and progress (**Markey 2020**), it is nonetheless interesting to note how many of the means through which such progress is achieved are direct products of chymical work, such as the furnace and the distillation tools visible in the foreground.

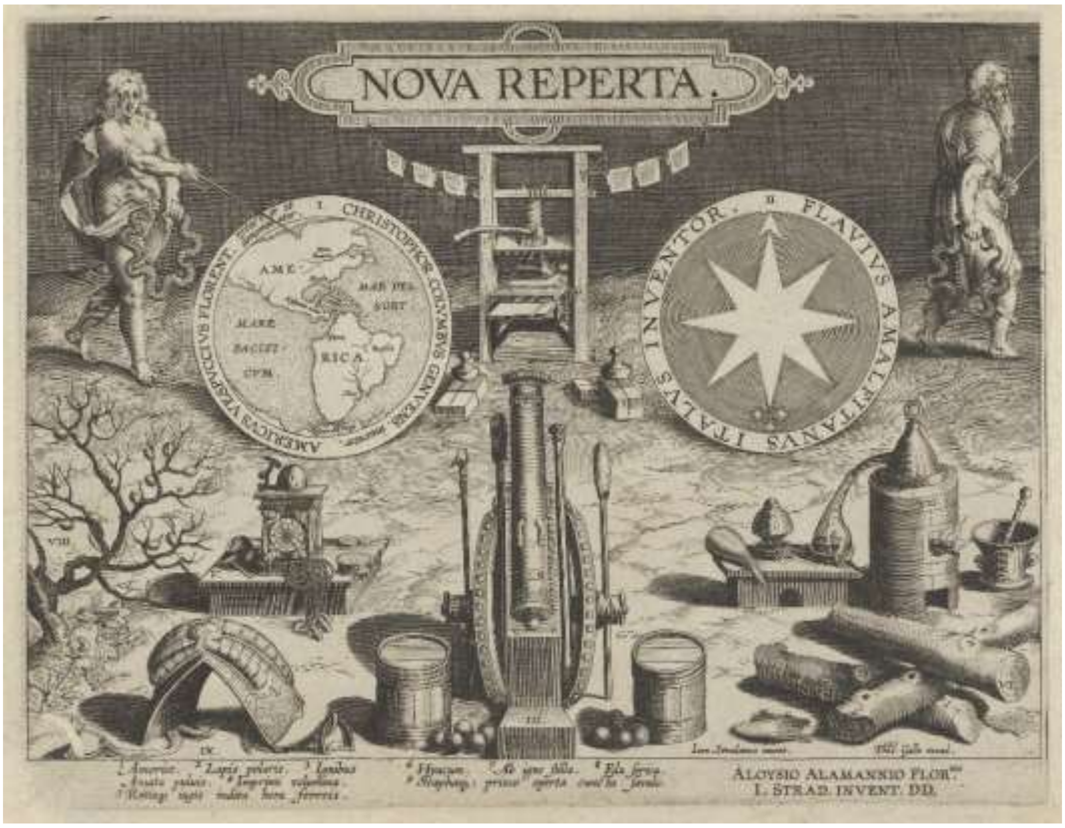


Figure 4.42. Nova Reperta, by Johannes Stradanus (ca. 1600).

Jamestown and the other early European colonies established in central and north America must be seen within this dynamic and fast changing context of shifting worldviews. The results presented in this chapter are largely in agreement with a scenario where expert artisans were at work amidst innumerable difficulties, which eventually rendered the efforts unsuccessful. Nonetheless, the chymists strove to collect, work with and make sense of a new and unknown natural environment which must have posed unprecedented challenges of adaptation to the European artisanal and scientific knowledge. After all, that the settlers were not lazy we are informed in a letter written by captain Smith himself to the Virginia Company, in which he passionately defends his men from the accusations of idleness and offers an accurate description of the tremendous efforts they have to make in order to keep things going (**Smith 1907: 147-150**). With the help of material culture, this chapter has thrown some new light on this side of the story. The next chapter will outline another material microhistory, one which takes place around a century after and a whole ocean apart from the early days at Jamestown, but which equally offers precious insights into material and practical aspects of early modern chymistry.

CHAPTER V

THE ASHMOLEAN LABORATORY

Figure 5.1. The Ashmolean Museum on broad street (1824 by H Slatter, Herald Office, Oxford)

5.1 INTRODUCTION

The Ashmolean Museum was opened in 1683 on Broad street, in Oxford, and it occupied the golden stone building that today houses - very conveniently – the History of Science Museum (**fig. 5.1**). The new institution was profoundly inspired by the work of Francis Bacon (1561-1626) and by his utopian community known as Solomon's House, after King Solomon and his proverbial wisdom. In the *New Atlantis* (1626), Bacon discusses an imaginary place where through the advancement of knowledge around the hidden causes of nature and the development of useful and fruitful technologies, the whole of the human race could be enhanced. The same overarching spirit had led to the foundation of the Royal Society in London in the 1660s, where scholars could gather, conduct experiments, keep a collection of rarities and curiosities and do what today we would call "research". The Ashmolean Museum took this agenda even further by including its activities within the context of Oxford University and of formal academic teaching. The building was developed on three floors, mirroring the three limbs that the institution was made of. The top floor was occupied by the collection of curiosities and the library, while the lecture theatre for the teaching of natural philosophy could be found on the ground floor. Last but not least was the *officina chimica*, the basement laboratory where the chymical work was carried out. Visitors to the laboratory were positively impressed and often report on how well planned and equipped it was (**Bennett et al 2000**). Besides the main space where the furnaces were operated, two more rooms are described, namely a library with books on chymical topics and a store room were various chemicals and ingredients were kept. It was clearly a state-of-the-art space for experimental chymistry and gave students the chance to learn natural philosophy from an overarching perspective which included hands-on practice with objects, substances and chemicals.

Chymistry was still a newcomer to university, having entered the curricula during the seventeenth century when an ever-increasing number of institutions throughout Europe had started to include this subject as part of medical education (**Debus 1990; Guerrini 1994**). The discipline was called *chymiatria* or *iatrochemistry*, and focused on the chemical preparation of medicinal substances from a wide spectrum of natural materials, as popularised by the Swiss physician and reformer Paracelsus. The bond between chymistry and medicine remained strong throughout the seventeenth century and in part characterises the teaching at the Ashmolean laboratory too, particularly during the following century when the laboratory was also used for anatomy teaching (**Bennett et al 2000; Hull 2003**). Chymistry was also the methodological backbone of the new experimental philosophy aimed at understanding the intimate causes of natural phenomena, at a time when Oxford was one of the most important hubs of such discipline. Figures of the calibre of Robert Boyle and Robert Hooke (1635-1703) had worked in Oxford during the seventeenth century (**Gunther 1923-1945; Williams et al 2009**), and the circle of natural philosophers active in town coalesced in the Oxford Philosophical Society, also created in this period. Among these men was Robert Plot (1640-1696), the author of the very popular *Natural History of Oxfordshire*, published in 1677 (**Gunther 1923-1945, vol. 12**). When the Ashmolean opened in 1683 Plot was appointed keeper of the museum and director of experiments, and he became the first official professor of chymistry at an English university. He lectured on natural history and philosophy three times per week and led laboratory demonstration of the principles addressed in class under the direction of Christopher White, his assistant and chemical operator of the laboratory. As Plot was not paid directly by the university, his income relied on student fees and on the sale of chymical preparations made in the laboratory (**Bennett et al 2000: 21**). Several documents suggest that Plot was very conscious of the importance of the entrepreneurial side of his work at the Old Ashmolean, and give us details of his entering in commercial contracts with several people during the course of his appointment (**Roos 2014**). He could count on high quality apparatus and on a modern space, and importantly also on the expertise of White who had been Boyle's laboratory assistant for ten years. He could use the *officina chimica* to manufacture medicines as well as any other marketable preparations that could increase his income and enhance his status as experimental natural philosopher, in full Baconian fashion. Indeed, the activities of the Ashmolean Museum adhere to the view of natural philosophy as a discipline that included – again following Bacon's definition - experiments of light and experiments of fruit.

Possibly because of the need to keep his profitable recipes secret Plot did not publish any books or major works related to his chymical activity, the little we know coming from letters and papers collected in manuscript form. In them, the natural philosopher discusses some well-known alchemical *arcana*, secrets of nature he was most interested in like the preparations of the universal solvent alkahest, capable to dissolve anything into their essential components (**Sherwood Taylor 1949; Roos 2014**). Further details of Plot's scientific interests can be gained from the frequent entries relative to the activity of the Oxford Philosophical Society and of the Royal Society where he was elected a council member in 1680 (see for example **Birch 1757 vol. 4**). As a natural historian Plot was especially interested in surveying territories and collecting and studying specimens of rocks, minerals and other curiosities that he would send to the Royal Society or present at one of the meetings. Moreover, and again following on Francis Bacon's tracks, Plot's interests were also directed towards arts, crafts and technological innovation, and he studied the improvements made in in England in his time. Plot remained in charge until 1690, when he resigned from his job and decided to retire to private life in the countryside, where he could concentrate on his natural historical interests. He was succeeded, as keeper and chemistry professor, by his former assistant Edward Lhwyd (1660-1709), who lectured on natural history and whose focus on stones and fossils presumably gave a different shape to the Ashmolean's teaching (**Gunther 1923-1945, vol. 14**). In 1710 a German visitor who visited the laboratory reported its state of semi-ruin and neglect, even though he praised it for being impressively equipped with "the finest instruments" (**Bennett et al 2000: 21**). During the 1720s, the new keeper of the Museum and chemistry professor was John Whiteside (1680-1729) a chymist with an interest in medical and pharmacological preparations of which we know from a series of lecture notes (**Guerrini 1994; Roos 2017**). Throughout the 18th century the *officina chimica* was also used for the teaching of anatomy until this was moved elsewhere in 1767, and eventually, in the 1770s the laboratory underwent complete refurbishment which involved new equipment for chemistry teaching. Finally, in the late 19th century the collection was moved to the current site in Beaumont Street, and the original building became the home of the History of Science Museum in the 1920s.

5.2 ARCHAEOLOGICAL BACKGROUND

In April 1999 the History of Science Museum underwent a major refurbishment project, which involved extensive excavations at the rear of the building and the removal of the limestone slabs used to cover the earth floor. Underneath the slabs it was discovered that the whole area had been used as a dump for materials which could be associated with the 17th and 18th century basement laboratory of the Ashmolean Museum (**Hull 2003**).

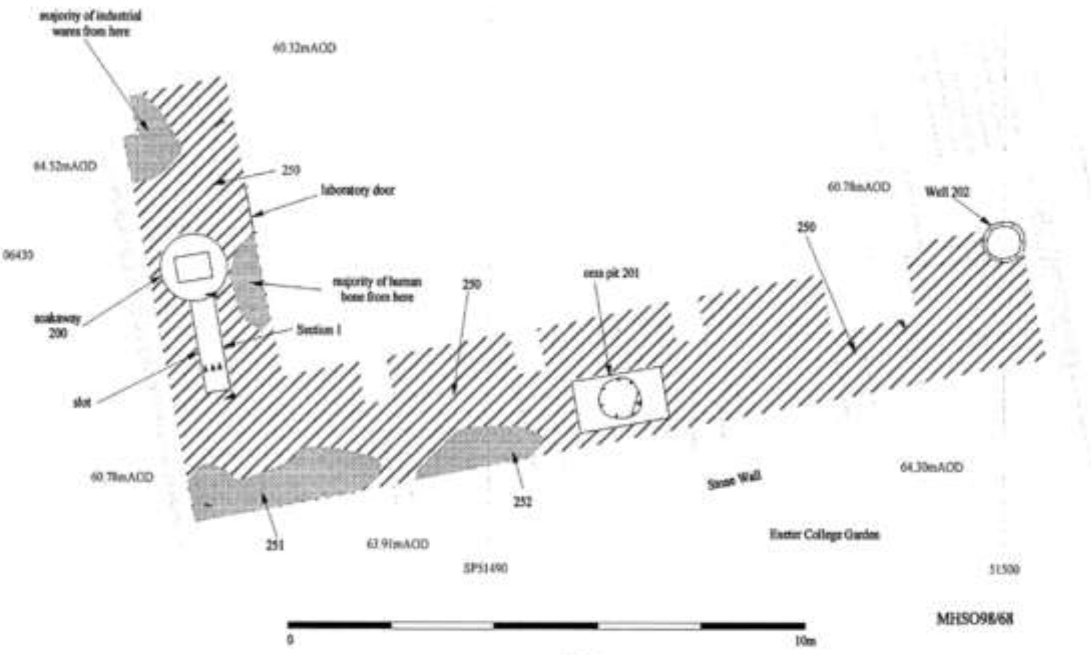


FIG. 2
Location of excavated area.

Figure 5.2. Map of the excavated area at the rear of the History of Science Museum in Oxford (after Bennett et al 2000: 35)

The vast majority of the artefacts recovered come from context 250, while a smaller number was excavated from the backfilling of smaller stratigraphic features like the soakaway 200 (**fig. 5.2**). The most numerous findings are several thousand fragments of human and animal bones, which probably relate to the anatomy teaching, as well as the assemblage of pottery which includes 110 sherds of "domestic" types, 31 fragments of clay tobacco pipes and dozens from laboratory equipment more strictly related to the chymical activity of the Ashmolean. The remaining artefacts include 46 glass fragments from windows and bottles, loose pieces of metal plus one copper alloy vial probably used in the laboratory, and then bricks, stones and shell (**Hull et al 2003**). Among the domestic wares are fragments of glazed and decorated pottery of various kinds, ranging from German stoneware to English-made tin-glazed stoneware, creamware and pearlware (**Hull 2003: 5-9**). The pieces of laboratory equipment proper (**fig. 5.3-4**) constitute the main focus of this section. The assemblage is composed of 25 mostly complete crucibles of recognisable type, plus several fragments not ascribable to any type, two retorts and the fragment of a base of uncertain type, possibly from a cucurbit or an aludel, the globular vessel forming the lower part of a distillation apparatus. There are then two flasks (one made of two fragments), one stoneware bottle, and the fragment of a lid. Most of the pieces show signs of exposure to high temperature and chemical attack, an indication that they had been used in furnace operations of sorts. The crucibles include both open and closed types (**fig. 5.3**). Examples of the former range from very small (H < 10cm) rounded specimens with a flat bottom (nos. 10-18 in **fig. 5.3A**), to larger ones (H > 10cm) of beaker shape with tapering profile and with or without a pouring spout (nos. 1-9 in **fig. 5.3A**), through to triangular crucibles of different sizes (H 5-10cm) (nos. 5-8 in **fig. 5.3B**). Closed-profile crucibles are the largest in the assemblage, with a height of over 15cm (nos. 2-4 in **fig. 5.3B**). The two retorts do not belong to the same type, the most relevant difference being the presence of an opening at the top of one of the two, which is absent in the other (nos. 1-2 in **fig. 5.3C**). The extra opening could be used to either facilitate the filling of the retort or to attach a second vessel for a further step of distillation of the more volatile matter. Possible evidence for the presence of an alembic is the fragment of what seems to be a cucurbit bottom (no. 11 **fig. 5.3B**), while no fragments from still heads have been found.

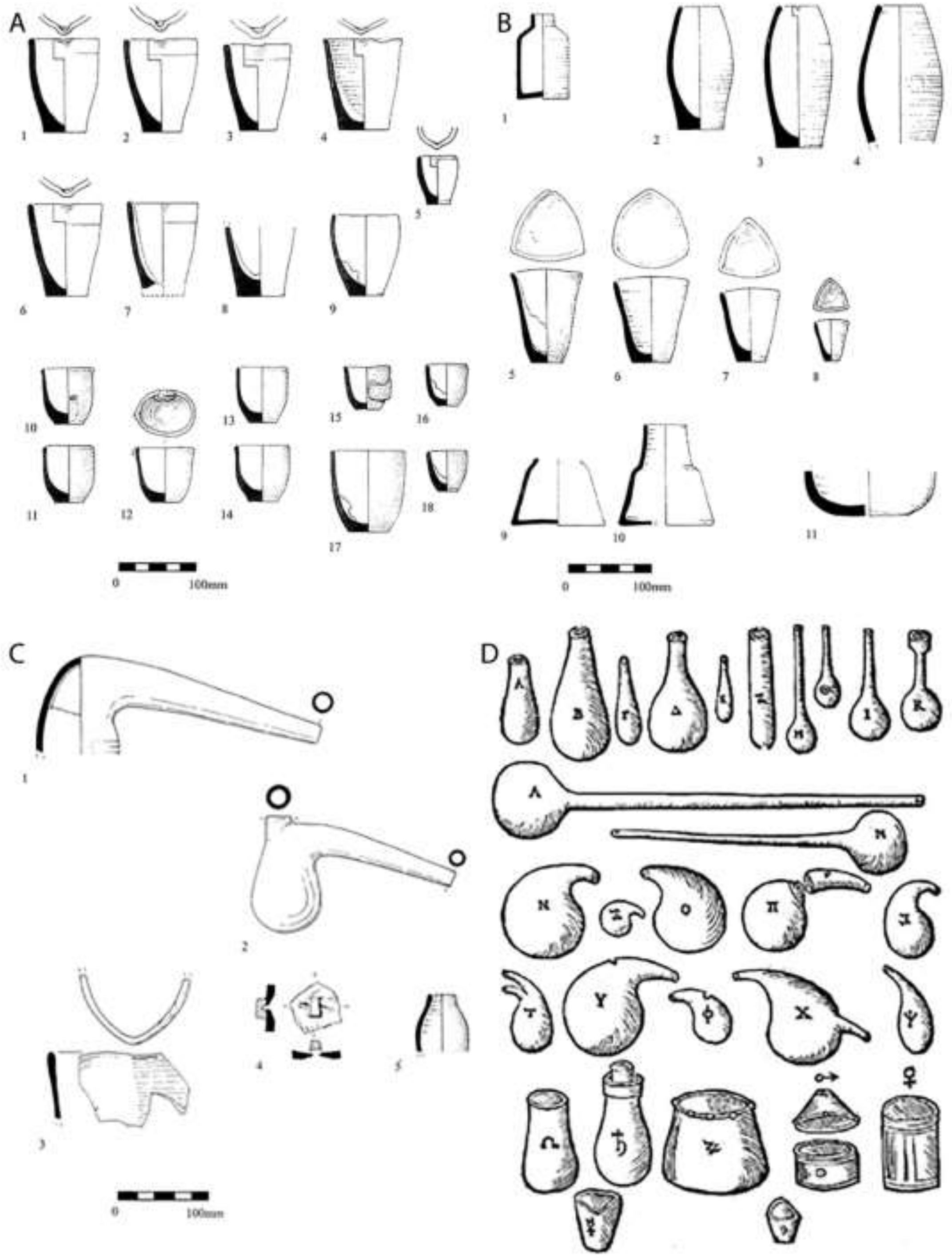


Figure 5.3. The ceramic apparatus excavated from the rear of the History of Science Museum in 1999 (after Hull 2003: 6-8). A: Rounded open crucibles of larger (no. 1-9) and smaller type (no. 10-18); B: Tall closed (no. 2-4) and triangular crucibles (n. 5-8), a bottle (no. 1), two flasks (no. 9-10) and a cucurbit's bottom (no. 11). C: The two retorts (no. 1-2), a larger triangular crucible (no. 3), the fragment of a lid (no.4) and a

small drug jar (no. 5). D: Drawings of laboratory apparatus from Andreas Libavius' 1606 Alchymia, showing glass distilling vessels and two triangular crucibles towards the bottom.

Providing a precise dating for the finds has proved problematic for two main reasons. First, the artefacts were retrieved from a secondary context relative to a dumping event and for this reason no useful stratigraphic information is available. Second, the pottery assemblage includes types that cover a very long period of time, with some of the laboratory tools such as the open and the triangular crucibles being produced, virtually unchanged, from the fourteenth until the nineteenth century (**Hull 2003: 9-10; Martinón-Torres and Rehren 2009**). The same is true for the retort whose shape did not change for centuries and is still pretty much the same today (**Taylor 1945; Moorehouse et al 1972; Booth 2016**). Some of the domestic wares have a more restricted timespan, but they too range from the late seventeenth century (e.g. the Staffordshire white salt-glazed ware) up to the first half of the nineteenth century (e.g. the pearlware). Graham Hull, who was the first to study the pottery, reports that "the assemblage [of domestic pottery] can only be dated to after 1770 and before 1825, with the weight of evidence suggesting that it most likely dates to c. 1780 and possibly as late as after 1790" (**Hull 2003: 9**). Besides ceramic typology, the history of the Ashmolean Museum can help better define the chronology of the dumping event. Indeed, in 1781 a major renovation programme took place in the basement laboratory following the appointment of Martin Wall (1747-1824) as reader in chemistry and this may have triggered the dumping of old equipment and the new flooring of the laboratory (**Bennett et al 2000: 23**). Finally, the range of chemical operations carried out inside of the crucibles and the other chemical vessels offers more clues as to the chronology of the assemblage. Marcos Martinón-Torres, who analysed a few of the crucibles, noted that "the chemical residues and experiments identified through scientific analyses [...] seem more consistent with the chymistry of the late seventeenth century." (**Martinón-Torres 2012a: 26**). In this sense, a potential *terminus ante quem* for the dating of the crucibles is the account written by Zacharias Conrad von Uffenbach, the German traveller who visited the laboratory in 1710 and found it in partial ruin, with broken equipment scattered all over the place and the ground covered in dirt (**Bennett et al 2000: 21; Martinón-Torres 2012a**). Parts of the assemblage of laboratory equipment had undergone initial exploratory analyses prior to the analytical work carried out for the present thesis. Shortly after the excavation, Chris Salter did some XRF screening on a large number of artefacts. On this basis, he proposed three main areas of activity: one related to zinc and lead, another to glass and glazes and finally one related to sulfur, which he interpreted as possibly connected with the production of fireworks (**Hull 2003: 11-13**). However, his data was mainly qualitative and was never reported in detail. In 2012, Marcos Martinón-Torres published the SEM-EDS analyses of some four crucible fragments from the Ashmolean laboratory, adding more detail to Salter's initial interpretation and hinting at the information potential of more extended invasive analyses (**Martinón-Torres 2012a**). Building on this, the present work therefore represents the third round of analyses performed on various segments of the assemblage, seeking to provide a more comprehensive account of the equipment and chemical processes at the laboratory.

5.3 SITE-SPECIFIC RESEARCH QUESTIONS

As for Jamestown in the previous chapter, a series of site-specific questions will be addressed in relation to the Ashmolean laboratory, which will subsequently feed into the broader research questions outlined in section 1.3. Specifically, the results discussed in the following pages will shed new light on the context of the Ashmolean Museum and its place within early modern chymistry and laboratory practice. The following questions will therefore be investigated:

1- From the perspective of the material culture, what is the spectrum of laboratory operations undergone at the *officina chimica*?
2- What is the range of ingredients, reagents and natural or artificial substances that were manipulated during these operations?

3- Can we establish connections emerging between the laboratory activities and broader chymical innovation and research themes prevalent in the late 17th and early 18th century in Europe?

4- How does the laboratory practice thus uncovered compares to the Baconian ideals of Solomon's House and the community of natural philosophers working towards the improvement of human knowledge?

5- Having addressed the previous questions, is it possible to bring a new contribution towards a more precise dating for the chymistry-related artefacts?

5.4 SAMPLE SELECTION AND MATERIALS ANALYSED

The majority of the chymistry-related objects described in section 5.2 are currently kept in store in the Ashmolean Museum. For many years, a small selection of the artefacts has been on display at the History of Science Museum, in the very room where the laboratory once operated. The objects have since been moved to the Ashmolean Museum, following the recent opening of a new room in celebration of the 400th anniversary of Elias Ashmole's (1617-1692) birth and the early days of the institution (**fig. 5.4**).

Figure 5.4. A selction of chymistry-related artefacts currently on permanent display at the Ashmolean Museum. On the shelves are crucibles of the various typologies, while below are the retort and the beaker type crucible with blue glass coating its inner wall.

Shortly before the opening of the new room, it was possible to carry out an initial pXRF screening of the entire assemblage of laboratory apparatus in the conservation department of the Ashmolean Museum. The screening was made necessary by the lack of detail relating the previous XRF analysis carried out in 1999 (cfr section 5.2) to specific artefacts. The results gathered, albeit only qualitative, offered nonetheless an initial idea regarding the nature of the crucible residues and were used to inform the choice of artefacts for invasive sampling and subsequent SEM-EDS microanalysis. The sampling aimed at encompassing all fabric groups and shapes discernible macroscopically, as well as the chemical groups based on the pXRF data and any residue that looked like intentionally-made glass. However, the most important factor affecting the sampling was the need to preserve the integrity of the objects, which limited the selection to those artefacts that were already in a fragmentary state. It was not therefore possible to obtain samples from the small rounded crucibles or from the stoneware bottle. In the end, a total of sixteen samples were taken using a small Dremel drill with a diamond-impregnated rotating blade attached; one of these (AN1999.205.17) comes from a crucible which had previously been analysed by Martinón-Torres in 2012 and corresponds to his ID OXn003 (**Martinón-Torres 2012a**). Finally, one additional sample (OX1409) that had been taken in the same occasion but never analysed was available for the present study. When possible, a section including the ceramic body and the residue was cut from the vessels. In two cases, only the internal residue was sampled by scraping it off with a scalpel. A detailed list of specimens is presented in **table 5.1**. Finally, the sample IDs employed here correspond to the accession numbers assigned by the Ashmolean Museum, even though IDs beginning with "OX" had already been given to the artefacts after the excavation in 1999 and these can be found in the 2003 publication (**Hull 2003**). However, the vast majority of the samples analysed here belong to a group of finds that due to its fragmentary state was not illustrated in the report and that was bulk-assigned the single ID OX1419 (**Hull 2003: 11**). Two of these not-illustrated samples have been analysed in 2012 by Martinón-Torres who re-names them by keeping the initial "OX" followed by "n00x". In some case, there is an overlap between initial IDs and accession numbers. Thus, to homogenise the nomenclature it was chosen here to employ the more recent museum accession numbers, the only exception being sample OX1409 for which no accession number was available.

Most of the samples analysed, twelve, were taken from crucibles. Of these, four are of the larger rounded type mentioned in section 5.2, another four of the triangular type, and one belongs to the closed taller type. Two fragments come from crucibles whose precise shape could not be reconstructed, namely the sample from graphitic crucible AN1999.205.28 and sample AN1999.205.19, the base of what may be a beaker type crucible. AN1999.205.14 and AN1999.205.27 are samples of, respectively, the glassy residue from the inside of what probably is a tall closed crucible and the white crust found adhering to the inside of a vessel of the same kind There are two fragments of distillation apparatus, one belonging to the bottom of a cucurbit and another to a retort. The last samples of equipment analysed are fragments from a stoneware flask and from a small bottle or drug jar. Finally, the last sample was taken from an amorphous lump of yellowish-white substance whose shape looks like it could be spillage from a container (**fig. 5.27**). Full details of all fragments analysed in this chapter can be found in **Appendix C**, while the specifics of the SEM-EDS analyses performed are outlined in section 3.2.3

Sample ID	**Type**	**Process**	**Hull 2003**	**Martinón-Torres 2012**
AN1999.205.9d	Beaker crucible	Lead crystal glass making/working		
AN1999.205.14	Greenish glassy residue (probably from tall closed 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?		
AN1999.205.29	Yellow lump	Uncertain		
AN1999.205.30	Beaker crucible	White opaque glass making		
AN1999.205.31	Triangular crucible	Uncertain		
AN1999.205.32	Retort	Zinc distillation?		
OX1409	Flask	N/A	OX1409	

Table 5.1. The Ashmolean samples analysed, with the type of laboratory vessel they come from, the chymical process to which they are associated and alternative nomenclatures when applicable. N/A indicates that no residue was discovered, while uncertain refers to instances when a residue was found but no conclusive interpretation can be given.

5.5 RESULTS AND TECHNICAL INTERPRETATION

The data indicates that a rather wide variety of laboratory operations were conducted in the *officina chimica* of the Ashmolean Museum. For this reason, and similarly to what done in the case of Jamestown, the presentation of the results is divided into three main areas according to the relevant spheres of chymical work. Section 5.5.1 reports the results on the ceramic equipment itself, its manufacture, material properties, possible provenance and contextualisation within European traditions of crucibles and other pieces of technical ceramics. The next sections cover the actual operations carried out inside of the vessels, 5.5.2 involving different technologies in glassmaking and 5.5.3 regarding the metallurgy of zinc. Further to this, a number of samples that could not be ascribed to any specific area and were labelled as “other” will be addressed in section 5.5.4. A more contextualised discussion based on the material culture will follow in section 5.6, which articulates the microhistory of chymical practice at the Ashmolean laboratory, and exposes the network of people and motivations making up Oxford’s chymical community.

5.5.1 THE CERAMIC EQUIPMENT

Apart from two samples taken from crucibles’ inside (AN1999.205.14 and 27) and the sample of yellow spillage (AN1999.205.29), the remaining specimens all include a cross-section containing the ceramic body. The composition of the chemical vessels is reported in **table 5.2**. The crucibles of triangular type have the highest levels of alumina in the assemblage (38.1-39.5 wt% Al_2O_3), and very low concentrations of alkali and alkali earth oxides (total < 3wt%), which suggests very good refractoriness of these vessels. Macroscopically, they display a light-coloured body and grainy texture of the surface, the result of sand tempering (cfr **Martinón-Torres and Rehren 2009**).

Sample	Type	Na_2O	MgO	Al_2O_3	SiO_2	Cl	SO_3	K_2O	CaO	TiO_2	FeO	ZnO	AgO
AN1999.205.9d	Beaker crucible	0.3	0.7	36.5	56.8	bdl	bdl	1.8	0.2	1.6	2.0	bdl	bdl
AN1999.205.17	Beaker crucible	0.3	0.9	34.8	58.3	bdl	bdl	2.1	0.2	1.3	2.2	bdl	bdl
AN1999.205.18	Drug jar	0.4	0.4	29.2	64.0	bdl	bdl	1.8	0.3	2.3	1.5	bdl	bdl
AN1999.205.19	Beaker crucible	0.3	0.8	32.6	60.2	bdl	bdl	2.2	0.3	1.3	2.3	bdl	bdl
AN1999.205.21	Cucurbit	0.9	0.4	30.5	63.6	bdl	bdl	2.2	0.2	1.4	1.0	bdl	bdl
AN1999.205.22	Tall closed crucible	0.2	0.5	32.1	60.8	bdl	bdl	2.2	0.3	1.4	2.1	0.3	bdl
AN1999.205.23	Triangular crucible	bdl	0.7	39.5	54.8	bdl	bdl	1.2	0.3	1.8	1.7	bdl	bdl
AN1999.205.24	Triangular crucible	bdl	0.6	38.1	55.7	bdl	bdl	1.9	0.3	1.8	1.7	bdl	bdl
AN1999.205.25	Triangular crucible	0.2	0.6	38.8	54.9	bdl	bdl	1.7	0.4	1.8	1.7	bdl	bdl
AN1999.205.28	Graphitic crucible	0.4	0.5	21.9	54.0	0.1	0.6	17.8	bdl	1.9	2.6	bdl	0.3
AN1999.205.30	Beaker crucible	0.2	0.6	37.2	56.3	bdl	bdl	2.1	0.4	1.3	1.8	bdl	bdl
AN1999.205.31	Triangular crucible	0.2	0.7	38.7	55.3	bdl	bdl	1.4	0.4	1.8	1.6	bdl	bdl
AN1999.205.32	Retort	0.3	0.7	34.0	59.3	bdl	bdl	2.6	0.2	1.2	1.7	bdl	bdl
OX1409	Flask	0.6	0.8	30.9	60.7	0.5	bdl	2.1	0.3	2.0	2.3	bdl	bdl

Table 5.2. Average chemical composition by SEM-EDS of the ceramic matrices of the fragments analysed.

Their microstructure is characterised by a highly vitrified matrix which nonetheless does not appear bloated nor severely distorted, another indication of the ceramic's high refractoriness. The matrix contains small rounded quartz grains as well as elongated voids parallel to the surface of the vessel, left by the expansion and subsequent shrinking of the quartz upon heating and cooling (**Martinón-Torres and Rehren 2014**) (**fig. 5.5**). These too would have contributed to the crucibles' resistance to thermal and mechanical stress, by preventing the propagation of catastrophic cracks during high-temperature operations (**Kilikoglou et al 1998; Tite et al 2001**). Occasional hollow iron-rich concretions and molten feldspars can be seen in the matrix, the latter as abnormally subspherical bloated areas (**fig. 5.5**) (**Martinón-Torres and Rehren 2005b; Martinón-Torres et al 2008a**). These features are consistent with crucibles made in the German region of Hesse.

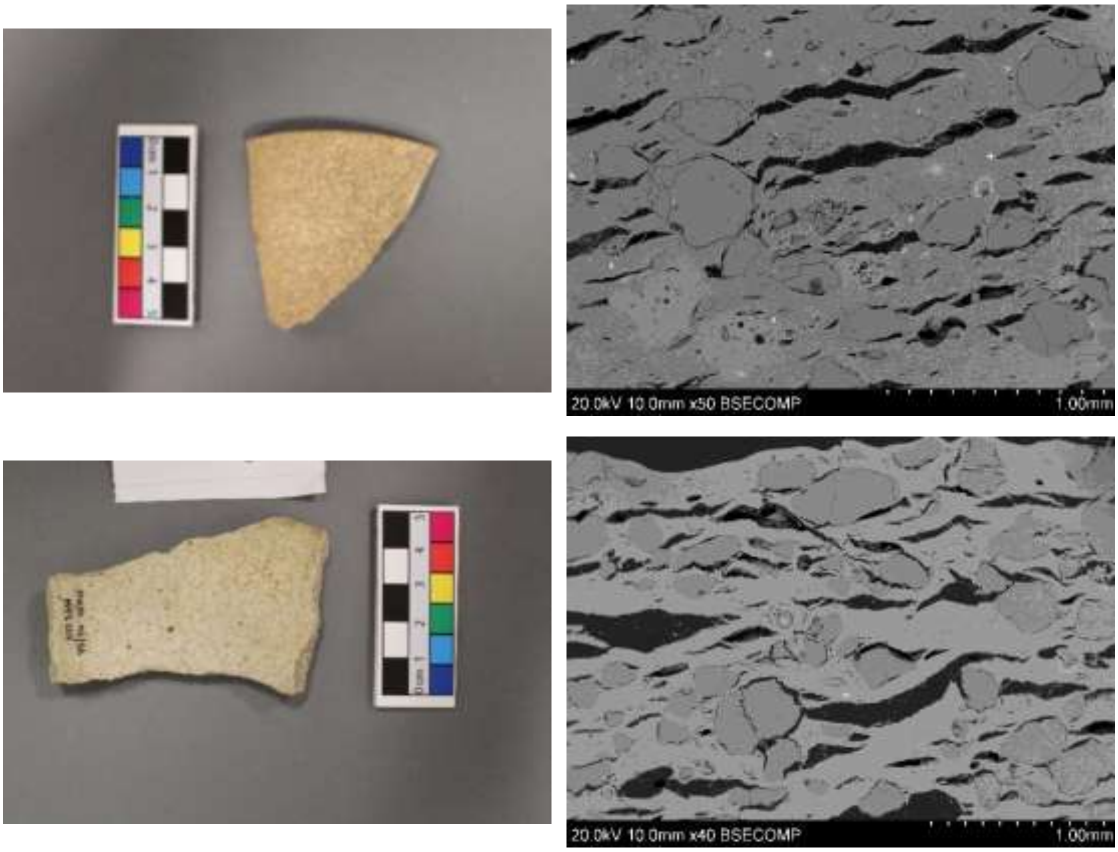


Figure 5.5. Photos and SEM micrographs (backscattered electron mode) of samples AN1999.205.24 (top) and AN1999.205.23 (bottom). Note the light coloured and grainy bodies typical of the Hessian crucibles as well as the presence of shattered quartz grains (mid-grey) and of elongated voids parallel to the

surface of the vessel. A more bloated area is visible on the bottom-left corner of the top-right image representing molten feldspars. Also note the hollow iron-rich concretion visible on the right of the top-right image as dark circular phase surrounded by a bright shell.

Except for the graphitic crucible AN1999.205.28 and the flask OX1409, which will be presented separately, the rest of the vessels share a 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. 5.6**). In the beaker crucibles, the tall one with closed profile and the retort, the clay was tempered with sand and grog, while in the stoneware bottle and in the cucurbit only quartz is present. The addition of grog, bits of crushed ceramic, is visible as pellets of different sizes scattered within the matrix (**fig. 5.6**). These appear microstructurally similar to the surrounding ceramic but often look more vitrified, a characteristic 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 in the post-Medieval period is found in contemporary texts (**e.g. Agricola in Hoover and Hoover 1950: 230; Antonio Neri in Cable 2006: 304; 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**; White and Kearns 2010; Paynter 2012**). Compared to the triangular crucibles, the non-triangular vessels generally show slightly higher content of alkali and alkali earth oxides (total >3wt%) and slightly lower alumina which may indicate a different clay (**table 5.2**). The levels of alumina, in particular, vary considerably across the samples, with the beaker crucibles showing the highest levels (32.6-37.2 wt% Al_2O_3), while the rest containing less of it (29.2-32.1 wt% Al_2O_3) and the two samples not tempered with grog having the lowest values.

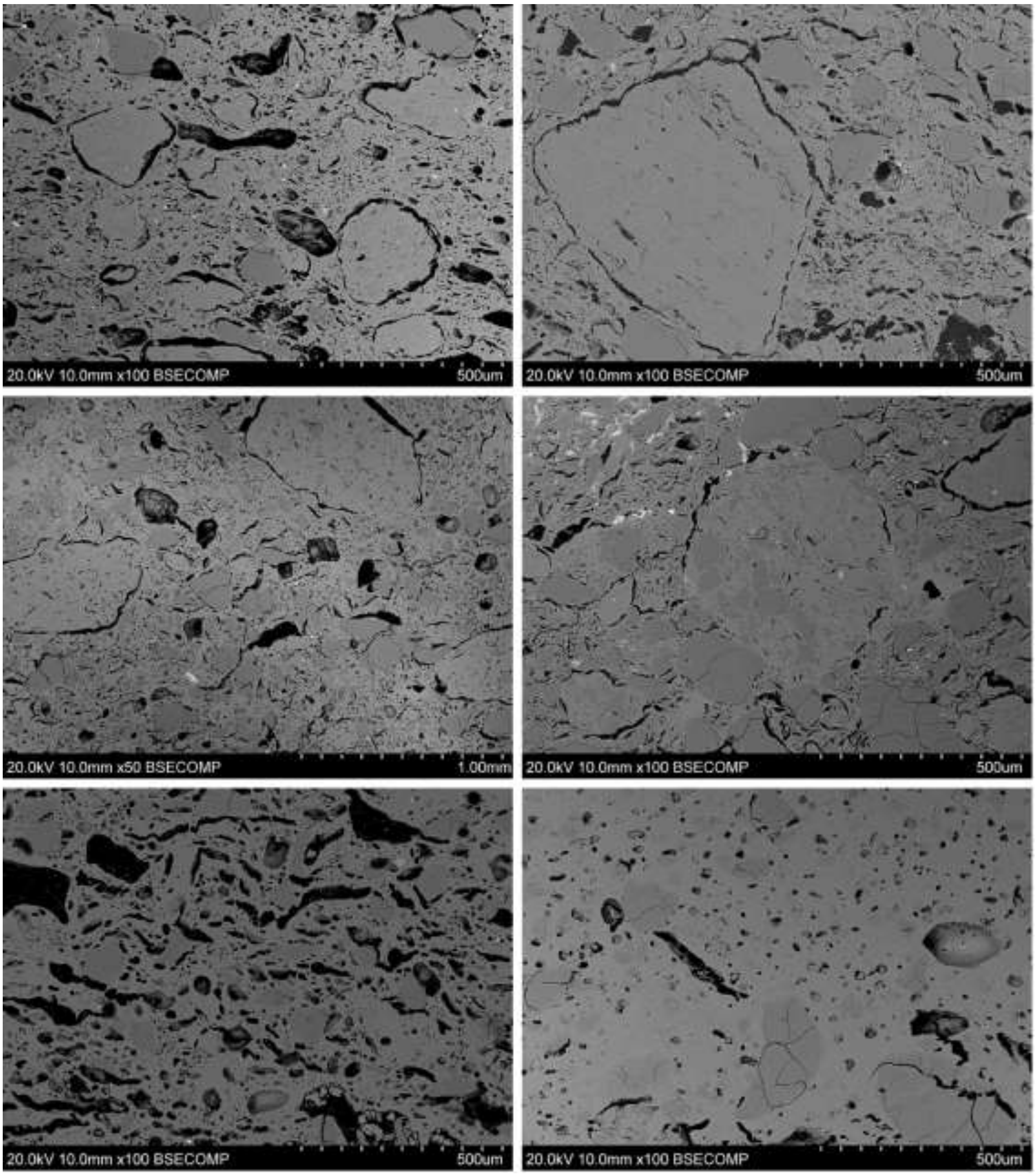


Figure 5.6. SEM backscattered micrographs showing the ceramic matrix of the non-triangular vessels. Note the distorted, variably melted ceramic and the frequent bloating pores developing throughout and particularly in the two bottom samples. The samples on top and middle row shows grog inclusions visible as discrete rounded phases with a higher degree of vitrification. From top-left: AN1999.205.17, AN1999.205.32, AN1999.205.30, AN1999.205.22, AN1999.205.21, AN1999.205.18.

The earthenware flask fragment OX1409 displays a microstructure that differs markedly from what seen so far, in that the ceramic is not vitrified, suggesting a lower temperature both in firing and in use. Moreover, its texture looks coarser with more diversified and more numerous inclusions in it (**fig. 5.7**). For instance, the majority of the quartz grains found in the matrix appear large, (sub-)angular and reddish-pink in cross-section, indicating that they did not come as sand but as some alternative type of siliceous temper crushed for the purpose (see also **Hull 2003: 11**). The grog inclusions vary widely in terms of size, from few tens of micron and up to around a millimetre across. Their chemical composition is consistent with what was found in the other samples tempered this way (**table 5.3**). A few seem to have slightly different levels of alumina and silica, but considering that the rest of the elements do not show significant differences this has more likely to do with the size of the areas analysed and the consequent amount of quartz in them than to actual differences in the source of grog. Smaller and rounded clay pellets probably coming with the sand can also be seen (**fig. 5.7**).

Sample	Type	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	FeO
AN1999.205.9d	Beaker crucible	0.3	0.7	37.5	55.9	1.8	0.3	1.6	1.9
AN1999.205.17	Beaker crucible	0.3	0.9	35.5	57.9	1.9	0	1.4	2.2
AN1999.205.22	Tall closed crucible	0.1	0.6	32.4	61.5	2.2	0.3	1.4	1.4
AN1999.205.30	Beaker crucible	0.2	0.7	38.5	54.7	2.1	0.4	1.6	1.9
AN1999.205.32	Retort	0.1	0.8	37.5	56.6	2.3	0.3	1.0	1.5
OX1409	Flask	0.3	0.7	31.2	61.8	2.0	0.1	1.6	2.1

Table 5.3. Average chemical composition by SEM-EDS of grog inclusions in the ceramic matrices of the samples analysed.

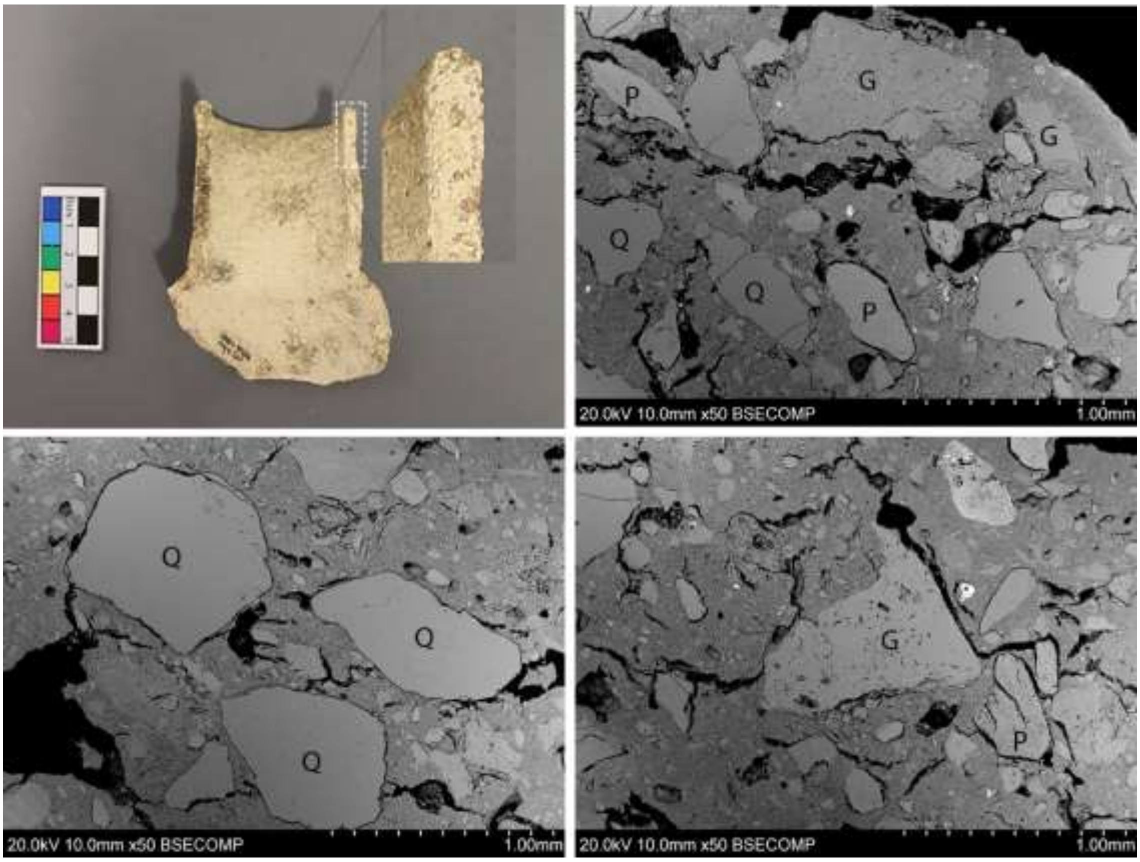


Figure 5.7. Image and SEM-EDS backscattered micrographs of flask fragment OX1409 showing the coarser texture of the ceramic matrix that does not appear vitrified. Note the pink quartz grains in the section (top-left), which can be seen as sub-rounded mid-grey phases in the ceramic matrix (Q). Grog (G) and clay pellets (P) inclusions of different sizes and shapes can also be seen as light-grey phases.

Finally, sample AN1999.205.28 also shows important differences in both its microstructural and compositional profile from the rest of the vessels analysed as well as from a macroscopic perspective. Indeed, the fabric appears black in cross-section due to the presence of graphite flakes added as temper to enhance the clay's refractoriness (**fig. 5.8**). The extraordinary heat resistance and inert qualities of graphite rendered these vessels resistant to extreme temperatures and corrosive chemical reactions. When seen against the rest of the assemblage, the chemical composition of the matrix reveals lower alumina (21.9 wt% Al_2O_3) and, unusually high levels of potash throughout its profile (17.8 wt% K2O). The latter is very unlikely to reflect the composition of the clay, and instead derives from interaction between the crucible and the potassium-rich residue found within it (cfr 5.5.4), which caused the vessel to partially melt as shown in **figure 5.26**. Graphitic crucibles of the same type were highly specialised technical ceramic mainly produced in Bavaria, but found at many sites throughout Europe and the colonial world (**Martinón-Torres and Rehren 2009**). This type is believed to appear in England during the 18th century at the Warmley brass works site near Bristol and linked to the distillation of zinc (cfr. 5.5.3) (**Dungworth and White 2007**). From the early 19th century graphitic crucibles were also being made in Cornwall (**Hull 2003: 10**). The ratio of alumina to silica of the sample analysed here is roughly in line with that of black crucibles from various other sites, including the one other fragment of graphitic crucible discovered at the Ashmolean laboratory and displaying a stamp from the Bavarian manufacturer (**Martinón-Torres 2012a**) (**fig. 5.8**).

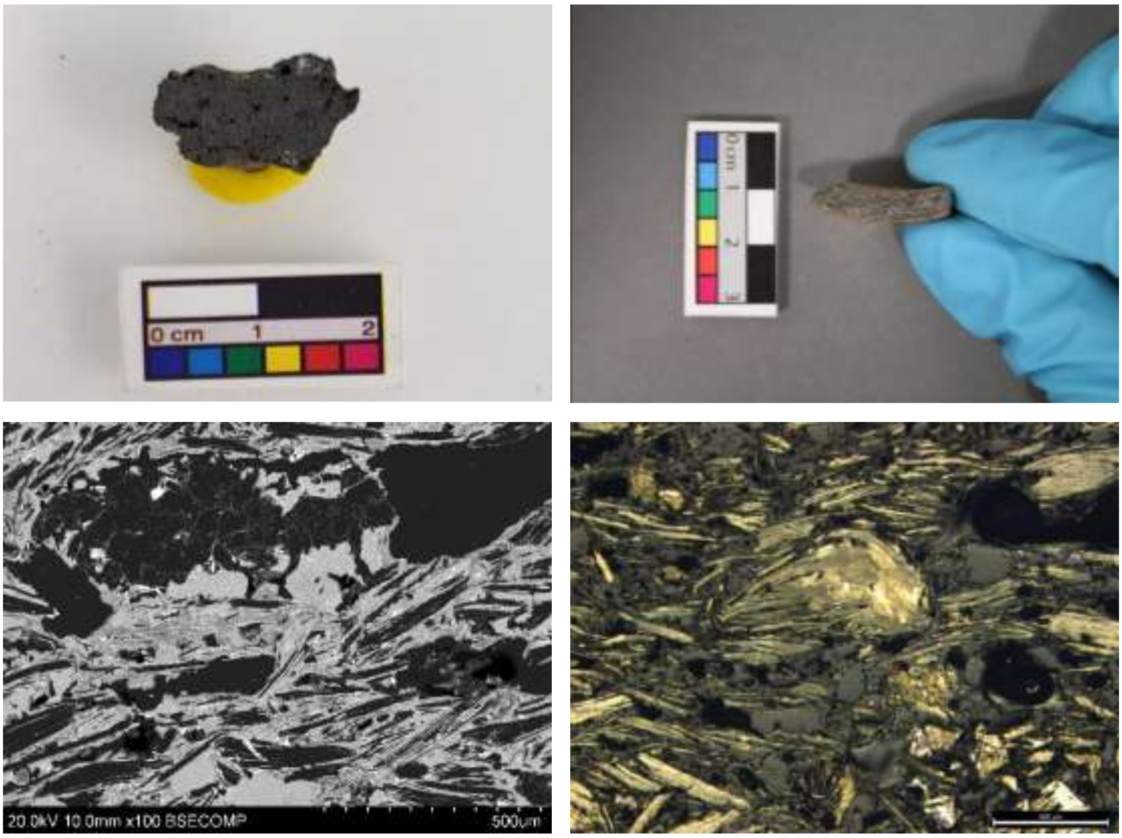


Figure 5.8. Top: Sections through the fragments of graphitic crucible AN1999.205.28 (left), analysed here, and OX1422 (right), analysed by Martinón-Torres in 2012 and belonging to a different vessel. Bottom: SEM-EDS micrograph showing the ceramic matrix of AN1999.205.28 (left). The black phases are bits of graphite, while the grey areas result strongly enriched in potash. On the right is an optical microscope micrograph of the same sample showing the flakes of graphite in yellow.

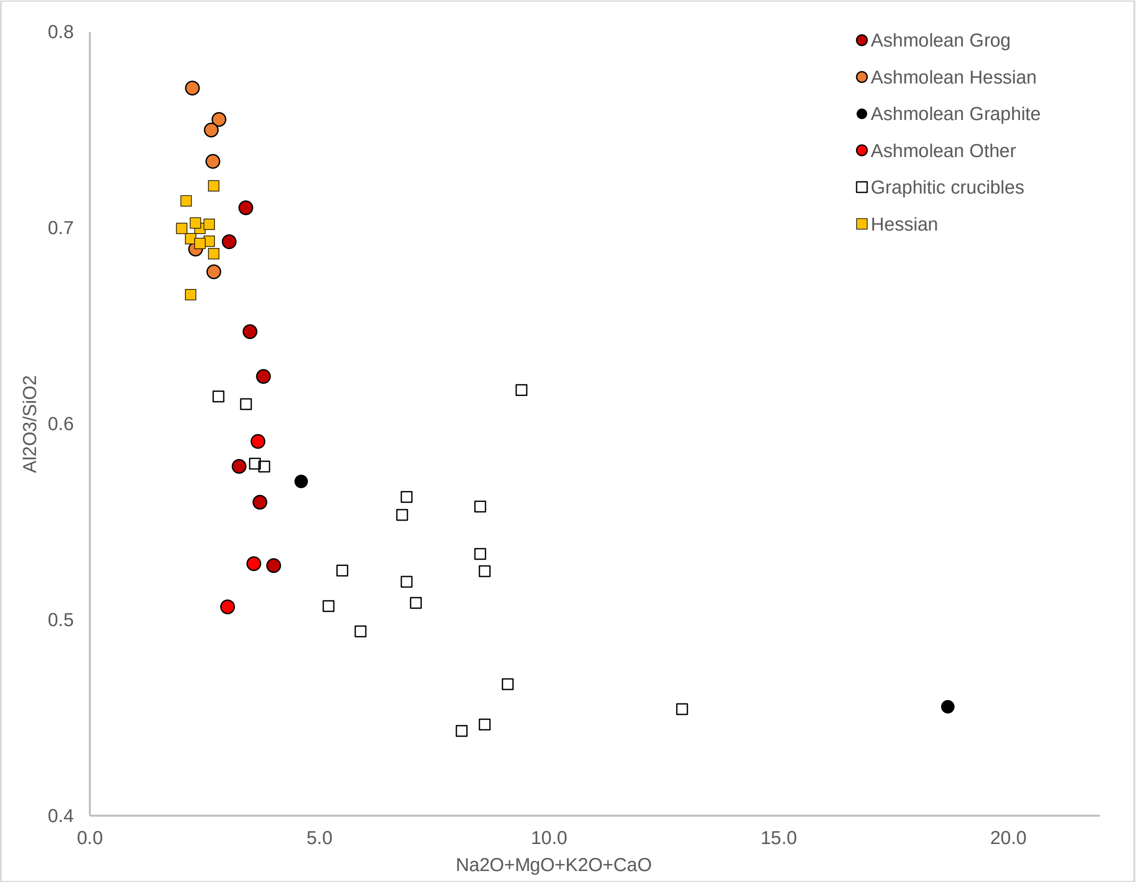


Figure 5.9. Alumina to silica ratio vs total alkalis in the Ashmolean vessels and in other post-Medieval crucibles of Hessian and Bavarian origin (data from Martinón-Torres and Verrocchio 2008; Martinón-Torres and Rehren 2009; Gardner et al 2018). The plot also includes the Ashmolean crucibles previously analysed in Martinón-Torres 2012a).

In **figure 5.9** the Ashmolean crucibles, divided according to their fabric type, are compared to German productions from Hesse and Bavaria found at many sites in and beyond Europe (**Martinón-Torres and Rehren 2009**). On top of the samples analysed here, the plot also includes the four fragments analysed in 2012, of which two are Hessian, one is grog-tempered and the last is graphitic (**Martinón-Torres 2012a**). As far as the total level of alkali and alkali earth oxides is concerned the samples analysed here do not show relevant oscillations, except for the graphitic crucible that confirms its unusual composition by falling away from everything else. The triangular crucibles from Oxford fall in the high-alumina region, close to the Hessian production, while the grog-tempered non-triangular vessels follow closely. Among them are the four glass-related samples (cfr section 5.5.2) as well as the one tall closed-profile crucible employed in zinc-related experiments (cfr section 5.5.3). Further down the alumina line are the vessels that were not tempered with grog and that include the two pieces of distillation equipment, also probably used in zinc-related metallurgical operations, as well as the flask and drug jar fragments. The composition makes these vessels the least heat-resistant in the assemblage which may be linked to the fact that they were used for laboratory operations involving gentler heating, like distillation, or even simply to store chemicals as is probably the case for the drug jar. Despite the internal differences, the vessels from the Ashmolean laboratory seem generally richer in alumina than other post-medieval crucibles from English glasshouses and metallurgical workshops, where the level rarely exceed the 30 wt% mark (**fig. 5.10**) (**Dungworth 2006; White and Kearns 2010**).

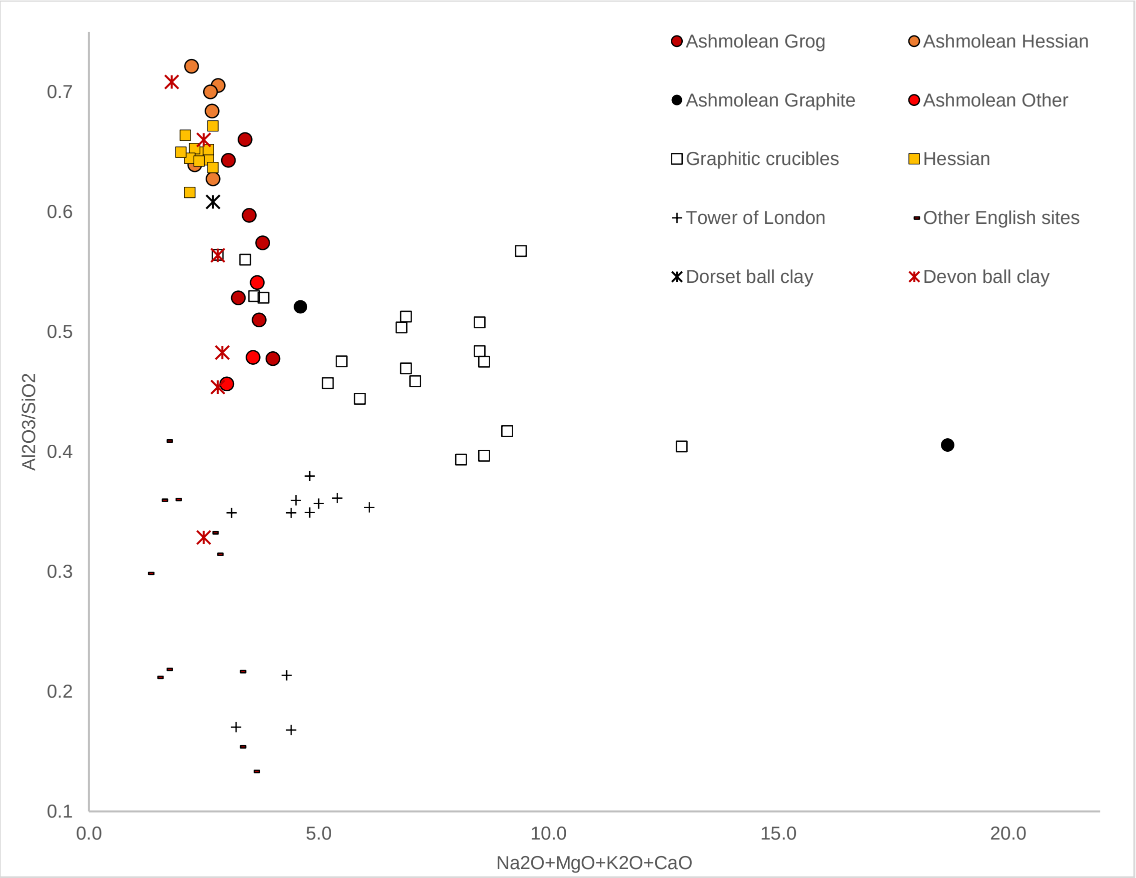


Figure 5.10. Alumina to silica ratio vs total alkalis in the Ashmolean vessels and in other post-Medieval crucibles, including Hessian and Bavarian examples (same data as before); the Tower of London (data from White and Kearns 2010) and various English glass houses (data from Vose 1994; Dungworth 2006). Data on Dorset ball clay comes from Tite et al 1986: 100, while data on Devon clays comes from http://www.clayheritage.org/aspects-of-the-ball-clay-industry/the-production-and-properties-of-devon-ball-clays (last accessed May 2020). Note that in most cases, the composition of English crucibles was obtained through the analyses of larger areas than those analysed here, and may therefore include a higher proportion of inclusions.

Clearly, the vessels employed in the Ashmolean laboratory were of exceptional quality. They were made using an especially high-alumina kaolinite-rich refractory clay of the same kind as that employed by German manufacturers. As a matter of fact, the very minor compositional differences between at least some grog-tempered crucibles and the Hessian ones does not allow to completely rule out a possible German origin (**Martinón-Torres 2012a: 31**). Nonetheless, the Ashmolean vessels may represent an English production, one that aimed to imitate the best crucibles on the market. During the 1670s, not long before the opening of the Museum, the potter and entrepreneur John Dwight had established a manufactory of high-quality stoneware and imitation porcelain in Fulham, near London (**Green 1999**). Dwight was a well-known figure in the English industry of specialised ceramic, so much so that Robert Plot, soon-to-be director of the laboratory, wrote in his *Natural History of Oxfordshire*:

> hath discovered the mystery of the stone or Cologne Wares [...] heretofore made only in Germany, and by the Dutch brought over into England in great quantities, and hath set up a manufacture of the same, which (by methods and contrivances of his own, altogether unlike those used by the Germans) in three or four years time he hath brought it to a greater perfection than it has attained where it hath been used for many ages, insomuch that the Company of Glass-sellers, London, who are the dealers for that commodity, have contracted with the Inventor to buy only of his English manufacture, and refuse the foreign. (**Plot 1677: 250**)

The site of Dwight's Fulham pottery has been the subject of archaeological excavation and thousands of fragments of brown stoneware and fine white imitation porcelain were recovered (**Green 1999**). Dwight also left us recipes in his laboratory notebooks where the chymist reports using Dorset ball clay for the ceramic bodies, a kaolinite-rich type of clay particularly fitting for his strong wares. According to the notebooks, Dwight added what he calls "white earth" to the clay. Some analyses have shown this to be a powdered frit made by sintering quartz and potash (**Tite et al 1986**). However, no grog seems to have been used in these productions. Plot also reports that Dwight had successfully cracked another German technological secret, namely that of Hessian crucibles:

> He [Dwight] hath discovered also the mystery of the Hessian wares, and makes vessels for retaining the penetrating salts and spirits of the chymists, more serviceable than were ever made in England, or imported from Germany it self. (**Plot 1677: 250**)

And interestingly, a number of pale grey triangular crucibles with coarse body were unearthed as well (**fig. 5.11**) (**Green 1999: 95**). Undoubtedly, these are the crucibles Plot talks about, the English-made reproduction which Dwight used in his own experiments and laboratory practice. Despite being virtually identical in appearance to Hessian vessels, Dwight's crucibles were found to contain crushed bottle glass alongside quartz (**Freestone 1991**). None of the triangular crucibles analysed here were found to contain crushed glass temper and their origin is therefore most likely German. However, it is possible that the rest of the crucibles were produced by Dwight or, if not by him, using the same clay. First, **figure 5.10** shows that the high levels of alumina are consistent with ball clays from Dorset and Devon. Second, beaker-type crucibles with remarkable typological similarities to the Ashmolean vessels were also found at the site of Dwight's pottery (**fig. 5.11**) (**Green 1999: 96-97**). No compositional data is available for these vessels however, and only further analyses on Dwight's materials can throw more light on the link between Plot's laboratory and the most renowned potter of the time. Thus, given the interest expressed by Robert Plot in such technological novelty, and given that Dwight had strong ties with the Oxford philosophical milieu having worked in Boyle's laboratory in the 1650s and 60s (**Bennett et al 2000: 31-32**), it is sensible to imagine that when Plot started his job at the *officina chimica* in1683, he would have turned to Dwight as a supplier of the laboratory's ceramic equipment (**Hull 2003: 24**).

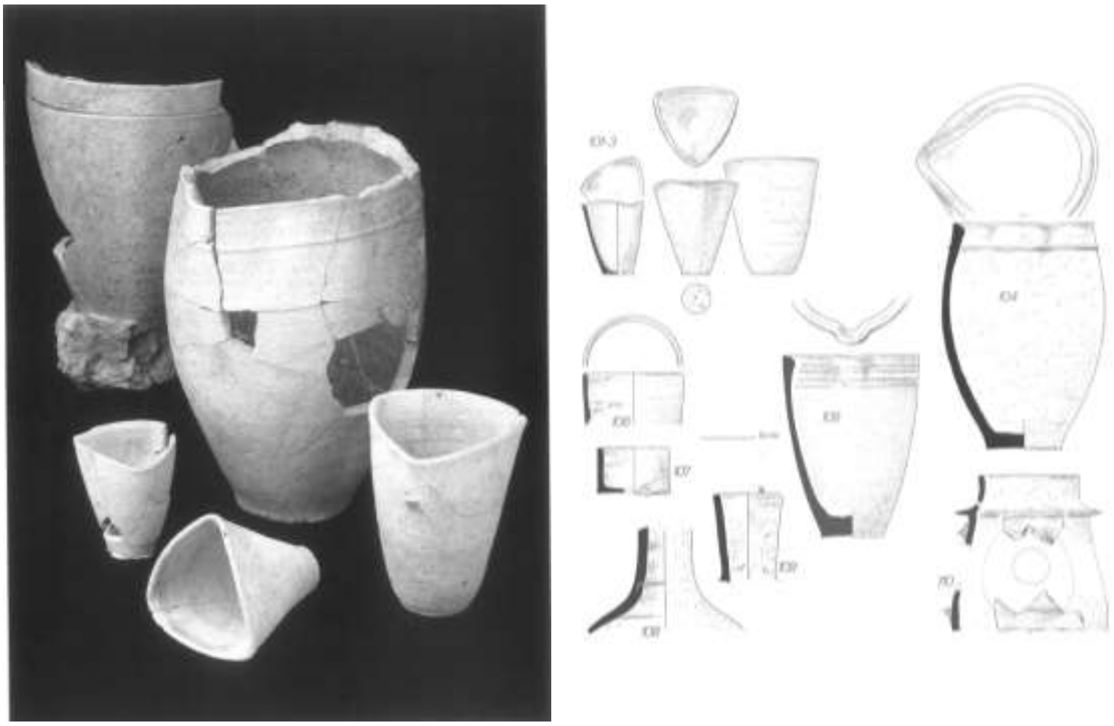


Figure 5.11. The crucibles recovered from John Dwight's Fulham pottery, including the triangular ones reproducing Hessian wares and non-triangular beaker and globular types (after Green 1999)

5.5.2 EXPERIMENTS WITH GLASS COMPOSITIONS

Five of the crucibles analysed from the Ashmolean assemblage were found to contain residues that can be related to the manufacture of glass-based products. There is, however, a substantial compositional variability across the samples, with important differences as to glassmaking ingredients employed and type of glass obtained. **Table 5.4** shows the chemical composition of the residues' glass matrices, avoiding larger inclusions where present. The most noticeable difference that can be appreciated is that between samples of lead-potash glass (AN1999.205.9d, AN1999.205.17 and AN1999.205.30) and one sample each of antimony-potash (AN1999.205.25) and soda-lead glass (AN1999.205.14).

Sample	Na_2O	MgO	Al_2O_3	SiO_2	Cl	SO_3	K_2O	CaO	TiO_2	FeO	Sn_2O	Sb_2O_3	PbO
AN1999.205.9d	bdl	bdl	0.1	51.3	0.2	bdl	11.1	bdl	bdl	bdl	bdl	bdl	37.4
AN1999.205.14	12.5	bdl	bdl	52.5	0.1	bdl	bdl	bdl	bdl	0.8	1.8	bdl	32.4
AN1999.205.17	0.8	bdl	bdl	59.9	0.3	bdl	11.8	bdl	bdl	bdl	bdl	bdl	27.1
AN1999.205.25	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	0.6	bdl	5.2	38.0	bdl	bdl	14.3	1.0	bdl	0.6	bdl	bdl	40.3

Table 5.4. Average chemical composition by SEM-EDS of the glass matrices of residues related to glass production.

5.5.2.1 LEAD-BASED GLASS

All but one samples of the glass-related residues show a high-lead composition. Two of them, namely AN1999.205.9d and AN1999.205.17, share a similar chemical composition indicating a potash-lead-silica glass of high purity (**table 5.4**). The latter is among the fragments analysed by Martinón -Torres (**2012**) and has been re-analysed here. Both come from beaker type crucibles with a transparent and glassy-looking residue stuck on their walls, the former showing a blue colour (**fig. 5.12A**). In SEM backscattered mode the glass layers appear very bright and dense and can be seen sitting on top of the crucible (**fig. 5.12B-C**). The ternary composition is made of silica (51.3 and 59.9 wt% SiO_2 respectively), lead (37.4 and 27.1 wt% PbO respectively) and potash (11.1 and 11.8 wt% K_2O respectively). Other elements resulting from the impurities present in the raw ingredients add up to less than 1 wt% of the total (**table 5.4**). Close to the interface area between crucible wall and residue are small newly formed crystals due to the interaction between crucible and glass (**fig. 5.12C**). Other inclusions in the glass phase include numerous residual grains of quartz and feldspars, with compositions ranging from potassium to sodium-potassium ones (**fig. 5.12D**) (**table 5.5**). The abundance, the grain size and the composition of such inclusions in the glass phase do not match the ceramic and may suggest that the vessels were used for primary glass making rather than for re-melting of cullet. However, both glasses appear too poor in alumina to have been made with such a feldspar-rich mix.

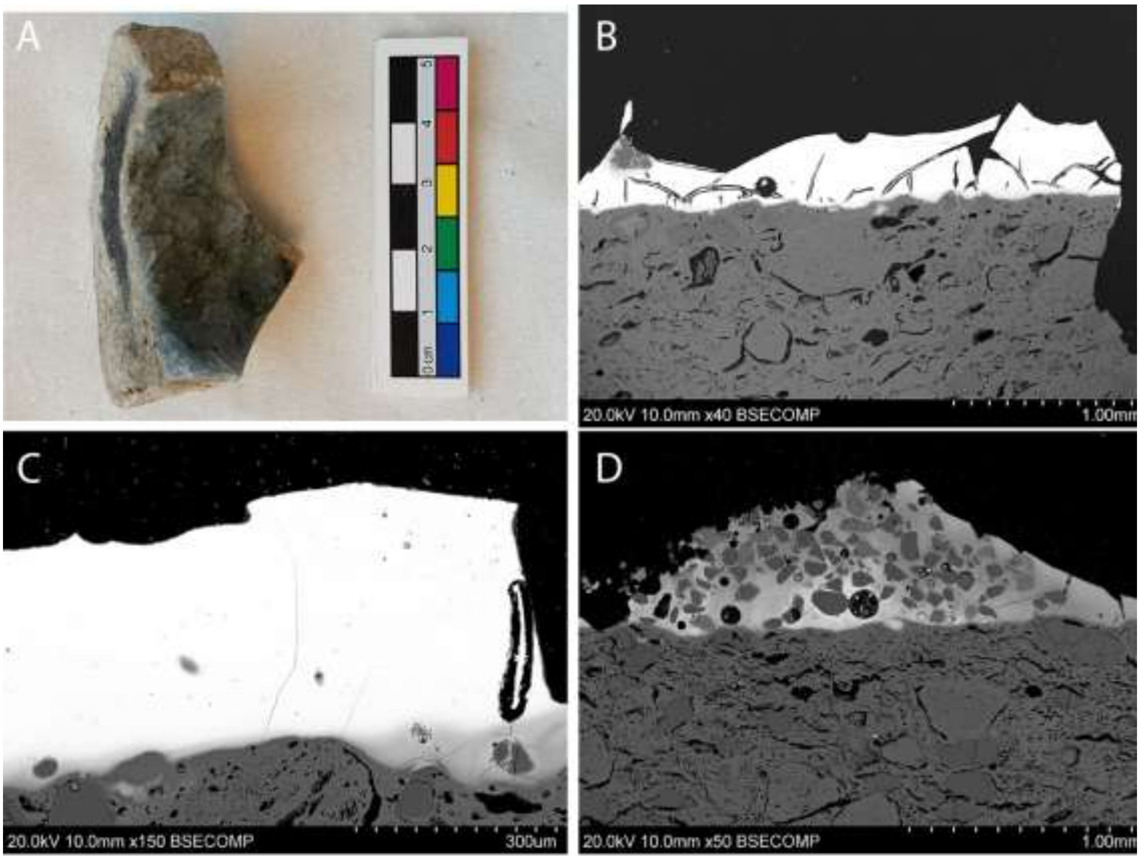


Figure 5.12. A-B: Image and SEM backscattered micrograph of crucible with blue glass AN1999.205.9d. C-D: SEM backscattered micrographs of the glassy residue on crucible AN1999.205.17. Note the area with residual rounded quartz and feldspars (right). The filament visible in the top-right and bottom-left pictures are corroded features.

Sample	Na_2O	MgO	Al_2O_3	SiO_2	SO_3	K_2O	TiO_2	FeO	BaO	PbO
AN1999.205.9d	bdl	0.5	19.7	59.0	bdl	18.0	0.3	0.8	bdl	1.7
AN1999.205.9d	0.3	0.9	25.9	50.6	bdl	19.4	bdl	2.1	bdl	0.9
AN1999.205.17	0.6	bdl	18.6	64.6	bdl	15.3	bdl	bdl	0.8	bdl
AN1999.205.17	7.5	bdl	18.5	66.5	bdl	6.2	bdl	1.4	bdl	bdl
AN1999.205.17	0.5	bdl	18.6	64.9	bdl	15.5	bdl	bdl	0.4	bdl
AN1999.205.17	6.5	bdl	19.9	67.6	bdl	5.0	bdl	0.9	bdl	bdl
AN1999.205.17	3.3	0.5	16.9	57.4	0.7	13.8	0.3	6.5	bdl	0.7

Table 5.5. Chemical composition by SEM-EDS of the residual grains of feldspar discovered in the glass matrices of samples AN1999.205.9d and AN199.205.17.

The chemical makeup of the two residues is consistent with lead crystal, a type of glass of recent invention patented in England by the entrepreneur George Ravenscroft

(1632-1683) in 1674 (**MacLeod 1987; Dungworth and Brain 2009; Brain and Brain 2016**). Ravenscroft wanted to create an alternative to the famed Venetian *cristallo*, a most desired product which owed its clear crystal-like appearance to a technology where ingredients were especially selected and thoroughly purified before being melted into the glass (**fig. 5.13**) (**Verità 2013; 2014**). The ingredients of lead crystal were also very pure and included sand or calcined flints (hence the alternative name of flint glass), saltpetre (potassium nitrate KNO_3) and litharge (PbO) (**Dungworth and Brain 2005**).

Figure 5.13. Left: Venetian cristallo goblet from the 16th century (from the Corning Museum of Glass); right: and examples of late 17th-century Ravenscroft's lead crystal (from the Victoria & Albert Museum).

Saltpetre was widely used in the early modern chemical industry (**Robertson 2016**) and it was particularly appreciated in glassmaking for being a very pure and strongly oxidising agent, which helped prevent the reduction of lead oxide into metallic lead (**Dungworth and Brain 2013**). Lead oxide was necessary to stabilise the glass, preventing it from quickly corroding in absence of impurities from the rest of the ingredients. It also gave the final product its characteristic brilliance while affording a soft glass ideal for carved decoration and elaborated tableware (**MacLeod 1987;**

Dungworth and Brain 2005). The final result was a ternary composition like the ones from the Ashmolean samples. However, there is uncertainty regarding the exact ingredients of the initial recipe by Ravenscroft as documents, including the patent itself, tend to be silent and rather confusing on this aspect due to the need of keeping the process as secret as possible. (**Moretti 2003; Dungworth and Brain 2009**). Lead crystal is discussed by Robert Plot in the 1677 *Natural History of Oxfordshire*, which confirms the profound interest he had towards what represented cutting-edge technology in late 17th-century England. Him and Robert Hooke both visited Ravenscroft's glasshouse and mention calcined flints, sand, tartar (potassium carbonate), niter (or saltpetre) and borax (sodium borate) among the ingredients used (**Dungworth and Brain 2009: 113**). Even though lead oxide was an essential ingredient for the reasons discussed above, neither Plot nor Hooke mention it in their reports, which may be due to the fact that the employment of litharge was kept secret by Ravenscroft. Lead oxide was probably part of the recipe from the very beginning, but what we know for sure is that the initial two decades of lead crystal production were characterised by a continuous adjustment of the quantity of litharge used (**Dungowrth and Brain 2013**). Chemical analyses carried out on assemblages of lead crystal show that there is a compositional trend towards increasing levels of lead oxide and decreasing levels of potash, which eventually settles at around 40 wt% of the former and 9 wt% of the latter and remains virtually unchanged throughout the 18th century (**Redknap et al 1995**). Documents tell us that the very initial production was severely affected by crizzling, a form of corrosion which resulted in a fine network of cracks developing on the glass. As a consequence, virtually all of the objects produced around the time of Ravenscroft's patent are believed to have been lost. The surviving objects probably date from 1676 onward when we know that the problem had been successfully addressed (**Dungowrth and Brain 2009**). Against this backdrop, sample AN1999.205.9d, with high lead oxide in excess of 35 wt% and potash levels of around 11 wt%, is similar to the post-1690s glasses belonging to Dungworth and Brain group 3/4 (**fig. 5.14**). On the contrary, sample AN1999.205.17 has levels of these oxides that are more similar to Dungworth and Brain group 2, which contains glasses dated to the

1680s (**Dungworth and Brain 2009; 2013**) (**fig. 5.14**). Both lead crystal samples display some important compositional differences when compared to the published data groups they were associated with. For instance, the silica to potash ratio of AN1999.205.17 is 5:1, versus an average of 4.7:1 of Dungworth and Brain group 2. In AN1999.205.9d the ratio is 4.6:1, while other high-lead flint glasses show sensibly higher ratios (**fig. 5.13**).

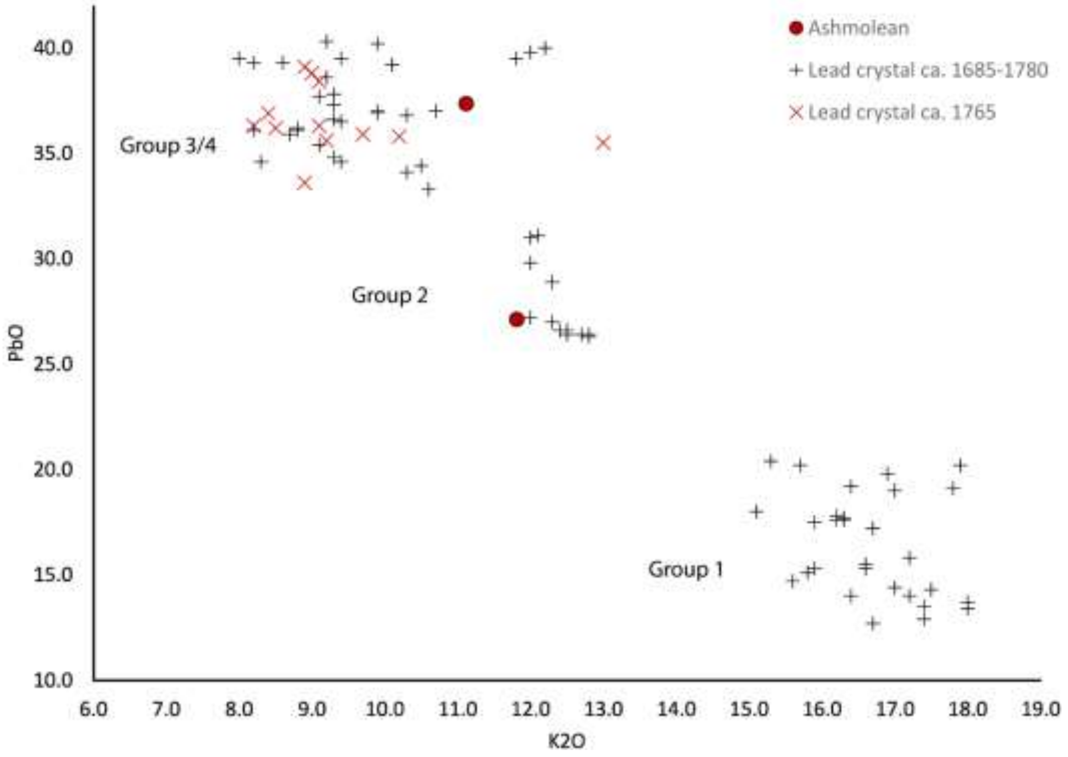


Figure 5.14. Potash vs lead oxide in the two samples of lead crystal discovered at the Ashmolean laboratory, compared to available data on lead crystal glass (from Dungworth and Brain 2009; 2013 and from Redknap et al 1995)

It is important to keep in mind that, as a result of the interaction with the ceramic, glass-on-crucible tends to be slightly more enriched in potash than finished objects (**Dungworth 2008**). Thus, differences between the Ashmolean samples and glasses from the literature may be less marked than what appears here. At any rate, the fact that the compositions do not exactly match anything known from the period is an indication that the chymists of the Ashmolean laboratory were experimenting with lead crystal recipes. This scenario is further substantiated by the presence of major compositional differences between the two very samples analysed here. The last notable aspect is the already mentioned blue colour of one of the two glasses. Considering the absence of impurities such as iron and manganese responsible for some of the most common naturally-occurring discolouration in glasses (**Weyl 1951: 89-131**), this sample must be intended as intentionally coloured. As an imitation of crystal, flint glass is by and large clear, but some coloured examples do exist in the archaeological record. An assemblage of purple, pink, green and blue lead crystal ingots was discovered in the cargo of the Albion ship that sank on the way to China in 1765 (**Redknap et al 1995**). The analysis shows that it has the same composition as Dungworth and Brain's group 3/4, confirming that the recipe had not changed in the later eighteenth century. The blue examples were found to contain ca. 0.04 wt% cobalt oxide and we can only assume that the same colourant was employed for the blue lead crystal in the Oxford crucible, but the very low levels needed to colour the glass fall below the detection limit of the SEM-EDS and it is therefore not possible to confirm this hypothesis. The pXRF screening performed on the sample signalled the presence of tiny amounts of cobalt as well as that of arsenic, a common association in cobalt ores from the mining districts of Saxony (**Gratuze et al 1992; 1996**). However, the data is qualitative only and does not allow to confirm the presence of cobalt beyond any reasonable doubt. Indeed, the same association of cobalt and arsenic shows up when testing other lead-rich residues too, a possible sign that the two elements came in with the lead source. As discussed in greater detail in section 5.6, cobalt coming from Germany was a matter of great curiosity among English natural philosophers, it was particularly appreciated as a blue pigment in vitreous materials and Robert Plot himself shows interest in the true nature of this substance (**Birch 1756 vol 4**).

The other two fragments with evidence of high-lead glass are AN1999.205.14 and 30, but unlike the samples previously addressed, they show evidence of tin-based opacification. However, substantial differences in the glassmaking recipes of these two samples confirms the ongoing experimentation and testing of recipes already seen in the case of the lead crystal. Sample AN1999.205.14 is a small fragment taken from the bottom of a beaker crucible, where two areas can be seen intermixing, one opaque white while the other, from which the fragment comes, dark green (**fig. 5.15**). The glass has a soda-lead-silica composition (12.5 wt% Na_2O, 32.4 wt% PbO, 52.5 wt% SiO_2) with an enrichment in tin oxide (1.8 wt% SnO_2) and only small traces of iron oxide. The absence of alumina, titania and alkali earths is an indicator that both the silica source and the soda flux were of very high purity. The tin oxide enrichment is coherent with the clusters rich in lead and tin oxide that in backscattered electron mode are visible as white rounded aggregates within the glass matrix (**fig. 5.15**). These crystals are mainly composed of tin oxide, whose levels nonetheless range from ca. 46 to ca. 93 wt% SnO_2. The balance is provided by lead oxide and silica, most likely absorbed from the surrounding lead glass and also highly variable (ca. 2 to 26 wt% PbO and ca. 4-27 wt% SiO_2) (**table 5.6**). In addition to these particles, large sub-rounded grains of partially reacted quartz can be seen in the glass matrix too.

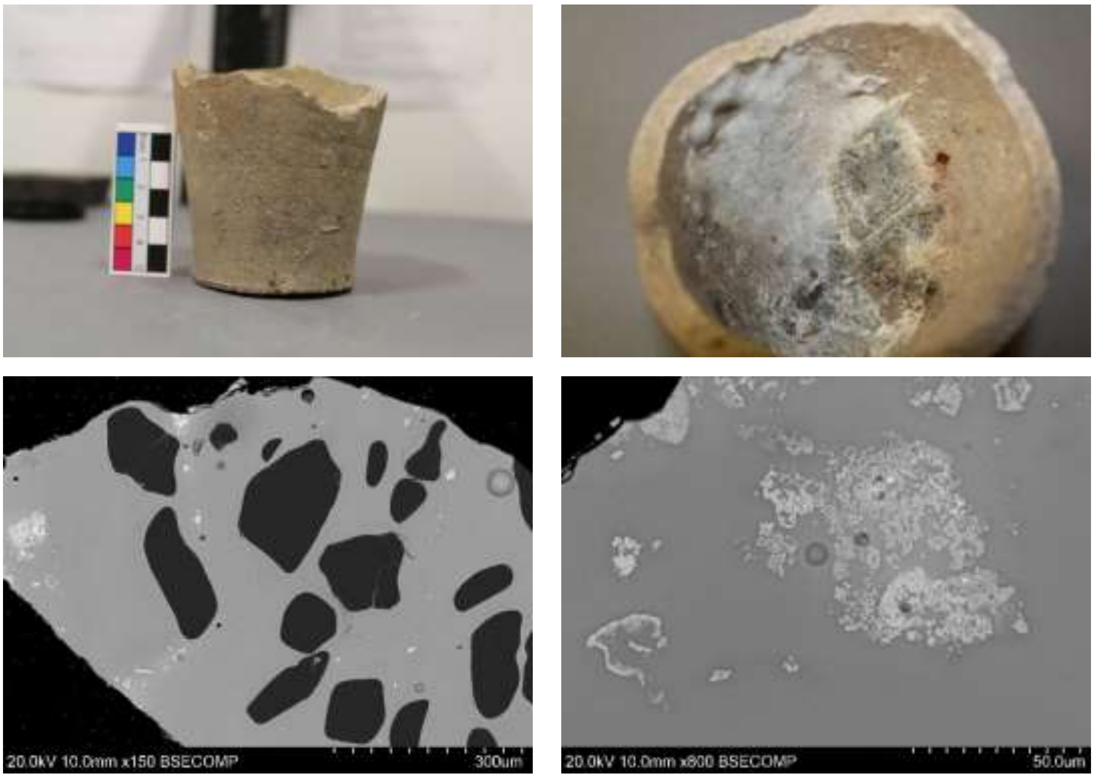


Figure 5.15. Sample AN1999.205.14. Top: Side view and inside of the crucible showing the glass coating on the inner wall. Note the two areas displayed by the glassy residue, the green one on the right and the opaque white on the left and down the wall. Bottom: SEM backscattered micrographs of the glass

sample showing the clusters rich in tin and lead oxide (white) and the grains of partially reacted quartz (black).

Sample	Na_2O	SiO_2	K_2O	FeO	SnO_2	PbO
AN1999.205.14	6.3	19.2	bdl	0.5	54.7	19.2
AN1999.205.14	3.7	12.3	bdl	0.5	77.1	6.5
AN1999.205.14	7.1	18.8	bdl	bdl	57.0	17.0
AN1999.205.14	6.3	19.8	bdl	bdl	47.3	26.6
AN1999.205.14	2.7	9.8	bdl	0.5	79.4	7.7
AN1999.205.14	1.8	6.3	bdl	bdl	83.9	8.1
AN1999.205.14	4.3	17.6	bdl	bdl	66.8	11.4
AN1999.205.14	3.8	15.5	bdl	bdl	71.7	8.9
AN1999.205.14	0.9	3.7	bdl	bdl	93.1	2.4
AN1999.205.14	6.6	27.2	bdl	0.6	52.5	13.2
AN1999.205.14	6.9	24.3	bdl	0.5	46.8	21.6
AN1999.205.14	2.2	9.5	bdl	bdl	83.0	5.3
AN1999.205.14	2.7	10.8	bdl	bdl	79.8	6.6
AN1999.205.14	2.1	8.2	bdl	bdl	84.4	5.3
AN1999.205.14	5.1	17.8	bdl	bdl	65.7	11.4
AN1999.205.30	bdl	42.5	21.4	bdl	35.8	bdl
AN1999.205.30	bdl	42.5	21.4	bdl	35.9	bdl
AN1999.205.30	bdl	42.3	21.0	bdl	34.4	2.3
AN1999.205.30	bdl	43.1	21.3	bdl	35.5	0.0
AN1999.205.30	bdl	42.5	21.2	bdl	35.7	0.7
AN1999.205.30	bdl	42.4	21.5	bdl	36.0	bdl
AN1999.205.30	bdl	42.1	21.8	bdl	35.7	bdl
AN1999.205.30	bdl	42.2	21.3	bdl	35.2	1.0
AN1999.205.30	bdl	42.1	21.5	bdl	36.4	bdl
AN1999.205.30	bdl	42.6	21.5	bdl	35.9	bdl
AN1999.205.30	bdl	42.4	21.5	bdl	36.1	bdl
AN1999.205.30	bdl	42.7	21.5	bdl	35.9	bdl

Table 5.6. Chemical composition by SEM-EDS of the opacifying crystals in samples AN1999.205.14 and AN1999.205.30

This sample is also remarkable for its substitution of soda for potash in the lead glass matrix. In the Venetian tradition, soda-rich lead glass was made by mixing calcined lead and *cristallo* frit, and it was employed 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 glass matrix of AN1999.205.30 has approximately 40 wt% lead oxide and 38 wt% silica, while the third main component here is potash (14.3 wt% K_2O). This is in contrast with the previously discussed sample where high levels of soda were detected and suggests the use of a different, potassium-based, type of fluxing agent, possibly saltpetre as in the case of the lead crystal samples noted before. A further difference with sample AN1999.205.14 is the substantial enrichment in alumina of the glass matrix (5.2 wt% Al_2O_3). This is most likely a consequence of the thinness of the residue analysed – only 100 to 200 µm – and of the absorption of ceramic material (**Dungworth 2008**). In order to test this hypothesis, a series of areas were analysed at increasing proximity to the ceramic-glass boundary (**fig. 5.16**). The results confirm that silica and alumina contents increase towards the glass-ceramic interface, while lead decreases correspondingly. In a similar way, it can be argued that the absence of tin oxide from the glass matrix, despite the tin-rich crystals found in it, can be explained by the same diluting effect, a phenomenon observed in other tin-opacified glasses (**Heck et al 2003**). Unlike what was observed by Dungworth (**2008**) in his study of chemical gradients at the interface between ceramic and glass, no gradient was recorded in alkali and alkali earth levels, though it should be noted that the glass layer analysed here is much thinner than that analysed by Dungworth, and very rich in potash throughout.

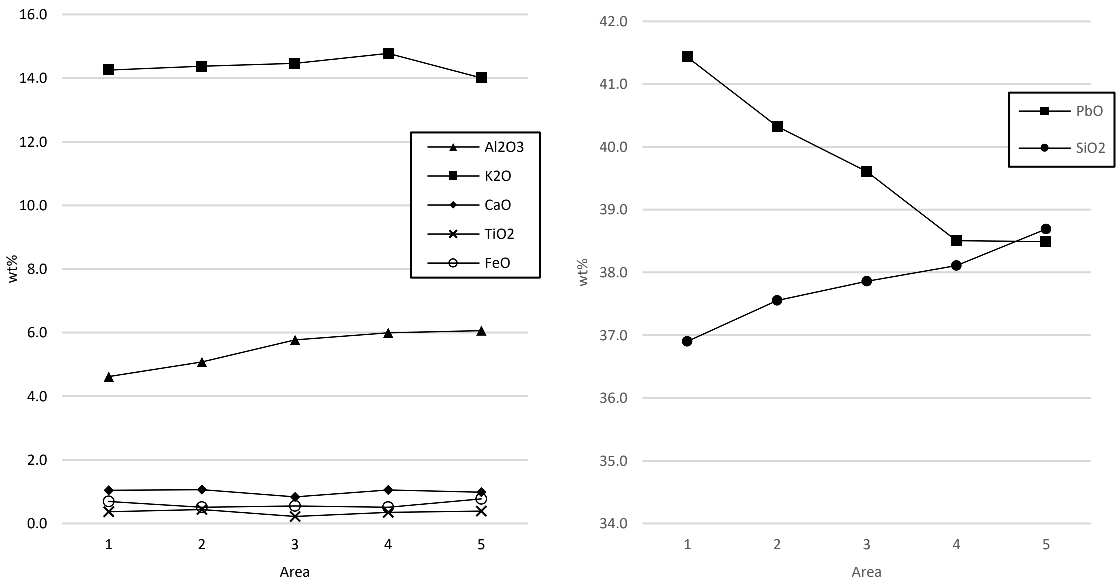


Figure 5.16. Differences in the chemical composition of the glass matrix of sample as a result of increasing proximity to the glass-ceramic border. The elements that are most affected are lead oxide, silica and alumina, while the rest seem to remain quite unchanged.

Non-homogeneously scattered throughout the glass matrix are angular particles rich in tin oxide (ca 35 wt% SnO_2) and representing the opacifying agent (**fig. 5.17**). Their composition is very constant and they also contain around 42 wt% silica and 20 wt% potash from the surrounding glass melt (**table 5.6**). its nearest mineral analogue is wadeite ($K_2ZrSi_3O_9$), with Zr instead of Sn in the formula, 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, not previously reported in historical glass samples (but see **Sakai et al 2000**), probably formed through the reaction of the added tin oxide with the surrounding glass melt. That the presence of silica and potash is to be related to interaction with the glass phase seems further confirmed by the presence, in some instances, of a bright tin oxide core within the grey crystals (similar to **Heck et al 2003: 40**) (**fig. 5.17**). All in all, the glass in sample AN1999.205.30 appears to have a chemical composition that is remarkably similar to that of the lead crystal samples AN1999.205.9d and 17, except the former is more contaminated by elements coming from the ceramic and has been opacified.

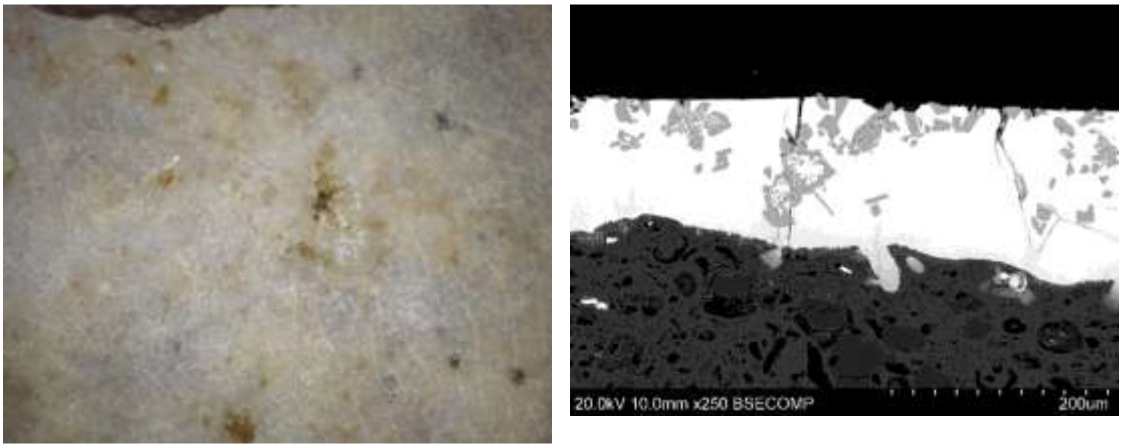


Figure 5.17. Sample AN1999.205.30. Left: close-up view of the glassy residue adhering to the inner wall of the vessel. Right: SEM backscattered micrograph showing the bright glassy layer on top of the ceramic. The angular tin-rich crystals can be seen scattered throughout the glass matrix (light grey). Note the bright tin-rich core visible in some of the crystals.

Starting from the 4th century AD, and particularly after the 9th century, tin-based opacifiers became the predominant reagents to achieve opaque white and yellow colours in mosaic tesserae (**Silvestri et al 2014; Neri et al 2017; Schibille et al 2018**), glazes (**Tite 2009; Matin et al 2018**) and in enamels (**Moretti and Hreglich 1984; Zecchin 1987; Henderson and Allan 1990; Freestone and Bimson 1995; Biron and Verità 2012**). In this period, there existed two main processes to opacify glass using tin-based reagents, namely *in situ* crystallisation and the addition of an intermediate product to a translucent base glass (**Lahil et al 2008; Biron and Chopinet 2013; Neri et al 2016**). In both cases, the first step was the preparation of a powder made by calcining lead and tin in various proportions. Differences in the ratio between lead and tin in the calx, firing temperature and heating time would be responsible for the white or yellow colour of the glass. Lead stannate particles produced a yellow colour (**Heck et al 2003**), whereas white develops when the lead oxide from the calx is absorbed by the glass melt and crystals of insoluble SnO_2 precipitate out of it (**Tite et al 2008; Matin 2019; Matin et al 2018**). While the *in situ* crystallisation entails the addition of the calx directly to the glass batch, causing the precipitation of opacifying crystals out of the high-temperature melt (**Matin 2019; Matin et al 2018**), in the alternative method, an intermediate glass known as *anime* is first made by melting the lead-tin calx with lead oxide and silica. This is then powdered and added to a translucent glass to colour and opacify it (**Tite et al 2008; Moretti and Hreglich 1986; 2013; Matin 2019**). This method was generally employed for intensely coloured glasses and is first mentioned in Venetian recipes from the mid-17th century but continues to be employed until the modern times (**Lahil et al 2008; Biron and Chopinet 2013**).

Both samples from the Ashmolean laboratory were opacified through the addition of a lead-tin calx and the subsequent formation of tin oxide particles in the glass melt. In the case of sample AN1999.205.14, partially reacted quartz grains further contributed to the opacification. It is important to point out that the fragment analysed comes from the green glass area visible in **figure 5.15**, and not from the white adjacent one (**Appendix C**). Indeed, the latter could have represented devitrified glass and it was deemed safer to avoid it at the sampling stage. In light of the results discussed here, the white area would probably have been more informative, while the green glass is likely to be a patch of poorly mixed ingredients with incomplete reaction. The chemical composition of the opacifying clusters supports this hypothesis since it does not match known compositions of lead-tin crystals found in the literature. In particular, the ratio between lead and tin oxides is much lower than in the crystals analysed by Matin and collaborators in Islamic opaque yellow glazes (table 9 in **Matin et al 2018**). Most likely, in the area analysed most of the lead from the calx had already been transferred to the glass melt, but the process was interrupted before the crystallisation of SnO_2 could happen. It is expected that an analysis of the white area would show a higher lead oxide content in the glass and fully formed SnO_2 particles dispersed within it. On the contrary, the opacifying crystals in sample AN1999.205.30 contain negligible amounts of lead, which had fully melted into the glass. However, the composition of these crystals is also severely distorted due to chemical interaction with the glass matrix. It seems sensible to assume that, if a thicker residue had been left on the vessel, one would have found purer and fully-formed cassiterite crystals where the reaction was complete.

The most important source of Venetian glassmaking technology is the treatise *L'Arte Vetraria*, written by the chymist/priest Antonio Neri and published in 1612. The book is the first entirely devoted to this art and comprises numerous recipes to make all sorts of coloured and crystal-like glasses in the tradition of Venice. In England, under the pressure of Robert Boyle who was deeply fascinated by glass, the Royal Society sponsored a translation which was carried out by Christopher Merrett in 1662. A few of the recipes of the *L'Arte Vetraria* deal with the production of opaque white glass. The first we encounter is for making *lattimo*, a famous type of milk-white glass (**Moretti and Hreglich 2013: 31**). About it Neri says:

> Take of crystal fritt twelve pound, of calcined lead and tin two pound, mix them well, of manganese prepared half an ounce, unite them all together and put them into a pot heated, let them stand twelve hours that the materials may be melted, and at the end of eight hours you may work it. This will be a fair white which I have often made. (**Cable 2006: 155**)

On the next page Neri provides another recipe:

> Take 400 weight of crystal fritt, and 60 pound of calcined tin, and two pound and a half of manganese prepared, powder and mix them all with the fritt, and set them in a furnace in a pot [...] and at the end of one day it becomes marvellous white. (**Cable 2006: 156**)

In both cases the outcome is the same, but the reagents differ slightly. The former reflects the usual mixture of calcined lead and tin (in a ratio of around 1:1), while in the latter only calcined tin is mentioned, indicating that lead was not mandatory when making opaque white glass (see also a recipe in **Moretti and Salerno 2006: 17**). The calx was mixed with a sinter of glassmaking ingredients (the crystal frit) and with manganese oxide, and fused in a crucible. The only ingredient that seems to be missing in the two samples analysed here is the manganese, even though with a ratio Sn:Mn of

60:2.5 the latter would have been so diluted that it may have fallen below the limit of detection of the SEM. Both recipes are mirrored almost exactly in entries of Robert Boyle's laboratory workbooks dated to 1656 and 1657 (**Boyle 2004**, work diary 14 and 15). Apart from making *lattimo*, Neri tells us that opaque white glass could be *the material werewith all enamels are made*:

> Take of fine lead 30 pound, of fine tin 33 pound, calcine them together in a kil and serce them [...] Take then of this fine calx, of crystal fritt made with tarso, ground and serced fine, of each 50 pound, of white salt of tartar eight ounces, powder, serce and mix them well: Then pour this stuff into a new earthen pot baked, giving it a fire for ten hours, then powder it and keep it in a dry covered place. Of this stuff are made all the enamels of whatsoever colours. (**Cable 2006: 204**)

In all these procedures, opacity is obtained through the precipitation of tin oxide particles in the glass matrix (i.e. *in situ* crystallisation). This last recipe is interesting because it shows that both sodium and potassium-based salts could be used when making these kinds of glasses, and this is reflected in the composition of the samples analysed here where both elements are present. At a time when in England lead crystal had just been discovered and its recipe was being perfected, it seems likely that Plot and the other chymists working at the Ashmolean laboratory were testing different recipes as well as ways of colouring and opacifying this new type of glass following the Venetian technological tradition.

5.5.2.2 ANTIMONY-BASED GLASS

The last glass-related sample is AN1999.205.25, a fragment belonging to a triangular crucible which shows a yellowish-green glassy drip going down its outer wall (**fig. 5.18**).

Figure 5.18. Sample AN1999.205.25, with the yellowish-green glassy residue adhering to the outside of the triangular crucible (left) and a detail of the sample taken from the corner of it (right).

The residue appears bright and dense in backscattered mode (**fig. 5.19**) and EDS analysis shows that it is a potash-antimony-silica glass (ca. 17 wt%, 31 wt% and 39 wt% respectively). The glass is also enriched in alumina, most likely from the crucible material (6.6 wt% Al_2O_3), and contains lime (3.3 wt% CaO), iron oxide (1.4 wt% FeO), sulfur (0.6 wt% SO_3) and titania (0.4 wt% TiO_2), plus negligible quantities of magnesia and soda. At the level of microstructure, small white crystals can be seen floating through the glass matrix (**fig. 5.19**). Spot analysis revealed they are particles of calcium antimonate with between 61-64 wt% antimony oxide and between 20-21 wt% lime, the balance being 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 (Sb_2S_3) as the source of antimony. Besides crucible contamination, the presence of stibnite in the melt, potentially carrying its own gangue, may be one of the reasons to explain the “dirtier” composition of this glass when compared to the other glass samples analysed. Equally, any calcium carried by the stibnite may also have triggered the crystallisation of calcium antimonate (**Shortland 2002; Lahil et al 2008**). Wood ashes can be ruled out as potential contaminants, as the levels of magnesia and phosphate are either absent or too low to account for this reagent often employed in glass making (**Wedepohl and Simon 2010; Dungworth 2011**).

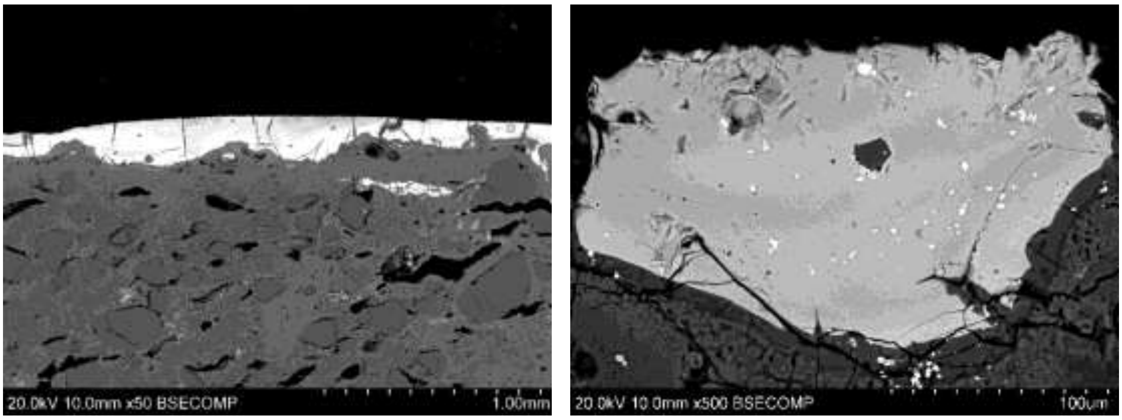


Figure 5.19. SEM backscattered micrographs of sample AN1999.205.25. Left: Bright glassy layer stuck on top of the crucible and seeping though the cracks in the ceramic matrix. Right: High-magnification detail of the glass phase with small calcium antimonate particles (white) scattered across the matrix.

Antimonates as opacifiers are known in glassmaking since the Late Bronze Age (**Shortland 2002**). At high-temperature, the antimony from calcined stibnite combines with the calcium contained in the glassmaking ingredients and forms the opacifying crystals of calcium antimonate by *in situ* crystallisation (**Moretti and Hreglich 2013**). This was the most common way to make opaque white glass throughout the Roman period and until the 4th century, when tin-based opacifiers take over (**Tite et al 2008; Schibille et al 2018: 419**). However, the employment of antimony compounds did not stop altogether with the end of the Roman Empire (**Lahil et al 2008**). Indeed, from the late sixteenth century both technologies coexisted in Venice with some recipes specifically calling for the use of stibnite and salt of tartar (**Moretti et al 2004: 27; Moretti e Salerno 2006; Biron and Verità 2012: 2711**). Again, the Venetian tradition reached Britain through Christopher Merrett's translation of Antonio Neri's treatise, and this sample may be an attempt at making an opaque white glass by following a different recipe from what has been found in the samples discussed above. In the commentary that follows the translation, Merrett reports a recipe for opaque white glass paste that can be used as base for coloured enamels:

> We shall be very brief in what follows, and shall onely deliver here one preparation come to my knowledge, whilst a secret of great value, but now commonly enough known to the furnaces, and 'tis this: Take of antimony and salt-peter well ground and mixed, of each

> twelve pound, together with 200 weight of the common materials for glass werewith this mixture of antimony and peter must be also well united, and then calcin'd in the calcar and made into a frit, or which is all one make regulus of antimony with crude antimony and peter, the manner every chymist knows, which being mixed with the metall afford a very white enamel, and serves with other mixtures for various colours. (**Cable 2006 :365**)

This is one recipe for the same white enamel with two variations. In the first, stibnite (antimony in the text) is mixed with saltpetre and the resulting powder is further mixed with common materials for glass, presumably silica and alkali salts, and fused together. In the second, metallic antimony (regulus of antimony) is first produced through calcination of stibnite (crude antimony) and saltpetre and then mixed with the glass frit (the term *metall* is used here for glass). Later in the commentary, Merrett refers back to this recipe stressing that it is a new way to make white enamels (**Cable 2006: 391**).

But early modern experimental chymistry was a multifaceted endeavour and often the same ingredients could be used in diverse laboratory operations. On top of that, the samples analysed here represent production residues and this makes it more complicated to know for certain what they were meant to become once the process had reached its end. This is particularly the case for a rather small patch of yellowish-green glassy matter as sample AN1999.205.25 is. It is therefore appropriate to consider whether a different interpretation may fit the same results obtained through SEM-EDS investigation. Among several possibilities, Martinón-Torres had proposed a link between this sample, which he had analysed as well, and the production of the substance known as glass of antimony (**Martinón-Torres 2012a**). Glass of antimony was a yellow-red vitreous form of antimony oxide, very well-known in early modern chymistry and pharmacology, whose popularity was greatly increased by the work of the German alchemist and physician known as Basil Valentine and his 1604 *The Triumphal Chariot of Antimony*. The book contains recipes to make glass of antimony by calcining stibnite and fusing the powder into a transparent yellow glass, sometimes adding borax (sodium borate) as a fluxing agent (**Valentine 1678**). Valentine does not mention other ingredients, but during replication experiments it was noted that in order for vitrification to occur the mixture needs some silica and that this most likely came as ore impurities and ceramic components absorbed from the crucible (**Principe 1987**). The glass was then used as a powerful emetic from which tinctures could also be extracted. We know from lecture notes and other pieces of documentary evidence that glass of antimony was indeed prepared by students at the Ashmolean sometime in the 1720s during John Whiteside's course on chymistry (**Roos 2017**). But glass of antimony also had chemical properties that were considered very attractive later in the 17th century. Building on Valentine's work, a new way of making antimony glass was published in the 1670s by the chymist Martin Lister (1639-1712). His process was essentially the same as exposed by Basil Valentine with the difference that Lister fluxed the calcined stibnite with barite, a by-product of English lead mines (**Roos 2010**). Interestingly, Lister introduces his recipe with these words:

> The several vitrifications of antimony are either opaque or transparent. To the first kind I shall add one, which is in itself very curious, and has these advantages above the rest, that is done with great ease and speed" (**Lister 1674**)

Even Isaac Newton showed great interest towards Lister's new glass of antimony as he hoped that the substance could make more reflective telescopic mirrors than those attained by metals (**Roos 2010**).

Nonetheless, the sample analysed here shows an important difference with glass of antimony as reported in the mentioned recipes, namely it contains almost 40 wt% of silica, definitely too much for it not to be intentional. For this reason, the interpretation of the residue as an attempt at making glass of antimony appears less likely. However, what is interesting to highlight here is that antimony-based glass products seem to have a relevant place in 17th-century chymistry, connecting medicinal endeavours to the field of optics and to the manufacture of valuable market commodities like enamels. In this sense, the chymists operating at the Ashmolean laboratory show an interest towards the effects of antimony in glass and the sample analysed here bears witness to a lively context where philosophical and practical endeavours intersected and cross-fertilisation occurred.

5.5.3 ZINC DISTILLATION

During the first XRF analysis carried out on the chymical tools recovered in 1999 Chris Salter reports that several of the artefacts bear traces of zinc-related metallurgical activity within them (**Hull 2003: 11-12**). However, the qualitative nature of the data obtained by Salter did not allow him to say anything else about the kind of operations that were going on in the vessels. Of the samples analysed here, five can be related to zinc metallurgy. The samples can be divided into two groups according to whether the evidence of zinc metallurgy is direct or else more speculative, but all belong to pieces of apparatus whose shape is suggestive of chemical reactions requiring a closed environment, a characteristic of zinc metallurgy (**Martinón-Torres and Rehren 2014: 115**). The most direct evidence is offered by two fragments belonging to the same tall closed-profile crucible of the type shown in **figure 5.3B**. Adhering to the crucible's internal wall is an unevenly distributed white crust (**fig. 5.20**) that during the pXRF screening prior to sampling revealed a strong enrichment in zinc, plus the presence of cadmium. One of the two fragments reported here (AN1999.205.22) represents a cross-section through the crucible and the white residue, while the second (AN1999.205.27) is a small lump of the white crust that was scraped off with a scalpel. Also targeted by the pXRF screening, but not sampled from SEM-EDS was the base of the same vessel, which showed a similarly high zinc signal (**fig. 5.20**). In the two cases when sampling was carried out, it was found that upon cutting through the white residue a fresh core of metallic zinc could be seen still preserved. SEM-EDS shows that in both samples the zinc phase is very pure, although minor amounts of cadmium (ca. 0.3 wt% Cd) and nickel (ca. 0.1-0.2 wt% Ni) were sometimes detected in the metal phase. These values are close to the instrumental limit of detection and should be taken as indicative only, but they are worth noting, especially the cadmium since it represents a common impurity in zinc ores, and like zinc is extremely volatile (**Dungworth and White 2007**). Tiny lead-rich droplets again containing small amounts of cadmium can be seen scattered in the metallic zinc (**fig. 5.21**). Developing all around the metal core, and particularly extended in sample AN1999.205.27, is a corroded area composed of zinc oxide and sulfate within which angular grey phases can sometimes be seen (**fig. 5.21**). These are either pure silica grains or crystals enriched in calcium, magnesium and sulfur, and are most likely of post-depositional origin.

Figure 5.20. Fragments of tall crucible with closed profile used in zinc-related metallurgy. Top: Fragment of the crucible wall (left) and sample taken from it (right). Note the layer of bright metallic zinc and the dark colour of the ceramic in contact with it due to the strongly reducing conditions within the vessel. Bottom: The bottom of the same crucible showing the zinc-rich white deposit and the dark-looking ceramic resulting from the reducing atmosphere.

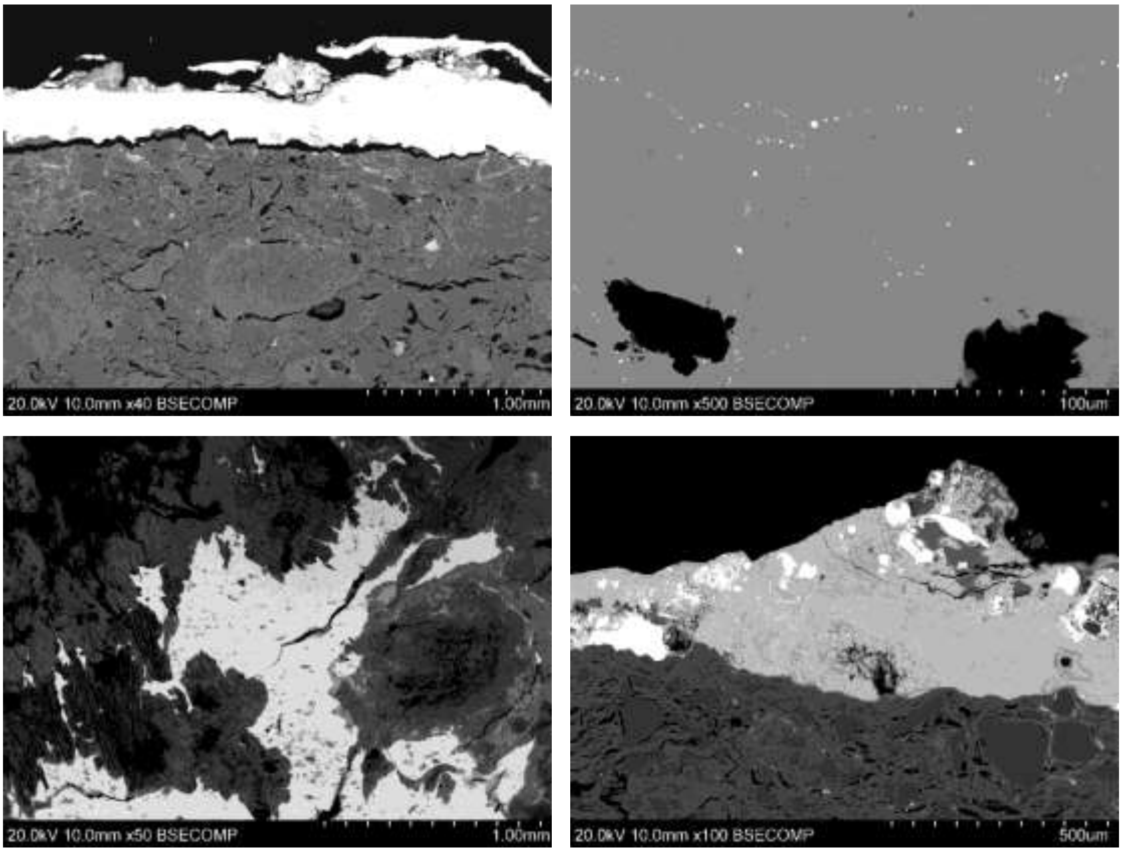


Figure 5.21. SEM backscattered micrographs of the zinc-rich samples. Top: Layer of metallic zinc in sample AN1999.205.22 (left) and tiny bright lead prills scattered across it (right). Bottom: Core of fresh zinc surrounded by zinc oxide/sulphate in sample AN1999.205.27 (left), and area of zinc oxide/sulphate with pools of metallic zinc (bright) and silica or lime-rich crystals (dark green) in sample AN1999.205.22 (right).

Some of the features found in these two samples, such as the segregation of tiny lead prills, the zinc oxide/sulfate crust covering the metallic areas and the presence of small quantities of cadmium in the zinc, find close parallel in the materials analysed from the 18th-century brass works near Bristol where zinc distillation was patented (**Dungworth and White 2007**). Bulk analysis of the ceramic body of AN1999.205.22 shows only minor contamination from zinc oxide (**table 5.2**), with very variable levels across it, reaching a maximum of around 2.5 wt%. Analyses of refractory materials used in similar operations often show higher levels of zinc oxide absorbed by the matrix (**Dungworth and White 2007; Etheridge and Dungworth 2012; Rehren 1999b**), but there are also instances where the contamination is minor and more similar to what was detected at the Ashmolean (**Craddock et al 1998**; **Rehren 1999a; Zhou et al 2012**). Possible reasons to explain the limited zinc oxide uptake in the ceramic matrix include the high refractoriness of the clay limiting the absorption of the zinc fumes, or the absence of a lid, which would have resulted in a great loss of volatile zinc.

The group with indirect evidence of zinc metallurgy includes the two fragments of distillation equipment, namely the fragments of cucurbit bottom (AN1999.205.21) and retort (AN1999.205.32), plus a small fragment taken from the bottom of a crucible with a black drip running down the wall (AN1999.205.19) (**fig. 5.22**). For these samples the connection with zinc can be hypothesised on the basis of the pXRF results and some SEM-EDS features, plus the fact that the first two belong to instrument types used in distillation.

Figure 5.22. Artefacts with indirect evidence of zinc metallurgy. Top: Fragment of a cucurbit's bottom showing a zinc-rich white glassy layer on the outside base probably deriving from its use as a lid (left), and base of crucible AN1999.205.19 with a dark residue dripping down its external wall. Bottom: Retort R1 (left) and sample AN1999.205.32 coming from it (right). Note the white slip that covers the internal wall in both the entire retort and the fragment.

Sample AN1999.205.21 represents a cross-section through the inside of the vessel's bottom and the dark-looking and glassy interaction layer that covers it (**fig. 5.23**). The layer is made of silica (57.5 wt% SiO_2), alumina (22.6 wt% Al_2O_3) and alkalis (respectively 9.1 wt% Na_2O and 4.8 wt% K_2O), plus smaller quantities of lime (2.9 wt% CaO), titania (1.1 wt% TiO_2), magnesia (1.6 wt% MgO), phosphate (0.3 wt% P_2O_5) and iron oxide (0.3 wt% FeO) (**table 5.7**). Small iron-rich prills can be seen dispersed in the glassy matrix (**fig. 5.23**), their composition including a host of elements among which phosphorus is the most abundant one (10.1-33 wt% P), followed by titanium (up to 6.3 wt% Ti), manganese (up to 5.1 wt% Mn), copper (up to 4.4 wt% Cu), vanadium (0.3-1.1 wt% V) and chromium (0-0.6 wt% Cr). Around 0.3 percent nickel was detected in only two of the prills analysed, while half a percent of zinc in one (**table 5.8**). The complex composition and the 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. Like for the previous sample, AN1999.205.21 mirrors quite closely the distillation apparatus from Warmley which has a very similar dark vitrified layer (see fig. 8 in **Dungworth and White 2007**) with small iron prills were also reported in the by-products from the zinc distillation site of Zawar in India (**Freestone et al 1985; Craddock et al 1998**). The main difference resides in the fact that, unlike what found in Bristol, no zinc was detected in the Ashmolean sample. More substantial evidence linking this sample to zinc metallurgy comes from the series of XRF analyses performed by Chris Salter soon after the 1999 excavation. He reports a strong zinc signal from the outside of the vessel's bottom, where a white vitrified layer can be seen (**fig. 5.22**), and suggests the possibility that the cucurbit may have been used as a lid (**Hull 2003: 11**). Unfortunately, the same area could not be targeted for invasive sampling, but the initial *in-situ* pXRF screening confirmed the strong zinc enrichment, and also indicated the presence of barium. Instead, neither sample AN1999.205.19 nor retort fragment AN.1999.205.32 gave any zinc signal when analysed with pXRF. For this reason, their connection to zinc metallurgy is only speculative.

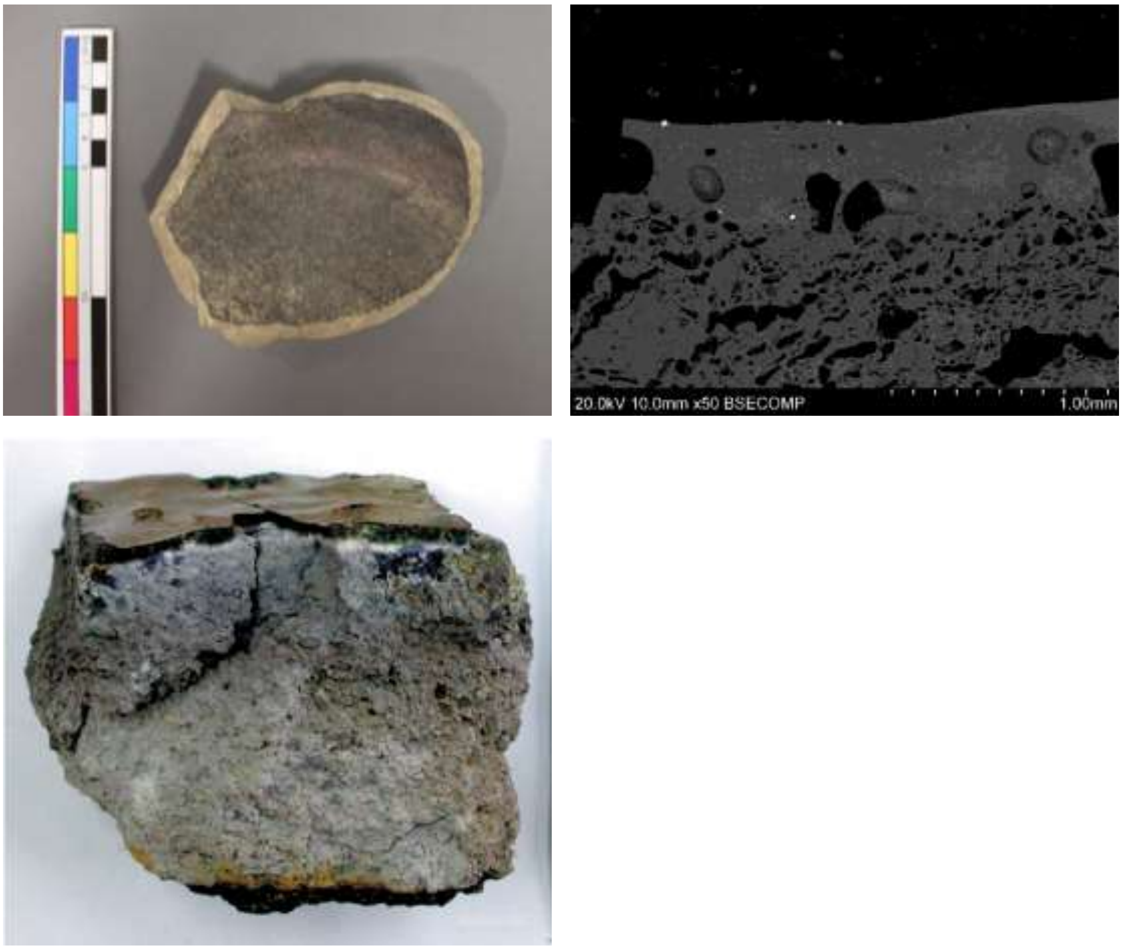


Figure 5.23. Top: Inside of sample AN1999.205.21 showing the dark vitrified layer (left) and SEM backscattered micrograph of the layer sitting on top of the ceramic (right, magnification 50x). Note the small iron-rich prills scattered across in (bright). Bottom: fragment of zinc distillation vessel from 18th-century Warmley brass works, showing a similar dark vitrified interaction layer (image from Dungworth and White 2007: 81).

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	FeO
AN1999.205.21	9.1	1.6	22.6	57.5	0.3	4.8	2.9	1.1	0.3
AN1999.205.19	0.5	1.3	21.5	57.5	0.4	1.9	2.9	1.2	12.8

Table 5.7. Average bulk chemical composition by SEM-EDS of the residue layers in sample AN1999.205.21 and AN1999.205.19.

AN1999.205.21	O	Si	P	Ca	Ti	V	Cr	Mn	Fe	Ni	Cu	Zn
Prill	bdl	0.5	10.1	bdl	bdl	0.3	bdl	bdl	88.7	bdl	bdl	0.5
Prill	bdl	bdl	21.7	bdl	1.6	1.1	0.5	0.7	71.9	0.3	2.2	bdl
Prill	bdl	bdl	21.9	bdl	1.6	1.1	0.6	0.5	72.2	bdl	2.0	bdl
Prill	bdl	bdl	32.4	bdl	5.2	0.5	0.4	3.9	56.4	bdl	1.2	bdl
Prill	5.4	bdl	20.7	bdl	3.1	0.6	0.5	3.8	61.4	0.3	4.4	bdl
Prill	bdl	bdl	21.6	0.2	6.3	0.5	0.4	5.1	61.9	bdl	4.0	bdl
Prill	bdl	bdl	33.0	0.2	4.9	0.4	0.4	4.3	56.1	bdl	0.8	bdl

Table 5.8. Chemical composition by SEM-EDS of the iron-rich prills found in the glassy residue of AN1999.205.21

Sample AN1999.205.19 comes from the base of a crucible whose type is difficult to ascertain. The shape of the foot appears to be compatible both with some open, beaker-type vessels (nos. 4, 6-8 in **figure 5.3A**) and with the closed-profile ones (nos. 2 and 3 in **figure 5.3B**). The focus of the chemical analysis was the characterisation of the black residue dripping down from the outside surface (**fig. 5.22**) (**table 5.7**). The three main components of the residue are silica, alumina and iron oxide (57.5 wt%, 21.5 wt% and 12.8 wt% respectively) and it shows an enrichment in fuel ash-related oxides (1.3 wt% MgO, 1.9 wt% K_2O, 2.9 wt% CaO and 0.4 P_2O_5). The residue matrix contains a large number of newly formed iron-rich crystals (ca. 40-70 wt% FeO), some of which with little amounts of nickel (0.3-1.2 wt% NiO) and zinc (0.2 and 0.4 wt% ZnO) (**fig. 5.24**) (**Appendix C**). This is the reason why AN1999.205.19 is discussed among the zinc-related samples, although its actual connection to zinc metallurgy cannot be ascertained.

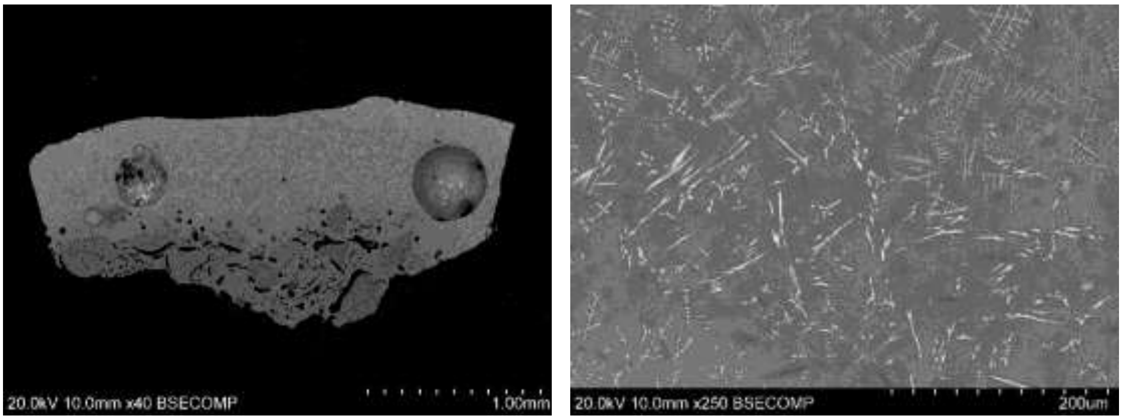


Figure 5.24. SEM backscattered micrographs of sample AN1999.205.19 showing the interaction area between the ceramic and the external black dripping residue (left, magnification 40x), and a close-up of the residue (right, magnification 250x). The white newly formed crystals that can be seen in the residues are rich in iron.

Similarly, the retort fragment AN1999.205.32 is also included in this section because of its connection with distillation activities, even though no definite link with specific zinc metallurgy could be established. The internal wall of the lower part of the retort is covered by a white and smooth slip (**fig. 5.22**). The pXRF screening performed on both the fragment and the almost entire retort highlighted an enrichment in barium in the internal slipped surface while it also detected mercury, lead, chromium, (plus some zinc and arsenic) towards the spout. Like zinc, metallic mercury is a highly volatile substance and requires a distillation technology in order to be recovered from the ore. However, the process was thermodynamically less demanding than in the case of zinc and for this reason it was known long before zinc distillation in western Europe (**Craddock 1995: 92-97**). Renaissance authors such as Agricola and Vannoccio Biringuccio provide detailed descriptions of how to heat cinnabar (HgS) in closed containers, allow the vapour to condense into mercury and finally retrieve the metal in various ways (**Hoover and Hoover 1950: 426-42; Smith and Gnudi 1990: 79-86**). They also provide detailed descriptions of the type of apparatus employed (**fig. 5.25**). It is likely that the technology of mercury distillation was a determining factor in envisioning and developing the principle of zinc distillation (**Craddock et al 1998: 48**) and the evidence found at the Ashmolean laboratory may indicate that both metals were being treated in the laboratory, potentially using the same apparatus.

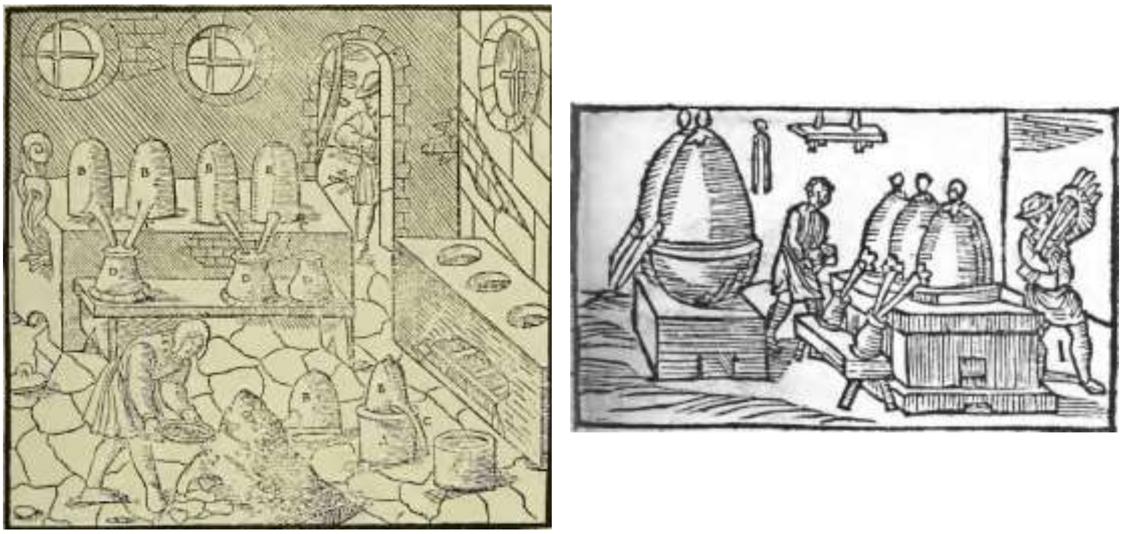


Figure 5.25. Woodcuts from Agricola's De Re Metallica (left) and Biringuccio's Pirotechnia (right), showing the apparatus for mercury distillation (images from Hoover and Hoover 1950: 429, and Smith and Gnudi 1990: 85)

Both Chris Salter and Marcos Martinón-Torres in their previous analytical work on the Ashmolean laboratory assemblage had reported the presence of zinc-related metallurgy going on in the laboratory (**Hull 2003; Martinón-Torres 2012a**). The new data discussed here offers further detail as to what sort of chemical operations the Ashmolean chymists were carrying out. First, the closed shape of the crucible analysed is in itself suggestive that such operations involved the need for achieving reducing conditions and, even more importantly, that any vapour be prevented from escaping, a characteristic of technical ceramics employed in zinc metallurgy (**Martinón-Torres and Rehren 2014: 115**). Second, the absence of any consistent trace of copper allows to discard brass cementation, in which closed-profile vessels were necessary to retain the zinc vapours and allow the copper to absorb it (cfr chapter 4) (**Bayley 1998; Day 1998a; 1998b; Rehren 1999a; Craddock 1998; Craddock and Eckstein 2003**). Instead, it seems that zinc was the sole focus of the laboratory practitioners' attention here, an interest that can be explained by the fact that this metal was still a rather puzzling substance to early modern metallurgists and natural philosophers who didn't recognise it as a metal in its own rights. The main reason lies in the peculiar thermodynamic behaviour of zinc, which owing to its high volatility could not be produced through the same high-temperature smelting process employed for other metals such as copper. Indeed, at around 907˚C zinc sublimes and escapes the reaction vessel as vapour, frustrating any chances of recovering the metal if a traditional furnace is being used. From at least the 12th century AD, Indian metallurgists seem to have used a distillation technology that allowed to catch the zinc vapour and condense it into metal droplets. The mineral was mixed with charcoal and other organic matter in closed vessels with a funnel-shaped condenser attached to the opening which were heated upside down and the fumes channelled towards a cooler area where condensation occurred (**Craddock et al 1983; Craddock et al 1998; Freestone 1985**) (left in **fig. 5.26**). In China, where zinc distillation begins at the latest in the 16th century AD, the same principle was applied, only the zinc vapour was allowed to rise to the upper part of tall vessels and condensed zinc was collected in special pockets (**Zhou 2007; Zhou et al 2012**) (right in **fig. 5.26**).

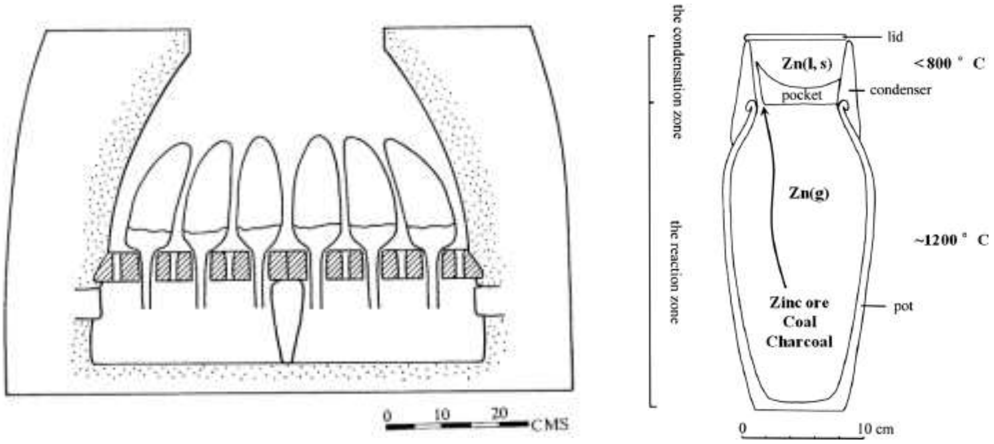


Figure 5.26. Left: Section of zinc distillation furnace from Zawar, India, with a set of retorts to perform the per descensum method in which the fumes were pushed downwards (after Craddock et al 1998). Right: Zinc distillation vessel from Fengdu, China, employed in the per ascensum method in which the zinc vapour was allowed to rise and condense in a pocket (after Zhou et al 2012).

Because in Europe metallic zinc was not generally available until the later 17th century, brass had for a long time been conceived as, to cite Biringuccio, “copper given a yellow color by art” (**Smith and Gnudi 1990: 70**), through the addition of powdered calamine. However, since classical antiquity metallurgists knew that when specific zinc-bearing silver and lead ores were smelted droplets of a silvery metal could be retrieved from the cracks in the furnace walls and that these droplets added to copper would produce brass (**de Rouette 1995; Craddock 1998; Craddock and Eckstein 2003**). For instance, the famous geographer Strabo, who lived between the 1st century BC and the 1st century AD, mentions a type of ore which "yields droplets of false silver" when heated in the furnace (**Caley 1964: 18**). Several centuries later the mining official and author of *De Re Metallica* Georgius Agricola reports the same phenomenon happening in the German lead-silver mines near Goslar and calls the metal recovered from the furnace walls *conterfei* (**Hoover and Hoover 1950: 408**). Also starting from around the 16th century, zinc ingots began to reach Europe from the east through trade routes, which further triggered the curiosity of chymists and natural philosophers towards this still mysterious substance (**de Ruette 1995; Rehren and Martinón-Torres 2008**). In the mid-17th century, Rudolf Glauber (1604-1670) discusses the recovery of zinc from the flues of German smelting furnaces and calls *calmei* the zinc retrieved, adding that by mixing this to copper one obtains brass (**Packe 1689: 319-320**). Glauber is a rather outspoken advocate of the need to better understand zinc and he regrets that "a great quantity is yearly burnt up and lost" and that "that ore might be melted with far greater profit, if they did not so burn up the zink and force it into fume" (**p.320**). Glauber's call for more experimentation with this substance seems to have been picked up in the Ashmolean laboratory where the increasing background knowledge around zinc in either mineral and metallic form may have been tested in the furnace. At any rate, the successful smelting of zinc via distillation, was only achieved in the 1730s when William Champion (1709-1789) of Bristol obtained a patent for a process that closely resembles the Indian one (**Day 1998a; 1998b; Etheridge and Dungworth 2012**). But it took much longer before speltering replaced cementation as the main method of making brass and before zinc was definitively classified within the metallic realm (**Rehren and Martinón-Torres 2008**). The documentary information regarding Champion's process is scarce and intentionally obscure, not unlike what was discussed concerning the invention of lead crystal (cfr section 5.7.2). We know that Champion had set up a copper, brass and zinc manufactory near Bristol and had developed a distillation technology following the Indian *per descensum* type, in which the zinc vapour was pushed down and condensed in water-filled vessels below (**Dungworth and White 2007**). Chemical analyses performed on a range of zinc-related materials from the Warmley brass works provides us with a detailed characterisation of the vessels and other residues of Champion's distillation (**Etheridge and Dungworth 2012**).

Within this context of growing scholarly curiosity and increasing technological knowledge around zinc the tall closed crucibles from the Ashmolean laboratory may represent an early attempt at distilling zinc in England not long before Champion's patent. Nonetheless, compared to pieces of zinc distillation equipment from India, China and England, the small size of Oxford's closed crucible is a rather clear indication of the experimental nature of the activity, most likely aimed at testing a novel technology rather than at actual production of zinc. In contrast with Warmley's materials, in the closed vessels from the *officina chimica* any distillation would have been performed with the vapour being sent upwards, like in the Chinese tradition. But the absence of the characteristic pocket found in the upper part of the vases and aimed at collecting the condensed metal, makes Oxford's exemplar also substantially different from the Chinese counterpart (**Zhou et al 2012**). The combination of pXRF and SEM-EDS data shows a large number of minor elements recurring in the samples analysed. These probably represent the suite of impurities associated with the zinc mineral tested as well as the input of other reagents added, like the charcoal necessary to maintain the right redox conditions within the vessel. Regarding the source of zinc ores, Robert Plot mentions the Mendip Hills district near Bristol as the place to find the best calamine and manganese in the world (**Gunther 1923-1945, vol. 12: 338**). This is also where William Champion mined the zinc minerals from for his brass works at Warmley (**Dungworth and White 2007**). The Mendip Hills deposit contains primary sphalerite and secondary smithsonite mineralisation associated with calcite, barite, gypsum and hematite (**Etheridge and Dungworth 2012**). In this sense, the grey calcium-rich crystals found in the white crust layer of AN1999.205.22 (**fig. 5.21**) may be a remnant of the gangue associated with the ore worked. On the basis of the information discussed here, we can imagine a scenario in which tall crucibles were filled with a mixture of powdered calamine (of the type described by Plot) and charcoal.

Heat was then applied and the vessels were closed using either proper lids or simply by placing another vessel on top. When the internal temperature reached around 900 ˚C zinc vapour started to rise towards the cooler upper part of the vessel where it would partly re-oxidise and stick to the walls (and lid). The resulting patches of white residue contained metallic zinc (or *conterfei*) which could be retrieved by getting rid of the oxidation coating. Such procedure has evident connections with the practices described by Agricola and Glauber but also shows more awareness around the nature of zinc as a metal in its own right, a still mysterious substance worth exploiting and studying.

5.5.4 OTHER CHYMICAL OPERATIONS

The remaining four samples cannot conclusively be ascribed to either glassmaking or distillation activities, and they point to a potentially wider range of activities carried out at the laboratory. 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 and seeping into the cross-section (**fig. 5.27 A-B**). The bulk composition of this residue layer is provided in **table 5.9**. Besides silica (52.8 wt% SiO_2), the residue is rich in alumina (18.8 wt% Al_2O_3) and potash (16.3 wt% K_2O), with other notable enrichments being the presence of some copper oxide (1.4 wt% CuO). Other elements include iron oxide (4.9 wt% FeO), lime (2.8 wt% CaO), titania (1.9 wt% TiO_2), phosphate (0.7 wt% P_2O_5) and magnesia (0.6 wt% MgO). The very diffuse interface between crucible and residue indicates a high degree of chemical interaction between the two. Indeed, elements such as alumina, potash, titania and part of the silica are likely to derive from molten crucible material. SEM backscattered images of the vitrified layer show a large number of sub-angular grey crystals, often clustered together and mainly composed of silica (39.3-43.3 wt% SiO_2), alumina (25.4-31.2 wt% Al_2O_3) and potash (24-28.2 wt% K_2O). Some of the crystals also contain sulfur (up to 1 wt% SO_3) and silver and copper oxides (respectively up to 1 and 1.6 wt%). The crystals are interspersed with numerous silver-rich globules, typically a few micrometres in diameter and with variable proportions of silver, copper and sulfur (**table 5.9**) (**fig. 5.27D**). One larger prill, of around 100 µm across, was found to be of almost pure silver but with 0.3 sulfur and some 1.8 wt% gold (**fig. 5.27C**) (**table C3**). Finally, the interstitial glassy background of the layer is enriched in silica, lime and iron oxide (74.4, 5.6 and 5 wt% respectively) when compared to the bulk, while it shows significantly less alumina and potash (9.2 and 2.1 wt% respectively). The process that led to the formation of the phases described is unclear. Given the presence of silver droplets (**table C3**), one possibility is the exploitation of silver-rich ores. However, the absence of significant enrichments in elements that often come in association with silver ores (e.g. lead) or of traces of a potential noble metal collector in the crucible seems to argue against a scenario of smelting/assay. Equally, no cupellation remains were found in the assemblage.

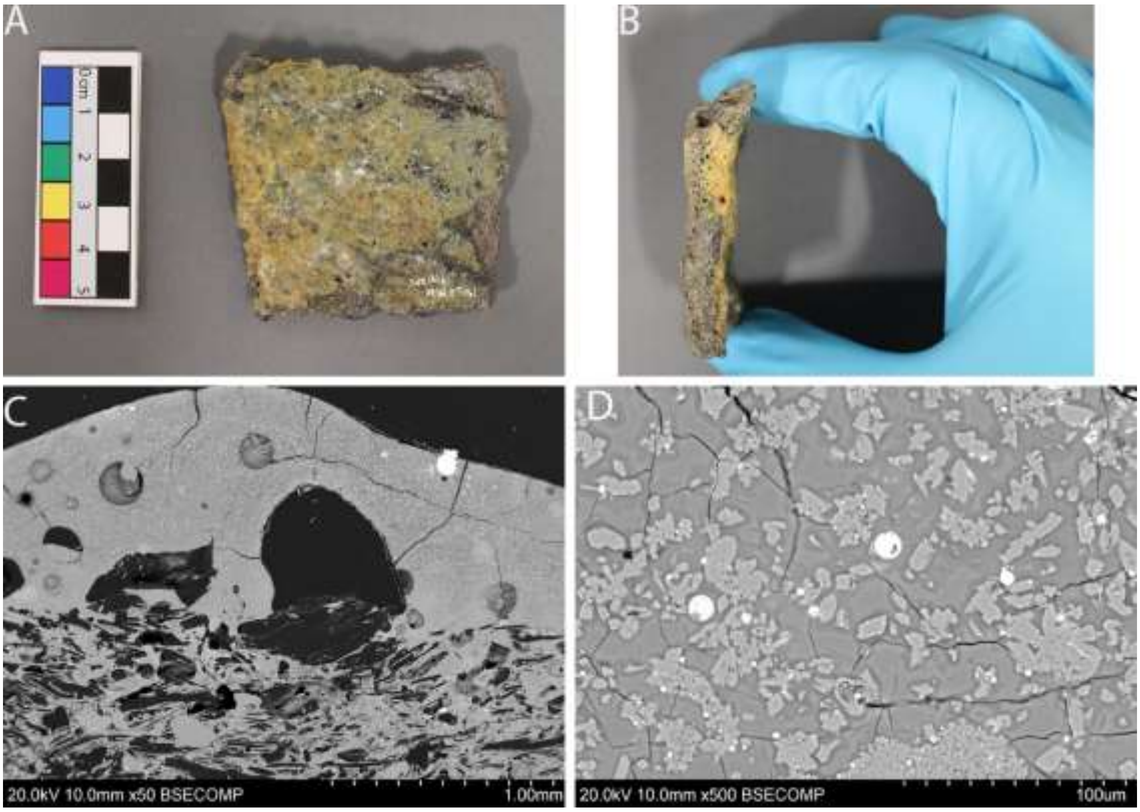


Figure 5.27. Top: Images of crucible fragment AN1999.205.28 showing the molten and discoloured ceramic on the inside wall (left) and the vitrification penetrating into the vessel (right). Bottom: SEM backscattered micrographs showing the microstructure of sample AN1999.205.28. The vitrified interaction layer can be seen adhering to the graphite-tempered crucible (left, magnification 50x). Note the large bright metallic globule towards the upper side. On the right, high-magnification image of the vitrified layer showing the kalsilite crystals (medium grey) and the metallic globules (bright) in a glassy matrix (dark grey). Some potential relics of the original mineral may be found in the clusters like the one in the bottom-right corner of this image.

AN1999.205.28	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	FeO	CuO	AgO
Bulk residue	bdl	0.6	18.8	52.8	0.7	bdl	16.3	2.8	1.9	bdl	4.9	1.4	bdl
Kalsilite	bdl	0.4	31.2	39.3	bdl	bdl	28.2	bdl	bdl	bdl	0.9	bdl	bdl
Kalsilite	bdl	0.3	30.9	39.6	bdl	bdl	28.2	bdl	bdl	bdl	1.1	bdl	bdl
Kalsilite	bdl	0.3	30.8	40.4	bdl	0.4	26.8	bdl	bdl	bdl	0.7	0.4	0.2
Kalsilite	bdl	0.5	27.3	42.5	bdl	1.0	24.0	bdl	0.2	bdl	1.9	1.6	1.0
Kalsilite	bdl	3.0	25.4	43.3	bdl	bdl	27.2	bdl	0.3	bdl	0.8	bdl	bdl
Glassy Background	bdl	0.6	9.2	74.4	0.7	1.0	2.1	5.6	1.6	bdl	5.0	bdl	bdl
Ceramic matrix	0.4	0.5	21.9	54.0	bdl	0.6	17.8	bdl	1.9	bdl	2.6	bdl	0.3

Table 5.9. Chemical composition of phases in the residue of sample AN1999.205.28.

The presence of silver may also suggest operations involving precious metals, potentially with an eye to alchemical goals of metallic transmutation. The discovery of a piece of elemental sulfur in the 1999 excavation would support this possibility since sulfur was an essential ingredient in metallic transmutation and was often combined with metals in order to manufacture the philosopher's stone. Robert Boyle's mentor, the chymist George Starkey, engaged in several transmutation experiments, mixing antimony with various other metals, of which he kept detailed descriptions in his laboratory notebooks (**Newman and Principe 2004**). Boyle himself, in his work diaries describes a process where he combines ground silver and copper with either sulphur or tartar (potassium bitartrate $KC_4H_5O_6$) in order to produce medicinal substances (**Boyle 2004, work diary 15**). In another section of the diaries the natural philosopher discusses how he made a solder by mixing gold, silver and copper (**Boyle 2004, work diary 17**). The potassium-rich grey crystals may therefore have resulted from the addition of a highly aggressive alkali salt such as tartar or saltpetre, which reacted with the ceramic to form kalsilite ($KAlSiO_4$), while also penetrating into the porous fabric causing it to vitrify and melt as seen in **figure 5.27**. This would also explain the extremely high levels of potash that were detected in the ceramic matrix (cfr section 5.5.1). Interestingly, in one of the pages of his notebooks, Starkey reports the failure of a Hessian crucible as a result of the extremely corrosive salts he employed (**Newman and Principe 2004: 217**).

The three remaining samples to be introduced and discussed in this section are AN1999.205.31, AN1999.205.18 and AN1999.205.29. The first represents the wall fragment of a crucible which the microstructure and chemical composition indicate as belonging to the triangular Hessian type. The surface appears severely altered by the chemical reactions taking place within it, with extensive melting and discolouration of the ceramic body (**fig 5.28A**). While the very high alumina content of the vitrified layer (16.6 wt% Al_2O_3) is due – in part or in total – to absorption of ceramic components from the crucible, other enrichments suggest an extra source (or sources) of lime (18.8 wt% CaO), magnesia (1 wt% MgO), phosphate (0.8 wt% P_2O_5) and the oxide of iron (4.9 wt% FeO) (**table 5.10**). The combination of lime, phosphate and magnesia suggests the presence of charcoal ashes in the crucible, but no other elements were detected which could help identify what substance was being worked. The high lime may also result from the melting of a calcareous rock, possibly to test the content of a metal, or from the presence of calcium-rich glass in the crucible's charge. In metallurgical operations, ground glass was sometimes added to the ingredients as a flux helping the formation of slag (**Hoover and Hoover 1950: 237**). However, there are no traces of metallurgical activity going on in this sample. Finally, some iron-rich bright clusters can be seen in the glassy matrix (**fig. 5.28B**), but their presence does not seem to offer clues as to the nature of the operations carried out in the vessel.

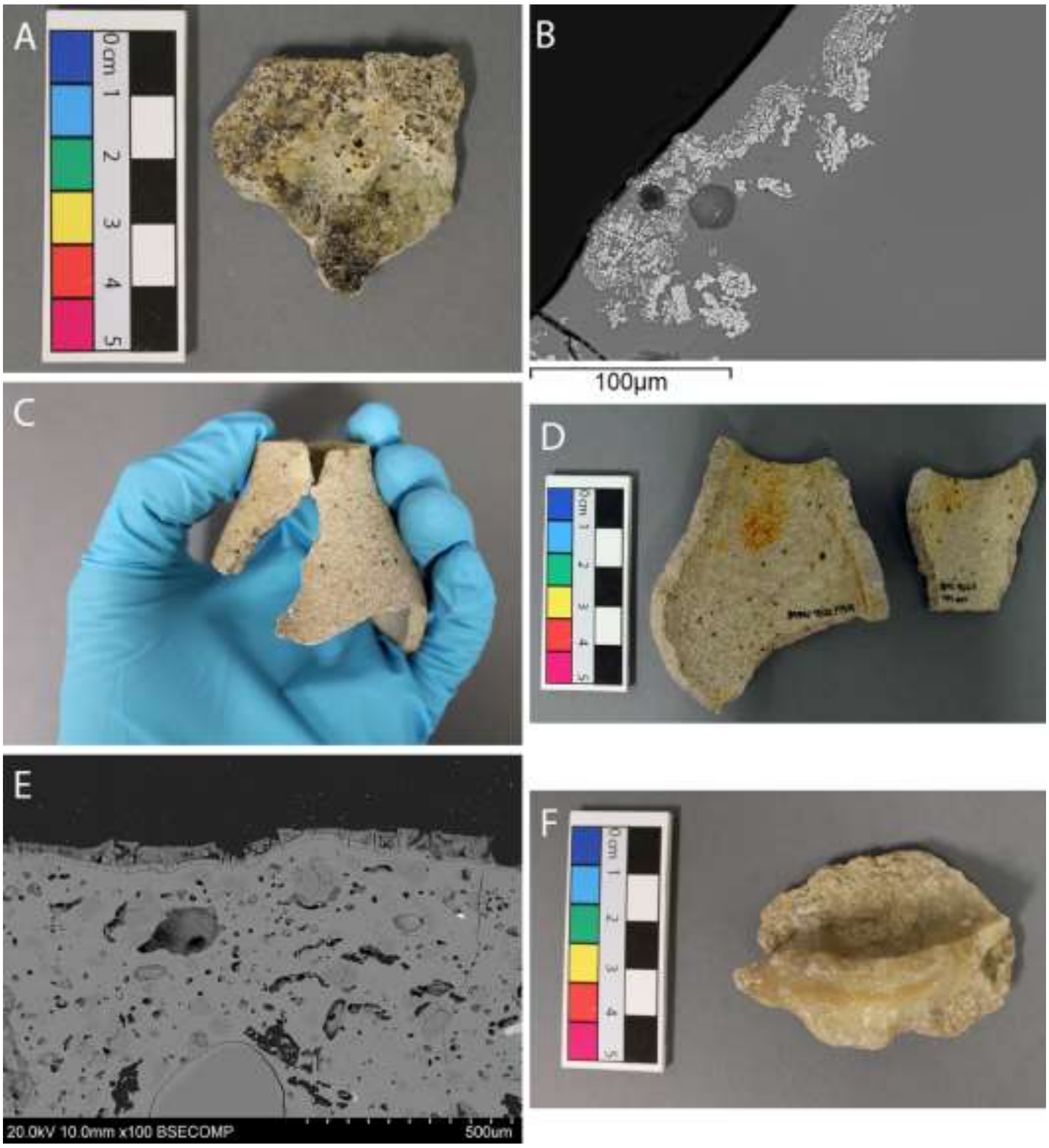


Figure 5.28. A-B: Crucible fragment AN1999.205.31 showing extensive surface vitrification and discolouration (left), and SEM backscattered micrograph of the vitrified layer (right). Note the bright iron-rich crystals. C-D: Images of the two fragments making the drug jar of sample AN1999.205.18 with orange spots visible on the inner surface. E: SEM backscattered micrograph of sample AN1999.205.18 showing the ceramic and the very corroded residue on adhering to it; F: image of the lump of yellow material from which sample AN1999.205.29 was taken.

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	FeO
AN1999.205.31 bulk residue	0.3	1.0	16.6	55.5	0.8	bdl	1.2	18.8	1.2	4.9
AN1999.205.18 bulk residue	0.1	bdl	17.7	62.1	0.5	bdl	1.2	8.9	0.9	8.6
AN1999.205.29 bulk	bdl	bdl	bdl	bdl	bdl	2.7	bdl	97.3	bdl	bdl

Table 5.10. Average bulk composition by SEM-EDS of the residue layers in sample AN1999.205.31 and AN1999.205.18 and of the bulk composition of sample AN1999.205.29.

AN1999.205.18 belongs to a small drug jar of the same type as no.5 in **figure 5.3C**. It is made of a white ceramic body with a thin orange deposit on its internal wall (**fig. 5.28D**). The deposit is very corroded and yielded very low analytical totals. The main components are silica and alumina from molten ceramic (62.1 and 17.7 wt% respectively), while enrichments that may derive from the jar's content include lime (8.9 wt% CaO) and iron oxide (8.6 wt% FeO) (**table 5.10**). Aside from the orange deposit, the vessel does not show other noticeable areas of discolouration, ceramic distortion or any visible sign of use in high-temperature operations. It is likely that jars like this one were only employed to store chemicals, either in the main room of the laboratory or possibly in the store room adjacent to it (cfr. 5.1).

Lastly, sample AN1999.205.29 is especially puzzling. It was taken from a yellowish lump of some undefined substance, whose slightly rounded shape suggests it may be the result of matter spilling from inside of a container (**fig. 5.28F**). SEM-EDS analysis shows it is made of ca. 97 wt% lime and ca. 3 wt% sulfate (**table 5.10**). However, the limited evidence available for this sample makes it difficult to establish whether it represents the product of a specific procedure or if instead it is some sort of intermediate material. Even though the composition is reminiscent of gypsum, the low amount of SO_3 points to a small proportion of this gypsum-like material, which may have been used as an additive or may simply be due to post-depositional contamination.

5.6 DISCUSSION: ARTISANS, PHILOSOPHERS AND ENTREPRENEURS

The previous section has attempted to provide a vivid picture of the laboratory operations and of the chymical practice carried out at the Ashmolean laboratory. The picture highlights that a relatively wide range of chemical operations were undertaken which required a host of ingredients, reagents, laboratory tools and operating conditions. Remarkably, such variety emerges from the analysis of only a selection of the objects recovered – and gives a clear sense that the laboratory's work was indeed wide-reaching, multifaceted and markedly experimental. The initial XRF screening of the whole assemblage performed by Chris Salter shortly after excavation noted the recurrence of specific enrichments in the vessels. In turn, Salter linked the presence of zinc to metallurgical operations involving this metal, that of lead to the production of glass and glazes, and finally the relative frequency of sulfates to fireworks. (**Hull 2003: 11-13**). While we found no further evidence to confirm Salter's claims to the latter area of chemical work, the results reported here shed new light on the intense activity of the Ashmolean laboratory in the chymistry of glass and zinc. And in particular, the experimental agenda of the *officina chimica* seems to be especially oriented in the direction of technological innovation in chymistry and materials, of new products and processes that between the second half of the 17th and the first half of the 18th centuries would play a major role in the English industry and help make the country more independent from the rest of Europe. These new technologies required experimentation and testing with recipes, and it is interesting to note here that although most of the reactions identified mirror codified practices of the time, the data also shows relevant discrepancies. This seems to point to the fact that Plot and the other chymists working in the Ashmolean laboratory were experimenting with recipes, trying to replicate and potentially improve more or less known procedures of their time as well as contemporary discoveries.

Glass technology is one of the industrial fields that features prominently in the chymical programme of the laboratory, and one where the interplay between established practice and experimental curiosity is most visible. Following the English translation of the treatise *L'Arte Vetraria*, sponsored by the Royal Society in 1662, cutting-edge glass technologies attracted erudite attention and stimulated experimental research and debate, rendering glass a current topic in scholarly circles. That Neri's treatise was a major source of speculation and enquiry is further indicated by the German translation of Johann Kunckel (1630-1703), where the author comments at length on the many recipes and significantly extends Neri's work (**Dupré 2017b; Hagendijk et al 2020**). A similar interest in the properties of glass had characterised Medici Florence, particularly in the 16th and 17th centuries, when chymical work on glass had become a major focus of the court laboratory at the Casino di San Marco (**Beretta 2017**). In England, such curiosity is mirrored in the meetings of the Royal Society where glass is a relatively frequent theme (**Birch 1756**) as well as in the fascination that natural philosophers such as Robert Boyle demonstrate towards a profession that he believed worth "to be made English" (**Hunter and Davis 1999, vol.6: 476**). *The Art of Glass* represented an effort in this direction as it brought to a wider public hundreds of recipes to produce all sorts of glasses, both coloured and clear, translucent and opaque, to manufacture vessels, enamels or to imitate precious stones (**Cable 2006**). The most notable achievement in this period of excitement for the English glass industry was the invention of lead crystal, patented less than a decade prior to the opening of the Ashmolean Museum. Scholarly curiosity around this new type of glass was very high, as demonstrated by the visits that both Robert Plot and Robert Hooke paid to George Ravenscroft's glasshouse at the Savoy, reporting the various ingredients they saw there (**Plot 1677: 253; Robinson and Adams 1935: 53; MacLeod 1987; Dungworth and Brain 2005; Brain and Brain 2016**). Importantly, the widespread interest in this new type of glass also shows that lead crystal was a matter of great economic relevance and indeed, Ravenscroft himself had obtained a monopoly over its production and distribution and had struck a deal with the London Company of Glass-sellers. That lead crystal had great economic potential on the market is further confirmed by Venetian authorities in London, who saw this new competitor as a threat to the Venetian production and wrote home with mounting preoccupation (**Moretti 2003**).

Besides encouraging experimentation with the most renown of Venetian products, Christopher Merrett's translation of the *Arte Vetraria* attracted more scientific and speculative interest around the wealth of technical information now available to the English audience. Robert Boyle as well as the members of the Royal Society seem eager to address issues of colour and opacity effects in glass in their experimental work. The use of cobalt – or *zaffera* - to tinge glass blue, for instance, is the centre of a lengthy comment made by Merrett in his observations to Antonio Neri's book in which he speculates on the nature of this reagent (**Cable 2006: 336-340**). Merrett begins by saying "what zaffer is I cannot find in any author, few there are that mention it" and goes on with a list of those author who mention it in their texts. Later in this discussion, he observes that *zaffer* must be made of a mixture of brass and sand (**Cable 2006: 338-339**). He comes to this conclusion partly by common sense: "the blew colour it gives, induceth me to think that 'tis from brass"; and partly by testing the substance, both physically: "a second ingredient into zaffer is sand your tongue and teeth may easily discover"; and through chemical analysis: "the reason I suppose that lapis calaminaris [zinc ore] may be admixt therewith is because neither aqua fortis [nitric acid] nor spirit of vitriol [sulphuric acid], poured over zaffer, have operation sensible thereupon". That the book had made cobalt a topic of interest among experimenters and natural philosophers is further confirmed by the information that Robert Plot sends a mineral sample to the Royal Society's repository (**Birch 1756 vol4: 179**) and by the long discussion that this ignites among the society members who, like Merrett, debate around the true nature of this substance and make experiments with it (**Birch 1756 vol4: 418-420**). The London-Oxford exchange on this issue continues, as shown by a piece of correspondence between the Royal Society and the Oxford Philosophical Society dated July 15 1685:

> Dr Slare [a member of the Royal Society] made some experiments with ye true kobalt or mineral of which ye zaffer is made, discovering what is ye true zaffer, which neither Dr. Merret nor any writer has yet done, it being nothing but this minera calcined [...] (**Gunther 1923-1945, vol.12: 99**)

A similar curiosity is reserved to the technology of opaque white glass for enamels. Robert Boyle seems to devote quite some energies to working on enamels of different colours. In some of his work diaries, dated to his time in Oxford in the mid-1650s, he reports some recipes, lists various ingredients and makes observations on what he sees happening in the crucibles (**Boyle 2004** work diary 14-15), demonstrating great familiarity with the work of Antonio Neri even before the English translation was published. In particular, Boyle details a procedure for making an opaque white glass paste from which "you may make all the other kinds of enamels" (**Boyle 2004** work diary 14) that mirrors one of Neri's recipes (**Cable 2006: 156**). And once again, it was noted how Merrett further contributed to this discussion by adding a recipe which does not feature in the Italian book (cfr section 5.5.2.2). In this recipe, Merrett substitutes the more common lead and tin calx with calcined stibnite (**Cable 2006: 365**). Indeed, antimony was another substance that sparked the curiosity of natural philosophical circles. Besides its possible use in making white enamels, stibnite could also be vitrified to make glass of antimony, whose multiple uses from pharmacology to optics, were discussed in section 5.5.2.2 too. Isaac Newton's interest towards this substance was already noted, but others express a similar attention. For instance, the chymist Robert Hooke too shows appreciation for the qualities of this substance, which he describes as "very dark and opaque" and therefore particularly suitable to make lenses for looking at the sun (**Gunther 1923-1945, vol. 8: 124-6**). Further, Plot includes glass of antimony in his *catalogue of bodies electricall* (**Gunther 1923-1945, vol. 4: 25**), while more than one source mentions a transparent red glass of antimony (see **Boyle 2004** work diary 24**; Valentine 1678; Principe 1987: 24**). Red glass, and particularly clear red glass, was in this period a sought product with the Royal Society showing interest towards the work of the German chymist Johann Kunckel who managed to make red glass through the addition of gold (**Birch 1756 vol. 4; Von Kerssenbrock-Krosigk 2008: 123-137**).

Against this background, the five glass-related crucible fragments from the Ashmolean laboratory each tell part of this story of renewed interest around glass in the second half of the 17th century. The two samples with evidence of lead crystal, whether relating to primary glassmaking or re-working of pre-existing glass, are both in line with the late 17th century state of the art. Clusters of residual quartz and feldspar grains were found preserved in some areas of both samples, and at least in the case of fragment AN1999.205.9d, these could have been brought in with the addition of the enigmatic blue colourant, probably a cobalt-rich ore or a silica-rich preparation (**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. Even in sample AN1999.205.14 the evidence for primary production as opposed to re-melting of pre-existing glass is not conclusive. If the numerous crystals of quartz partly dissolved in the matrix may support the former, these seem more likely to have been added as opacifiers. Further, the sharp contact of the melt to the ceramic body, and the generally clean composition of the base glass seem to point more towards re-melting. Regardless, the presence in the glass matrix of clusters of minute tin-rich crystals clearly indicates that the main aim of the operations was obtaining an opaque white glass (**Shortland 2002**), whether this was done by opacifying a pre-existing glass or by making it from scratch. 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 17th century. As mentioned in section 5.5.2.1, 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, $K_2ZrSi_3O_9$. 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. Lastly, sample AN1999.205.25 bears us witness to the experimentation with antimony that was going on within scholarly circles of the period. What is interesting here is that the glass, unlike the two samples just discussed, is based on more than 30 wt% antimony oxide rather than on lead oxide. It is quite possible that the red-yellow litharge typically used for lead glass preparations was replaced with the similarly red-yellow *glass of antimony*, the amorphous antimony oxide. Taken together, these findings reinforce the idea of the Ashmolean laboratory as a space for cutting-edge research on processes that may represent both technological innovation of the period or already known procedures whose interest to natural philosophers had grown in the course of the 17th century.

More links between the chymical programme of the Ashmolean and the work of innovators in English industry come to the fore in the assemblage analysed here, showing the extent to which Plot's laboratory operated at the forefront of scientific and technological research. If the contents of the vessels represent direct evidence of the link Ashmolean-industrial innovation, the ceramic apparatus itself represents indirect evidence. Indeed, specialised pottery is an important protagonist of the chemical technology between the 17th and the 18th centuries, characterised by intense experimental efforts and notably those of the potter John Dwight. There are remarkable parallels between Dwight and the contemporary glassmaking work of George Ravenscroft. Like Ravenscroft in his field, Dwight represented the very latest in ceramic technology and he had been capable to make imitation Chinese porcelain and German stoneware, and to have cracked the secret of Hessian crucibles. These achievements were so noteworthy to contemporaries that both Plot and Hooke visited the Fulham establishment and report on Dwight's work (**Plot 1677: 250-251**), just like they had done with Ravenscroft. And also like Ravenscroft, Dwight struck a deal with the London Company of Glass-sellers which sourced stoneware exclusively from his factory (**Plot 1677: 250**). Under this light, it seems likely that Plot turned to Dwight when stocking up his laboratory and indeed it was noted how at least part of the vessels may be of Dwight's production.

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 and in dealing with its extreme volatility. William Champion's patent for the extraction of zinc through distillation was a ground breaking discovery in the first half of the 18th century. The achievement represented the apex of an artisanal tradition stretching back centuries and characterised by an ever-increasing knowledge around zinc's elusive nature, a knowledge reached through experience and experimentation as well as through growing exposure to this "new" metal coming from China and India (**Martinón-Torres and Rehren 2002**). The patent itself was intentionally misleading as to the type of ore to be distilled, a practice that was all but uncommon in this period and was aimed at preserving the industrial secret against competition (**Etheridge and Dungworth 2012**). For a long time and again in the decades leading to the development of Champion's process, zinc had attracted a great deal of scholarly attention. As outlined in section 5.5.3, at the core of this curiosity was a generalised confusion in relating zinc minerals such as calamine, well-known and rather unproblematic substances, to the silvery metal that was being traded in from India or to the droplets that miners had been retrieving from silver smelting furnaces since classical antiquity (**de Rouette 1995; Craddock 1998; Craddock and Eckstein 2003; Rehren and Martinón-Torres 2008**). Such confusion is very well pictured by the host of names by which the various minerals and sublimates were known in antiquity and in the early modern period (**de Rouette 1995**). The work on zinc carried out at the Ashmolean laboratory must be seen within such context of experimentation with this substance before or around the time of the 1738 patent. Furthermore, after lead crystal, stoneware, porcelain and English-made Hessian crucibles, the distillation of zinc is the story of another foreign technology made in England through a combination of scholarly study and laboratory testing, it shows once more that scholarly endeavours of this period were very often fuelled by technological progress.

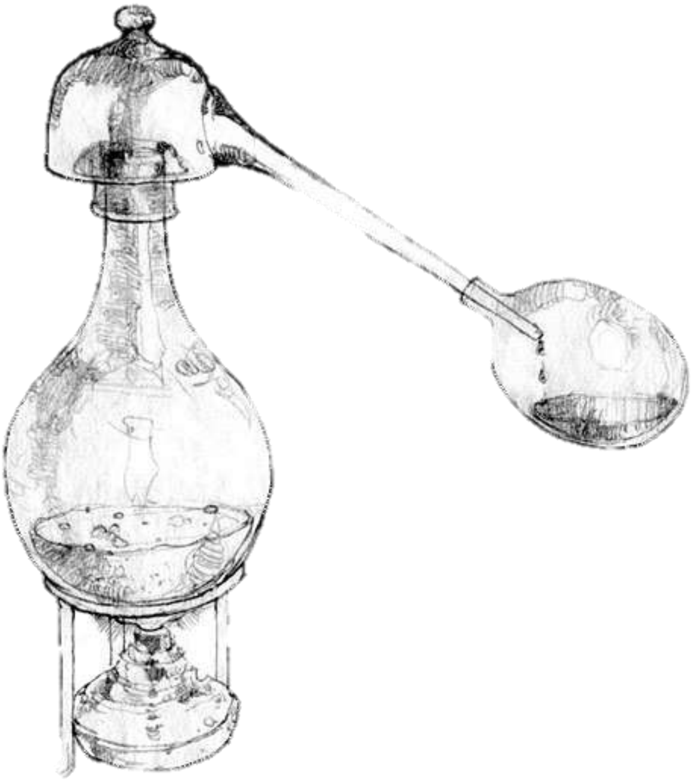
In this respect, the network of materials and chymical practices that the Ashmolean assemblage exposed, provides us with a connecting thread running through some of the most influential figures of early modern English chymistry. The work of Plot, Boyle, Hooke, Newton, Merrett and the circle of natural philosophers of the Royal Society and of the Oxford Philosophical Society had indeed very intimate connections to that of chymists-entrepreneurs who brought major technological innovation to the English industry. John Dwight and George Ravenscroft are two such examples, and their innovative products seem to have been central to the experimental programme of the Ashmolean laboratory. Robert Plot, the laboratory's first director and a contemporary of Dwight's and Ravenscroft's, follows their activity closely and in his written work he shows a profound interest towards the technological innovations of his time. What emerges from following the thread is a network, 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 products that came out of the Oxford's *officina chimica* had significance as both objects of philosophical enquiry as well as economically valuable commodities. Making coloured glasses and successfully distilling zinc were both ways of investigating the inner workings of nature and also of testing procedures and recipes with their own value in the market, and whose importance cannot be overestimated. The story of flint glass is as much about industrial espionage and profitable deals with regulating bodies like the Company of Glass Sellers of London as it is about understanding the chemical reasons of the catastrophic weathering that affected the early attempts. But working with glass, producing effects of colour and opacity or obtaining a softer glass that could be carved more easily were also intimately connected to philosophical speculation around the structure of matter, the debates around transmutational alchemy and the value of art and human artifice in imitating nature (**Beretta 2004; 2009; 2017; Von Kerssenbrock-Krosigk 2008**). Robert Plot himself was aware of the economic potential of the activities carried out in his laboratory and we know that "a large quantity of prepared chemicals […] may be purchased for money" (**Bennett et al 2000: 21**). He too entered deals with potential sponsors and buyers of the chymical preparations he and Christopher White made (**Roos 2014**). At the same time, the laboratory operated within the scholarly, erudite context of academic teaching and quickly became a reference point for the philosophical societies of the time and the epicentre of Oxford's chymical community. The mixing of more scholarly and more industrial endeavours, as revealed by the reconstruction of the Ashmolean's experimental agenda, very well reflects the spirit of the 17th century and the ideas of Francis Bacon on which the institution had been founded. Bacon's belief that the best way to obtain knowledge around the causes of things is by turning the attention to those who actively engage with nature (**Rossi 2009; Pastorino 2017**), gives a decisive shape to the chymical work carried out at Ashmolean Museum. In the intentions of its founders, the institution was supposed to be akin to the Solomon's house described by Bacon in his *New Atlantis*, a place where empirical chymistry served the purpose of exploring the sensible world while also producing useful things. Similar ideas can be found in the other central figure of 17th century chymistry, Robert Boyle, who in the fourth essay of his 1671 *Usefulness of Natural Philosophy* claims that "an insight into trades may improve the naturalist's knowledge" while at the same time "the naturalist, as well by the skills thus obtain'd, as by other parts of his knowledge, may be enabled to improve trades” (**Hunter and Davis 1999, vol.6: 467**). Cooperation between artisans and philosophers will not only result in a deeper understanding of nature’s secrets but also in a significant improvement of the industry, for everyone’s gain. The material culture of the Ashmolean laboratory gives a very clear sense of such integration of artisanal values into natural philosophical enquiry, a characteristic of the new experimental sciences of the late 17th century.

5.6.1 A NOTE ON CHRONOLOGY

As already addressed earlier in this chapter (cfr 5.2), the dating of the assemblage analysed is a debatable issue and this is due to 1) the materials coming from a secondary context and 2) the long-lived formal type the crucibles and other vessels belong to (**Bennett et al 2000; Hull 2003**). On the basis of the domestic pottery, and particularly the presence of pearlware, Graham Hull proposes a late 18th century dating for the deposition of the materials, though he reports sherds of ceramic types with an earlier date, like German stoneware (**Hull 2003: 5-10**). Shifting the focus on the chymistry-related vessels, Marcos Martinón-Torres indicates the possibility of an earlier date of at least part of the assemblage based on the consistency between the content of some crucibles and known chymical practices of the late 17th or early 18th century (**Martinón-Torres 2012**). The larger quantity of apparatus that could be examined here supports this observation. Indeed, the results outlined and discussed in 5.6 and in the present section very closely mirror chymical operations and underlying interests that are generally consistent with a chronology spanning from the opening of the Museum in 1683 to the first quarter of the 18th century, probably before William Champion’s zinc distillation process had become widely known. It may be argued that lead crystal and white glass for enamels were important products throughout the 18th century and after (**Charleston 1963; Redknap et al 1995**). However, it was shown how the late 17th century had been a period of particular innovation in these fields, and a moment of great scholarly interest towards glassmaking. These new technologies generated great curiosity in the circle of natural philosophers gravitating around the Ashmolean Museum, including the director Robert Plot. Moreover, one of the lead crystal samples analysed in this thesis shows a composition that is similar to others dated to the more experimental initial phase during which the recipe was elaborated (cfr 5.5.2.1). Thus, still maintaining a necessary note of caution, the new data reported here is a further piece of evidence that goes in the direction of an earlier chronology for the Ashmolean laboratory assemblage, at least as far as the chemical vessels are concerned, even if these may well have been deposited later in the context where they were recovered.

CHAPTER VI

ARCHAEOLOGY AND THE CHYMICAL LABORATORY



6.1 A DIFFERENT SET OF PRIMARY SOURCES

Chapters 4 and 5 reconstructed the details of laboratory practice at two early modern chymical contexts of very high profile. What emerges is a material landscape, a mental map made of the many substances manipulated at the sites and the various tools the chymists used in different high-temperature operations (**fig. 6.1**). While the focus of historiography is generally on people and people's networks, it is argued here that material maps such as the one discussed here provide an alternative way of following the shifting aims that drove chymistry and dictated agendas across different realities. Moreover, materials and their analysis become a fundamental contribution to the history of the two sites under investigation, both reinforcing and re-assessing what we know about them. We see, for instance, that material culture challenges the traditional historiographic narrative that blames the failure of Jamestown on the incompetence and idleness of the settlers, a narrative that is, at best, simplistic. Instead, the chymists knew what they were doing and came equipped with some of the best apparatus available at the time. They were required to find sources of gold and silver, to establish long-lasting industries and new trade routes, but were confronted by a new environment that must have severely challenged their expertise. Even so, materials show how they strove to use their technical knowledge in assessing the quality of the minerals of Virginia, prospecting for noble metals and also searching for potential sources of zinc and tin for copper alloys. The types of mineral bodies they found during exploration trips up the river James were of complex nature, they contained several metals and were for the most part rich in sulphur, adding further difficulty to the smelting and assaying process. The ores contained some silver - though gold seems largely absent – but also frequently included copper, iron and lead, and less frequently other metals such as tin, zinc, arsenic, antimony, nickel and cobalt. The minerals were collected, crushed and worked in crucibles together with the necessary reagents, such as charcoal to attain the correct redox conditions and a host of substances to flux the mixture and promote separation of the slag. Metallic lead was probably added to the crucibles too, as a noble metal collector, although most of the ores tested were already rich in lead. Potential zinc and tin ores were allowed to react with scrap copper in an attempt to test their suitability for making brass and bronze. These are all common procedures in early modern metallurgy and fire assay and can be found in contemporary technical writings (see, for example, **Sisco and Smith 1949; Hoover and Hoover 1950: 219-265**). However, being in a new natural environment, the chymists must have faced some degree of unknown and must have dealt with substances that looked and possibly behaved in unexpected ways. Thus, they had to move through unexplored terrain, make tests, experiment. Such strongly experimental character coexists with the colony's fundamentally profit-driven nature, where chymistry was carried out in the name of exploiting the riches of the new world and boost English power on the international market.

The industrial emphasis frames Jamestown's chymical agenda in a decisive way, and makes the work carried out here different from that of Oxford's Ashmolean laboratory, which instead operated within a more scholarly context, one intimately connected to the town's natural philosophical circles and to the university education. Here, it was research and teaching that shaped the chymical agenda much more than the productive needs of an industrial enterprise. As a consequence, we can see curiosity and scientific interest as the main factors fostering the chemical reactions pursued and the substances worked. Instead of testing minerals and assessing their yield, the Ashmolean chymists experimented with technological innovation of their time in fields such as glass production and zinc metallurgy. Instead of moving within an established tradition like that of fire assay, they worked on very novel processes which still required adjusting and testing. This is the case of lead crystal, destined to great success but still in its infancy between the end of the 17th and the beginning of the 18th century. The Ashmolean practitioners enter this arena, trying recipes while also investigating broader aspects of scientific interest such as the use of reagents to obtain colour effects in the glass body. Given the link of the Ashmolean laboratory to the teaching of chemistry at Oxford's university, it is likely that alongside fulfilling entrepreneurial and philosophical purposes, the experimentation with lead crystal may also have served as a case in point to discuss issues of interest to students, such as matter and its transformations or the production of colour in glass (**Roos 2017**). These processes were of philosophical interest to the circles of natural philosophers who gravitated around the newly established Ashmolean Museum and sparked discussion during the meeting of philosophical societies. Nonetheless, the chymists also knew that perfecting glass recipes and making a more stable, more beautiful product would have opened up new chances of profit and would have inserted the laboratory in the glass market. The same of course applies to the distillation of zinc, which was not to become an established technology for some time. Robert Plot was very conscious of the economic relevance of the work of his laboratory and he was always on the lookout for potential sponsors and buyers.

Thus, chymistry at the two sites presented here differs in what are the ultimate motivations and aims behind it. The Ashmolean laboratory is more varied and international, the tools come from different suppliers and many of the ingredients and reagents were imported from abroad as well. The second-floor repository was stocked with natural specimens from various places, materials that could equally be studied and employed in chymical preparations. Jamestown is more productive and somewhat less varied in the range of operations, even though chymists necessarily needed to try and test the substances they worked with. Moreover, despite the prevalence of fire assay remains, we know that some glass was also made in the same years and that apothecaries were present too. Further differences involve the profile of the chymists who left the materials analysed. While mostly artisanal experts operated in Jamestown, who were trained in metallurgy, glass making and pharmacology, the laboratory operation of the Ashmolean were led by people whose technical expertise was probably broader and who worked simultaneously on different recipes. Students populated this space as well, observing the teachers but also presumably trying things themselves. The Ashmolean was a more organised and settled space, it had specific rooms for specific procedures and for storage purposes, it was large and made for accommodating several people both working and watching. We do not know much about where chymistry happened in Jamestown, but we know that assay operations were carried out both in some sort of metallurgical workshop and sometimes outdoors too, during upriver expeditions and in order to quickly get a sense of the minerals collected. We can imagine a less organised space, possibly used for multiple purposes as settlers had to make do with much more limited resources. And yet, in spite of all these important differences, once we turn to the material landscape of the two sites, multiple connections and overlaps come to the fore, and what one perceives is a fundamental proximity much more than a distance. The tools, the array of substances found within them and the chemical reactions these underwent speak of the same underlying chymical knowledge, articulated according to the specific needs. To follow material connections like those that emerge here is to create a network of microhistories on early modern chymistry and chymical practice.

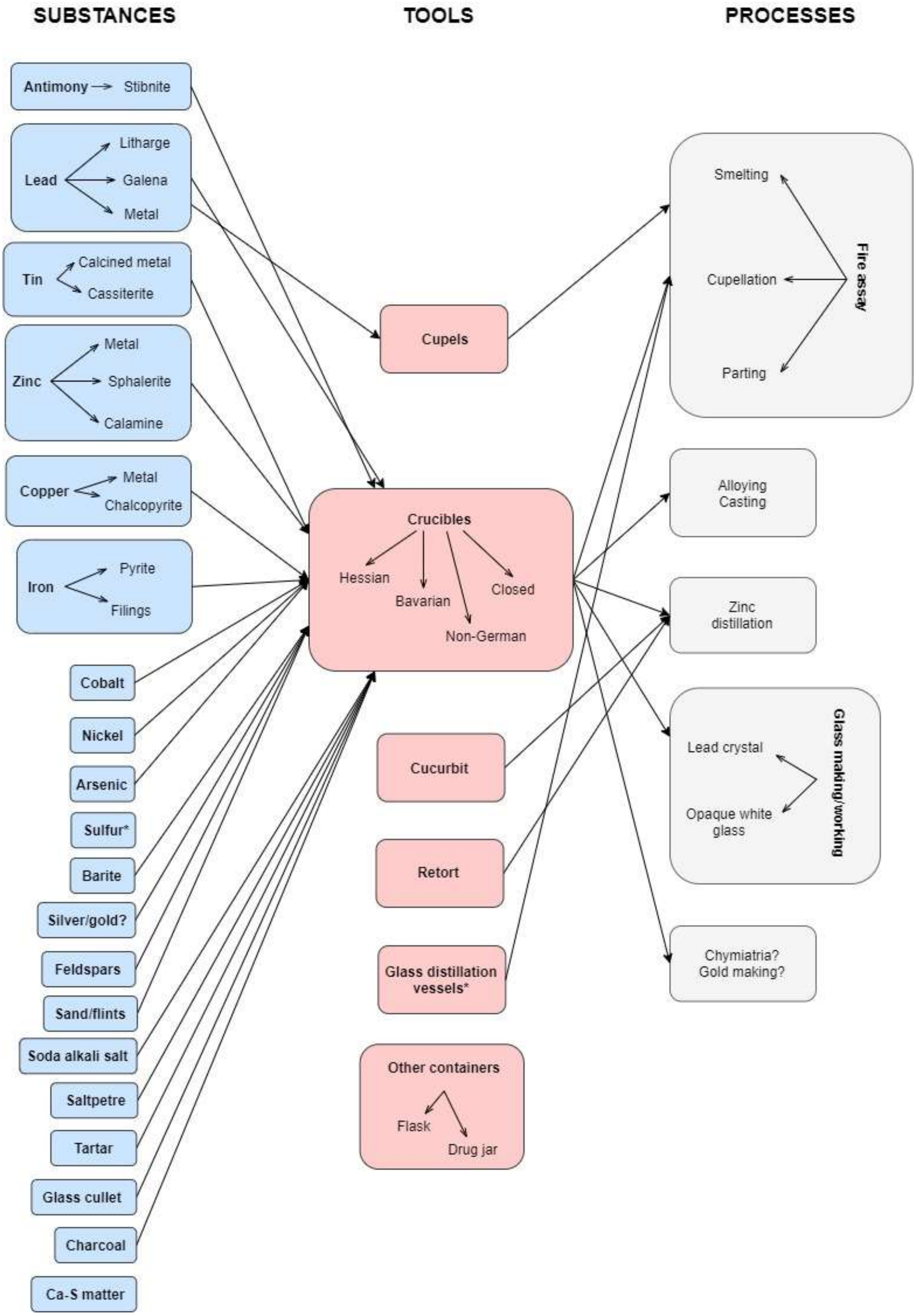


Figure 6.1. Material map showing the substances and tools identified in Jamestown and the Ashmolean laboratory, as well as the processes these underwent. Asterisk indicates materials that have been found but not analysed in this thesis.

6.1.1 CRUCIBLES, CUPELS AND THE ROLE OF HIGH-QUALITY TOOLS

The material network of **figure 6.1** shows how prominent the role of ceramic crucibles was in the two assemblages analysed here as nearly every operation carried out happens in these high-quality pieces of apparatus. The Ashmolean laboratory was reportedly among the best equipped in Europe with state-of-the-art furnaces as well as glass, ceramic and metallic vessels for all kinds of chemical reactions (**Bennet et al 2000: 21**). A visitor reported that the laboratory was “Perchance one of the most beautiful and useful in the world, furnished with all sorts of furnaces and all other necessary materials in order to use and practice” (**Chamberlayne 1684: 327**). The Ashmolean seems to have sourced its apparatus from various suppliers. While some of the crucibles were imported from Hesse, in Germany, and show the sandy texture characteristic of these objects (**Martinón-Torres and Rehren 2009**), other vessels may have been bought from a local English manufacturer instead. At any rate, they are different enough to suggest a non-German origin.

The region of Hesse, in Germany, was in this period the epicentre of high-quality crucibles manufacture, with a handful of production centres in the district of Großalmerode supplying a large number of laboratories and workshops worldwide (**fig. 6.2**). Exceptionally resistant, we have seen how these crucibles were ideal for the highly reactive substances and the extreme temperatures of many chymical activities. Hessian crucibles are more predominant in Jamestown, where they constitute the vast majority of the ceramic apparatus. They have also been found at other sites of European colonies, from Quebec to Colombia, where analyses have shown that settlers by and large employed them in fire assay and other metallurgical operations (**Martinón-Torres 2010; Martinón-Torres et al 2018; Veronesi et al 2019**). In Europe, Hessian crucibles have been found at numerous sites in England, Norway (**Saunders 2001; Ulseth et al 2015**), Germany (**Martinón-Torres 2005**), Portugal (**Dordio et al 1997; Hsu and Martinón-Torres**) and Spain (**Amores Carredano and Lloret Marín 1995**). The most comprehensive assemblage of German-made triangular crucibles comes from the 16th-century chymical laboratory of Oberstockstall in lower Austria (**Sokup et al**

1997; von Osten 1998). As briefly mentioned earlier in this thesis (cfr. section 2.3), the various chymistry-related materials discovered there have gone through several rounds of analytical investigation, and our knowledge of the site is now rather comprehensive.[14] The crucibles span from tiny to huge examples and were employed in metallurgical activities of mineral testing (**Mongiatti et al 2009a**). The vessels are of the black type from the small village of Obernzell in Bavaria, equally high-quality and also enormously popular in post-Medieval Europe (**fig. 6.2**) (**Martinón-Torres and Rehren 2009**). Outside of Germany, similar crucibles have been discovered at chymical contexts in England (**Cotter 1992; Martinón-Torres 2005; 2012a; Veronesi et al 2021**), Switzerland, Austria (**Friedl 2006; Mongiatti 2009**), Czech Republic (**Bauer 1983**), Italy (**Martinón-Torres and Verrocchio 2008**) and Croatia (**Gardner et al 2018**), as well as in the colonial world at the first ever European settlement of la Isabela in the Dominican Republic (**Deagan and Cruxent 2002; Thibodeau et al 2007**) and in Brazil (**Lima and da Silva 2003**). For the most part, Hessian and Bavarian crucibles were employed in metallurgical contexts such as goldsmith's workshops and mints where small-scale operations of quality control and refining required great precision.

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[14] An in-depth introduction to the site falls out of the scope of the present thesis. Further details are discussed in section 6.1.2, but the reader who would like to know more about the history of Oberstockstall's laboratory, including information on the owners and patrons of chymical activities, as well as details of the high-temperature operations carried out can turn to Marcos Martinón-Torres' and Aude Mongiatti's PhD theses.

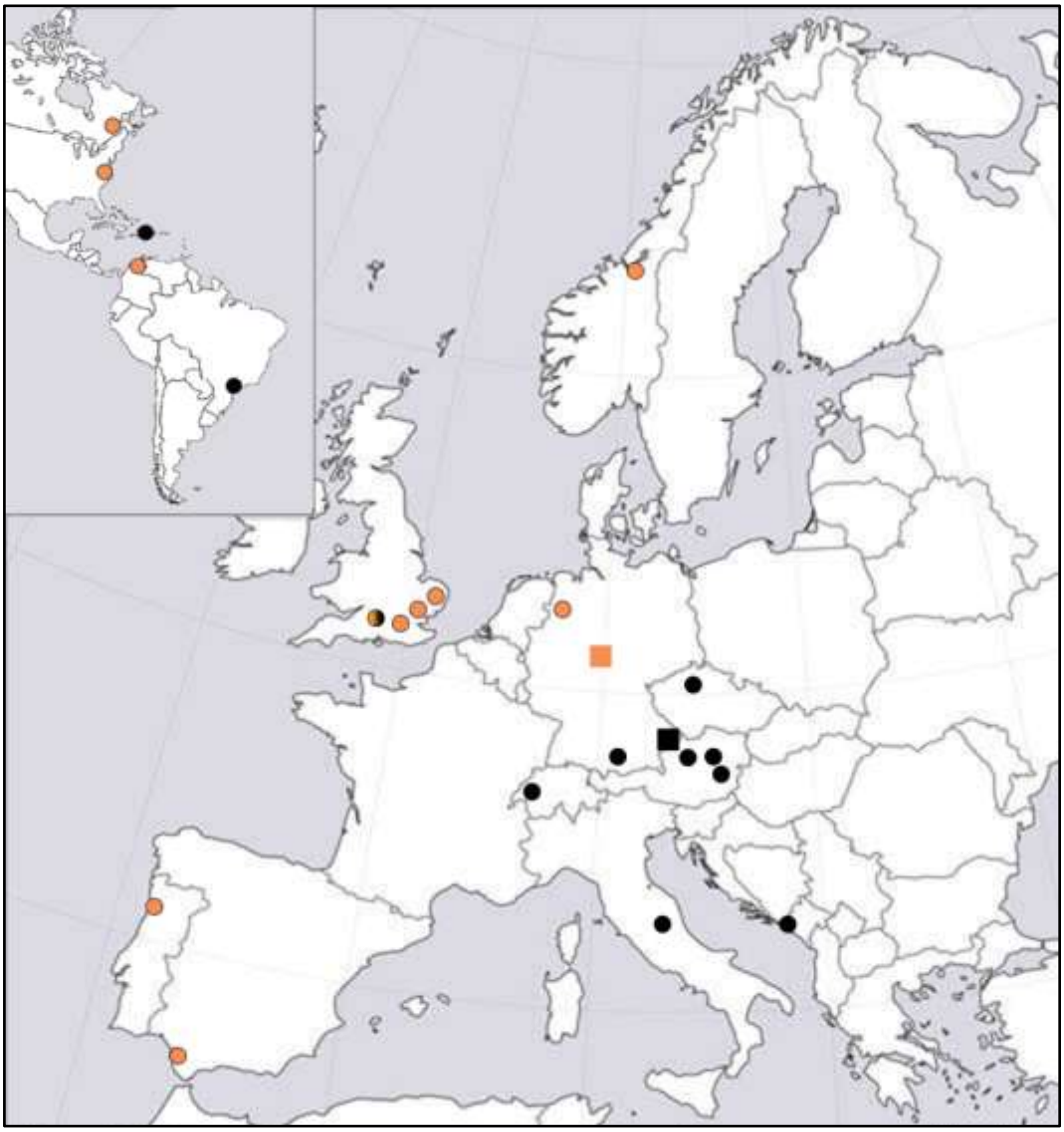

Figure 6.2. Sites in the post-Medieval world where German crucibles have been found in the archaeological record. Orange circles refer to Hessian crucibles while the black circles graphitic vessels from Bavaria. So far, both types only appear at the Ashmolean laboratory. The two squares represent the main production centres of Großalmerode (Hesse) and Obernzell (Bavaria). For a detailed list of most sites in the map see Martinón-Torres and Rehren 2009: 52-53).

And triangular crucibles feature heavily also in Renaissance genre depictions of alchemical laboratories as well as in contemporary literature related to metallurgy (**Principe and De Witt 2002**) (**fig. 6.3**). As it was mentioned when discussing sources of information on chymical practice (cfr. section 2.3), these images have their own biases and cannot be taken at face value as representative of the reality of laboratories. However, the fact that these crucibles are so ubiquitous in artistic depictions of the time is at least suggestive that they must have been a common component in chymical contexts.

Figure 6.3. An alchemist, Hans Weiditz, c.1520

Evidence therefore suggests that in this period German crucibles largely dominated the market of specialised laboratory-grade ceramic instruments. Their technical quality was so well-known and the volume of production enough that they could be exported pretty much everywhere. Chymists knew that these instruments would perform reliably well during high-temperature operations and had a lower chance of failure due to melting, cracking or excessive contamination from the charge. And as Robert Plot writes, German crucibles were considered something of an early modern technological mystery whose reproduction was highly desirable (**Plot 1677**). Nonetheless, ordinary potters also made crucibles and so did assayers themselves as we are informed by George Agricola, whose *De Re Metallica* contains how-to instructions for this purpose (**Hoover and Hoover 1950: 230-231**). At a time when systemic wars and shifting diplomatic relations made importing expensive and unreliable, local alternatives were often a cheaper, more viable option embraced by many workshops and laboratories. In both case studies examined in this thesis we have examples of non-German crucibles of high technical quality. The presence of these objects in archaeological assemblages bears witness to the tension between the need for the best equipment, which often required importing from abroad, and the strong political and economic desire of being more self-sufficient.

At the Ashmolean, around half of the ceramic apparatus does not appear to be a German import. Circumstantial evidence points to the experimental potter John Dwight as the possible suppliers of these vessels, as indicated by the mentions of his success in replicating Hessian crucibles (cfr section 5.5.1; **Plot 1677: 250**). Even though further analyses are needed to ascertain where the vessels were made, the Ashmolean materials indicate that high-quality apparatus could be obtained from different suppliers, and that chymists often chose to source their crucibles locally. At the Royal Mint in London, for instance, we known that a potter was employed on-site and made crucibles to be used in high-precision operations of assay and refining (**White and Kearns 2010**). Local productions are by and large the most popular solution for glassmakers in post-Medieval England, and assemblages recovered from glasshouses dating between the 16th and the 18th century show that the vessels varied greatly in shape and size and did not include triangular examples (**fig. 6.4**). Composition-wise, these crucibles tend to be much more variable and, although still technically good for chymical use, somewhat less refractory than their German counterparts.

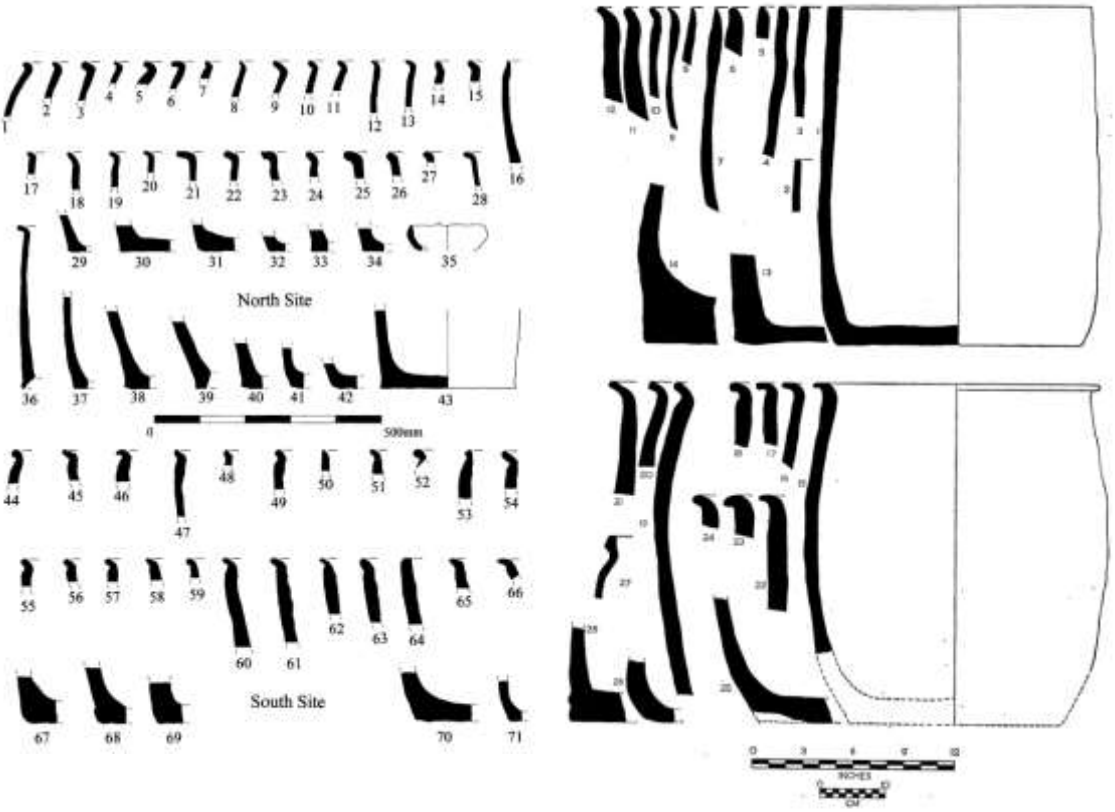

Figure 6.4. Examples of non-German crucibles recovered at the site of English glasshouses of Wolseley (left, after Welch 1997) and Bagot's park in Staffordshire (right, after Crossley 1967). Note the wide variety of types.

A notable example of alternative production is the French workshop of Louviers, which is believed to have provided the first settlers of Quebec with the crucibles to assay Canadian ores in the early colonisation attempts of the 1540s (**Monette 2013**). The assemblage of crucibles discovered at the colonial site of Cartier-Roberval (1541-1543) strongly resemble the Louviers type, and were probably brought along during the voyage from France (**Martinón-Torres 2010**) (**fig. 6.5**).

Figure 6.5. Crucibles from the French colonial site of Cartier-Roberval (left) and from the pottery workshop in Louviers (right), from which the former came (after Monette 2013)

In some cases, metalworking crucibles were made directly at the colonial sites using local raw materials, as revealed at the Spanish settlement of Santa Cruz de Mompox (Colombia). The analysis of a number of crucibles used in goldsmithing activities show that while the vast majority are most likely German imports, one vessel was definitely made locally using rocks of the area as temper in the clay paste (**Martinón-Torres et al 2018**).

Further specialised tools used in fire assay include scorifiers and cupels, which feature much less prominently than crucibles in the two assemblages analysed in this thesis. Scorifiers were ceramic open dishes whose uses for refining were varied but most notably served to pre-concentrate the noble metal in the lead bullion before cupellation (**Martinón-Torres and Rehren 2005b**). Only a few of such objects were found in Jamestown, none of which has been analysed yet, while no examples are known from the Ashmolean laboratory where fire assay does not seem to have been practiced. Again, the remains from Oberstockstall offer what is the largest surviving assemblage of scorifiers, with dishes of different sizes (**fig. 6.6**). Mostly, the vessels have been used and display a residue layer on the inner surface, not too dissimilar to the crucible residues discussed in the previous chapters only this time resulting from oxidising rather than reducing reactions. Analyses on some samples show lead oxide as the main enrichment present, with variable traces from the mineral tested or of the metals concentrated in the lead bullion (**Martinón-Torres 2005**).

Figure 6.6. Scorifiers in the archaeological record. Left: two examples from Jamestown (photo by Marcos Martinón-Torres); right: the assemblage from Oberstockstall, showing different sizes and shapes as well as residues and signs of use in high-temperature operations (after Mongiatti 2009).

Cupels were key instruments in early modern chymistry, employed on a daily basis at sites such as mints and assay workshops (cfr section 3.1; **Hsu and Martinón-Torres 2019; Ulseth et al 2015; White and Kearns 2010**) (**fig. 6.7**). Unlike what seen for crucibles, there are no renown manufacturers in this period, users generally making their own cupels by pressing the compacted ash mix into moulds (**Hoover and Hoover 1950: 228-230**). As a consequence, recipes are less standardised and the degree of technical quality oscillates accordingly (**Martinón-Torres et al 2009**). A good cupel had to be porous and inert, to allow the best possible separation between the noble metal and the lead in which it had been concentrated after crucible reduction. Impurities were likely to promote unwanted reactions with the lead oxide, ending in potential losses and a biased quantification. The cupel from Jamestown was made with pure bone ashes and little else, a recipe that granted the least amount of impurities in the vessel and that was therefore thought of as the most appropriate (cfr section 4.5.2).

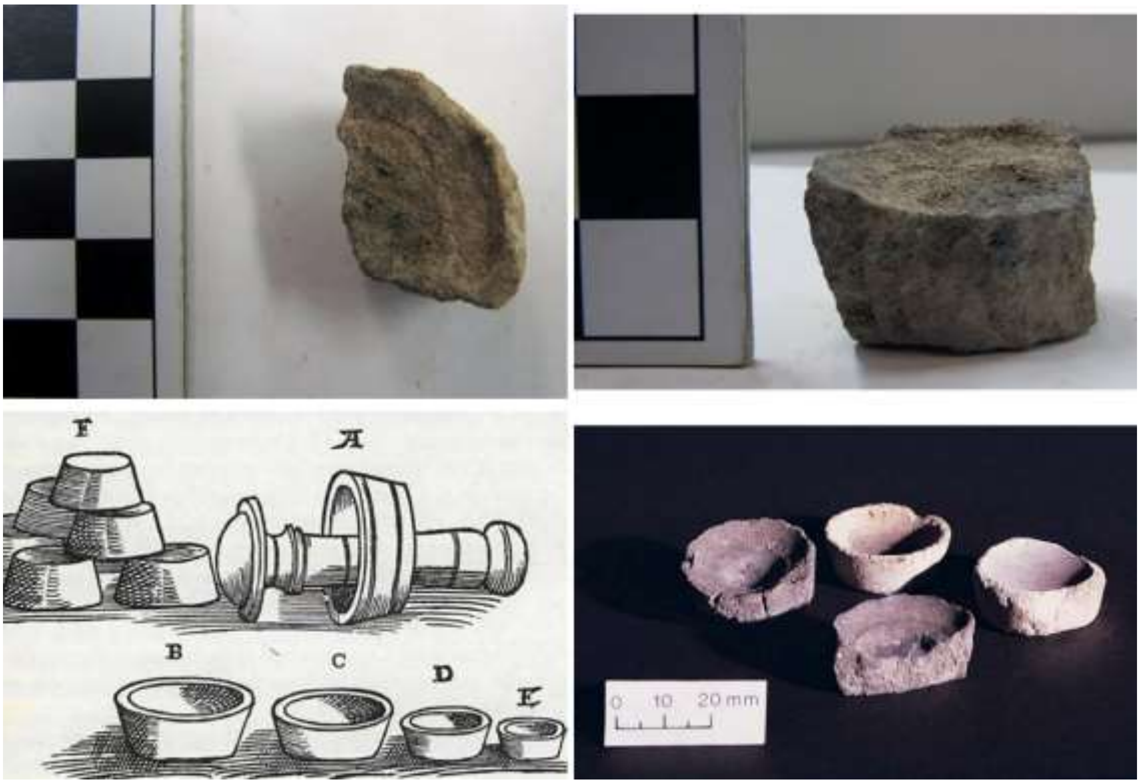

Figure 6.7. Cupel from Jamestown (top, photo by Marcos Martinón-Torres) and cupels from the Tower of London mint (bottom-right, after White 2010). Note their grey-ish colour due to lead oxide absorption. Bottom-left: cupel making in Ercker's treatise (after Sisco and Smith 1951: 33)

Cupellation on a larger scale could be performed by lining hearths with the bone ash mixture, the overall procedure unaltered in its technical aspects. At La Isabela, the Spanish colonists carried out this type of cupellation, but they lined the hearths with a sand-rich mixture that gave a very poor outcome. The sand reacted heavily with the lead oxide to form a highly viscous lead-silicate glass and some of the lead did not oxidise but got trapped in it, together with the silver it contained (**Thibodeau et al 2007**). Agricola warns about sand and prescribes to get rid of it as much as possible from the ashes that will be used to make cupels (**Hoover and Hoover 1950: 229**). At other early modern European sites bone ash is generally the main ingredient, as in the mints of London and Trondheim, for instance, where the cupels analysed are very pure (**White 2010; Ulseth et al 2015**), and in the mint of Porto where wood ashes were added as well (**Hsu and Martinón-Torres 2019**). Cupellation remains feature abundantly also in already mentioned the 16th-century chymical laboratory of Oberstockstall (**Sokup et al 1997; von Osten 1998; Rehren 1998; Martinón-Torres and Rehren 2005b; Mongiatti**

2009). Here, too, bone ashes were mixed with purified wood ashes and analyses show that the cupels performed exceptionally well and silver losses were very low and very consistent (**Martinón-Torres et al 2009**).Whether working on more standardised processes or innovating, the availability of instruments and tools of high technical quality was key to the successful outcome of the operations undergone. In procedures such as glass making/working or the casting of molten metal the crucible needed to withstand hot and highly reactive ingredients without melting or interacting too much with the charge. However, it was in small-scale processes of high precision where the quality of the apparatus became critical. Specialised tools such as the small crucibles and the cupels discovered at many sites of early modern chymistry made it possible to accurately estimate the yield of mines, the quantity of precious metals in an ore sample or in an alloy, and even assess the quality of alchemical gold. Any failure on the tool's part, any excessive interference with the reactions going on, would lead to errors of quantification and to a less reliable result. Ultimately, it is in fire assay laboratories and during transmutation attempts that quantitative analytical chemistry begins, thanks to the increasingly reliable, increasingly advanced apparatus produced across Europe.

6.1.2 GLASS: FROM DISTILLATION TO TRANSMUTATION

Glass apparatus is perhaps the most iconic of laboratory tools and is inextricably linked to the chymical processes of distillation. However, there seems to be a discrepancy between how widespread these objects were at the time and how little survives in the archaeological record. Linking small glass fragments to distillation vessels can be very hard, particularly when the archaeological context cannot be readily related to chymical activities, so that often such fragments are classified more broadly as unspecified glass vessel fragments. Even more importantly, glass corrodes much more quickly than ceramic, with an evident impact on what we find, identify and study today. Finally, while used and broken crucibles were disposable objects, broken glass got recycled rather than discarded. Crushed cullet from broken windows was brought to Jamestown where it could be re-melted to form objects or even employed as an alkali-rich reagent in metallurgical operations (cfr section 4.5.5). Similar bits of broken glass were used in ceramic production as well. The Hessian-like crucibles of John Dwight, mentioned above, were found to contain glass as temper, as did the so-called coadstone, a type of decorative building material resembling limestone popular in the later 18th century (**Freestone 1991**). This varied role of glass explains why only a fraction of distillation glass waste survives in contexts like those from which the various assemblages discussed in this thesis come. This is why, for instance, so little glass has ended up with the many broken crucibles and bits of bones from the Ashmolean assemblage. According to the excavators, of the forty-seven glass fragments recovered only one may come from a piece of laboratory equipment, while the rest is either from windows or wine bottles (**Hull 2003: 20**). And since glass vessels are mentioned in descriptions of the laboratory, we must assume that such scarcity is due to the issues outlined above rather than reflecting their limited use. Very little evidence of distillation vessels was found in Jamestown, such as the fragment of a still head and what looks like a cucurbit, the bottom part of the apparatus (**fig. 6.8**). However, one would expect more abundant finds of this type, considering the relevance of assay activities which normally required the distillation of mineral acids for further refining and parting gold from silver.

Figure 6.8 Glass distillation equipment from Jamestown, including a fragment of still head on top of a ceramic cucurbit (left) and potential cucurbit (right) (photos by Marcos Martinón-Torres).

Therefore, in spite of glass' importance to our understanding of chymical practice, remarkably little is known about the chemical composition of specialised glass and its patterns of production, distribution and use. It would be interesting to know whether specific producers were considered superior, and whether glass vessels travelled as widely as German crucibles across post-medieval and early modern laboratories. Like with crucibles and cupels, quality was essential in distillation apparatus too, and not every type of glass was advisable to make laboratory equipment. Due to glass' inertness and the generally lower temperatures reached during distillation, interaction between the vessel and its content was not as much of an issue to chymists as it was in other processes. Instead, the impervious nature and transparency of glass made the difference. Even when heated up, the vessel should not break too easily, while the clearer and thinner the glass, the easier it was for the chymist to keep an eye on the distillation's progress. These preoccupations show through the pages of early modern documents, which indicate that even the glass market had its renown producers. For instance, Daniel Sennert (1572-1637), a prominent 17th century physician, complained that the war was preventing him from obtaining laboratory glassware from his favourite suppliers, and he thus had to work with a lower-quality material that constantly shattered (**Klein 2016: 299**). Similarly, Hieronymus Brunschwig (1450-1512) and Johann Rudolf Glauber (1604-1670) both gave advice on the subject in their treatises, the former explicitly mentioning Venetian or Bohemian products (**Anderson 2000: 14**) while the latter prescribing a "strong and firme glass" that can better retain the distillation vapours (**Glauber 1651: 2**). Venetian glass and its imitations, known as *façon de Venise*, was certainly very expensive, but its strength made it preferable for vessels that had to go through repeated cycles of heating. Even more importantly, the thinness and the higher transparency of this type allowed practitioners to monitor more effectively what was going on inside. Indeed, being able to recognise a small colour change or the formation of particles in the liquid being distilled could make the difference between success and failure (**Principe 1987: 21**). Vannoccio Biringuccio writes that cucurbits should be of a glass "as clear and uniform as possible" with no "bubbles and indentations" (**Smith and Gnudi 2015: 183**). In order to begin exploring along these lines of enquiry, a number of fragments from the apparatus in Oberstockstall have been recently analysed (**Veronesi and Martinón-Torres 2018**) (**fig. 6.9**). Based on their shape the fragments can be ascribed to both specialised vessels for distillation and more general-purpose domestic forms. The former includes a distillation column, a still head, and several receivers, vials and other vessels of transparent glass, while the latter are mostly bottles and flat dishes.

Figure 6.9. Glass apparatus from Oberstockstall. Left: Some of the fragments analysed, showing different colours according to vessel typology; right: an alembic (height 41.8cm) and a bottle with ribbed decoration (after Veronesi and Martinón-Torres 2018).

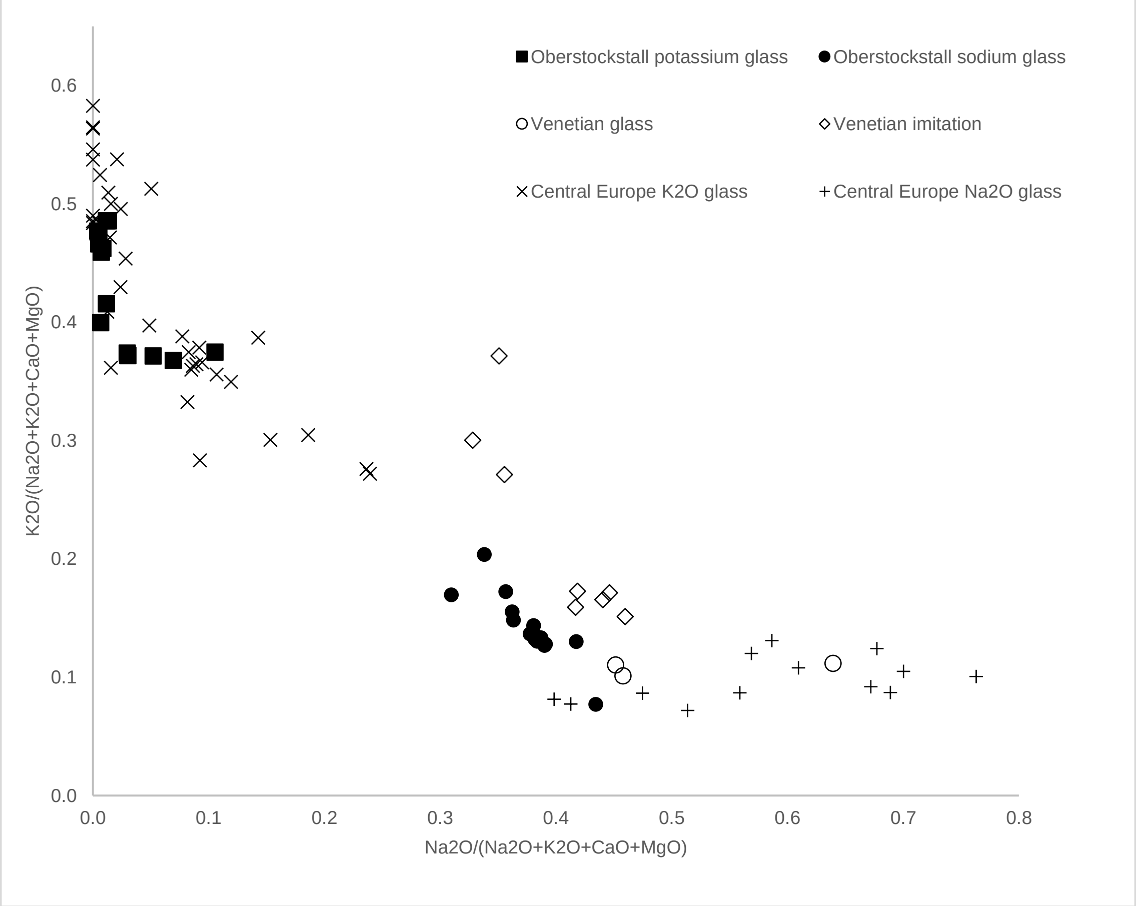

Figure 6.10. Scatterplot comparing the concentrations of alkalis between Oberstockstall and contemporary European glasses. Data for Venetian and Venetian imitation glass was taken from Janssens et al 2013 while data for Central European glass from Sedlackova et al 2014.

The initial questions revolved around which type of glass had been used for the fragments analysed and whether we could observe any pattern in the assemblage, composition-wise. Were specialised glasses specifically chosen for the purpose? Was it local or imported? The results (**Appendix D**) revealed that the specialised forms for distillation were all decolourised through the addition of manganese and all belong to the sodium-rich Venetian or Venetian imitation type. On the other hand, bottles, flat discs and other non-specialised forms were made of potassium-rich forest glass, invariably darker in colour and certainly easier to source locally (**fig. 6.10**). Thus, important distinctions were made when it came to buying the glass for the laboratory needs, with a more expensive glass type being specifically chosen for the distillation vessels. The special status of Oberstockstall's chymical glass is indicated by the fact that in central and north-western Europe sodium-rich glasses normally only occur as fine tableware and expensive ornamental items (**Sedlackova et al 2014**). The case of Oberstockstall reinforces the critical role of quality instruments in early modern chymical practice, introduced with the ceramic crucibles in the previous section.

The materials analysed tell us about glass from another perspective as well, namely its role as an object of both economic and philosophical interest (**Beretta 2004; 2009; von Kerssenbrock-Krosigk 2008**). Jamestown's 1608 trial of glass was supposed to be the beginning of a profitable enterprise for England, whose production lagged behind that of other places and forced the country to rely heavily on imports. In a similar way, lead crystal was intended as the English answer to the famous *cristallo* from Venice and central Europe. Again, what we see in glass production is the same push towards foreign technologies, the same attempt at making them English as seen with high-quality crucibles in the section above. Absorbing foreign artisanal knowledge involved a movement of people. The successful production of lead crystal had ultimately been possible thanks to glassmakers who emigrated from Italy and set up partnerships with entrepreneurs in England and Ireland (**MacLeod 1987**), German experts led the first glass making attempts in Jamestown and Italians run the later glasshouse of the 1620s (**Harrington 1972**). On the other hand, some of the experiments of the Ashmolean targeted new or peculiar aspects of glass production. In particular, processes involving the colouring and opacifying of glass remind us how this material was intimately linked to ideas of transformation and transmutation, as colour was the oldest and the most important theme in alchemical practice (**Newman 2004; Martelli 2014**). The product of humble ingredients, glass could take virtually any colour by the addition of a number of reagents, it changed into beautiful objects and imitated gems and semi-precious stones (**Beretta 2004; 2009; Dupré 2018; Bycroft and Dupré 2019; Hagendijk et al 2020**). Vannoccio Biringuccio defines it as “born from the speculations of good alchemistic savants” and “one of the fruits of the art of fire” (**Smith and Gnudi 2015: 126**). Chymical laboratories of European princely courts often included glassworks, while rulers sponsored and even joined the chymists in experimenting with glass (**Smith 1994; Beretta 2017**). Glass’ peculiar physico-chemical properties and changeable nature fascinated natural philosophers and offered the basis for theoretical considerations on the structure of matter. Jan Baptist van Helmont (1580-1644) was able to prove experimentally that the initial amount of sand used to make a piece of glass could be retrieved intact, by dissolving it in acids and re-precipitate sand out of the liquid (**Newman and Principe 2002: 77**). The Flemish chymist used his result to show that during glass making sand simply changes the shape and dimension of its corpuscles, and that a perfectly homogeneous-looking mixture was possible to separate into its initial components. He also introduced the concept of mass balance, whereby “nothing comes into being from nothing hence weight comes from another body weighting just as much” (**Newman and Principe 2002: 69**). Similar experiments where substances are reduced to their initial state were used by Robert Boyle in articulating the corpuscular theory of matter at the core of his mechanical philosophy (**Newman 1996; 2006; 2014**). Maybe more than the other materials of early modern chymistry, glass had multiple layers of meaning. As the material of quality instruments, it made better observation of reactions possible, and not only during distillation. Indeed, clearer and more resistant glass was also used for telescopic and microscopic lenses, which allowed discoveries of enormous impact (**Verità 2014**). As a material in its own right, glass provided chymists with both a valuable product of commerce and a source of knowledge on nature and matter.

6.1.3 FORMS AND ROLES OF MINERALS AND METALS IN CHYMICAL PRACTICE

Looking at the material map in **fig. 6.1**, one can see that a variety of metallic and mineral substances entered the crucibles analysed, and they did so in different capacities and for different purposes. When the same map is divided according to what features at each of the two sites, numerous material connections emerge with a wide array of substances appearing at both (**fig. 6.11**). Such connections bring to the fore the idea, fundamental to this thesis, of material connections and overlaps among spheres of early modern chymical practice, while also exposing important differences as to the role that the same substances may have had in different contexts.

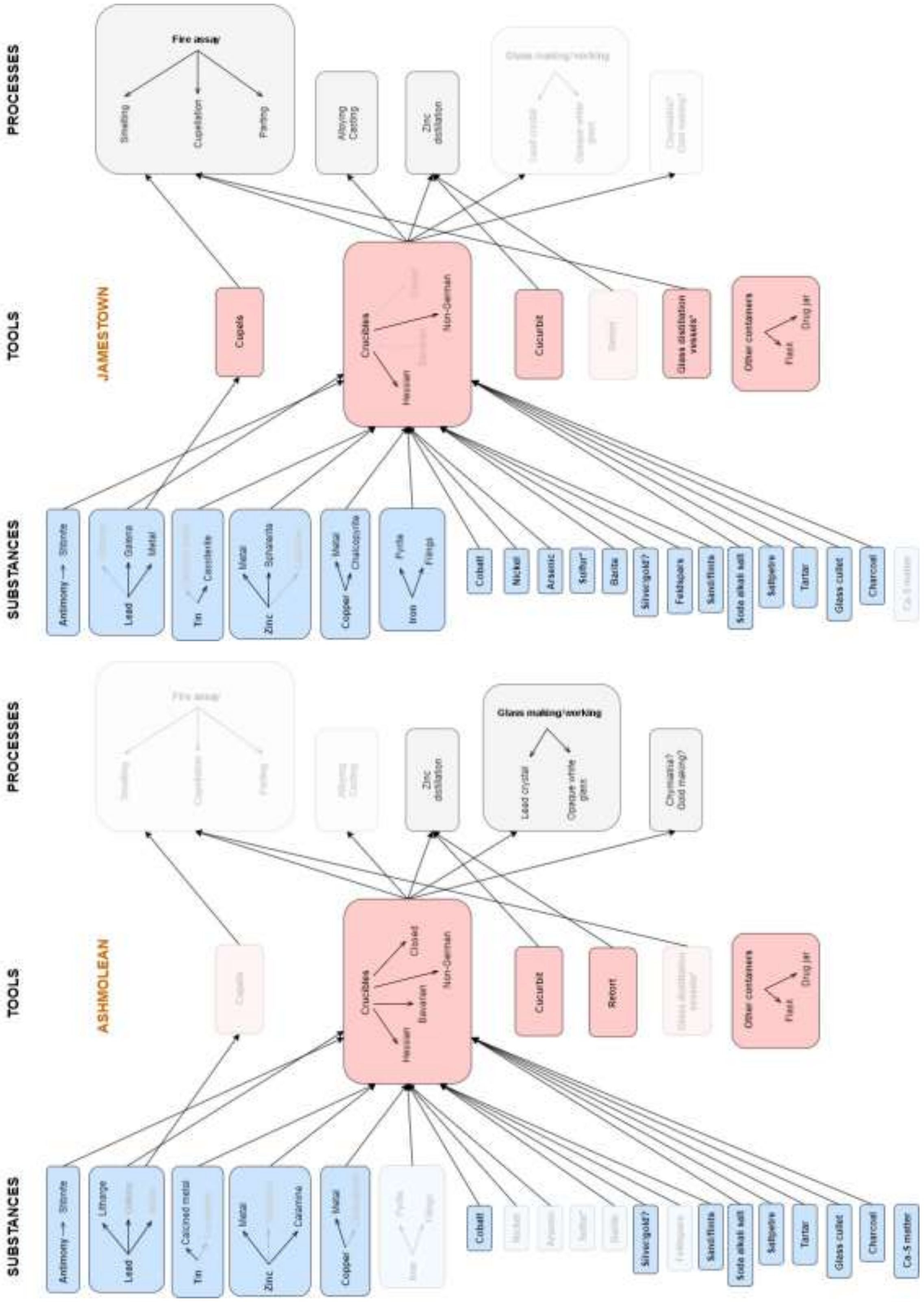

Figure 6.11. The material network from figure 1, divided by site. Faded boxes represent substances, tools and processes not mirrored by the material culture.

Lead, for instance, features prominently among the materials. In the form of the oxide litharge (PbO), it is a key component of most glass-related samples from the Ashmolean laboratory. Mixed with sand/flint and saltpetre it gave lead crystal chemical stability, while calcined with tin it contributed as colouring and opacifying agent. The lead sulfide galena (PbS), often in association with a host of other silver-bearing minerals, is among the ores assayed by the chymists of Jamestown. During the same assay operations metallic lead (Pb) was presumably employed too, added to the crucibles as a noble metal collector. The resulting bullion would then undergo cupellation and end up absorbed by the porous cupel as lead oxide. Small lumps of lead are often indicative of assay activity and we know of such finds in other colonial contexts where settlers looked for precious metals. Moreover, isotopic analyses on lead samples from La Isabela and from French and English sites in Quebec show that these were brought along from Europe specifically to be used as reagents (**Beaudoin and Auger 2004; Martinón-Torres 2010; Monette 2010**). Whether metallic, as a powdered oxide or as a sulfide rock, the element lead appears in different forms in early modern chymistry and laboratory activities. Most importantly, it assumes different roles across different operations but also sometimes within the same one. It can be concurrently an element in the rock analysed and the reagent that is instrumental to the process' success.

Other substances have multiple values and physico-chemical states. Tin has been mentioned in relation to glass opacification at the Ashmolean laboratory. Following common recipes of the period, the metal was calcined (with or without lead) and the powder added to the crucible with the glass melt. The map indicates that tin was an important mineral resource in the form of cassiterite (SnO_2), which Jamestown chymists crushed and mixed with scrap copper to test whether any good bronze could be made. Indeed, copper in Jamestown tells a similar story to the lead from the colonial site mentioned above. As waste from other processes, these materials had travelled from Europe together with the settlers, so that they may be of use to the colony's needs. The very same story is shared by the bagful of broken glass discussed in the previous section. Copper also features as the mineral chalcopyrite ($CuFeS_2$), one of the several

Virginian minerals that undergo fire assay in order to assess their silver content. But the most puzzling occurrence of this material is perhaps in the residue of the graphitic crucible from the Ashmolean, where it probably entered in metallic form together with silver and sulfur in what may be a transmutation experiment or a medical preparation.

Also related to copper metallurgy is zinc, which like tin plays different roles and was found in different forms. In Jamestown, the zinc sulfide sphalerite ($(Zn,Fe)S$) underwent a similar treatment as cassiterite, it was crushed and mixed with scrap copper to try and make brass by cementation. A similar zinc ore was tested by the French colonists of the Cartier-Roberval site, further testimony to the connections running through colonial chymical practices. Evidence of metallic zinc is found in some Ashmolean crucibles, a very rare occurrence in early modern Europe as this was still a poorly known material around which was much confusion (cfr section 5.5.3). The data indicates that some zinc, either in metallic or mineral form, was first heated in closed crucibles to provoke its evaporation and the subsequent condensation of metallic droplets on the vessel's walls. The experiment must have been aimed at studying the nature of this material, and perhaps the crucibles had been used during a demonstration of zinc's peculiar thermodynamic behaviour to chemistry students. Whether from a more productive or speculative starting point, the zinc-centred reactions investigated here tell the same underlying story of increasing knowledge around an early modern puzzle. Was zinc a metal or a semi-metal/semi-mineral? Could it be extracted from calamine ($ZnCO_3$)? And how, given its tendency to evaporate so quickly? Similar questions were extended to brass too since brass was understood more in terms of a transmutation of copper than of an alloy. "Just as steel is iron converted by art into almost another kind of metal" writes Vannoccio Biringuccio in *De La Pirotechnia*, "so also brass is copper given a yellow colour by art" (**Smith and Gnudi 2006: 70**). Biringuccio is the heir of a long tradition that had tried to come to terms with this material and that had considered it a species of copper. The Greeks called it *ὄρείχαλκος* (oreichalkos), literally copper of the mountain, while the Romans transformed the word in *aurichalcum* (gold-copper) placing the accent on brass' colour (**Craddock 1998**). However, knowledge shifts rapidly in the course of the

16th and 17th century, experimental work increasingly suggesting that something else besides colour change takes place during brass cementation, that indeed there is an increase in weight, a physical addition to copper (**Martinón-Torres and Rehren 2008;** see also **Newman and Principe 2002: 35-91**). In this respect, the materials analysed here bear witness to the unfolding of zinc's story in early modern chymistry. In the early 17th century, zinc is still mostly seen as the agent of colour change than transmutes copper into brass, and indeed the chymists are after zinc minerals for brass production. By the time of the Ashmolean experiments, zinc is more commonly found in metallic form and the beginning of its distillation in Western Europe is only a few years away (**Etheridge and Dungworth 2012**). Much in the same way, terms such as *zinck* or *zink* had become commonplace in the works of chymists such as Glauber and Boyle who appear to use them to indicate zinc in its metallic form (**Packe 1689: 319-320; Boyle work diary 7**). And yet, a 1701 paper in the Philosophical Transactions of the Royal Society discussing cementation is titled "The method, manner and order of the transmutation of copper into brass" (**Povey 1701**). Accepting zinc as a metal in its own right would have had major consequences on the understanding of the cosmos as governed by tight connections and interdependencies between its components. If each of the seven metals had a celestial counterpart in one of the seven planets where would zinc sit in the scheme? A new metal zinc entailed a necessary review of knowledge that transcended the field of metallurgy alone. It was part of a wider phenomenon of changing worldviews that characterises early modern European culture at large and includes, among other things, making room for the new plants, animals, minerals and peoples that Europeans had to face as a result of the discovery voyages.

Other mineral substances with multiple meaning are antimony and cobalt, which appear in the Ashmolean assemblage as reagents in glass working experiments. In the form of stibnite (Sb_2S_3), antimony was the main ingredient in an alternative recipe for opaque white glass, while a cobalt ore was presumably used for colouring lead crystal blue. The cobalt-based pigment, known as *zaffera* or *safre* (CoO), came from German mines as a mixed ore of cobalt, arsenic, nickel and bismuth (**Gratuze et al 1992; 1996**).

Notably, this is also an association of elements characterising one of the ores that Jamestown chymists tested for silver, and that equally contain nickel and arsenic. More connections with central European mining and metallurgy are the residues of a complex sulfidic ore known as fahlore found in the crucibles of Oberstockstall (**Mongiatti et al 2009a**). This fahlore is remarkably similar to the polymetallic sulfides tested in Jamestown, which suggests that the assayers must have been somewhat familiar with the geology of Virginia, all the more if we consider that German specialists were among the settlers. Agricola writes that silver ore "is mixed with all kinds of earth and stone compounds" and he goes on listing "pyrites, *cadmia metallica fossilis*, galena, *stibium*, and others" (**Hoover and Hoover 1950: 114**). Pyrites, galena and stibium refer respectively to the sulfides of iron, lead and antimony. The term c*admia fossilis* was normally used for zinc carbonate calamine, while *cadmia metallica* should refer to cobalt (**Hoover and Hoover 1950: 113**). At any rate, it seems clear that what Agricola describes is a type of ore closely related to those worked in Oberstockstall and Jamestown.

Sulfur is indeed an element that recurs frequently at both sites analysed in this thesis and, more generally, in early modern chymical practice. We have seen that most minerals assayed in Jamestown occur in the form of sulfides, which usually required a first oxidising treatment to drive off the sulfur in smoke. An un-roasted ore would lead to the formation of matte, a sulfur-rich intermediary substance that could trap some of the noble metals and potentially bias their accurate quantification during fire assay. The lack of any traces of matte in the assemblage from Jamestown is suggestive of roasting, which is unsurprising since this was part of the codified practice of the period (**Hoover and Hoover 1950: 242; Sisco and Smith 1951:114**). But in Oberstockstall the chymists followed a different process whereby the sulfidic ore was not roasted before being melted in the crucible, and matte was indeed produced and refined in a later step (**Mongiatti et al 2009a**). This was probably due to the desire of trying variants of the commonplace procedure and experiment with different options. Sulfur appears as a lump of the pure element in the Ashmolean assemblage, although it is difficult to say how this would have been used. During the first round of analyses, Chris Salter linked the presence of sulfur and of sulfates to the production of fireworks, as this was a rather common chemical technology in the 18th century (**Hull 2003:24; Werrett 2010**). A few white bits of the same substance from the colonial site of Cartier-Roberval were equally puzzling and may tentatively be linked to the manufacture of gunpowder through the addition of saltpetre (**Martinón-Torres 2010**). Nonetheless, as one of the fundamental elements out of which metals and other substances were believed to be made, sulfur also had a powerful philosophical meaning in early modern chymistry (**Principe 2013; Newman 2014b**). Thus, its presence among the materials worked at the Ashmolean is consistent with the research and teaching programme of the laboratory, where it may have been used during attempts at metallic transmutation, or kept as an ingredient in medical preparations. Various forms of sulfur feature in the collection of nearly 700 specimens of *materia medica* that belonged to the chymist John Francis Vigani (1650-1712). They include elemental sulfur like the one from the Ashmolean as well as various sulfates known as vitriols and labelled as *vitriolum Romanum* (copper sulfate, $CuSO_4$), *vitriolum viride* (iron sulfate, $FeSO_4$) and *vitriolum album* (zinc sulfate, $ZnSO_4$). Originally from Verona, Vigani was interested in the practical more than philosophical side of its work as apothecary. He travelled throughout Europe, building an expertise in metallurgy and pharmacology, until he settled in England in 1683, where he started teaching chemistry, first privately and from 1703 as Cambridge's first professor of chemistry. This story is especially interesting as it runs parallel to the events happening in Oxford, with the opening of the Ashmolean Museum and Plot's chemistry teaching in the very same years. Vigani's collection, still mostly preserved in the original 18th-century cabinet, is vast and ranges from seeds, roots, fruits, animals and resins to shells, fossils, minerals, metals and pigments (**fig. 6.12**). What is worth noting here is that many of the substances collected and used by Vigani are the same as or are closely related to those identified in the crucibles of Jamestown and the Ashmolean, as well as in the other chymical assemblages discussed so far. Cobalt, arsenic, iron, copper and lead all appear as minerals and mineral-based pigments of various types, namely the already mentioned cobalt oxide *zaffera*, orpiment (As_2S_3), *verdigris* ($Cu_2CO_3(OH)_2$), white lead ($2PbCO_3{\cdot}Pb(OH)_2$) and red minium (Pb_3O_4). A sample of the lead oxide litharge comes *ex separatione argenti a plumbo*, the by-product of separating silver from lead through cupellation in large hearths. Antimony is present too, and takes the form of the mineral stibnite with samples coming from England and Hungary, the latter variety mentioned by Basil Valentine in his *Triumphal Chariot*. Finally, zinc is present as *lapis calaminaris*, which may refer to the zinc carbonate smithsonite ($ZnCO_3$) or the silicates hemimorphite ($Zn_4Si_2O_7(OH)_2{\cdot}H_2O$). As we have seen previously, these minerals collectively known as calamine found ample room in both philosophical discussion and chymical practice in early modern Europe. Vigani's cabinet contains a sample of metallic zinc too, labelled as *spelter*, again showing that by this time metallic zinc was becoming more common.

Figure 6.12. Four drawers from the cabinet of professor John Francis Vigani containing pigments (A), minerals and gums (B), animals (C) and flowers and fruit (D).

Vigani's collection also includes examples of crystalline substances such as *lapis specularis* (selenite), a form of the calcium sulfate gypsum ($CaSO_4 \cdot 2H_2O$), and two more labelled *talc* and *amianthus*. These are worth noting here because of the connection with the lump of calcium sulfate from the Ashmolean laboratory for which no satisfactory explanation was found (cfr section 5.5.4). In chymical practice, these stones were considered a kind of naturally-occurring glass and due to their tendency to flake they were sometimes worked in thin sheets and used as slides for microscopic observations (**Gunther 1923-1945, vol. 8: 313**). Among those who discuss the nature and properties of *lapis specularis* are chymists gravitating around Oxford and the Ashmolean Museum, such as Robert Plot (**1677: 81-85**; **Gunther 1923-1945, vol. 12: 350-351**) and Edward Lhwyd, who succeeded Plot as director of experiments in the 1690s (**Gunther 1923-1945, vol.14: 406**). Another one is Boyle who mentions talc in some recipes where he mixes it with saltpetre, tartar, minium, antimony and glass and melts the mixture (**Boyle 2004, work diary 23**).

6.2 BETWEEN WORKSHOP AND LABORATORY: CLASSIFICATION OF CHYMICAL SPACES

Prior to the 19th century, the term "laboratory" designated a place where some sort of manual work (*labor, laborare*) was carried out through chemical technologies (**Hannaway 1986; Klein 2008; Smith 2013; Saunders et al 2013**). Whether this work involved distillation, glass making, fire assay, metal making, chymiatria or the manufacture of pigments, the central idea was that of experiment and observation, the combination of practice and theory and the transformation by means of high-temperature manipulation of substances. As a place of material production where "the study of nature and technical innovation went hand in hand" (**Dupré 2014**), a laboratory was characterised by the presence of specialised equipment. Different types of furnaces for smelting, assay, distillation and other processes are hallmarks of the chymical laboratory, together with the various pieces of apparatus introduced and discussed so far. What follows is that in the early modern period there is no neat distinction between those two distinct categories that today we call laboratory and workshop, as both entailed the transformative action on materials that defines chymical activity. Such "linguistic peculiarity", as Ursula Klein calls it (**Klein 2008: 771**), has a lot in common with the terminological issue of alchemy vs chemistry discussed in section 2.1. Just as traditional historiography has artificially separated alchemy and chemistry on the basis of a presentist bias, the tendency was also to consider laboratory and workshop as spaces of different actions and mindsets, the distinction ultimately resting on the practical vs philosophical split. But separating practical from philosophical is a risky operation when discussing chymistry as it has often come to imply that those who engaged in crafts did not "think" or generate knowledge worth including in the historiography of science. The practical vs philosophical distinction underlies the taxonomy of early chemical laboratories proposed by the historian Robert Anderson, who distinguishes between the categories of investigation (modern-day research), production (industrial work), testing (quality control, i.e. assay) and teaching (from apprenticeship to universities) (**Anderson 2013**). If we were to adhere to Anderson's taxonomy, the case studies presented here would seem to fall into separate categories, with Jamestown being an example of production/testing context while the Old Ashmolean belonging to the investigation/teaching category. However, inferences grounded on practice suggest that things are not as clear-cut, and that there was much more interaction between categories. The previous sections have shown that materials cross boundaries and reveal connections that go beyond the type of activities or the profile of the practitioners. What they ultimately give back is the image of a rich spectrum with its gradients rather than of closed and readily distinguishable categories. Thus, a taxonomy that does not take the material element into account is apt to miss a crucial element of connection, alongside diversity.

But we also have seen how differences, even important ones, existed between Jamestown and the Ashmolean, and between sites of chymistry at large. As in every spectrum there are end points characterised by aspects such as the profile of the chymists, the spaces where chymistry happened and ultimately the aims behind the chymical work itself. Some laboratories solely focused on producing a commercial product, with little if any interest in the knowledge-making value of the manipulative, transformative work carried out. In discussing *aqua fortis* (nitric acid), Robert Boyle writes that it is “distilld by men of several professions, as chymists, refiners, goldsmiths etc. Yet they have had hitherto so little curiosity to enquire into the nature of it, or vary the wayes of making it” (**Hunter and Davis 1999, vol.6: 475**). At the other end of the spectrum are those individuals, like Boyle himself, whose experimental efforts were mostly moved by curiosity and personal interests instead of economic consideration. And yet this does not mean that Boyle did his chymical work in a vacuum. On the contrary, it was already shown how Boyle believed in an active role of the natural philosopher that would foster technological improvements (cfr section 5.6). To Boyle, the work of artisans is valuable because of its philosophical significance, because it is during production that one can see “nature in motion”, that state when nature is “put out of her course, by the strength or skill of man” which represents “the most instructive condition, wherein we can behold her” (**Hunter and Davis 1999: 468**).

In between the ends of the spectrum is a mosaic of chymical microhistories, cases where research often intermixed with commerce without clear demarcation. Thus, in acknowledging that some form of classification is necessary when studying such rich and varied spectrum, it is perhaps more useful to find ways to classify and compare that keep the focus on materials, evaluating overlaps and following connections. A new material-driven taxonomy of chymical contexts should revisit Anderson’s one by taking its start from the shifting aims fuelling early modern chymistry, but avoiding anachronistic separations of the workshop vs laboratory kind. Given the enormous importance of tools discussed above, a good starting point for classifying is to ask what type of apparatus the chymists employed in different spaces. What do the instruments tell us about the range of operations carried out? Is there evidence of distillation? Are there cupels? What was the quality of the equipment? Did they import or buy locally (or both)? Generally, there seems to be a difference between operations that required high precision and others where this was less of an issue. In mints and assay laboratories, where quantification was critical, German crucibles feature heavily even when these had to be imported at a high cost (**Martinón-Torres and Rehren 2009**). On the other hand, spaces like glasshouses tended to employ local vessels. Yet, quality crucibles were very important in glass making too as they had to withstand high temperatures and corrosion from the silicate melt (**Dungworth 2008; Paynter 2012**). The universe of substances manipulated during chymical activities offers another source of comparison between laboratories. It was shown how, more often than not, chymists from different contexts handled the same or directly comparable substances, but these were seen in different ways, or came in different forms according to the aims underpinning the work. Clearly, John Francis Vigani had no personal connection with the assayers working in Jamestown, and yet a focus on materials makes us realise that the two can and should be discussed together. Lead, which is so often present within the crucibles, was used in a host of different processes. Calcined, it was a reagent in glass making/working, for both structural and aesthetic purposes. In metallic form, it could be employed as a noble metal collector during fire assay. Thus, drawing such maps reveals many connections and overlaps that would otherwise remain invisible, and foster new material-led questions and topics. In turn, material-based considerations can be employed to introduce ways to classify, compare and contrast different chymical contexts, while reviewing the practical-productive vs philosophical-inquisitive paradigm that underpins Anderson's classification. Three such arenas of discussion are addressed in the next subsections, namely scale, experimentation and patronage.

6.2.1 ISSUES OF SCALE AND EXPERIMENTALISM

An important question to ask is whether chymists sought to sell their products and how economic needs interact (if at all) with more scholarly interests and with the production of knowledge. The need to make a profit was a huge factor in shaping chymical agendas, as exemplified by Robert Plot and John Francis Vigani who, despite teaching chemistry at prestigious universities, still needed to make a living with their chymical products. However, teaching and research were equally strong drives. The Ashmolean laboratory had ties with the most important philosophical societies in England and the variability encountered within the crucibles hints at a significant element of curiosity. And indeed, from the later 17th century, the scientific societies of Europe became a powerful catalyst for experimental chymistry, their purpose the advancement of knowledge on a broad spectrum of technological and philosophical issues. Therefore, variability in the range of substances and of processes identified is a useful element for classification, as it can tell us something about experimentalism. Can we see different approaches to a recipe, or relevant changes in the variables of a chymical process? The marked element of variability at the Ashmolean has been stressed already, the reagents at play were many and the operations reconstructed had different desired outcomes across different craft and philosophical traditions. Experimental work at this site has a strong element of philosophical curiosity and practitioners could choose, to some extent at least, what to work with. The repository two stories up was stocked with all sorts of interesting substances which could be studied or used in productive processes. The collection has not survived to this day, but it must have been as rich and as varied as Vigani's, quite possibly even more comprehensive if we consider that Plot was a natural historian and he often sent boxes of specimens to the Royal Society (**Birch 1756, vol. 4; Gunther 1939, vol. 12**). For different reasons, chymical practice in Jamestown had a strong experimental character too. Chymists had to work with what was available and under the pressure of the Virginia Company. The combined effect of an unpredictable environment and the need to "deliver" must have acted as a stimulus, albeit a forced one, to experiment and innovate. And indeed, what we see is that the fire assay operations involved a wide range of substances, both in terms of minerals tested and of reagents used.

In other cases, circumstances allowed or in fact required more standardisation. At the smelting site of Angertal in Austria, materials show that ores rich in gold and silver, quite similar to the sulfides tested in Virginia, were processed in standardised operations that left little room to variation (**Mongiatti et al 2009a**). Similar instances where production and the market were the main drivers, and where operations were standardised, are the zinc distillation sites discovered in China and India, where large amounts of ore were processed in batteries of furnaces (**Zhou 2007; Zhou et al 2012**). The same applies to the 18th-century zinc and brass manufactory of Warmley, near Bristol, the site of William Champion's patent for zinc distillation in 1738 (**Day 1998; Etheridge and Dungworth 2012**). So, variability in the host of materials identified at a site of chymistry gives us interesting information on the ultimate reasons that motivated the activity and where it sits within the chymical spectrum. Nevertheless, even material variety, experimentation and standardisation do not define closed categories and overlaps remain. Much to our loss, we have no material evidence of a laboratory like the 17th-century Viennese "workhouse" of Johann Joachim Becher, where the chymist carried out experimental work on various crafts (**Smith 1994**). If we had materials, they would likely show us both variability and standardisation since the products were meant to be sold on the market, but were also sources of knowledge on natural creation and transformation. Standardisation alongside variability is mirrored in the materials from John Dwight's Fulham pottery (**Green 1999**). Besides the specialised wares produced and sold in large amounts, the assemblage discovered at the site includes the remains of Dwight's tests in search of the correct recipe to reproduce Chinese porcelain.

All of these examples also show that from a material perspective, productivity and standardisation issues tend to result in large-scale operations. In contrast to the small crucibles, glassware and tools from Jamestown and the Ashmolean, the sites mentioned above feature larger apparatus and handled larger batches (**fig. 6.13**). While highly experimental or philosophy-oriented chymistry could happen in large spaces (e.g. the court laboratories of the 16th and 17th century), shifting the focus on the actual size of the objects and of the quantities handled in a single operation offers an alternative to the often too simplistic association small-philosophical vs large-industrial. More zinc ore was processed in Bristol, Zawar (India) or 16th-century Fengdu (China) than it was in the Ashmolean laboratory, where distillation technology was still under development. In the imaginary assemblage from Becher's workhouse, we would probably see small crucibles for testing craft recipes, but also probably larger pieces of apparatus for making larger amounts of certain products that would end up on the market.

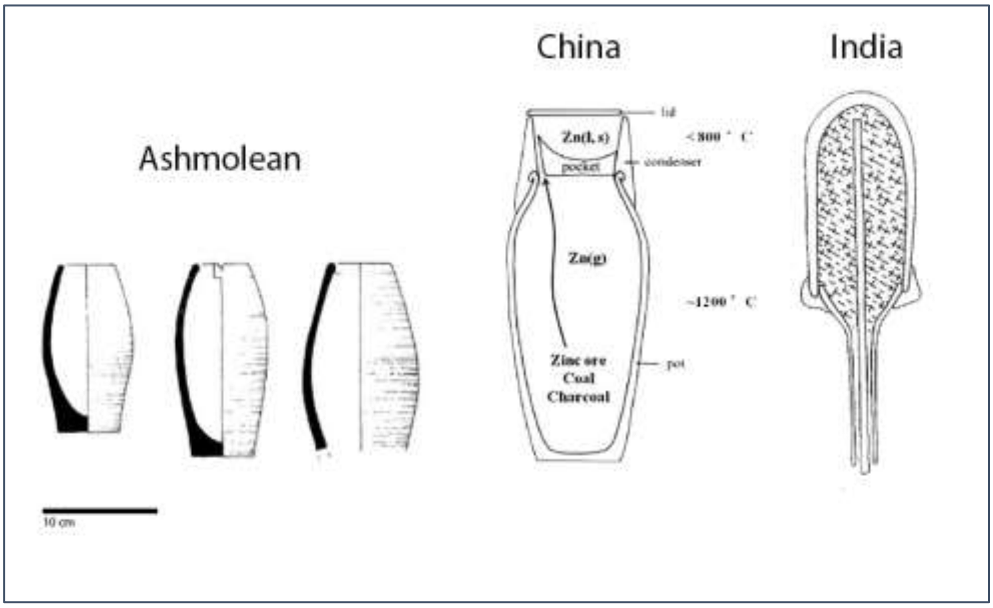

Figure 6.13. Comparison of zinc distillation crucibles from the Ashmolean laboratory (after Hull 2003), Fengdu (after Zhou et al 2012) and Medieval Zawar (after Craddock 2018)

Scale also applies to furnaces, an absolutely key feature of early modern chymical contexts alongside the tools discussed so far. Furnace design and size were dependant on the type of operations carried out in the laboratory, and the many case studies discussed in this thesis showcase the wide array of sizes and shapes that furnaces could take, from America to Europe and all the way to Asia. Early modern chymical authors often address the topic of furnaces, often using their work to promote new and improved furnaces, of which the portable ones were perhaps the most popular (see for instance the case of Boerhaave in **Hendriksen and Verwaal 2020**). However, furnaces are also some of the least archaeologically visible remains of early modern laboratories, and what we normally end up, when lucky, are just remains of the foundations, with relatively little indication of what the furnaces would have looked like. This has significant implications for the material-based taxonomy of laboratories proposed in this chapter, as only rarely can this count on such evidence. The scale of materials themselves, therefore, tells us about what were the aims at the heart of chymical activities, and further help navigate the many facets of chymical laboratories.

6.2.2 PATRONAGE AS DRIVER IN CHYMICAL PRACTICE

Mechanisms of patronage are a useful way to frame the different motives fuelling chymical practice at different sites as well as an arena where the classification criteria discussed above come together. Who sponsored the work in the laboratory and who enjoyed the fruits of such work? The different agendas of different patrons set expectations, while defining the spaces and often also the actors at play, playing a fundamental role in shaping the different identities of early modern chymistry (**Nummedal 2007**). These identities emerge clearly from the material approach to laboratories discussed here. Several members of the Virginia Company were also shareholders in the English copper monopolies and it is them who must have provided the first settlers of Jamestown with the scrap copper found in the assemblage, which chymists then used to test minerals for copper alloys. One of the founders of the Company of Mineral and Battery Works was the instrument maker, goldsmith and assayer Humphrey Cole (1530-1591). As an entrepreneur, he was interested in the technology of brass for his mathematical instruments, and while he was involved in mining calamine in Somerset, he must have been attracted to the possibility of finding viable sources in the New World too. Incidentally, Cole was employed at the Royal Mint when Martin Frobisher explored the Canadian arctic, and performed tests on the load of worthless pyrite brought back (**Jardine 2018**). That the exploitation of Virginia was also an exciting opportunity to broaden existing natural knowledge is shown by the mathematician and natural philosopher Thomas Hariot, who participated in Walter Raleigh's voyages of the 1580s. In Roanoke, Hariot made observations and collaborated with the chymists Joachim Gans in activities that must have resembled those of Jamestown (**Hariot 1588; Nöel-Hume 1994; Beaudoin and Auger 2004**).

In early modern Europe, patronage very often came from emperors and princes, who put up court laboratories and hired chymists in a multitude of tasks (**Smith 1994;**

Nummedal 2007; Beretta 2014; Dupré 2014; Klein 2014). The type of operations changed according to the needs and the desires of the patrons, who may have been interested in exploiting mines, engaging in metallic transmutation, making glass and medicines, or work on mysterious and potentially profitable technologies like porcelain. As mentioned, court laboratories may be very large, like the Casino di San Marco in Medici Florence, where "the investigation of natural phenomena was intimately connected with arts and crafts" (**Beretta 2014: 131**) (**fig. 6.14**).


Figure 6.14. The Medici laboratory depicted by Johannes Stradanus, 1570

All of these granted financial gain, power increase and health benefits, but it also fundamentally shaped aspects of governance and modes of production (**Roberts and Werrett 2017**). Other times private entrepreneurs set their own agenda, as in the case of John Dwight and George Ravenscroft, to cite two figures whose careers partly intersect with the Ashmolean laboratory (**MacLeod 1987; Green 1999**). Like Dwight's, Ravenscroft's chymical efforts were at once experimental and industry-oriented, he made large amounts of glass and was an innovator in the field. Both relied on secrecy and struck profitable deals with the London Glass Sellers Company as the sole official manufacturers of stoneware and lead crystal. The role of scientific institutions in sponsoring chymical work has been addressed in the previous section 5. In this case, work was more varied and included a wider spectrum of interests. That of patronage, it should be noted, is a rather elusive topic to address with archaeology alone, and one where the synergy with historical information is especially important.

In light of what has been discussed in this thesis, Anderson's distinction so heavily dependent on fixed categories of craft vs science inevitably downplays the multifaceted reality of early modern chymical spaces so strongly reflected in their material culture. What we need is a more balanced material-based classification that evaluates the complex nature of chymical practice and shows that the terminological unity between laboratory and workshop is made of interconnecting substances, tools, spaces, people and motivations.

6.3 ARCHAEOLOGY AND THE HISTORY OF SCIENCE: IDEAS FOR A CROSS-DISCIPLINARY AGENDA

Materials connect, they are part of a network of tools, of substances and of processes, and provide a thread running through places of chymical practice. When we allow materials to create context and we proceed from the bottom up, what emerges is a spectrum made of different activities and people and of shifting motivations and desires. We see that the emergence of scientific ideas and new technologies was something collaborative and messy, a process which entailed experiments, errors, repetitions and plenty of interactions. We see that the new reactions, more accurate quantifications and better observations made possible by high-quality tools allowed key developments in natural philosophy. The approach and the type of arguments made in this thesis are very significant to a historiography of science that is increasingly sensitive to material culture and more inclusive of different specialisms. This thesis also argues that historians, archaeologists and archaeological scientists often explore along similar lines and ask similar questions, but use different sources of information and different frameworks of interpretation. Sometimes even the frameworks largely correspond and only terms or points of view change. More and more historians rely on ideas such as object biographies and concepts that find a close parallel in the anthropology of technology and in the *chaîne opératoire* approach commonly adopted in archaeology (cfr section 2.3). Technical art history gives relevance to the context, both technological and social, within which an object was created and used or became what we see now. Moreover, this approach also shares with archaeological science a scientific methodology of investigation, and both significantly engage with conservation and conservation science (**Dupré 2017a**). Historians are also increasingly adopting an experimental approach to their narratives, through performative methods that aim to investigate non-verbal, sensory knowledge otherwise invisible (**Smith 2007; Hagendijk 2018; Hendriksen 2020**). Experimental archaeology has long employed the restaging of technological processes in order to better understand how people made things or what kind of marks specific actions left on materials. These methodological overlaps are notable and highlight that the gulf between the two disciplines is not as wide as it may seem, that in fact there is room for relevant exchange.

By choosing a materials-centred approach to the history of chymistry and of chymical laboratories, this thesis reinforces such proximity, and argues that more cross-disciplinary exchange is not only promising but also timely. At the same time, the cases discussed in the thesis also show that some current narratives in the historiography of science can act as a different framework for an added element of texture and complexity. This is especially true for studies on past technology that investigate ways in which people made and used things, and how materials and objects travelled. As a matter of fact, these same questions were at the very core of the analysis of the case studies presented here. What did the chymists of Jamestown and the Ashmolean laboratory work on? What substances did they manipulate and to what end? The results were then fed into broader cross-disciplinary narratives that threw more light on the intersection of science, craft and knowledge in early modern Europe and beyond. Archaeological scientists could benefit from a higher familiarity with text-based sources, engage with them in ways that complement information from materials and that may lead to significant and unexpected insights. In this sense, if historical research should - and increasingly does - place more emphasis on practice over ideas, to bring some of those ideas into the picture could be good practice in archaeological science.

This thesis has offered examples of how such interaction can happen. A number of examples from the literature were brought up throughout the present chapter, each discussed in light of their contribution to a material network defining chymical practice. The diachronic analysis of brass making remains from various European sites led the way to the discussion how the understanding of this alloy changed in relation to changes in metallurgical technology (**Martinón-Torres and Rehren 2008**). This in turn became a way to look at interactions between artisanal practices and shifting theories in natural philosophy. The analysis of glassware from Oberstockstall and the rationale behind sourcing the laboratory apparatus proved insightful in exploring the role of high-quality tools in the history of experimental science and analytical chemistry. However, given the richness of the chymistry spectrum, more assemblages can be approached this way and of course the scope can be broadened beyond the specificity of the early modern period. In *The Alchemy of Glass* Marco Beretta traces a history of chymistry from the peculiar point of view of glass technology (**Beretta 2009**). One of the most insightful arguments regards the intimate connection between technological innovation and philosophical interest. Following technological breakthroughs such as the massive production of coloured glass for mosaic tesserae during the Byzantine period, curiosity around this puzzling material surged, with natural philosophers investigating its nature and its many transformations. The book shows that a specific material can become the theme around which a history is built and in this respect Beretta's work is very significant to the kind of approach brought forward here. However, even this work misses the large amount of information on glass technology available from archaeological science (**Martinón-Torres 2012c**). While Beretta mostly bases his research on texts, one could interrogate materials to see if they are equally vocal in showing us the connection between the flourishing glass technology and philosophical questions of transmutation. Incidentally, mosaic technology, as well as glazes and enamels, are fields that have traditionally received extended attention in archaeological science with specific focus on colour production (cfr section 5.5.2). We know what reagents glass specialists employed to colour and opacify the glass, we know where these came from and how they were sourced and treated in the production centres. The available data adds to the material connections of chymistry since many of the substances from the map in **figure 6.1** tend to recur in the same or in similar forms. We may want to cast new light on these contexts by asking if mosaic makers worked within the boundaries of official patronage, for instance. After all, mosaic decoration was very strongly linked to power, to sacred and generally high-profile buildings, and this may have shaped the aims behind the chymical work in significant ways. However, what we also may want to address, adding a new level of interpretation, is the philosophical value of glass itself, as a material that undergoes radical transformations during the high-temperature processes involved in such technologies. Some exciting work in this direction has been done with reference to the earliest glass made in Egypt and Mesopotamia during the Late Bronze Age. After reconstructing how glass had been made and coloured, these studies used the data in conjunction with documentary evidence to explore the conceptualisation of glass as an artificial product, the generative and transformative action that glass making entailed, and the relationship between glasses and the precious stones they represented through colour terminology (**Duckworth 2012; Shortland 2012; Walton et al 2012**). It is interesting to notice how at a philosophical level there seems to be little difference between dark blue glass and the stone lapis lazuli, their apparent identity given by sharing the same colour. The artificial creation of colours was a crucial element to the invention of glass as a high-temperature technology, but it was simultaneously a means to reproduce, observe and investigate natural phenomena. Over three thousand years after the first glassmakers of Egypt and Mesopotamia, we see the very same interaction between glass technology and natural philosophy in the material culture of the Ashmolean laboratory.

CHAPTER VII

FROM HERE: CONCLUSIONS AND FUTURE WORK

In 1845, the English scientist Michael Faraday was busy working on his experiments with light and magnetism. He believed that the two forces are related and tried to test this hypothesis by shining a beam of polarised light through a magnetically charged medium, to see if he could observe any changes in the light itself. He made several attempts using different transparent media but no visible changes in the light occurred, until he decided to try with a piece of heavy glass that he himself had made nearly twenty years before. Back then, a younger Faraday was working on a project to find an improved formula for optical glasses to be used in scientific instruments, mainly telescopic lenses. He had devoted the years 1827-1829 to the task in the laboratory of the Royal Institution, where he had tried many glassmaking recipes, had become a glass expert and had personally overseen the production. However, the project had not brought any significant results and Faraday came to consider it as little more than a waste of his precious time. As a reminder of the many exciting experiments, he could have embarked on had he not been assigned to the optical glass project, Faraday had kept a piece of glass, the same piece that now stood on the poles of his electromagnet. Upon starting the current Faraday observed that the light changed intensity according to the strength of the magnetic force as a result of its polarisation, what is now known as the magneto-optic or Faraday effect. The souvenir of a past failure had become the carrier of a new discovery, one of great relevance to Faraday’s field theory of electro-magnetism. In a sense, what Faraday had discovered was what many chymists had been postulating since the Middle Ages as the web of correspondences, of sympathies and antipathies, the invisible force that regulates the transformation of matter, the force of God and His presence in nature. Even though he would never be able to prove it, what Faraday had discovered was the existence of the electromagnetic field, which as it happens is a powerful force with a huge influence on our world. And what had ultimately allowed Faraday to achieve such scientific breakthrough was the outcome of an almost forgotten applied development project of many years prior, the product of artisanal and experimental efforts and, even more interestingly, of a failure story (**James 1991**).

This thesis presented an attempt at investigating the rich and complex relationship between intersecting spheres of knowledge in the early modern period, through a materials and practices-oriented approach. In aligning with the definition of chymistry as a way "to understand and manipulate matter" (**Newman 2000: 49**), the work started with addressing how this manipulation happened, what chymists did in their laboratories and why. What was chymistry about? This is one of the "surprisingly basic questions" (**Nummedal 2011: 332**) that recent historiography has recently been striving to address. Through the analysis of two high-profile case studies this thesis has provided some answers to such questions, pointing to the information potential of material culture as a different set of primary sources on early modern chymistry.

The laboratory remains from Jamestown have shown us that doing chymical work revolved around the desire (and the duty) of making a colonial venture profitable. However, the many tests conducted at the site were also framed by the desire (and the need) of making sense of a new environment which profoundly challenged established natural knowledge in Europe. Results on the residues analysed in chapter 4 show a host of minerals being assayed in just a handful of crucibles, a testimony to the curious and markedly trial-and-error attitude of early colonial chymistry. If noble metals were certainly the most desirable outcome of mineral prospection, materials tell us that other substances were of high relevance too, such as viable sources of zinc and tin to be employed in the manufacture of copper alloys. Thus, in following the trail of materials this thesis is also an argument in favour of including those chymical contexts with a more production-oriented character in the history of science. At sites such as Jamestown and the other early European colonies discussed, chymists had to be creative, they had to adapt their knowledge to a new environment and work under strain. Equally, chemical technology and productive endeavours entailed observable chemical reactions that may spark ideas and theories. More than one constraint also shaped the chymical programme of the Ashmolean laboratory, the epicentre of chymistry in early modern Oxford and one of the most up-to-date sites for chymical practice in Europe. On the one hand, the materials point to a strong sense of curiosity towards chemical technologies, industrial innovation and links with current natural philosophical interests. And even though the archaeometric analyses could not provide direct evidence in favour of teaching activities going on in the laboratories, it is likely that students interested in chymistry did engage with some of the procedures carried out and that these may have been used as the basis for a discussion around the structure and the transformations of matter. Indeed, operations such as evaporating and distilling zinc or opacifying and colouring glass would have offered some useful demonstration of how to manipulate substances and conduct high-temperature chemical reactions. On the other hand, the very same procedures, and the many more that must have taken place in the laboratory, were a source of profit to Plot and his assistant who did not receive a formal salary from the university. This helps explaining the lack of detailed documentary information regarding what was being worked in the *officina chimica* and for what purposes. The outcomes of the experiments were precious sources of income and as such they had to be protected, their access regulated.

What has emerged most prominently from answering the questions of what doing chymistry meant is a network of materials that exposes the many souls of this early modern phenomenon, the reality of its complexity. When telling the story of early modern chymistry materials and their networks are as relevant a source of information as networks of people are, and as such they require more attention. Jamestown and the Ashmolean laboratory appear to have little in common, being as they are a century and an ocean apart from each other. The type of activity carried out at the two sites is as significantly different as are the motives that drove it, according to a distinction production/testing vs research/experimenting that we may be tempted to apply. But the landscape of substances, tools and processes that appears when we look at the materials surviving from the two laboratories tells a more complex story, a story of numerous connections not just between the two sites discussed here, but also across a variety of chymical contexts more broadly. As this thesis argues, a history of chymistry can be built from the bottom up, from broken crucibles and the residues within them, from shards of glass and bits of slag left behind. Archaeology is inherently prone to reconstructing microhistories, well-contextualised single realities, often covering restricted periods of time and lesser-known parts of the picture. More often than not, archaeological remains of chymical contexts give visibility to lesser-known or in fact anonymous practitioners and everyday operations. They remind us that science happened as much in book-filled rooms as in messy laboratories and people's kitchens all over the world. As "something people *did*" (**Moran 2005: 10**), science was fuelled by myriads of single experiments and trials and it was fundamentally shaped by the different hopes and needs behind them (**Roberts and Werrett 2017**). But even when materials speak of high-profile institutions and renown individuals, as it is largely the case here, the different point of view they represent offers us insights that would otherwise be impossible to obtain.

More immediately, the case studies discussed in the previous chapters raise some additional questions and point to possible pieces of new research. It would be interesting, for example, to go back to the crucibles discovered at the site of John Dwight's Fulham pottery to establish if the rounded ones found there (cfr. fig. 5.11) are consistent with the non-Hessian crucibles from the Ashmolean. This would shed further light on the supply of high-quality apparatus at Oxford's laboratory and add an important link to the chymical community gravitating around it. Additional work on Jamestown materials should target those crucibles and other remains that can be linked to glass production, a major aim for the colony of which we know virtually nothing. Previous work on what seemed to be glass making residues has recently been reassessed as metallurgy-related, but more convincing candidates for glassmaking were also pointed out (**Rehren et al 2019; Veronesi et al 2019**). Moreover, future studies adopting a materials perspective on early modern chymistry could expand in directions that the assemblages analysed here could only in part address. Indeed, due to the specific interests and expertise of the few scholars involved in it so far, the archaeology of chymistry has by and large focused on metallurgy and its relationship with natural philosophy (see cited works by Marcos Martinón-Torres, Thilo Rehren, Aude Mongiatti and Nicholas Thomas). And even when tools were targeted these were mostly pieces of apparatus used in metallurgical processes. Thanks to these studies we now know quite a lot about Hessian and Bavarian crucibles of post-Medieval Europe, how and where they were made and what made them so popular features in the material landscape of early modern chymical laboratories. Nevertheless, it was pointed out how alternative productions existed in this period and how chymists often chose a cheaper local solution over the expense of buying imported vessels. A striking case is that of English glasshouses discussed in section 6.1.1. A look at some publications of archaeological collections is enough to see that German crucibles are not commonly found and that a huge variety of forms and types was used instead (**Crossley 1967; Vose 1994; Welch 1997; Dungworth et al 2013**). One is tempted to ask why such a marked distinction between different spheres of chymistry. Was it because the different material constraints and technical challenges set by glass technology made German crucibles not quite a game changer as in high-precision metallurgy? Were glass making/working crucibles adapted for those specific challenges? A comprehensive analysis of crucibles across English glasshouses would bring more insight into the theme of chymical apparatus discussed in this thesis, and would diversify it as well.

As we have seen here, chymistry comes in many shapes and the amount of potential material assemblages and questions to address is broad. In this respect, another major lead that this thesis offers to future studies is that of glass as an especially promising topic to investigate within the framework of the archaeology of chymistry. The intimate link between glass and chymistry is by no means new and has been discussed in detail in section 6.1.2. Glass has numerous layers of meaning, being at the same time the material of some of the most important laboratory instruments, a valuable market commodity and a deeply fascinating substance which had attracted the interest of natural philosophers and experts for centuries. From the day of the alchemist Mary the Jewess, who in the 3rd century AD praised glass vessels because they allowed to "see without touching" (**Patai 2014: 61**), to the ground-breaking discoveries made through telescopes and microscopes, glass can rightly claim its position as one of the prime sources in the history of science and technology, just as much as metals can. But the archaeological science literature lacks comprehensive studies of glass laboratory apparatus which, like for ceramic crucibles, likely prompt specific questions. Did the special needs of the object demand specific glass types or a tweaking of traditional recipes? The alchemist Thomas Norton (1532-1584), for instance, calls for the addition of recycled glass when manufacturing distillation vessels, as this would make them stronger (**Anderson 2000: 13**). Similar pieces of advice from technical texts were reported in section 6.1.2, strength and clarity understandably the most important parameters for good tools. The example of the glass apparatus from Oberstockstall presented in this thesis (cfr 6.1.2), where data shows that vessels were sourced differently according to their use, has but scratched the surface of the issue (**Veronesi and Martinón-Torres 2018**). But in spite of glass' more problematic survival in the archaeological record, some important assemblages do exist whose study could further contribute to charting the many roles of glass in chymical practice. What may be the most exceptional in terms of quantity, has recently come to light from the site of a 17th-century chymical laboratory discovered in Wittenberg and tentatively linked with the chymist Daniel Sennert (**Klein 2016; Stephan 2016**) (**fig. 7.1**). Unlike what was seen in Oberstockstall, where distillation vessels are invariably of clear Venetian glass, the extensive assemblage comprises of mostly green glass. So, was clear glass essential? Something must have made the green glass preferable here, what was it? And where was it sourced?

Figure 7.1. 17th-century Glass distillation apparatus from Wittenberg (photo by Joel Klein)

These first steps into a poorly known area inevitably raise more questions than they provide answers, and more systematic analyses of distillation equipment are needed. It is hoped that the initial results discussed here may encourage archaeological scientists to explore new questions in glass studies, questions that will both be informed by and contribute to parallel work in the historiography of science (**Beretta 2009; 2017; Dupré 2018; Bycroft and Dupré 2019**).

There are also broader themes for which materials networks in early modern chymistry may offer a powerful focus. The discussion around text-object interaction in cultural studies is a crucial topic which scholarship has gone back to time and again (**Andrén 1998; Moreland 2001; Martinón-Torres 2008;**). Nonetheless, the most recent developments in the historiography of chymistry, the focus on practice and on artisans, the replication of recipes, all demand a new wave of studies on what interesting information can be obtained by letting textual and material sources interact. The sheer volume of documentary evidence available, coupled with an equally rich archaeological record, makes the early modern period and the study of chemical technologies especially fit to integrate approaches. Artefacts materialise the elusive knowledge of craftspeople, so difficult to articulate in writing but also so relevant to the new sciences of the 17th century, and so increasingly claiming a large share of historiographic research (cfr section 2.2 and 2.3). In chapter 5 it was discussed how the material landscape of Oxford's early modern chymistry gives us a connecting thread through the somewhat patchy knowledge on the many figures operating in town. In doing so, materials put in sharp relief the interactions among these figures and unveil the overlapping interests, the awareness of each other's work and even hinted at the competition that must have been going on. We know that Robert Plot did not list litharge among the raw materials for lead crystals that he sees at the site of George Ravenscroft's glasshouse and this has often been explained with the latter's need for protecting a valuable trade secret (**MacLeod 1987**). However, we have seen how litharge was a major ingredient for the lead crystal produced in Plot's own laboratory (cfr section 5.5.2.1). In a similar way, the residues from the crucibles in

Jamestown tell us that the metallurgists knew what they were doing and that they were trying to apply their knowledge on minerals to Virginia's geology. Materials and their analysis have readdressed the traditional historiographic view blaming the ultimate failure to find viable sources of metal to the settlers' laziness and incompetence.

Coming out of the immediate finds presented in this thesis, a pointer for studies to come that want to give fresh perspectives on the artefacts-texts relation are those instances where we have both laboratory notebooks and material assemblages associated with the work of experimenters. John Dwight, abundantly mentioned throughout this thesis in relation to his experimental efforts on ceramics, kept details of his porcelain tests in a notebook. Incidentally, excavations at his Fulham pottery have unearthed a number of test chips that he had produced using different recipes (**Green 1999**) (**fig. 7.2**). A similar case is that of one of the most famous potters of all times, Josiah Wedgwood, whose archives contain a mine of information of his experimental work in pursuit of fine wares[15] (**Blake-Roberts and Rawsthorn 2017**). Another example is that of Michael Faraday's optical glass project which opened this chapter. Here too, we have an extensive collection of test glass squares on display at the Royal Institution (**fig. 7.2**), while the Royal Society holds Faraday's notebooks in which he kept thorough details of his experiments (**James 1991**). Taken separately, case studies like these hold a tremendous information potential on laboratory practice in the contexts of industry, a theme with major relevance in current historiography of science and technology. One could explore the kind of background that these figures came from as some, like Faraday, had to learn from scratch what glass making entailed while others, like Dwight, were artisanal experts. In turn, the experiments and the discoveries they made fed into broader scientific knowledge of the time. Taken together, the three collections could also provide exciting new paths to chart the development of how laboratory experiments were recorded from the late 17th to the first half of the 19th century. An analytical study of the material remains and a parallel scrutiny of the notebooks could point to what parameters the different authors

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[15] http://www.wedgwoodmuseum.org.uk/archives (Last accesses June 2020)

deemed more important, they would allow to explore issues of reproducibility and to tell stories of success and failure. Finally, studies of this kind could also bring a further and much needed contribution to enriching the spectrum of aims driving chymical practice at different sites, which would argue once more for the overlaps and diffuse boundaries in early science, in research and development that this thesis has explored.

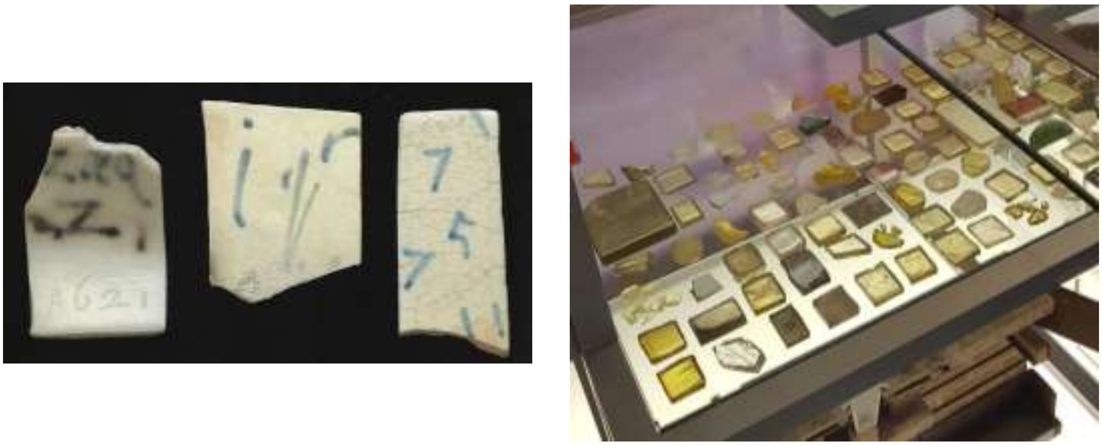

Figure 7.2. Left: John Dwight's porcelain test chips (from Green 1999); right: glass squares made by Michael Faraday in his optical glass experiments, on display at the Royal Institution.

On a broader methodological level, this thesis is an attempt at engaging present-day specialisms in mutual conversation by defining one possible arena where such conversation can successfully happen. In this respect, the fundamentally open character of chymistry that this thesis argues for was the basis for proposing - in section 6.2 - an alternative classification of laboratories. Rather than insisting on anachronistic divisions that fail to capture the fluid boundaries between multiple chymistries, the proposed taxonomy is based on materials and reactions instead. Thus, while still offering a point of view for comparing and contrasting different chymical contexts it also acknowledges continuity and connection emerging from the object-scapes[16] reconstructed. It is felt that this way of classifying is much more attuned to the increasing historiographic attention to the laboratory as a trading zone between the

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[16] For the idea of object-scapes as an interpretive framework for the role of materials in human history see **Van Oyen and Pitts 2017; Pitts 2019 and Pitts and Versluys 2021**.

worlds of making and of knowing (see **Klein 2008; Smith 2013; Saunders et al 2013; Dupré 2014**). Categories that were taken into account here include the type of apparatus employed at different sites and the scale of chymical activities, but also the role that the same or very similar substances may have had in different contexts. And since the very same categories are commonly found in archaeological science and technological studies, it is expected that the framework discussed here will encourage more crossing the disciplinary boundaries and more acknowledging of the benefits that come with it. It is also expected that, as more of such interaction takes place, further material-based categories of significance to the history of high-temperature laboratories will be laid out, and that these will become relevant to historians too.

The archaeology of chymistry is not in itself a new concept that the present work has introduced. It was inaugurated in the past two decades or so and showed the exciting potential for broadening the scope of traditional approaches in archaeological science (**Martinón-Torres 2011**). The first generation of such studies have laid the tracks for integrating material culture, scientific analysis and textual information in meaningful ways. For future works that will continue on these tracks it is believed that integration should happen following two channels. The first is that of active collaborations between scholars of different backgrounds. Examples are the increasing number of research projects adopting this approach, some of which have been mentioned in this thesis (cfr section 2.3). Researchers involved in similar projects are able to acquire new skills, become more aware of the importance of crossing disciplinary boundaries and of the potential for new and exciting insights that arise from such "contamination" (**Rehren 2014**). The results are promising and as more projects of the kind will no doubt appear in the future, it is hoped that more room for archaeological science and its contribution to the cause may be found. The analysis of archaeological assemblages could accompany the re-enactment of historical recipes, an approach that is ever so rapidly gaining momentum among science and art historians (cfr. section 2.3). If it is true that "the question no longer appears to be whether performative methods have a place within history and philosophy of science research, but what their place is, could, or should be" (**Hendriksen 2020: 314**), one may also argue that disciplines such as archaeological science with a well-developed experience in performative methods could bring a significant contribution towards reconstructing technological recipes and exploring (at least some) questions on artisanal knowledge (**Doonan and Dungworth 2013**). Since to replicate a process is not necessarily the same as to perform an experiment, more thorough testing of the final products and comparing the data with historical materials and sources may become an important addition in future projects.

The second channel for inter-disciplinary communication is what has been attempted here, and involves using one methodology (i.e. archaeometry) to look at some of the questions and narratives of a different field (i.e. the history of science). In other words, to re-frame one discipline within the scope of another, thereby widening the horizons of both by creating room for new layers of interpretation of various aspects of the human experience (**Rehren 2014**). For obvious reasons, this type of interaction necessarily implies a bias towards one of the disciplines at play and is dependent on the researcher's background. The present work is first of all a contribution to the field of archaeological science, as it begins by asking technology-related questions and uses a standard archaeometric methodology. However, this thesis shows that in spite of this bias the results can be framed in such a way as to become meaningful to complement current historiographic research. Venturing into different disciplinary realms is not without risks and inevitably results in some gaps and limitations. This thesis has kept its scope as broad as possible, moving within a space that included seemingly very distant themes. Despite losing some penetration depth, it was important to address such a diverse range of topics as this is precisely what allowed to build a stronger argument for the connection provided by a materials-focused approach. Having said that, there are possible – and perhaps even obvious – developments that have only been touched upon and that could have had a bigger role. Some may argue, for instance, that the data from Jamestown could trigger more insight into the history of European colonisation and the discovery voyages of Elizabethan England. More in-depth research in this direction could unveil interesting material-lead connections among the figures who one way or another were involved in the Virginia Company or the English copper industries (cfr section 6.2.2.

Hopefully, the initial results discussed in the previous chapters will provide future projects in both disciplines with inspiration to go back to the loose ends that could not be tied up here, and to come up with fresh questions. After all, the archaeology of chymistry is still in its infancy, not so much in terms of its actual age but because sectorial boundaries and disciplinary divisions have discouraged researchers from engaging in it on a regular basis. The excitement of young disciplinary branches lies in the fact that their scope is initially broad, as are the research questions that they address. The present work was no exception, and the initial reconstruction of chymical practice at Jamestown and the Ashmolean laboratory triggered and fed into a series of further questions and potential leads to follow, some of which were expected while others less so. To some this thesis has tried to do justice, but others have been left unattended, for now. However, a path has been opened, a path where two disciplines can come together and mix some of their research narratives in light of an enhanced awareness of each other's agendas. To the history of science, the archaeology of chymistry represents something like a fresh pair of eyes, the chance to bring an element of balance to a research field that has traditionally been heavily dependent on texts and skewed towards a few prominent individuals and their ideas, rather than the actions of many. As historians seem to be increasingly sensitive to including material culture among their sources, an approach that takes its start from artefacts and the information within them is apt to become a relevant player in the future developments of the field. On the other hand, in a fast-moving academic world where we are constantly looking for new ways to elaborate on disciplines, what the history of science framework has to offer to the archaeometric study of past technologies is a new layer of investigation, a way to widen its scope and make new questions relevant. Having opened new channels for cross-disciplinary dialogue this thesis has attempted to put new questions on the agenda - in fact even to try and imagine a new agenda. Last but not least, the work presented in this thesis represents another player within the broader historiographic narrative that aims to replace the enduring view of alchemy as an irrational endeavour. The fact that making gold artificially or manufacturing the universal medicine have been proven an impossible task by later developments of knowledge does not allow us to see the alchemist as a sorcerer-like figure, forever doomed to chase ghosts. After all, some of the scientific truths we give for granted today may well become the myths of the future.

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APPENDIX A

SEM-EDS ANALYSIS OF REFERENCE MATERIALS

A number of certified and non-certified reference materials were analysed with SEM-EDS in order to assess the accuracy and precision of the data generated. The analytical protocols followed are those outlined in section 3.2.3. A basalt standard was chosen as comparative material for the ceramic matrix of the crucibles and other ceramic vessels, while three glass standards were used for the composition of the slag layers and for the glass finds. Finally, three metal standards helped assess the quality of the data for metallic phases within the slag layers.

A.1 BASALT

The basalt reference material BHVO-2, provided by the United States Geological Survey in the form of fused glass, mounted in resin and polished was used to monitor SEM-EDS data quality. The results are presented in table A.1 and refer to four rounds performed at different times during the analyses. The precision is generally very good, all elements except for phosphorus showing a coefficient of variation consistently below 5%. Equally, the accuracy of the data, indicated by the relative error, is within 5% for the majority of the elements and in any case below 10%, again with the exception of phosphorus as well as of manganese, both falling around the machine's limit of detection and for which the error is consequently higher.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	Fe_2O_3
12/12/2016										
Measurement 1	2.12	7.39	13.83	50.96	0.32	0.48	10.98	2.78	bdl	12.25
Measurement 2	2.28	7.32	13.77	51.11	0.18	0.50	10.94	2.87	bdl	12.13
Measurement 3	2.16	7.38	13.94	51.14	0.17	0.47	10.96	2.82	bdl	12.05
Measurement 4	2.34	7.20	13.93	51.36	0.12	0.51	10.80	2.75	bdl	12.08
Measurement 5	2.17	7.15	13.95	51.00	0.13	0.47	10.98	2.82	bdl	12.46
Average normalised data	**2.22**	**7.29**	**13.88**	**51.12**	**0.18**	**0.49**	**10.93**	**2.81**	**bdl**	**12.19**
Standard deviation	0.08	0.10	0.07	0.14	0.07	0.02	0.07	0.04		0.17
Coefficient of variation (%)	3.67	1.31	0.51	0.27	39.15	3.39	0.61	1.37		1.36
Normalised reference values	**2.22**	**7.23**	**13.50**	**49.90**	**0.27**	**0.52**	**11.40**	**2.73**	**0.17**	**12.30**
Absolute error	0.00	0.06	0.38	1.22	-0.09	-0.03	-0.47	0.08		-0.11
Relative error (%)	**-0.16**	**0.80**	**2.84**	**2.44**	**-31.80**	**-6.25**	**-4.12**	**2.92**		**-0.86**
10/01/2017										
Measurement 1	2.05	7.38	13.79	50.75	0.19	0.52	10.99	2.82	bdl	12.67
Measurement 2	2.21	6.97	13.94	50.71	0.21	0.57	11.12	2.85	bdl	12.57
Measurement 3	2.09	7.09	13.53	50.93	0.26	0.55	11.22	2.87	bdl	12.60
Measurement 4	2.16	7.09	13.77	50.80	0.14	0.50	11.15	2.88	bdl	12.66
Measurement 5	2.12	7.18	13.61	50.80	0.19	0.54	11.17	2.88	bdl	12.66
Average normalised data	**2.13**	**7.14**	**13.73**	**50.80**	**0.20**	**0.53**	**11.13**	**2.86**	**bdl**	**12.63**
Standard deviation	0.06	0.14	0.14	0.07	0.04	0.02	0.08	0.02		0.05
Coefficient of variation (%)	2.62	1.90	1.05	0.15	18.79	4.52	0.69	0.76		0.36
Normalised reference values	**2.22**	**7.23**	**13.50**	**49.90**	**0.27**	**0.52**	**11.40**	**2.73**	**0.17**	**12.30**
Absolute error	-0.09	-0.09	0.23	0.90	-0.07	0.01	-0.27	0.13		0.33
Relative error (%)	**-4.20**	**-1.24**	**1.68**	**1.80**	**-26.25**	**2.77**	**-2.36**	**4.85**		**2.69**
12/06/2018										
Measurement 1	2.28	7.36	13.85	51.09	0.19	0.48	10.97	2.77	bdl	12.12
Measurement 2	2.14	7.11	13.94	51.26	0.33	0.48	11.00	2.75	bdl	12.09
Measurement 3	2.20	7.34	13.52	51.35	0.22	0.54	10.78	2.68	bdl	12.51
Measurement 4	2.30	7.11	13.88	51.13	0.14	0.48	11.02	2.79	bdl	12.27

Average normalised data	**2.23**	**7.23**	**13.80**	**51.21**	**0.22**	**0.49**	**10.94**	**2.75**	**bdl**	**12.25**
Standard deviation	0.06	0.12	0.16	0.10	0.07	0.03	0.10	0.04		0.19
Coefficient of variation (%)	2.81	1.66	1.19	0.20	32.45	5.14	0.88	1.49		1.56
Normalised reference values	**2.22**	**7.23**	**13.50**	**49.90**	**0.27**	**0.52**	**11.40**	**2.73**	**0.17**	**12.30**
Absolute error	0.01	0.00	0.30	1.31	-0.05	-0.03	-0.46	0.02		-0.05
Relative error (%)	**0.34**	**0.02**	**2.22**	**2.62**	**-19.48**	**-5.15**	**-4.02**	**0.64**		**-0.42**
20/06/2018										
Measurement 1	2.19	7.3	13.8	50.91	0.1	0.51	11.12	2.77	0.2	12.188
Measurement 2	2.17	7.29	13.72	50.8	0.29	0.5	10.97	3.01	0.22	12.122
Measurement 3	2.24	7.14	13.93	50.91	0.2	0.5	11.07	2.74	0.17	12.21
Measurement 4	2.09	7.31	13.89	50.56	0.27	0.49	11.06	2.97	0.21	12.254
Measurement 5	2.23	7.18	13.9	50.73	0.25	0.52	10.9	2.74	0.25	12.408
Average normalised data	**2.18**	**7.24**	**13.85**	**50.78**	**0.22**	**0.50**	**11.02**	**2.85**	**0.21**	**12.24**
Standard deviation	0.05	0.07	0.08	0.13	0.07	0.01	0.08	0.12	0.03	0.11
Coefficient of variation (%)	2.45	0.97	0.56	0.26	30.60	2.02	0.71	4.17	12.42	0.88
Normalised reference values	**2.22**	**7.23**	**13.50**	**49.90**	**0.27**	**0.52**	**11.40**	**2.73**	**0.17**	**12.30**
Absolute error	-0.04	0.01	0.35	0.88	-0.05	-0.02	-0.38	0.12	0.04	-0.06
Relative error (%)	**-1.62**	**0.19**	**2.58**	**1.77**	**-17.78**	**-3.08**	**-3.30**	**4.25**	**23.53**	**-0.52**

Table A.1. SEM-EDS data for basalt standard BHVO-2. Not detected: Ba, Ce, Co, Cr, Cu, Ga, Hf, La, Nd, Ni, Rb, Sc, Sr, V, Y, Zn, Zr.

A.2 GLASS

The glass standards analysed are Corning A, with a soda-lime-silica composition, Corning C, a high-lead high-barium glass and Corning D, of the potash-lime-silica type. They were originally provided by the Corning Museum of Glass and are mounted in resin blocks, ground and polished for SEM-EDS analysis. Tables A.2-A.4 show the results of analyses, again performed during more than one session, against published values for these reference glasses (**Brill 1999** for all elements except Cl where updated values by **Adlington 2017 were used instead**). As a general trend, the precision tends to be poor (> 25% cv) for heavy elements in low concentrations such as CuO, PbO, Sb_2O_3 and CoO, while other elements in low concentrations such as TiO2 and Fe_2O_3 have better values (< 15% cv). All other elements display very good precision and a cv consistently below 10%. In terms of accuracy of the data, most elements display a relative error well within the 10% mark (Na_2O, MgO, SiO2, K_2O, CaO, MnO, Fe_2O_3), with slight oscillations, while Al_2O_3 appears more problematic when in concentrations below 5 wt% (10-25% error), with a substantial improvement in Corning D (< 5% error). Again, heavier elements in low concentrations such as CuO, CoO, Sb_2O_3 and PbO have poorer accuracy, as does TiO_2 (> 20% error).

06/02/2017	Na_2O	MgO	Al_2O_3	SiO_2	Cl	K_2O	CaO	TiO_2	MnO	Fe_2O_3	CuO	Sb_2O_3
Measurement 1	14.80	2.70	0.90	68.80	bdl	3.00	4.80	1.00	1.10	1.10	0.80	1.20
Measurement 2	14.30	2.70	0.80	68.60	bdl	2.80	5.10	1.10	1.00	0.99	0.90	1.80
Measurement 3	14.40	2.50	0.90	68.80	bdl	3.00	5.00	0.90	1.20	1.10	0.70	1.40
Measurement 4	14.30	2.60	0.90	69.40	bdl	2.90	4.90	1.00	1.00	1.32	0.40	1.40
Average normalised data	**14.50**	**2.60**	**0.90**	**68.90**	**bdl**	**2.90**	**5.00**	**1.00**	**1.10**	**1.13**	**0.70**	**1.40**
Standard deviation	0.24	0.10	0.05	0.35		0.10	0.13	0.08	0.10	0.14	0.22	0.25
Coefficient of variation (%)	1.64	3.68	5.56	0.50		3.30	2.58	8.16	8.70	12.28	30.86	17.98
Normalised reference values	**14.30**	**2.66**	**1.00**	**66.56**	**0.09**	**2.87**	**5.03**	**0.79**	**1.00**	**1.09**	**1.17**	**1.75**
Absolute error	0.20	-0.06	-0.10	2.34		0.03	-0.03	0.21	0.10	0.04	-0.47	-0.35
Relative error (%)	**1.40**	**-2.26**	**-10.00**	**3.52**		**1.05**	**-0.60**	**26.58**	**10.00**	**3.44**	**-40.17**	**-20.00**
16/02/2017												
Measurement 1	14.37	2.70	0.80	68.94	0.08	3.02	4.99	1.10	0.91	1.07	0.69	1.45
Measurement 2	14.34	2.71	0.79	68.41	0.13	2.84	4.97	1.07	1.00	1.08	1.35	1.42
Measurement 3	14.06	2.61	0.85	69.14	0.11	3.05	5.15	0.93	1.05	1.10	0.42	1.64
Measurement 4	14.08	2.67	0.82	69.60	0.05	3.09	5.10	0.97	0.99	1.29	0.21	1.24
Average normalised data	**14.21**	**2.67**	**0.81**	**69.02**	**0.09**	**3.00**	**5.05**	**1.02**	**0.99**	**1.13**	**0.67**	**1.44**
Standard deviation	0.17	0.05	0.03	0.49	0.03	0.11	0.09	0.08	0.06	0.10	0.50	0.16
Coefficient of variation (%)	1.16	1.69	3.27	0.71	37.84	3.68	1.71	7.90	5.85	9.14	73.97	11.37
Normalised reference values	**14.30**	**2.66**	**1.00**	**66.56**	**0.09**	**2.87**	**5.03**	**0.79**	**1.00**	**1.09**	**1.17**	**1.75**
Absolute error	-0.09	0.01	-0.19	2.46	0.00	0.13	0.02	0.23	-0.01	0.04	-0.50	-0.31
Relative error (%)	**-0.63**	**0.38**	**-19.00**	**3.70**	**2.78**	**4.53**	**0.40**	**29.11**	**-1.00**	**3.94**	**-42.74**	**-17.71**
21/08/2018												
Measurement 1	13.92	2.54	0.82	70.32	0.11	3.24	5.03	1.06	1.25	1.10	0.56	bdl
Measurement 2	13.74	2.58	0.74	70.45	0.13	3.20	4.99	1.09	1.09	1.01	0.91	bdl
Measurement 3	13.69	2.59	0.73	70.33	0.06	3.23	4.89	1.09	1.16	1.14	1.02	bdl
Measurement 4	13.83	2.36	0.80	70.66	0.13	3.09	4.94	1.00	1.11	1.12	0.95	bdl
Measurement 5	13.85	2.49	0.87	70.44	0.10	3.28	5.01	1.09	1.14	1.18	0.78	bdl
Average normalised data	**13.81**	**2.51**	**0.79**	**70.44**	**0.11**	**3.21**	**4.97**	**1.07**	**1.15**	**1.11**	**0.84**	**bdl**
Standard deviation	0.09	0.09	0.06	0.14	0.03	0.07	0.06	0.04	0.06	0.06	0.18	

Coefficient of variation (%)	0.66	3.73	7.35	0.19	27.18	2.24	1.14	3.66	5.40	5.60	21.57	
Normalised reference values	**14.30**	**2.66**	**1.00**	**66.56**	**0.09**	**2.87**	**5.03**	**0.79**	**1.00**	**1.09**	**1.17**	**1.75**
Absolute error	-0.49	-0.15	-0.21	3.88	0.02	0.34	-0.06	0.28	0.15	0.02	-0.33	
Relative error (%)	**-3.43**	**-5.64**	**-21.00**	**5.83**	**17.78**	**11.85**	**-1.19**	**35.44**	**15.00**	**1.93**	**-28.21**	
27/02/2017												
Measurement 1	14.37	2.70	0.79	68.94	0.08	3.02	4.99	1.10	0.91	1.07	0.68	1.45
Measurement 2	14.34	2.71	0.79	68.39	0.13	2.84	4.96	1.06	1.00	1.07	1.38	1.42
Measurement 3	14.06	2.61	0.85	69.14	0.11	3.05	5.15	0.93	1.05	1.10	0.41	1.64
Measurement 4	14.08	2.68	0.82	69.61	0.05	3.09	5.11	0.96	0.99	1.29	0.19	1.25
Average normalised data	**14.21**	**2.67**	**0.81**	**69.02**	**0.09**	**3.00**	**5.05**	**1.02**	**0.99**	**1.13**	**0.66**	**1.44**
Standard deviation	0.16	0.05	0.03	0.50	0.03	0.11	0.09	0.08	0.06	0.11	0.52	0.16
Coefficient of variation (%)	1.16	1.70	3.53	0.73	36.68	3.73	1.78	7.89	5.74	9.44	78.00	11.07
Normalised reference values	**14.30**	**2.66**	**1.00**	**66.56**	**0.09**	**2.87**	**5.03**	**0.79**	**1.00**	**1.09**	**1.17**	**1.75**
Absolute error	-0.09	0.01	-0.19	2.46	0.00	0.13	0.02	0.23	-0.01	0.04	-0.51	-0.31
Relative error (%)	**-0.62**	**0.48**	**-18.66**	**3.70**	**3.07**	**4.53**	**0.46**	**28.56**	**-1.30**	**3.94**	**-43.33**	**-17.79**
20/09/2017												
Measurement 1	14.56	2.49	0.81	68.64	0.09	2.92	4.78	0.92	1.08	1.16	0.90	1.53
Measurement 2	14.54	2.61	0.76	68.64	0.08	2.87	4.85	0.95	1.06	1.02	1.19	1.43
Measurement 3	14.53	2.59	0.76	68.74	0.06	2.86	4.89	0.97	1.00	1.12	0.90	1.50
Measurement 4	14.36	2.63	0.70	69.05	0.12	2.91	5.07	1.04	0.99	1.17	0.57	1.27
Measurement 5	14.37	2.62	0.76	68.67	0.06	2.90	4.95	0.87	1.05	1.22	0.85	1.51
Average normalised data	**14.47**	**2.59**	**0.76**	**68.75**	**0.08**	**2.89**	**4.91**	**0.95**	**1.04**	**1.14**	**0.88**	**1.45**
Standard deviation	0.10	0.06	0.04	0.17	0.02	0.03	0.11	0.06	0.04	0.07	0.22	0.11
Coefficient of variation (%)	0.70	2.14	5.43	0.25	31.43	1.00	2.20	6.50	4.00	6.44	25.23	7.28
Normalised reference values	**14.30**	**2.66**	**1.00**	**66.56**	**0.09**	**2.87**	**5.03**	**0.79**	**1.00**	**1.09**	**1.17**	**1.75**
Absolute error	0.17	-0.07	-0.24	2.19	-0.01	-0.02	-0.12	0.16	0.04	0.05	-0.29	-0.30
Relative error (%)	**1.20**	**-2.74**	**-24.23**	**3.29**	**-11.97**	**0.67**	**-2.47**	**20.19**	**3.54**	**4.35**	**-24.62**	**-17.30**

Table A.2. SEM-EDS data for glass standard Corning A. Not detected: P_2O_5, SO_3, *CoO, NiO, ZnO,* SnO_2, *BaO, PbO,* LiO_2, B_2O_3, V_2O_5, Cr_2O_3, Rb_2O, *SrO,* ZrO_2, Ag_2O, Bi_2O_3.

	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	Fe_2O_3	CoO	CuO	BaO	PbO
12/12/2016												
Measurement 1	1.10	2.60	0.80	34.30	2.80	4.70	1.00	0.33	0.20	1.20	12.30	38.80
Measurement 2	1.00	2.60	0.70	34.40	2.90	4.80	1.20	0.44	0.10	1.00	11.80	39.10
Measurement 3	1.10	2.70	0.80	34.00	2.80	4.90	0.80	0.22	0.10	1.30	12.10	39.30
Measurement 4	1.10	2.60	0.70	34.10	2.90	4.90	0.90	0.33	0.20	1.10	12.00	39.20
Measurement 5	1.10	2.60	0.70	34.30	2.80	5.00	1.00	0.22	0.20	1.10	12.10	39.20
Average normalised data	**1.10**	**2.60**	**0.70**	**34.20**	**2.80**	**4.90**	**1.00**	**0.31**	**0.20**	**1.10**	**12.10**	**39.10**
Standard deviation	0.04	0.04	0.05	0.16	0.05	0.11	0.15	0.09	0.05	0.11	0.18	0.19
Coefficient of variation (%)	4.07	1.72	7.82	0.48	1.96	2.33	14.83	29.88	27.39	10.37	1.50	0.49
Normalised reference values	**1.07**	**2.76**	**0.87**	**34.87**	**2.84**	**5.07**	**0.79**	**0.34**	**0.18**	**1.13**	**11.40**	**36.70**
Absolute error	0.03	-0.16	-0.17	-0.67	-0.04	-0.17	0.21	-0.03	0.02	-0.03	0.70	2.40
Relative error (%)	**2.80**	**-5.80**	**-19.54**	**-1.92**	**-1.41**	**-3.35**	**26.58**	**-9.41**	**11.11**	**-2.65**	**6.14**	**6.54**
02/02/2017												
Measurement 1	1.10	2.70	0.80	34.10	2.80	4.90	0.90	0.22	0.30	1.40	11.80	38.90
Measurement 2	1.00	2.70	0.80	34.20	2.80	4.80	0.70	0.33	0.10	1.30	12.30	39.10
Measurement 3	1.00	2.60	0.70	34.20	2.70	4.90	1.00	0.33	0.20	1.30	11.80	39.20
Measurement 4	1.00	2.60	0.80	34.10	2.70	4.80	1.00	0.33	0.10	1.20	12.20	39.10
Average normalised data	**1.00**	**2.60**	**0.80**	**34.10**	**2.80**	**4.80**	**0.90**	**0.30**	**0.20**	**1.30**	**12.00**	**39.10**
Standard deviation	0.05	0.06	0.05	0.06	0.06	0.06	0.14	0.05	0.10	0.08	0.26	0.13
Coefficient of variation (%)	5.00	2.22	6.25	0.17	2.06	1.20	15.71	18.18	47.87	6.28	2.19	0.32
Normalised reference values	**1.07**	**2.76**	**0.87**	**34.87**	**2.84**	**5.07**	**0.79**	**0.34**	**0.18**	**1.13**	**11.40**	**36.70**
Absolute error	-0.07	-0.16	-0.07	-0.77	-0.04	-0.27	0.11	-0.04	0.02	0.17	0.60	2.40
Relative error (%)	**-6.54**	**-5.80**	**-8.05**	**-2.21**	**-1.41**	**-5.33**	**13.92**	**-11.03**	**11.11**	**15.04**	**5.26**	**6.54**
21/06/2018												
Measurement 1	0.97	2.67	0.68	34.14	2.74	4.82	0.83	0.20	bdl	1.12	12.20	39.66
Measurement 2	0.99	2.47	0.67	34.17	2.75	4.89	0.73	0.37	bdl	1.15	11.93	39.9
Measurement 3	1.03	2.62	0.70	34.19	2.83	4.88	1.15	0.35	bdl	1.19	11.79	39.31

Measurement 4	1.08	2.62	0.65	34.29	2.88	4.98	1.02	0.19	bdl	1.12	11.82	39.39
Measurement 5	1.04	2.63	0.74	34.47	2.78	4.87	0.94	0.10	bdl	1.3	11.95	39.19
Average normalised data	**1.02**	**2.6**	**0.69**	**34.25**	**2.8**	**4.89**	**0.93**	**0.24**	**bdl**	**1.17**	**11.94**	**39.49**
Standard deviation	0.04	0.08	0.03	0.13	0.06	0.06	0.16	0.12		0.08	0.16	0.29
Coefficient of variation (%)	4.24	2.95	4.96	0.39	2.09	1.19	17.55	48.43		6.41	1.35	0.73
Normalised reference values	**1.07**	**2.76**	**0.87**	**34.87**	**2.84**	**5.07**	**0.79**	**0.34**	**0.18**	**1.13**	**11.40**	**36.70**
Absolute error	-0.05	-0.16	-0.18	-0.62	-0.04	-0.18	0.14	-0.10		0.04	0.54	2.79
Relative error (%)	**-4.67**	**-5.80**	**-20.69**	**-1.78**	**-1.41**	**-3.55**	**17.72**	**-28.82**		**3.54**	**4.74**	**7.60**

Table A.3. SEM-EDS data for glass standard Corning C. Not detected: P_2O_5, SO_3, *Cl, MnO, NiO, ZnO,* SnO_2, Sb_2O_5, LiO_2, B_2O_3, V_2O_5, Cr_2O_3, Rb_2O, *SrO,* ZrO_2, Ag_2O, Bi_2O_3.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO	Fe_2O_3	CuO	Sb_2O_3	PbO
12/12/2016															
Measurement 1	1.33	4.08	5.15	57.52	3.67	0.37	0.17	11.58	14.54	0.47	0.55	0.64	0.00	bdl	bdl
Measurement 2	1.46	3.89	5.20	57.20	3.85	0.26	0.17	11.62	14.52	0.46	0.66	0.43	0.31	bdl	bdl
Measurement 3	1.41	3.86	5.17	57.56	3.78	0.32	0.17	11.53	14.43	0.41	0.55	0.54	0.32	bdl	bdl
Measurement 4	1.41	4.08	5.07	57.34	3.75	0.37	0.14	11.52	14.35	0.53	0.52	0.69	0.29	bdl	bdl
Measurement 5	1.35	4.02	5.21	57.43	3.73	0.39	0.18	11.54	14.38	0.66	0.56	0.40	0.20	bdl	bdl
Average normalised data	**1.39**	**3.99**	**5.16**	**57.41**	**3.76**	**0.34**	**0.17**	**11.56**	**14.44**	**0.51**	**0.57**	**0.54**	**0.22**	**bdl**	**bdl**
Standard deviation	0.05	0.10	0.06	0.14	0.07	0.05	0.02	0.04	0.08	0.10	0.05	0.13	0.13		
Coefficient of variation (%)	3.75	2.63	1.08	0.25	1.76	15.39	9.14	0.36	0.58	18.99	9.43	23.84	59.78		
Normalised reference values	**1.20**	**3.94**	**5.30**	**55.24**	**3.93**	**0.30**	**0.16**	**11.30**	**14.80**	**0.38**	**0.55**	0.52	**0.38**	**0.97**	**0.24**
Absolute error	0.19	0.05	-0.14	2.17	-0.17	0.04	0.01	0.26	-0.36	0.13	0.02	0.02	-0.16		
Relative error (%)	**16.00**	**1.17**	**-2.64**	**3.93**	**-4.43**	**14.00**	**3.75**	**2.28**	**-2.41**	**33.16**	**3.27**	**3.65**	**-41.05**		
02/02/2017															
Measurement 1	1.50	4.20	5.20	57.50	3.70	bdl	0.20	11.50	14.30	0.50	0.60	0.44	0.30	0.10	bdl
Measurement 2	1.30	4.10	4.90	57.90	3.80	bdl	0.20	11.60	14.30	0.50	0.60	0.55	0.20	0.10	bdl
Measurement 3	1.40	4.00	5.20	57.60	3.80	bdl	0.20	11.60	14.40	0.60	0.50	0.44	0.10	0.20	bdl
Measurement 4	1.30	4.10	5.20	57.50	3.90	bdl	0.20	11.70	14.50	0.50	0.50	0.55	0.10	0.10	bdl
Measurement 5	1.30	4.10	5.20	57.20	3.70	bdl	0.20	11.60	14.60	0.60	0.60	0.44	0.00	0.40	bdl
Average normalised data	**1.40**	**4.10**	**5.10**	**57.50**	**3.80**	**bdl**	**0.20**	**11.60**	**14.50**	**0.50**	**0.60**	**0.48**	**0.10**	**0.20**	**bdl**
Standard deviation	0.09	0.07	0.13	0.25	0.08		0.00	0.07	0.13	0.05	0.05	0.06	0.11	0.13	
Coefficient of variation (%)	6.39	1.72	2.63	0.44	2.20		0.00	0.61	0.90	10.95	9.13	12.45	114.02	65.19	
Normalised reference values	**1.20**	**3.94**	**5.30**	**55.24**	**3.93**	**0.30**	**0.16**	**11.30**	**14.80**	**0.38**	**0.55**	0.52	**0.38**	**0.97**	**0.24**
Absolute error	0.20	0.16	-0.20	2.26	-0.13		0.04	0.30	-0.30	0.12	0.05	-0.04	-0.28	-0.77	
Relative error (%)	**16.67**	**4.06**	**-3.77**	**4.09**	**-3.31**		**25.00**	**2.65**	**-2.03**	**31.58**	**9.09**	**-6.92**	**-73.68**	**-79.38**	
06/02/2017															
Measurement 1	1.36	4.12	5.13	57.21	3.76	0.17	0.21	11.51	14.39	0.57	0.54	0.52	0.21	0.08	0.28
Measurement 2	1.35	4.02	5.23	57.39	3.52	0.41	0.21	11.36	14.57	0.66	0.51	0.48	0.07	0.15	0.10

Measurement 3	1.32	4.06	5.08	57.20	3.82	0.27	0.18	11.54	14.32	0.65	0.58	0.64	0.06	0.25	0.10
Average normalised data	**1.34**	**4.07**	**5.14**	**57.27**	**3.70**	**0.28**	**0.20**	**11.47**	**14.43**	**0.62**	**0.54**	**0.55**	**0.11**	**0.16**	**0.16**
Standard deviation	0.02	0.05	0.08	0.11	0.16	0.12	0.02	0.10	0.13	0.05	0.04	0.08	0.08	0.09	0.10
Coefficient of variation (%)	1.55	1.24	1.49	0.19	4.29	43.06	8.66	0.84	0.89	7.96	6.50	14.84	76.24	53.40	64.95
Normalised reference values	**1.20**	**3.94**	**5.30**	**55.24**	**3.93**	**0.30**	**0.16**	**11.30**	**14.80**	**0.38**	**0.55**	**0.52**	**0.38**	**0.97**	**0.24**
Absolute error	0.14	0.13	-0.16	2.03	-0.23	-0.02	0.04	0.17	-0.37	0.24	-0.01	0.03	-0.27	-0.81	-0.08
Relative error (%)	**11.67**	**3.30**	**-3.02**	**3.67**	**-5.85**	**-6.67**	**25.00**	**1.50**	**-2.50**	**63.16**	**-1.82**	**5.06**	**-71.05**	**-83.51**	**-33.61**
27/02/2017															
Measurement 1	1.34	3.94	5.08	57.49	3.85	0.31	0.21	11.54	14.43	0.53	0.67	0.43	0.21	bdl	bdl
Measurement 2	1.52	4.02	5.10	57.24	3.76	0.45	0.17	11.58	14.42	0.55	0.57	0.57	0.08	bdl	bdl
Measurement 3	1.17	3.96	5.11	57.64	3.77	0.29	0.18	11.54	14.67	0.44	0.63	0.54	0.12	bdl	bdl
Measurement 4	1.22	4.07	5.09	57.67	3.59	0.32	0.24	11.59	14.58	0.46	0.55	0.53	0.14	bdl	bdl
Average normalised data	**1.31**	**4.00**	**5.10**	**57.51**	**3.74**	**0.34**	**0.20**	**11.56**	**14.53**	**0.50**	**0.61**	**0.52**	**0.14**	**bdl**	**bdl**
Standard deviation	0.16	0.06	0.01	0.20	0.11	0.07	0.03	0.03	0.12	0.05	0.06	0.06	0.05		
Coefficient of variation (%)	11.88	1.48	0.25	0.34	2.92	21.40	15.81	0.23	0.83	10.65	9.03	11.91	38.85		
Normalised reference values	**1.20**	**3.94**	**5.30**	**55.24**	**3.93**	**0.30**	**0.16**	**11.30**	**14.80**	**0.38**	**0.55**	**0.52**	**0.38**	**0.97**	**0.24**
Absolute error	0.11	0.06	-0.20	2.27	-0.19	0.04	0.04	0.26	-0.27	0.12	0.06	0.00	-0.24		
Relative error (%)	**9.17**	**1.52**	**-3.77**	**4.11**	**-4.83**	**13.33**	**25.00**	**2.30**	**-1.82**	**31.58**	**10.91**	**-0.58**	**-63.16**		
21/08/2018															
Measurement 1	1.33	3.81	5.01	56.63	3.76	0.36	0.22	11.98	14.94	0.54	0.58	0.62	0.29	bdl	bdl
Measurement 2	1.06	4.01	4.98	57.27	3.65	0.27	0.20	11.98	15.03	0.47	0.62	0.51	0.00	bdl	bdl
Measurement 3	1.34	3.91	5.12	56.81	3.87	0.21	0.18	11.96	14.97	0.52	0.56	0.46	0.12	bdl	bdl
Measurement 4	1.19	3.81	5.14	57.28	3.63	0.31	0.18	12.09	14.87	0.44	0.55	0.45	0.12	bdl	bdl
Measurement 5	1.34	3.87	4.90	56.95	3.89	0.22	0.22	11.99	14.82	0.50	0.54	0.48	0.32	bdl	bdl
Average normalised data	**1.25**	**3.88**	**5.03**	**56.99**	**3.76**	**0.27**	**0.20**	**12.00**	**14.93**	**0.49**	**0.57**	0.50	**0.17**	**bdl**	**bdl**
Standard deviation	0.12	0.08	0.10	0.29	0.12	0.06	0.02	0.05	0.08	0.04	0.03	0.07	0.13		
Coefficient of variation (%)	9.97	2.14	1.99	0.50	3.20	22.88	10.00	0.43	0.55	8.05	5.55	13.14	78.26		
Normalised reference values	**1.20**	**3.94**	**5.30**	**55.24**	**3.93**	**0.30**	**0.16**	**11.30**	**14.80**	**0.38**	**0.55**	0.52	**0.38**	**0.97**	**0.24**
Absolute error	0.05	-0.06	-0.27	1.75	-0.17	-0.03	0.04	0.70	0.13	0.11	0.02	-0.02	-0.21		

Relative error (%)	**4.33**	**-1.47**	**-5.09**	**3.16**	**-4.33**	**-8.67**	**25.00**	**6.19**	**0.85**	**30.00**	**3.64**	**-3.12**	**-55.26**

Table A.4. SEM-EDS data for glass standard Corning D. Not detected: CoO, NiO, ZnO, SnO_2, BaO, LiO_2, B_2O_3, V_2O_5, Cr_2O_3, Rb_2O, SrO, ZrO_2, Ag_2O, Bi_2O_3.

A.3 METAL

Three reference metals, mounted in resin block, ground and polished, were analysed. AGA 2 and 3 (tables A.5 and A.6) are in-house silver standards, whose values need to be considered nominal and not officially certified. They contain some copper and small amounts of other elements. On the other hand, the CURM brass standard 42.23-2 is certified, and provided by the Bureau of Analysed Samples and contains small quantities of several other elements (table A.7). Combined, these standards cover most of the compositions of metallic prills and other residues from the crucibles analysed in this thesis (cfr section 4.5.2, 4.5.3 and 4.5.4 and 5.5.4). The data for the two AGA standards shows good precision (cv within or slightly above 10%) for copper, silver (present at high concentrations), lead and gold (present at low concentrations), and poor precision for tin, gold and zinc (all present at low concentrations). A good precision (cv below 10%) is recorded for Cu, Zn (present at high concentrations) and tin (present at lower concentrations) in CURM 42.23-2, but poor precision characterises phosphorus, nickel, arsenic, iron, antimony and lead (all present at low levels). The accuracy tends to vary between different standards. The accuracy for copper and tin, for instance, is poor (or very poor) for the AGA standards, but improves considerably to values well below 10% relative error for CURM 42.23-2. Lead, zinc, antimony and iron show values within 20% error, but markedly lower in some cases. Silver has been quantified accurately in both AGA standards, while gold only in AGA 2, with a higher relative error in AGA 3. It is worth noting that the presence of oxygen in the two AGA standards raises a note of caution on the reliability of the accuracy values reported. However, this is not problematic for the sake of the interpretation of the samples and contexts analysed in this thesis. Finally, elements found in low concentrations, such as nickel, arsenic and phosphorus, display a relative error below 20%, except in one case where the error for arsenic reaches around 48%.

	O	Cu	Zn	Ag	Au	Pb
24/02/2017						
Measurement 1	0.80	17.39	0.43	79.77	0.54	1.07
Measurement 2	0.29	19.37	0.38	78.03	0.55	1.38
Measurement 3	1.77	17.69	0.31	78.54	0.58	1.11
Measurement 4	0.94	16.53	0.54	80.39	0.50	1.09
Measurement 5	0.00	16.89	0.57	80.76	0.42	1.36
Measurement 6	0.70	16.09	0.38	80.93	0.62	1.28
Average normalised data		**17.33**	**0.44**	**79.74**	**0.54**	**1.22**
Standard deviation		1.15	0.10	1.20	0.07	0.14
Coefficient of variation (%)		6.66	23.21	1.51	12.94	11.65
Normalised reference values		**10.00**	**0.50**	**87.00**	**0.51**	**1.02**
Absolute error		7.33	-0.07	-7.26	0.03	0.20
Relative error (%)		**73.27**	**-13.35**	**-8.35**	**5.52**	**19.12**

Table A.5. SEM-EDS data for metal standard AGA 2.

	O	Cu	Zn	Ag	Sn	Au	Pb
Measurement 1	0.36	6.69	0.62	89.85	0.24	0.20	2.04
Measurement 2	bdl	6.01	0.92	90.62	0.00	0.45	2.01
Measurement 3	bdl	6.23	0.86	90.16	0.06	0.47	2.23
Measurement 4	0.38	6.22	0.81	89.81	0.32	0.39	2.08
Measurement 5	0.61	5.70	0.92	90.00	0.22	0.52	2.03
Average normalised data		6.17	0.83	90.09	0.17	0.41	2.08
Standard deviation		0.36	0.12	0.33	0.13	0.12	0.09
Coefficient of variation (%)		5.86	15.01	0.36	79.24	30.60	4.27
Normalised reference values		4.91	0.82	90.61	0.92	0.26	1.89
Absolute error		1.26	0.01	-0.52	-0.75	0.15	0.19
Relative error (%)		**25.66**	**1.23**	**-0.58**	**-81.76**	**57.36**	**9.95**

Table A.6. SEM-EDS data for metal standard AGA 3.

	P	Fe	Ni	Cu	Zn	As	Sn	Sb	Pb
04/05/2018									
Measurement 1	0.15	0.37	0.25	74.78	21.28	0.29	1.78	0.38	0.72
Measurement 2	0.19	0.45	0.24	75.03	21.28	0.09	1.80	0.24	0.67
Measurement 3	0.14	0.39	0.11	75.34	21.09	0.45	1.69	0.38	0.40
Measurement 4	0.08	0.45	0.26	75.16	21.11	0.19	1.58	0.45	0.71
Measurement 5	0.12	0.30	0.13	74.88	21.56	0.24	1.99	0.41	0.35
Average normalised data	**0.14**	**0.39**	**0.20**	**75.04**	**21.27**	**0.25**	**1.77**	**0.37**	**0.57**
Standard deviation	0.04	0.06	0.07	0.22	0.19	0.13	0.15	0.08	0.18
Coefficient of variation (%)	28.84	16.05	35.95	0.30	0.89	53.25	8.56	21.40	31.55
Normalised reference values	**0.13**	**0.35**	**0.17**	**74.36**	**22.13**	**0.17**	**1.63**	**0.36**	**0.58**
Absolute error	0.01	0.04	0.03	0.68	-0.86	0.08	0.14	0.01	-0.01
Relative error (%)	**9.38**	**10.17**	**19.05**	**0.91**	**-3.89**	**48.81**	**8.59**	**3.93**	**-0.87**
05/07/2018									
Measurement 1	0.15	0.35	0.22	74.88	21.40	0.19	1.72	0.34	0.76
Measurement 2	0.19	0.54	0.15	74.58	21.54	0.24	1.71	0.19	0.87
Measurement 3	0.12	0.50	0.16	74.85	21.71	0.18	1.55	0.24	0.70
Measurement 4	0.04	0.30	0.13	74.92	21.70	0.19	1.80	0.35	0.56
Measurement 5	0.12	0.40	0.22	74.73	21.75	0.18	1.73	0.37	0.50
Average normalised data	**0.12**	**0.42**	**0.18**	**74.79**	**21.62**	**0.20**	**1.70**	**0.30**	**0.68**
Standard deviation	0.06	0.10	0.04	0.14	0.15	0.03	0.09	0.08	0.15
Coefficient of variation (%)	45.87	23.95	23.11	0.18	0.68	12.55	5.41	26.18	22.02
Normalised reference values	**0.13**	**0.35**	**0.17**	**74.36**	**22.13**	**0.17**	**1.63**	**0.36**	**0.58**
Absolute error	-0.01	0.07	0.01	0.43	-0.51	0.03	0.07	-0.06	0.11
Relative error (%)	**-6.25**	**18.64**	**7.14**	**0.58**	**-2.30**	**19.05**	**4.29**	**-15.73**	**18.26**

Table A.7. SEM-EDS data for metal standard CURM 42.23-2. Not detected: Si, Mn, Bi, Al, S.

APPENDIX B

JAMESTOWN

B.1 SAMPLES DETAILS

A brief description of the samples analysed during the present work is provided in the following table B.1, with pictures of the artefacts and reference to the relevant thesis section where they have been discussed. As the assemblage was found in dumping contexts, no detailed stratigraphic information is available.

	**A-630** Base and wall of rounded crucible with tapering profile and bulging foot. The vessel is the only non-Hessian one among those analysed here. The ceramic appears heavily distorted due to heat exposure. It was used in fire assay of iron-rich ores, discussed in section 4.5.1
	**JR2G** Fragment of flat clear window glass of high lime low alkali type. Part of the Fort's reservoir of waste glass brought from England. Discussed in section 4.5.5
	**JR329A** Wall of Hessian crucible with black glassy residue adhering to the internal wall. In the residues are abundant grains of unreacted feldspar and charcoal imprints. Discussed in section 4.5.4.

	**JR741A** Crucible fragment with white corroded residue on the internal wall and glassy intermediate layer. The fragment may belong to one of the large triangular crucibles that were probably used in glass making (see figure 4.34). Discussed in section 4.5.5.
	**152-JR** Base and wall of very small Hessian crucible of probable triangular shape. Discussed in section 4.5.5.
	**J-crucibles** Wall fragments of seemingly unused Hessian crucibles collected under the same ID. Discussed in section 4.5.1.
	**JR965B** Wall of Hessian crucible with glassy residue adhering to the internal wall and a white corroded layer on the surface. It was used in fire assay of iron and copper-rich ores. Discussed in section 4.5.3

	**JR1115B** Slag lump formed inside of a triangular crucible from the cementation of copper and a tin ore. Discussed in section 4.5.3.1.
	**JR1339C** Wall of Hessian crucible with black and glassy residue adhering to both the internal and the external walls, where it appears much thicker. The vessel was used to assay silver-bearing ores. Discussed in section 4.5.2.
	**JR1400B I and II** Respectively the base and spout of a Hessian crucible used to assay silver-bearing ores. The spout shows a thick greenish copper-rich residue corresponding to the tapping of slag. Discussed in section 4.5.2.
	**JR1416C** Base of Hessian crucible with thin reddish residue adhering to the internal surface. It was used in the fire assay of silver-bearing tin-rich ores. Discussed in section 4.5.2.
	**JR1545C** Wall of Hessian crucible showing a thin black glassy residue on both surfaces. It was used to assay feldspar-rich ores for their content in silver. Discussed in section 4.5.4.

	**JR1957A I** Wall of Hessian crucible showing a grainy black residue on the inner surface. It comes from context “Pit 12” together with numerous other similar fragments, all potentially belonging to the same vessel. It was used to assay feldspar-rich ores for their content in silver. Discussed in section 4.5.4.
	**JR1957A II** Cupel fragment made of pure bone ash and used during the assay activities. Discussed in section 4.5.2.
	**JR2361D** Wall of Hessian crucible with corroded green prills embedded in a think residue on the internal surface. The vessel was used in brass cementation experiments. Discussed in section 4.5.3.1.
	**JR2436B** Wall of Hessian crucible with black grainy residue adhering to the internal surface. Probably used to assay iron-rich minerals. Discussed in section 4.5.5.

	**JR2718J** Wall of Hessian crucible with thin reddish residue on both surfaces. Probably used for casting copper. Discussed in section 4.5.3
	**JR2718M** Wall of Hessian crucible with black grainy residue adhering to the internal surface. Probably used to assay feldspar-rich minerals for their silver content. Discussed in section 4.5.5.
	**JR2718N** Bits of black and white frothy residue probably coming from inside a crucible. Possibly resulting from the same assay operations of feldspar-rich ores found in other samples. Discussed in section 4.5.5.
	**JR2718W** Wall of Hessian crucible of triangular shape with a thick corroded residue on its internal surface. The fragment comes from towards the bottom of the crucible and it is most likely where the lead bullion sank during assay operations. Discussed in section 4.5.5.

B.2 BULK COMPOSITIONS

Table B.1 shows the bulk chemical composition of the ceramic matrices of the crucibles analysed and refers to the results discussed in section 4.5.1. The data in table B.2 contains the bulk composition of the residue layers found within the vessels.

Sample	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	FeO
JR1957A I	**0.67**	**0.52**	**39.28**	**54.19**	**1.50**	**0.28**	**1.90**	**1.35**
Std. dev. (4 ms)	0.16	0.04	0.35	0.54	0.52	0.09	0.16	0.31
Min.	0.54	0.46	38.78	53.40	0.99	0.19	1.73	0.90
Max.	0.91	0.56	39.59	54.59	2.23	0.40	2.05	1.63
JR392A	**0.16**	**0.60**	**39.16**	**54.88**	**1.45**	**0.23**	**1.87**	**1.64**
Std. dev. (3 ms)	0.02	0.04	0.46	0.41	0.19	0.06	0.06	0.17
Min.	0.15	0.57	38.64	54.60	1.33	0.20	1.81	1.52
Max.	0.18	0.65	39.47	55.35	1.67	0.31	1.93	1.83
JR1339C	**0.26**	**0.61**	**38.31**	**55.54**	**1.36**	**bdl**	**1.74**	**2.18**
Std. dev. (3 ms)	0.08	0.06	0.31	0.27	0.11		0.09	0.08
Min.	0.17	0.55	37.98	55.27	1.24		1.65	2.13
Max.	0.34	0.67	38.57	55.81	1.47		1.82	2.26
JR2436B	**1.19**	**0.48**	**37.77**	**55.21**	**2.13**	**0.22**	**1.74**	**1.05**
Std. dev. (3 ms)	0.11	0.12	0.34	0.66	0.15	0.04	0.03	0.06
Min.	1.11	0.36	37.40	54.75	2.04	0.17	1.72	0.99
Max.	1.31	0.59	38.07	55.96	2.31	0.25	1.77	1.09
JR1545C	**0.31**	**0.62**	**37.64**	**56.34**	**1.11**	**0.40**	**1.91**	**1.66**
Std. dev. (3 ms)	0.03	0.03	0.30	0.34	0.03	0.02	0.08	0.18
Min.	0.28	0.60	37.31	56.13	1.07	0.39	1.83	1.50
Max.	0.34	0.65	37.89	56.72	1.13	0.42	1.98	1.86
JR1400BI	**0.25**	**0.56**	**39.31**	**55.01**	**1.21**	**0.25**	**1.82**	**1.59**
Std. dev. (3 ms)	0.04	0.04	0.87	1.23	0.13	0.02	0.05	0.12
Min.	0.20	0.51	38.31	54.21	1.08	0.23	1.77	1.47
Max.	0.28	0.60	39.87	56.43	1.33	0.27	1.88	1.71
JR1400BII	**0.29**	**0.57**	**38.75**	**55.59**	**1.11**	**0.31**	**1.82**	**1.56**
Std. dev. (3 ms)	0.07	0.05	0.26	0.23	0.12	0.02	0.11	0.06
Min.	0.24	0.51	38.51	55.33	1.00	0.29	1.70	1.50
Max.	0.37	0.61	39.03	55.78	1.24	0.32	1.91	1.61
JR1416C	**0.15**	**0.65**	**36.41**	**57.73**	**1.20**	**0.30**	**1.87**	**1.70**
Std. dev. (3 ms)	0.04	0.06	0.91	0.91	0.09	0.02	0.14	0.24
Min.	0.12	0.58	35.44	56.83	1.13	0.28	1.78	1.47
Max.	0.20	0.70	37.23	58.65	1.29	0.32	2.02	1.95
JR965B	**0.26**	**0.61**	**36.78**	**57.63**	**1.06**	**0.35**	**1.75**	**1.56**
Std. dev. (3 ms)	0.02	0.05	1.26	1.40	0.01	0.04	0.09	0.10
Min.	0.25	0.57	35.42	56.38	1.05	0.30	1.67	1.50
Max.	0.28	0.66	37.90	59.14	1.06	0.38	1.85	1.68
JR2361D	**bdl**	**0.64**	**38.33**	**55.67**	**1.47**	**0.19**	**1.81**	**1.90**
Std. dev. (3 ms)		0.05	0.71	0.73	0.21	0.06	0.08	0.10

Min.		0.58	37.78	54.85	1.29	0.12	1.72	1.78
Max.		0.67	39.13	56.22	1.70	0.22	1.88	1.98
JR2718J	**0.18**	**0.56**	**38.87**	**55.11**	**1.40**	**0.28**	**1.84**	**1.76**
Std. dev. (3 ms)	0.08	0.04	0.58	0.61	0.04	0.01	0.11	0.10
Min.	0.09	0.52	38.21	54.72	1.37	0.27	1.72	1.64
Max.	0.25	0.59	39.25	55.82	1.44	0.29	1.94	1.83
JR2718W	**0.21**	**0.60**	**37.17**	**56.69**	**1.58**	**0.35**	**1.85**	**1.54**
Std. dev. (3 ms)	0.06	0.09	0.16	0.39	0.66	0.09	0.05	0.35
Min.	0.15	0.50	36.98	56.35	1.19	0.26	1.81	1.13
Max.	0.26	0.67	37.28	57.12	2.34	0.43	1.91	1.78
JR2718M	**bdl**	**0.57**	**38.89**	**55.79**	**1.12**	**0.29**	**1.76**	**1.57**
Std. dev. (3 ms)		0.02	0.34	0.46	0.05	0.01	0.05	0.08
Min.		0.55	38.64	55.25	1.08	0.28	1.71	1.48
Max.		0.59	39.28	56.05	1.17	0.31	1.80	1.63
JR741A	**0.51**	**0.95**	**25.38**	**64.56**	**3.15**	**0.24**	**1.24**	**3.98**
Std. dev. (3 ms)	0.00	0.11	1.69	1.61	0.46	0.09	0.07	0.22
Min.	0.51	0.86	23.60	63.58	2.88	0.14	1.20	3.74
Max.	0.52	1.07	26.96	66.42	3.68	0.31	1.33	4.17
JR152	**1.55**	**0.41**	**31.45**	**62.66**	**0.25**	**bdl**	**1.84**	**1.83**
Std. dev. (4 ms)	0.74	0.13	0.85	1.15	0.06		0.12	0.22
Min.	0.88	0.28	30.56	61.00	0.20		1.67	1.53
Max.	2.26	0.52	32.39	63.68	0.31		1.92	2.05
J-crucibles	**bdl**	**0.55**	**37.09**	**57.69**	**1.06**	**bdl**	**1.84**	**1.77**
Std. dev. (3 ms)		0.03	0.41	0.43	0.05		0.05	0.05
Min.		0.52	36.82	57.19	1.02		1.79	1.73
Max.		0.57	37.57	58.01	1.12		1.90	1.82
A-630	**0.22**	**0.47**	**23.09**	**70.64**	**1.77**	**0.35**	**1.90**	**1.55**
Std. dev. (3 ms)	0.09	0.05	1.27	1.27	0.17	0.07	0.28	0.08
Min.	0.11	0.41	21.64	69.60	1.66	0.30	1.66	1.48
Max.	0.28	0.51	24.02	72.05	1.97	0.43	2.21	1.64

Table B.1. Average bulk ceramic composition. All results in wt% and normalised to 100%

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO	FeO	NiO_2	CuO	ZnO	As_2O_3	SrO	AgO	SnO_2	BaO	PbO
JR1339C int	**3.90**	**bdl**	**9.27**	**27.70**	**bdl**	**0.97**	**0.51**	**2.13**	**7.92**	**0.43**	**bdl**	**7.99**	**bdl**	**bdl**	**bdl**	**4.42**	**bdl**	**bdl**	**bdl**	**33.97**	**0.80**
Std. dev. (3 ms)	0.81		0.35	1.04		0.11	0.02	0.46	0.68	0.75		1.02				0.25				0.94	0.04
Min.	3.37		8.98	27.07		0.85	0.00	1.78	7.13	1.30		6.82				4.18				32.99	0.78
Max.	4.83		9.65	28.90		1.06	0.00	2.65	8.37	1.30		8.66				4.68				34.86	0.85
JR1339C ext	**4.67**	**2.97**	**13.48**	**45.56**	**0.37**	**bdl**	**0.24**	**5.08**	**8.92**	**0.81**	**0.29**	**6.63**	**0.16**	**bdl**	**bdl**	**0.76**	**bdl**	**bdl**	**bdl**	**9.38**	**0.73**
Std. dev. (2 ms)	0.47	0.21	0.29	0.30	0.01		0.06	0.01	0.21	0.02	0.04	0.36	0.22			0.06				0.11	0.05
Min.	3.05	0.29	13.27	45.34	0.36		0.00	5.07	8.77	0.79	0.26	0.64	0.31			0.52				9.30	0.69
Max.	5.00	3.11	22.37	59.51	0.37		0.00	8.94	9.07	1.67	0.31	6.88	0.31			0.80				9.46	0.76
JR1416C	**bdl**	**0.34**	**12.87**	**56.49**	**1.49**	**0.75**	**bdl**	**bdl**	**5.59**	**0.68**	**bdl**	**6.08**	**bdl**	**3.40**	**1.42**	**bdl**	**bdl**	**0.26**	**3.93**	**1.65**	**5.00**
Std. dev. (3 ms)		0.01	1.18	3.49	0.16	0.16			0.30	0.14		0.73		1.17	0.54			0.45	0.66	0.46	0.25
Min.		0.33	11.51	53.76	1.31	0.63			5.36	0.52		5.38		2.08	0.81			0.00	3.18	1.16	4.74
Max.		0.35	13.56	60.43	1.62	0.93			5.93	0.76		6.83		4.28	1.85			0.78	4.38	2.06	5.23
JR1400BI base	**bdl**	**bdl**	**15.51**	**55.03**	**2.01**	**0.12**	**bdl**	**0.18**	**4.57**	**1.25**	**0.10**	**11.63**	**bdl**	**1.04**	**7.95**	**bdl**	**bdl**	**0.60**	**bdl**	**bdl**	**bdl**
Std. dev. (2 ms)			2.96	3.85	0.38	0.21		0.16	0.68	0.13	0.18	7.32		0.14	3.05			0.17			
Min.			12.70	50.98	1.72	0.00		0.00	4.18	1.12	0.00	4.76		0.93	4.88			0.43			
Max.			18.60	58.65	2.44	0.37		0.28	5.36	1.38	0.31	19.33		1.20	10.97			0.76			
JR1400BII spout	**bdl**	**1.21**	**5.60**	**43.25**	**bdl**	**bdl**	**bdl**	**bdl**	**2.76**	**0.68**	**bdl**	**3.57**	**bdl**	**29.56**	**1.15**	**bdl**	**bdl**	**0.25**	**1.59**	**bdl**	**11.53**
Std. dev. (2 ms)		0.14	0.21	0.03					0.23	0.09		0.01		0.72				0.35	0.10		0.51
Min.		1.11	5.45	43.23					2.60	0.61		3.56		29.05				0.00	1.52		11.17
Max.		1.31	5.75	43.27					2.92	0.74		3.57		30.07				0.50	1.66		11.89
A-630	**0.57**	**2.59**	**7.21**	**48.28**	**1.01**	**bdl**	**bdl**	**4.32**	**9.69**	**0.69**	**0.69**	**24.99**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (2 ms)	0.08	0.25	0.08	0.59	0.02			0.11	0.25	0.11	0.06	1.29									
Min.	0.51	2.41	7.15	47.86	0.99			4.24	9.51	0.61	0.64	24.08									
Max.	0.62	2.76	7.26	48.69	1.02			4.40	9.87	0.77	0.73	25.90									

JR1957A II	**0.32**	**0.27**	**0.24**	**1.37**	**15.68**	**bdl**	**0.98**	**bdl**	**18.67**	**bdl**	**bdl**	**0.37**	**bdl**	**bdl**	**bdl**	**bdl**	**0.68**	**bdl**	**bdl**	**bdl**	**61.42**
Std. dev. (3 ms)	0.18	0.15	0.09	0.14	1.31		0.08		0.27			0.31					0.12				1.99
Min.	0.17	0.14	0.13	1.21	14.40		0.89		18.37			0.04					0.58				59.26
Max.	0.52	0.43	0.29	1.49	17.02		1.02		18.91			0.64					0.81				63.19
JR965B	**9.02**	**1.03**	**3.72**	**42.23**	**1.62**	**bdl**	**0.86**	**6.19**	**4.26**	**0.59**	**0.23**	**22.46**	**bdl**	**6.43**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**1.37**
Std. dev. (4 ms)	0.96	0.11	0.15	3.93	0.16		0.11	1.18	0.60	0.08	0.05	2.38		4.69							0.86
Min.	7.70	0.90	3.55	36.60	1.51		0.69	4.77	3.39	0.50	0.17	20.73		2.57							0.81
Max.	10.00	1.16	3.89	45.10	1.85		0.95	7.65	4.72	0.67	0.28	25.76		13.04							2.62
JR2718J	**1.69**	**1.33**	**6.63**	**61.99**	**0.19**	**bdl**	**bdl**	**17.31**	**7.15**	**0.96**	**0.28**	**2.15**	**bdl**	**0.32**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (5 ms)	0.15	0.18	0.32	3.00	0.21			0.57	1.90	0.11	0.05	0.97		0.46							
Min.	1.53	1.10	6.16	57.98	0.00			16.52	4.94	0.87	0.24	1.06		0.00							
Max.	1.97	1.53	7.09	65.74	0.43			18.07	9.87	1.15	0.38	3.96		1.18							
JR2361D	**2.70**	**3.70**	**7.00**	**53.09**	**0.95**	**0.54**	**0.18**	**10.52**	**17.90**	**0.82**	**0.44**	**0.79**	**bdl**	**0.36**	**1.02**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (4 ms)	0.38	1.31	1.22	0.93	0.15	0.21	0.02	3.31	4.54	0.06	0.08	0.18		0.36	0.16						
Min.	2.31	2.51	5.92	52.27	0.80	0.34	0.15	7.37	13.28	0.76	0.36	0.68		0.00	0.83						
Max.	3.20	5.00	8.30	54.43	1.14	0.79	0.19	13.68	21.87	0.90	0.52	1.06		0.82	1.21						
JR1115B	**0.65**	**0.56**	**15.50**	**59.73**	**bdl**	**bdl**	**bdl**	**2.95**	**0.23**	**1.39**	**bdl**	**3.12**	**bdl**	**13.05**	**bdl**	**bdl**	**bdl**	**bdl**	**2.00**	**bdl**	**0.83**
Std. dev. (4 ms)	0.10	0.08	1.55	1.69				0.95	0.11	0.46		0.45		2.54					1.04		0.37
Min.	0.55	0.46	14.03	57.85				2.10	0.11	0.97		2.47		10.42					1.05		0.45
Max.	0.77	0.64	16.87	61.88				3.85	0.37	1.97		3.48		16.22					3.08		1.27
JR1957A I	**8.55**	**1.79**	**8.71**	**57.81**	**bdl**	**bdl**	**0.75**	**13.96**	**2.62**	**0.70**	**bdl**	**4.48**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**0.47**
Std. dev. (3 ms)	1.74	0.03	0.49	0.59			0.06	2.00	0.60	0.06		0.30									0.02
Min.	6.62	1.76	8.15	57.13			0.68	11.89	1.97	0.65		4.23									0.45
Max.	9.98	1.82	9.03	58.16			0.79	15.88	3.13	0.76		4.81									0.49
JR1545C	**8.73**	**0.63**	**12.20**	**60.41**	**bdl**	**0.23**	**0.61**	**13.14**	**0.88**	**0.75**	**bdl**	**2.43**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (3 ms)	0.63	0.24	2.81	0.91		0.21	0.14	0.21	0.49	0.30		0.94									
Min.	8.06	0.35	9.75	59.58		0.00	0.45	12.89	0.32	0.41		1.35									

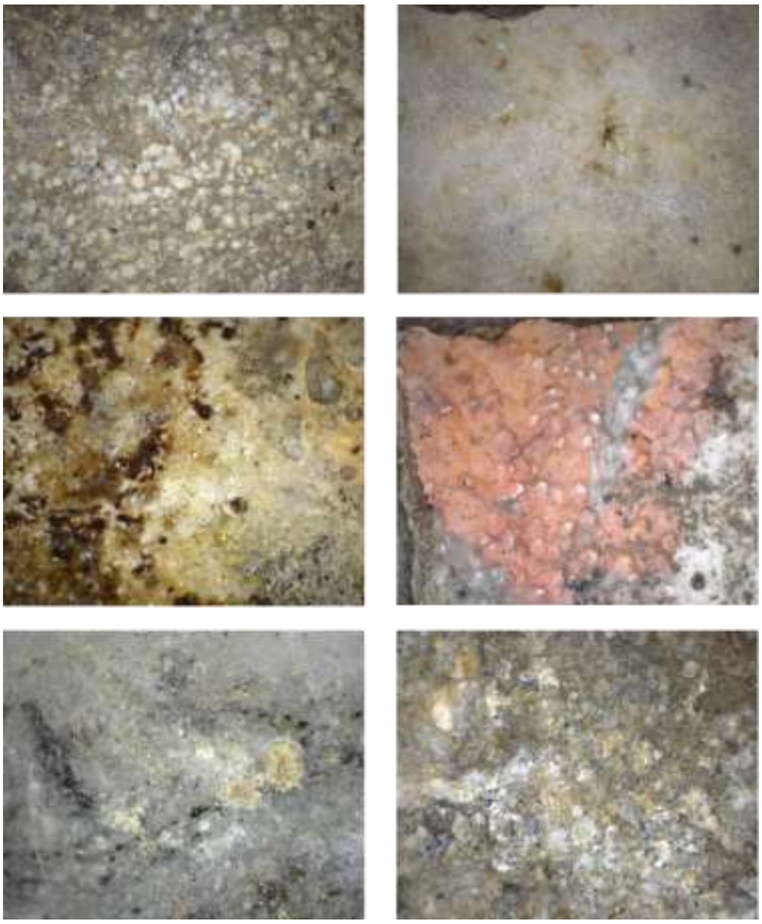
Max.	9.32	0.80	15.27	61.39		0.42	0.72	13.27	1.24	0.95		3.10									
JR392A	**5.31**	**2.40**	**11.16**	**52.09**	**0.41**	**0.65**	**0.48**	**17.85**	**4.59**	**0.85**	**bdl**	**2.52**	**bdl**	**bdl**	**bdl**	**0.41**	**bdl**	**bdl**	**bdl**	**bdl**	**1.28**
Std. dev. (3 ms)	0.46	0.15	0.34	2.08	0.09	0.07	0.02	1.04	0.53	0.10		0.16				0.06					0.24
Min.	4.78	2.24	10.78	50.71	0.33	0.60	0.47	16.66	4.01	0.78		2.43				0.32					1.01
Max.	5.57	2.55	11.45	54.49	0.51	0.73	0.50	18.51	5.03	0.96		2.70				0.45					1.44
JR2G	**0.84**	**2.80**	**1.63**	**63.54**	**1.91**	**0.35**	**bdl**	**4.50**	**22.60**	**0.26**	**0.69**	**0.88**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (3 ms)	0.13	0.08	0.06	0.20	0.09	0.05		0.03	0.06	0.05	0.12	0.05									
Min.	0.75	2.73	1.57	63.33	1.86	0.30		4.48	22.55	0.22	0.57	0.82									
Max.	0.98	2.88	1.68	63.73	2.01	0.40		4.53	22.67	0.32	0.80	0.91									
152-JR?	**bdl**	**0.33**	**1.53**	**bdl**	**14.51**	**4.32**	**0.31**	**bdl**	**5.63**	**bdl**	**bdl**	**36.15**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**19.84**	**bdl**	**0.87**	**16.51**
JR2436B	**1.18**	**3.95**	**14.88**	**50.34**	**1.30**	**bdl**	**bdl**	**3.17**	**20.15**	**0.93**	**0.61**	**3.49**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (3 ms)	0.14	1.09	1.97	1.37	0.52			0.27	4.07	0.09	0.11	2.02									
Min.	1.08	2.86	13.00	49.34	0.76			2.87	15.71	0.84	0.49	1.58									
Max.	1.35	5.04	16.92	51.90	1.80			3.38	23.73	1.02	0.72	5.60									
JR2718M	**1.81**	**1.60**	**14.76**	**67.02**	**0.12**	**0.33**	**0.33**	**4.36**	**5.15**	**0.64**	**bdl**	**3.88**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (3 ms)	0.33	0.17	1.21	1.51	0.21	0.29	0.05	1.60	0.19	0.19		0.63									
Min.	1.45	1.41	13.74	65.32	0.00	0.00	0.30	2.95	4.95	0.42		3.20									
Max.	2.11	1.70	16.09	68.22	0.37	0.53	0.38	6.10	5.32	0.78		4.44									
JR2718N	**1.03**	**0.58**	**6.26**	**82.96**	**bdl**	**bdl**	**0.19**	**2.51**	**3.15**	**0.79**	**bdl**	**2.55**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (2 ms)	0.06	0.04	0.52	0.43			0.03	0.42	1.10	0.06		0.26									
Min.	0.99	0.55	5.89	82.65			0.17	2.21	2.37	0.75		2.36									
Max.	1.07	0.60	6.62	83.26			0.21	2.81	3.93	0.83		2.73									
JR741A (white)	**1.24**	**7.57**	**7.86**	**58.99**	**1.94**	**bdl**	**0.22**	**5.02**	**11.97**	**1.44**	**1.20**	**2.55**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
JR741A (intermediate)	**2.08**	**0.87**	**18.04**	**60.61**	**bdl**	**bdl**	**0.10**	**8.18**	**8.17**	**0.96**	**bdl**	**0.98**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (3 ms)	0.19	0.24	1.16	0.64			0.02	0.25	0.75	0.12		0.13									
Min.	1.90	0.60	17.26	59.89			0.08	7.98	7.31	0.83		0.89									

Max.	2.28	1.04	19.37	61.13			0.12	8.46	8.71	1.05		1.13

Table B.2. Average bulk residue composition. All results in wt% and normalised to 100%

APPENDIX C

ASHMOLEAN LABORATORY



C.1 SAMPLE DETAILS

Below is a brief description of the samples analysed for the present work, with pictures of the artefacts after sampling and reference to the relevant section where they have been discussed.

	**AN1999.205.9d** Wall of rounded beaker-type crucible with tapering profile showing a blue lead crystal residue adhering to its internal wall. Discussed in section 5.5.2.1.
	**AN1999.205.14** Base and wall of rounded beaker-type crucible with tapering profile showing a thick glassy residue adhering to its internal base and wall and probably related to an opaque white glass recipe. The sample analysed in section 5.5.2.1 comes from the green area. No analyses are available for the white area.

	**AN1999.205.17** Wall and rim of rounded beaker-type crucible with tapering profile showing a thin layer of yellowish-green glass of lead crystal composition adhering to its internal wall. Discussed in section 5.5.2.1. The same sample had been previously analysed by Martinón-Torres in 2012.
	**AN1999.205.18** Wall and rim of drug jar with rounded body and closed profile showing areas of orange discolouration on its internal walls. Discussed in section 5.5.4
	**AN1999.205.19** Base and wall of rounded beaker-type crucible with tapering profile showing a black dripping residue running down the external wall. Discussed in section 5.5.3

	**AN1999.205.21** Base and wall of ceramic cucurbit potentially used for zinc distillation experiments, showing a continuous black glassy layer covering its internal surface. Discussed in section 5.5.3.
	**AN1999.205.22** Wall of tall closed-profile crucible used in zinc distillation experiments showing patches of a white crust of zinc oxide with metallic zinc core. Discussed in section 5.5.3
	**AN1999.205.23** Wall and rim of triangular crucible most likely made in Hesse, Germany, showing no signs of use. Discussed in section 5.5.1

	**AN1999.205.24** Wall and rim of triangular crucible most likely made in Hesse, Germany, showing no signs of use. Discussed in section 5.5.1
	**AN1999.205.25** Wall and rim of triangular crucible most likely made in Hesse, Germany, showing a greenish-yellow glassy residue running down the external wall. The residue is an antimony-silicate glass and may relate to the production of opaque white glass through the addition of stibnite (Sb_2S_3). Discussed in section 5.5.2.2
	**AN1999.205.27** Wall and rim of tall closed-profile crucible used in zinc distillation experiments showing patches of a white crust of zinc oxide with metallic zinc core. Only a small piece of the crust was scraped off in this case, since the crucible is the same as AN1999.205.22. Discussed in section 5.5.3.

	**AN1999.205.28** Wall of graphitic crucible with characteristic black ceramic paste, most likely a German import from Bavaria and showing extended distortion of the ceramic due to the presence of corrosive salts in the charge. The vessel may have been used in transmutation experiments or for a medical recipe. Discussed in section 5.5.4
	**AN1999.205.29** Lump of a yellowish-white substance possibly the result of spillage from a vessel. The calcium sulfate composition is of uncertain origin. Discussed in section 5.5.4
	**AN1999.205.30** Wall of rounded beaker-type crucible with tapering profile showing a thin continuous lead-based glassy residue adhering to its internal wall and probably related to an opaque white glass recipe. Discussed in section 5.5.2.1

	**AN1999.205.31** Wall of triangular crucible most likely made in Hesse, Germany, showing extended distortion of the ceramic. Discussed in section 5.5.4
	**AN1999.205.32** Wall of ceramic retort potentially used for zinc distillation experiments and showing a white slip covering the internal surface. Discussed in section 5.5.3

C.2 BULK COMPOSITIONS

Table C.1 shows the bulk chemical composition of the ceramic matrices from the vessels analysed. The data in the following table C.2 refers to the bulk composition of the residue layers within the ceramic vessels.

Sample	Na_2O	MgO	Al_2O_3	SiO_2	Cl	SO_3	K_2O	CaO	TiO_2	FeO	ZnO	AgO
AN.1999.205.9d	**0.32**	**0.66**	**36.55**	**56.84**	**bdl**	**bdl**	**1.82**	**0.24**	**1.59**	**1.99**	**bdl**	**bdl**
Std. dev. (3 ms)	0.04	0.06	1.03	1.01			0.22	0.01	0.13	0.07		
Min.	0.29	0.59	35.36	56.10			1.59	0.23	1.44	1.92		
Max.	0.36	0.70	37.19	58.00			2.02	0.26	1.67	2.07		
AN.1999.205.17	**0.34**	**0.85**	**34.79**	**58.26**	**bdl**	**bdl**	**2.08**	**0.21**	**1.27**	**2.20**	**bdl**	**bdl**
Std. dev. (3 ms)	0.06	0.11	3.03	2.79			0.35	0.01	0.25	0.18		
Min.	0.28	0.74	31.61	55.60			1.85	0.21	1.03	2.04		
Max.	0.38	0.97	37.64	61.16			2.48	0.22	1.53	2.39		
AN.1999.205.18	**0.43**	**0.41**	**29.21**	**63.97**	**bdl**	**bdl**	**1.82**	**0.34**	**2.31**	**1.51**	**bdl**	**bdl**
Std. dev. (4 ms)	0.07	0.25	4.97	3.83			0.31	0.14	0.16	0.79		
Min.	0.36	0.17	21.82	61.88			1.63	0.23	2.15	0.82		
Max.	0.53	0.71	32.14	69.71			2.28	0.54	2.50	2.27		
AN.1999.205.19	**0.33**	**0.78**	**32.56**	**60.18**	**bdl**	**bdl**	**2.23**	**0.32**	**1.29**	**2.30**	**bdl**	**bdl**
Std. dev. (3 ms)	0.03	0.07	4.08	3.21			0.27	0.13	0.16	0.52		
Min.	0.29	0.71	27.99	57.77			2.03	0.22	1.14	1.99		
Max.	0.35	0.85	35.84	63.82			2.54	0.46	1.45	2.89		
AN.1999.205.21	**0.85**	**0.37**	**30.45**	**63.61**	**bdl**	**bdl**	**2.17**	**0.18**	**1.36**	**1.02**	**bdl**	**bdl**
Std. dev. (3 ms)	0.25	0.06	0.62	0.39			0.33	0.02	0.25	0.12		
Min.	0.59	0.34	29.95	63.18			1.96	0.16	1.07	0.91		
Max.	1.07	0.44	31.15	63.93			2.55	0.19	1.51	1.15		
AN.1999.205.22	**0.24**	**0.51**	**32.15**	**60.84**	**bdl**	**bdl**	**2.17**	**0.33**	**1.42**	**2.07**	**0.28**	**bdl**

Std. dev. (3 ms)	0.03	0.08	1.41	1.85			0.25	0.03	0.24	0.32	0.25	
Min.	0.22	0.42	30.63	59.58			1.91	0.31	1.23	1.88	0.00	
Max.	0.27	0.57	33.41	62.96			2.41	0.36	1.69	2.44	0.50	
AN.1999.205.23	**bdl**	**0.65**	**39.51**	**54.76**	**bdl**	**bdl**	**1.24**	**0.34**	**1.83**	**1.67**	**bdl**	**bdl**
Std. dev. (3 ms)	bdl	0.06	0.35	0.28			0.05	0.03	0.05	0.05		
Min.		0.59	39.24	54.44			1.19	0.31	1.78	1.64		
Max.		0.72	39.90	54.94			1.29	0.38	1.89	1.73		
AN.1999.205.24	**bdl**	**0.58**	**38.08**	**55.67**	**bdl**	**bdl**	**1.85**	**0.25**	**1.83**	**1.73**	**bdl**	**bdl**
Std. dev. (3 ms)		0.08	0.81	0.56			0.77	0.13	0.14	0.18		
Min.		0.51	37.27	55.06			1.41	0.09	1.70	1.53		
Max.		0.67	38.89	56.16			2.74	0.33	1.97	1.88		
AN.1999.205.25	**0.20**	**0.58**	**38.75**	**54.93**	**bdl**	**bdl**	**1.66**	**0.37**	**1.81**	**1.70**	**bdl**	**bdl**
Std. dev. (3 ms)	0.03	0.08	0.51	0.36			0.27	0.03	0.09	0.24		
Min.	0.18	0.49	38.16	54.61			1.38	0.35	1.72	1.42		
Max.	0.23	0.64	39.08	55.33			1.93	0.41	1.91	1.89		
AN.1999.205.28	**0.35**	**0.54**	**21.94**	**53.95**	**0.05**	**0.63**	**17.81**	**bdl**	**1.89**	**2.55**	**bdl**	**0.29**
Std. dev. (3ms)	0.34	0.06	2.18	1.09	0.19	0.09	1.89		1.16	0.65		0.28
Min.	0.00	0.51	20.40	52.74	0.50	0.00	15.75		1.18	2.04		0.00
Max.	0.67	0.61	24.44	54.84	0.84	0.16	19.46		3.23	3.28		0.55
AN.1999.205.30	**0.22**	**0.64**	**37.19**	**56.31**	**bdl**	**bdl**	**2.13**	**0.40**	**1.35**	**1.76**	**bdl**	**bdl**
Std. dev. (4 ms)	0.08	0.09	3.16	3.36			0.15	0.09	0.19	0.15		
Min.	0.12	0.57	32.60	53.56			1.95	0.30	1.18	1.62		
Max.	0.31	0.76	39.31	60.98			2.29	0.50	1.62	1.91		
AN.1999.205.31	**0.21**	**0.66**	**38.70**	**55.29**	**bdl**	**bdl**	**1.39**	**0.38**	**1.78**	**1.58**	**bdl**	**bdl**
Std. dev. (3 ms)	0.04	0.06	0.46	0.57			0.06	0.05	0.08	0.10		
Min.	0.16	0.60	38.40	54.64			1.33	0.32	1.71	1.51		
Max.	0.24	0.71	39.23	55.66			1.45	0.42	1.86	1.70		
AN.1999.205.32	**0.26**	**0.74**	**34.04**	**59.28**	**bdl**	**bdl**	**2.57**	**0.21**	**1.19**	**1.71**	**bdl**	**bdl**
Std. dev. (4 ms)	0.05	0.17	3.80	3.62			0.45	0.16	0.23	0.52		
Min.	0.21	0.50	30.40	54.38			2.12	0.00	0.97	1.24		
Max.	0.31	0.89	38.93	62.52			3.19	0.38	1.47	2.46		

OX1409	**0.59**	**0.78**	**30.93**	**60.65**	**0.45**	**bdl**	**2.05**	**0.28**	**2.01**	**2.25**	**bdl**	**bdl**

Table C.1. Average ceramic compositions. All results in wt% and normalised to 100%

Sample	Na2O	MgO	Al_2O_3	SiO_2	P_2O_5	Cl	SO_3	K_2O	CaO	TiO_2	FeO	CuO	AgO	SnO_2	Sb_2O_3	PbO
AN.1999.205.9d	**bdl**	**bdl**	**0.08**	**51.27**	**bdl**	**0.18**	**bdl**	**11.12**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**37.35**
Std. dev. (3 ms)			0.14	0.26		0.09		0.28								0.43
Min.			0.00	51.12		0.13		10.80								36.96
Max.			0.25	51.57		0.28		11.34								37.81
AN.1999.205.17	**0.85**	**bdl**	**bdl**	**59.90**	**bdl**	**0.32**	**bdl**	**11.81**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**27.12**
Std. dev. (3 ms)	0.14			1.41		0.05		0.14								1.53
Min.	0.71			58.29		0.28		11.71								26.11
Max.	0.99			60.91		0.37		11.97								28.87
AN.1999.205.14	**12.51**	**bdl**	**bdl**	**52.51**	**bdl**	**0.07**	**bdl**	**bdl**	**bdl**	**bdl**	**0.77**	**bdl**	**bdl**	**1.77**	**bdl**	**32.38**
Std. dev. (3 ms)	0.06			0.45		0.12					0.16			0.46		0.32
Min.	12.44			52.11		0.00					0.62			1.36		32.13
Max.	12.55			53.00		0.20					0.94			2.28		32.74
AN.1999.205.25	**0.07**	**0.32**	**6.65**	**38.86**	**bdl**	**bdl**	**0.56**	**17.34**	**3.30**	**0.45**	**1.37**	**bdl**	**bdl**	**bdl**	**31.09**	**bdl**
Std. dev. (6 ms)	0.12	0.17	0.43	0.80			0.17	1.00	1.44	0.13	0.19				1.80	
Min.	0.00	0.00	6.07	37.88			0.36	15.73	1.50	0.22	1.18				28.13	
Max.	0.30	0.46	7.28	39.99			0.78	18.54	5.72	0.55	1.66				32.96	
AN.1999.205.30	**0.65**	**bdl**	**5.23**	**37.97**	**bdl**	**bdl**	**bdl**	**14.29**	**1.00**	**bdl**	**0.58**	**bdl**	**bdl**	**bdl**	**bdl**	**40.29**
Std. dev. (3 ms)	0.05		0.55	0.80				0.23	0.06		0.06					1.44
Min.	0.60		4.89	37.26				14.07	0.94		0.52					38.66
Max.	0.69		5.86	38.84				14.53	1.06		0.64					41.39
AN.1999.205.21	**9.05**	**1.55**	**22.62**	**57.50**	**0.26**	**bdl**	**bdl**	**4.78**	**2.88**	**1.07**	**0.30**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (6 ms)	0.94	1.10	2.11	2.06	0.20			0.86	2.26	0.27	0.26					
Min.	7.16	0.58	20.93	55.12	0.00			3.69	0.90	0.89	0.00					
Max.	10.20	3.03	26.69	61.35	0.54			6.25	6.21	1.66	0.77					

AN.1999.205.19	**0.51**	**1.31**	**21.53**	**57.49**	**0.44**	**bdl**	**bdl**	**1.86**	**2.91**	**1.15**	**12.80**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (3 ms)	0.03	0.08	0.06	0.28	0.04			0.02	0.06	0.09	0.29					
Min.	0.48	1.25	21.47	57.31	0.40			1.85	2.84	1.06	12.47					
Max.	0.53	1.40	21.59	57.82	0.49			1.88	2.96	1.22	13.02					
AN.1999.205.28	**bdl**	**0.58**	**18.84**	**52.78**	**0.67**	**bdl**	**bdl**	**16.13**	**2.77**	**1.91**	**4.93**	**1.41**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (3 ms)		0.09	0.50	0.55	0.07			0.37	0.10	0.26	0.54	0.48				
Min.		0.51	18.36	52.43	0.59			15.71	2.69	1.61	4.37	0.89				
Max.		0.68	19.36	53.42	0.72			16.41	2.88	2.06	5.45	1.84				
AN.1999.205.18	**0.07**	**bdl**	**17.68**	**62.06**	**0.47**	**0.07**	**bdl**	**1.23**	**8.87**	**0.92**	**8.65**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (4 ms)	0.14		1.90	1.07	0.55	0.13		0.14	0.30	0.15	2.40					
Min.	0.00		15.77	60.48	0.00	0.00		1.02	8.51	0.77	6.38					
Max.	0.28		19.88	62.80	1.08	0.27		1.30	9.20	1.12	11.85					
31	**0.31**	**0.97**	**16.58**	**55.47**	**0.82**	**bdl**	**bdl**	**1.15**	**18.82**	**0.97**	**4.91**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**

Table C.2. Average bulk residue composition. All results in wt% and normalised to 100%

C.3 COMPOSITION OF METALLIC PHASES

Data in table C.3 refers to the metallic phases found in the residues of the crucibles analysed. AN1999.205.22 and AN1999.205.27 represent the core of metallic zinc found inside the white crust stuck to the vessels' walls, and for this reason an average of several measurements from the same pool of metal is presented. On the other hand, sample AN1999.205.28 contains many small prills, and their single composition is given here.

Sample	O	P	S	Fe	Ni	Cu	Zn	Ag	Cd	Ba	Au	Pb
AN.1999.205.22	**1.27**	**bdl**	**bdl**	**bdl**	**0.04**	**bdl**	**98.69**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**
Std. dev. (4 ms)	0.22				0.07		0.23					
Min.	1.10				0.00		98.42					
Max.	1.58				0.15		98.88					
AN.1999.205.22 (segregation)	**9.44**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**11.40**	**bdl**	**bdl**	**bdl**	**bdl**	**78.60**
AN.1999.205.27	**1.92**	**bdl**	**bdl**	**bdl**	**0.02**	**bdl**	**97.96**	**bdl**	**0.10**	**bdl**	**bdl**	**bdl**
Std. dev. (5 ms)	0.18				0.05		0.28		0.14			
Min.	1.65				0.00		97.56		0.00			
Max.	2.08				0.11		98.35		0.26			
AN.1999.205.27 (segregation)	**5.30**	**bdl**	**bdl**	**bdl**	**bdl**	**bdl**	**27.25**	**bdl**	**0.31**	**bdl**	**bdl**	**66.71**
Std. dev. (2 ms)	1.56						19.20		0.05			21.35
Min.	4.19						13.67		0.27			51.61
Max.	6.40						40.82		0.34			81.80
AN.1999.205.28	**bdl**	**bdl**	**0.27**	**bdl**	**bdl**	**bdl**	**bdl**	**97.98**	**bdl**	**bdl**	**bdl**	**bdl**
	9.14	**bdl**	**12.24**	**0.28**	**bdl**	**0.98**	**bdl**	**75.59**	**bdl**	**bdl**	**1.76**	**bdl**
	23.37	**bdl**	**7.26**	**0.94**	**bdl**	**26.33**	**bdl**	**37.34**	**bdl**	**bdl**	**bdl**	**bdl**
	34.51	**0.13**	**7.91**	**1.26**	**bdl**	**8.40**	**bdl**	**44.88**	**bdl**	**1.28**	**bdl**	**bdl**
	2.00	**bdl**	**14.12**	**0.34**	**bdl**	**0.58**	**bdl**	**81.92**	**bdl**	**bdl**	**bdl**	**bdl**
	9.98	**0.38**	**16.91**	**2.83**	**bdl**	**41.67**	**bdl**	**23.43**	**bdl**	**bdl**	**bdl**	**bdl**
	8.34	**bdl**	**12.14**	**0.89**	**bdl**	**47.56**	**bdl**	**27.65**	**bdl**	**bdl**	**bdl**	**bdl**
	40.66	**0.47**	**6.64**	**2.55**	**bdl**	**20.97**	**bdl**	**9.56**	**bdl**	**bdl**	**bdl**	**bdl**

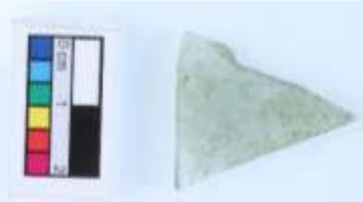
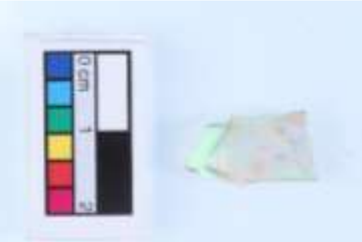
Table C.3. Composition of metallic phases in crucible residues.

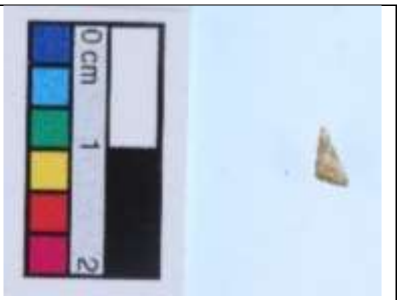
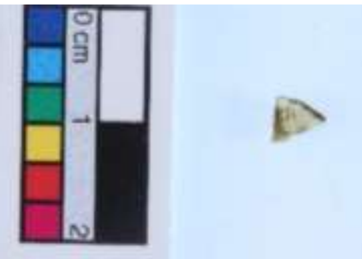
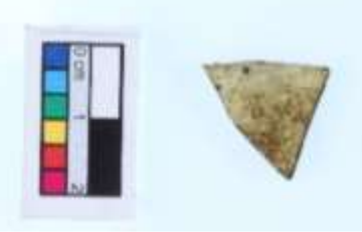
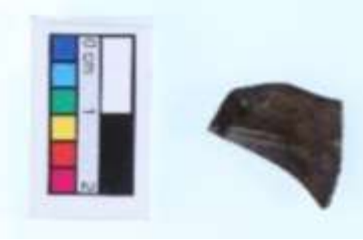
APPENDIX D

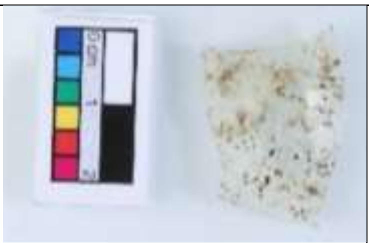
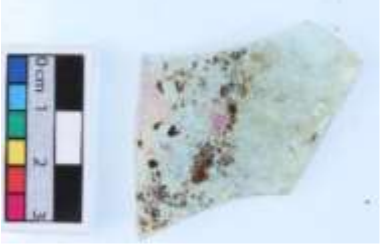
OBERSTOCKSTALL GLASS

D.1 SAMPLE DETAILS

A total of 34 samples were analysed with SEM-EDS, following the analytical protocols outlined in section 3.2.3. Pictures of the glass fragments prior to sampling are reported in the following table. Samples OB 1, OB3, OB6, OB 15, OB 21 and OB 25 were already embedded in resin blocks when the present work started and consequently no pre-sampling picture is available for these. Sample OB 20 displays a thick layer of clay, known as lute, adhering on its external surface and meant to protect the vessel from breaking during distillation.

OB 1	 OB 2	OB 3
 OB 4	 OB 5	OB 6
 OB 7	 OB 8	 OB 9

 OB 10	 OB 11	 OB 12
 OB 13	 OB 14	OB 15
 OB 16	 OB 17	 OB 18
 OB 19	 OB 20	OB 21
 OB 22	 OB 23	 OB 24

OB 25	 **OB 26**	 **OB 27**
 **OB 28**	 **OB 29**	 **OB 30**
 **OB 31**	 **OB 32**	 **OB 33**
 **OB 34**		

D.2 BULK GLASS COMPOSITION

The following table D.2 displays the bulk chemical composition of the glass fragments recovered at the site of the laboratory. The data is discussed in section 6.1.2 of this thesis.

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	Cl	SO_3	K_2O	CaO	TiO_2	MnO	FeO
OB 1	**0.22**	**3.17**	**1.34**	**51.56**	**0.94**	**bdl**	**0.26**	**21.12**	**20.27**	**bdl**	**0.64**	**0.47**
Std. dev. (4 ms)	0.10	0.04	0.09	0.34	0.06		0.02	0.21	0.09		0.10	0.11
Min.	0.09	3.13	1.21	51.15	0.86		0.24	20.97	20.16		0.53	0.32
Max.	0.33	3.22	1.40	51.97	1.01		0.29	21.41	20.35		0.73	0.57
OB 2	**2.32**	**2.67**	**1.71**	**62.62**	**0.79**	**0.16**	**bdl**	**12.28**	**16.13**	**bdl**	**0.82**	**0.51**
Std. dev. (4 ms)	0.04	0.09	0.06	0.16	0.04	0.01		0.16	0.08		0.07	0.06
Min.	2.26	2.55	1.64	62.41	0.75	0.15		12.12	16.02		0.73	0.42
Max.	2.34	2.76	1.77	62.78	0.85	0.17		12.42	16.22		0.88	0.56
OB 3	**10.78**	**3.39**	**1.63**	**66.13**	**0.27**	**0.25**	**0.59**	**4.62**	**10.97**		**0.75**	**0.63**
Std. dev. (4 ms)	0.04	0.12	0.07	0.39	0.04	0.06	0.02	0.11	0.15		0.08	0.05
Min.	10.74	3.28	1.53	65.79	0.21	0.18	0.56	4.48	10.81		0.67	0.55
Max.	10.83	3.56	1.71	66.48	0.30	0.32	0.61	4.74	11.10		0.83	0.66
OB 4	**0.39**	**2.97**	**1.99**	**49.55**	**1.17**	**bdl**	**0.32**	**21.08**	**21.15**	**bdl**	**0.77**	**0.60**
Std. dev. (4 ms)	0.05	0.09	0.07	0.24	0.09		0.05	0.09	0.12		0.05	0.06
Min.	0.34	2.88	1.90	49.36	1.07		0.27	20.95	21.02		0.73	0.54
Max.	0.44	3.09	2.06	49.87	1.27		0.38	21.16	21.26		0.83	0.68
OB 5	**10.12**	**3.22**	**1.21**	**66.54**	**0.17**	**0.27**	**0.56**	**6.09**	**10.5**	**bdl**	**0.76**	**0.56**
Std. dev. (4 ms)	0.01	0.07	0.05	0.09	0.04	0.08	0.02	0.06	0.11		0.07	0.05
Min.	10.11	3.16	1.17	66.46	0.14	0.17	0.54	6.04	10.4		0.71	0.49
Max.	10.13	3.31	1.27	66.63	0.23	0.33	0.58	6.17	10.6		0.85	0.6
OB 6	**11.18**	**3.35**	**1.24**	**66.94**	**0.22**	**0.32**	**0.60**	**3.94**	**10.77**	**bdl**	**0.81**	**0.63**

Std. dev. (4 ms)	0.09	0.05	0.07	0.21	0.04	0.07	0.04	0.05	0.08		0.02	0.07
Min.	11.07	3.29	1.15	66.71	0.19	0.26	0.55	3.87	10.65		0.77	0.56
Max.	11.27	3.41	1.30	67.16	0.26	0.40	0.66	3.98	10.83		0.83	0.72
OB 7	**11.06**	**3.39**	**1.22**	**66.9**	**0.21**	**0.29**	**0.61**	**3.99**	**10.84**	**bdl**	**0.93**	**0.56**
Std. dev. (4 ms)	0.20	0.07	0.05	0.30	0.06	0.05	0.06	0.06	0.11		0.07	0.05
Min.	10.89	3.30	1.15	66.6	0.12	0.24	0.59	3.95	10.73		0.83	0.5
Max.	11.33	3.47	1.28	67.2	0.25	0.36	0.63	4.08	10.99		1	0.63
OB 8	**0.57**	**2.58**	**3.5**	**50.29**	**1.03**	**bdl**	**0.29**	**20.95**	**19.04**	**0.29**	**0.41**	**1.05**
Std. dev. (4 ms)	0.05	0.13	0.10	0.21	0.05		0.03	0.09	0.12	0.05	0.02	0.08
Min.	0.53	2.45	3.40	50.08	0.99		0.25	20.85	18.94	0.24	0.39	0.96
Max.	0.63	2.72	3.62	50.50	1.09		0.33	21.06	19.20	0.35	0.44	1.15
OB 9	**10.75**	**3.26**	**1.45**	**66.57**	**0.24**	**0.24**	**0.5**	**4.38**	**11.19**	**bdl**	**0.84**	**0.59**
Std. dev. (4 ms)	0.15	0.11	0.07	0.16	0.05	0.1	0.01	0.07	0.07		0.02	0.07
Min.	10.6	3.14	1.36	66.4	0.17	0.11	0.48	4.28	11.13		0.82	0.5
Max.	10.93	3.41	1.51	66.75	0.28	0.33	0.51	4.42	11.27		0.86	0.68
OB 10	**11.06**	**3.38**	**1.32**	**67.11**	**0.24**	**0.26**	**0.61**	**3.83**	**10.7**	**bdl**	**0.91**	**0.6**
Std. dev. (5 ms)	0.13	0.09	0.03	0.26	0.09	0.09	0.02	0.02	0.08		0.04	0.04
Min.	10.88	3.28	1.29	66.73	0.15	0.13	0.57	3.8	10.64		0.85	0.53
Max.	11.24	3.51	1.36	67.35	0.37	0.37	0.62	3.85	10.84		0.95	0.64
OB 11	**0.19**	**2.93**	**1.25**	**52.58**	**0.88**	**bdl**	**0.33**	**20.8**	**19.77**	**bdl**	**0.85**	**0.41**
Std. dev. (5 ms)	0.06	0.06	0.02	0.11	0.04		0.06	0.13	0.15		0.10	0.06
Min.	0.13	2.84	1.22	52.44	0.84		0.23	20.60	19.55		0.70	0.36
Max.	0.26	2.98	1.28	52.75	0.92		0.39	20.94	19.90		0.93	0.5
OB 12	**0.22**	**2.83**	**1.20**	**52.45**	**0.85**	**bdl**	**0.30**	**20.91**	**19.92**	**bdl**	**0.87**	**0.45**
Std. dev. (4 ms)	0.03	0.08	0.03	0.16	0.06		0.04	0.08	0.14		0.10	0.05
Min.	0.20	2.74	1.16	52.23	0.82		0.26	20.85	19.72		0.79	0.40
Max.	0.26	2.92	1.22	52.59	0.94		0.34	21.02	20.03		1.02	0.52
OB 13	**0.51**	**2.61**	**3.52**	**50.24**	**1.01**	**bdl**	**0.28**	**20.98**	**19.10**	**0.28**	**0.40**	**1.06**
Std. dev. (4 ms)	0.07	0.02	0.11	0.22	0.08		0.06	0.07	0.17	0.04	0.05	0.06
Min.	0.45	2.59	3.41	49.93	0.90		0.19	20.91	18.88	0.23	0.34	0.97
Max.	0.59	2.64	3.67	50.41	1.06		0.32	21.08	19.25	0.31	0.45	1.11

OB 14	**12.47**	**3.54**	**1.07**	**67.87**	**0.17**	**0.31**	**0.76**	**2.21**	**10.48**	**bdl**	**0.56**	**0.57**
Std. dev. (4 ms)	0.10	0.15	0.04	0.26	0.05	0.08	0.02	0.07	0.07		0.05	0.06
Min.	12.36	3.32	1.04	67.61	0.11	0.25	0.74	2.13	10.38		0.50	0.51
Max.	12.59	3.62	1.11	68.15	0.23	0.42	0.78	2.28	10.53		0.61	0.64
OB 15	**11.32**	**3.42**	**1.29**	**67.04**	**0.24**	**0.28**	**0.63**	**3.68**	**10.6**	**bdl**	**0.93**	**0.57**
Std. dev. (4 ms)	0.05	0.13	0.03	0.13	0.05	0.05	0.04	0.04	0.09		0.09	0.05
Min.	11.26	3.31	1.26	66.91	0.19	0.23	0.58	3.65	10.48		0.83	0.52
Max.	11.37	3.6	1.33	67.18	0.28	0.32	0.67	3.74	10.67		1.05	0.64
OB 16	**11.23**	**3.49**	**1.20**	**67.45**	**0.18**	**0.29**	**0.64**	**3.67**	**10.34**	**bdl**	**0.88**	**0.62**
Std. dev. (4 ms)	0.17	0.08	0.11	0.06	0.02	0.05	0.04	0.08	0.11		0.04	0.07
Min.	11.07	3.42	1.06	67.38	0.16	0.23	0.61	3.62	10.22		0.84	0.55
Max.	11.38	3.61	1.29	67.52	0.20	0.35	0.70	3.78	10.45		0.92	0.69
OB 17	**10.53**	**3.33**	**1.32**	**66.57**	**0.24**	**0.32**	**0.59**	**5.08**	**10.59**	**bdl**	**0.89**	**0.54**
Std. dev. (4 ms)	0.11	0.09	0.04	0.22	0.07	0.07	0.04	0.17	0.15		0.03	0.07
Min.	10.41	3.20	1.28	66.36	0.21	0.23	0.56	4.90	10.41		0.86	0.45
Max.	10.65	3.41	1.35	66.86	0.34	0.40	0.65	5.24	10.76		0.92	0.61
OB 18	**0.29**	**4.1**	**1.64**	**51.17**	**1.18**	**bdl**	**0.28**	**17.61**	**22.1**	**0.21**	**0.83**	**0.58**
Std. dev. (4 ms)	0.04	0.11	0.09	0.14	0.06		0.07	0.18	0.13	0.06	0.05	0.09
Min.	0.23	3.95	1.55	51.03	1.09		0.20	17.41	21.94	0.16	0.76	0.52
Max.	0.33	4.21	1.75	51.32	1.23		0.35	17.78	22.24	0.29	0.88	0.72
OB 19	**0.51**	**2.94**	**3.87**	**48.77**	**1.39**	**bdl**	**0.24**	**18.23**	**22.20**	**0.19**	**1.03**	**0.62**
Std. dev. (4 ms)	0.08	0.13	0.10	0.10	0.08		0.04	0.07	0.15	0.06	0.06	0.07
Min.	0.37	2.75	3.76	48.6	1.32		0.21	18.16	22.06	0.11	0.94	0.51
Max.	0.59	3.10	4.02	48.86	1.53		0.32	18.32	22.41	0.25	1.09	0.67
OB 20	**11.16**	**3.29**	**1.27**	**66.9**	**0.22**	**0.29**	**0.59**	**3.96**	**10.83**	**bdl**	**0.85**	**0.64**
Std. dev. (4 ms)	0.08	0.06	0.03	0.18	0.07	0.03	0.03	0.06	0.11		0.08	0.03
Min.	11.05	3.22	1.22	66.72	0.18	0.27	0.57	3.90	10.67		0.78	0.61
Max.	11.23	3.36	1.30	67.15	0.32	0.34	0.63	4.02	10.94		0.93	0.67
OB 21	**1.40**	**4.50**	**2.60**	**48.40**	**1.60**	**bdl**	**0.20**	**16.70**	**22.30**	**0.20**	**1.00**	**1.10**
Std. dev. (4 ms)	0.10	0.10	0.00	0.30	0.10		0.10	0.20	0.10	0.20	0.00	0.1
Min.	1.20	4.40	2.50	48.20	1.50		0.10	16.5	22.2	0.30	1.00	1.00

Max.	1.40	4.60	2.60	48.80	1.80		0.30	16.90	22.50	0.30	1.00	1.10
OB 22	**1.35**	**4.55**	**2.70**	**48.94**	**1.60**	**bdl**		**16.60**	**22.14**	**bdl**	**1.01**	**1.12**
Std. dev. (4 ms)	0.05	0.04	0.06	0.20	0.10			0.17	0.06		0.06	0.10
Min.	1.31	4.51	2.64	48.65	1.49			16.38	22.06		0.94	1.04
Max.	1.42	4.6	2.77	49.07	1.74			16.78	22.22		1.07	1.26
OB 23	**0.24**	**3.29**	**1.99**	**48.45**	**1.18**	**bdl**	**0.22**	**21.69**	**21.31**	**bdl**	**0.76**	**0.87**
Std. dev. (4 ms)	0.03	0.07	0.12	0.12	0.05		0.07	0.17	0.09		0.05	0.09
Min.	0.19	3.23	1.91	48.36	1.15		0.15	21.54	21.20		0.72	0.76
Max.	0.27	3.38	2.17	48.62	1.26		0.29	21.94	21.43		0.83	0.97
OB 24	**0.31**	**4.27**	**1.74**	**51.2**	**2.90**		**0.34**	**19.59**	**18.5**	**bdl**	**0.50**	**0.65**
Std. dev. (4 ms)	0.04	0.05	0.04	0.08	0.03		0.07	0.12	0.03		0.02	0.08
Min.	0.27	4.22	1.69	51.08	2.87		0.24	19.42	18.45		0.49	0.56
Max.	0.36	4.33	1.77	51.26	2.95		0.39	19.68	18.53		0.53	0.75
OB 25	**1.32**	**4.41**	**2.61**	**48.62**	**1.6**			**16.69**	**22.23**	**0.23**	**1.09**	**1.20**
Std. dev. (4 ms)	0.06	0.03	0.08	0.20	0.04			0.09	0.13	0.04	0.04	0.06
Min.	1.25	4.38	2.54	48.35	1.56			16.59	22.07	0.20	1.06	1.13
Max.	1.38	4.44	2.73	48.8	1.66			16.79	22.36	0.27	1.15	1.26
OB 26	**1.80**	**2.51**	**1.61**	**61.49**	**0.77**	**0.20**	**0.24**	**12.84**	**17.42**	**bdl**	**0.70**	**0.43**
Std. dev. (4 ms)	0.07	0.07	0.03	0.07	0.07	0.06	0.02	0.04	0.06		0.07	0.04
Min.	1.71	2.43	1.58	61.41	0.70	0.13	0.21	12.80	17.33		0.65	0.37
Max.	1.86	2.59	1.66	61.57	0.83	0.28	0.27	12.88	17.48		0.80	0.47
OB 27	**12.1**	**3.36**	**1.15**	**67.39**	**0.23**	**0.3**	**0.77**	**3.77**	**9.75**	**bdl**	**0.75**	**0.54**
Std. dev. (3 ms)	0.10	0.13	0.06	0.08	0.03	0.06	0.04	0.08	0.10		0.05	0.03
Min.	12.00	3.20	1.10	67.30	0.21	0.25	0.74	3.69	9.67		0.69	0.51
Max.	12.2	3.45	1.21	67.47	0.26	0.34	0.81	3.84	9.87		0.79	0.57
OB 28	**11.00**	**3.40**	**1.30**	**67.20**	**0.20**	**0.20**	**0.70**	**4.20**	**10.40**	**bdl**	**0.90**	**0.60**
Std. dev. (3 ms)	0.10	0.00	0.10	0.10	0.10	0.20	0.00	0.10	0.20		0.00	0.10
Min.	11.00	3.40	1.20	67.10	0.20	0.30	0.60	4.10	10.20		0.80	0.50
Max.	11.10	3.50	1.30	67.30	0.30	0.40	0.70	4.20	10.60		0.90	0.70
OB 29	**11.60**	**3.40**	**1.20**	**68.70**	**0.20**	**0.20**	**0.70**	**4.00**	**10.90**	**bdl**	**0.80**	**0.60**
Std. dev. (3 ms)	0.10	0.10	0.10	0.10	0.00	0.20	0.00	0.10	0.10		0.10	0.00

Min.	11.60	3.40	1.10	68.60	0.20	0.30	0.70	3.900	10.80		0.80	0.60
Max.	11.70	3.60	1.30	68.70	0.30	0.30	0.70	4.10	11.00		0.90	0.60
OB 30	**11.20**	**3.40**	**1.20**	**67.10**	**0.20**	**0.30**	**0.70**	**3.80**	**10.60**	**bdl**	**0.90**	**0.70**
Std. dev. (4 ms)	0.10	0.10	0.00	0.20	0.00	0.10	0.00	0.10	0.10		0.00	0.00
Min.	11.10	3.30	1.20	66.90	0.20	0.20	0.70	3.70	10.50		0.80	0.60
Max.	11.30	3.50	1.30	67.30	0.30	0.30	0.70	3.80	10.60		0.90	0.70
OB 31	**11.20**	**3.40**	**1.20**	**67.00**	**0.20**	**0.30**	**0.70**	**3.80**	**10.70**	**bdl**	**0.90**	**0.60**
Std. dev. (3 ms)	0.10	0.00	0.00	0.10	0.00	0.10	0.00	00	0.00		0.00	0.00
Min.	11.10	3.40	1.20	67.00	0.20	0.20	0.70	3.80	10.70		0.90	0.50
Max.	11.20	3.40	1.30	67.20	0.30	0.40	0.70	3.80	10.80		0.90	0.60
OB 32	**3.10**	**2.10**	**1.20**	**67.00**	**0.70**	**0.40**	**Bdl**	**110**	**13.20**	**bdl**	**0.90**	**0.40**
Std. dev. (4 ms)	0.10	0.10	0.10	0.10	0.00	0.00		0.00	0.10		0.10	0.00
Min.	3.00	2.00	1.10	66.90	0.60	0.40		11.00	13.10		0.80	0.30
Max.	3.20	2.20	1.30	67.10	0.70	0.50		11.10	13.30		0.90	0.40
OB 33	**9.20**	**3.40**	**1.50**	**660**	**0.40**	**0.20**	**0.50**	**5.00**	**12.10**	**bdl**	**0.90**	**0.70**
Std. dev. (3 ms)	0.10	0.00	0.00	0.10	0.10	0.00	0.00	0.00	0.00		0.00	0.00
Min.	9.10	3.40	1.50	65.90	0.40	0.20	0.50	5.00	12.10		0.90	0.60
Max.	9.20	3.40	1.60	66.00	0.50	0.30	0.60	5.10	12.10		0.90	0.70
OB 34	**0.20**	**2.90**	**1.20**	**52.60**	**0.90**	**bdl**	**0.40**	**20.80**	**19.80**	**0.20**	**0.70**	**0.50**
Std. dev. (3 ms)	0.00	0.10	0.00	0.10	0.00		0.10	0.10	0.10	0.10	0.00	0.10
Min.	0.10	2.80	1.20	52.50	0.80		0.30	20.70	19.70	0.10	0.70	0.40
Max.	0.20	2.90	1.20	52.60	0.90		0.50	20.90	19.80	0.30	0.70	0.60

Table D.1. Average glass composition by SEM-EDS. All results in wt% and normalised to 100%