Ceramic materials in fire assay practices: a case study of 16th-century laboratory equipment

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ABSTRACT   During the European Renaissance, the techniques for quantitative metallurgical analysis or fire assay were developed to remarkable extents of precision and accuracy. These small-scale reactions were of great relevance for chymists, metallurgists and similar specialists, due to their economic, scientific and even philosophical implications. This paper presents the analytical study by optical microscopy, SEM-EDX and ICP-AES of some fire assay equipment recovered in the laboratory of Oberstockstall (Austria) and dated to the second half of the 16th century. By studying the ceramic material and temper, the manufacturing processes and particular functions of some of these tools, it will be argued that the significant development of fire assay in the Renaissance was only possible because of this range of technical ceramics especially designed and maximised to fit their respective functions. However, at the same time, the analytical investigation shall reveal the limits of the technical knowledge and the understanding of nature of Renaissance assayers, as well as the presence of technological choices that appear to originate from outside modern technical concepts. Although the paper largely concentrates on the triangular crucibles, some discussion of the analyses of scorifiers and bone-ash cupels is presented as a counterpoint.

1. Introduction

Fire assay is the quantitative determination of metals of economic interest present in samples of ores, alloys or other metallurgical products. When assaying for gold and/or silver, the process normally comprises a sequence of specialised high-temperature reactions to first liberate the precious metals from their original host matrix and transfer them into a quantity of lead metal, which is acting as a collector, before separating them from the lead in an oxidising process, and finally parting gold from silver.

The practice of fire assay was greatly developed during the late Middle Ages achieving high degrees of precision and accuracy. This analytical tool was relevant for extractive metallurgy as much as for chymistry[3], for quality control in coin production and metals trade, and even for small-scale recycling and refining in jewellery. By the 16th century, hundreds of fire assays were conducted across Europe daily, by different professionals who shared the very same tools and techniques, but pursuing diverse aims. It is in this context that the laboratory appears as a specific workplace for the first time in history. This is the most important root of modern analyical chemistry and, moreover, the observations carried out during these experiments would lead, in the long run, to the rejection of the traditional Aristotelian worldview. Thus, the study of developments in the practice and purpose of fire assay is crucial to understand the history of chemistry and the emergence of the modern understanding of nature (Sisco and Smith, 1949, 1951; Smith and Forbes, 1969; Halleux, 1986;

Rehren, 1996, 2001; Newman, 2000; Newman and Principe, 2002, p. 35-49; Martinón-Torres, 2003, forthcoming; Martinón-Torres and Rehren, forthcoming a, forthcoming b).

The conduction of fire assays required the use of several specialised ceramic and glass wares, the most fundamental of which were triangular crucibles, shallow dishes or scorifiers, and bone ash cupels (Fig. 1). This basic set was normally complemented by a plethora of other specialised pieces of equipment such as alembics, receivers, sublimation apparatus, aludels, retorts, adopters, to name but a few. This paper is based on the analytical study by optical microscopy, SEM-EDX (following analytical procedures detailed in Martinón-Torres and Rehren, 2002) and ICP-AES of the late-16th-century laboratory equipment recovered in the excavations at the sacristy of the church at the manor house of Oberstockstall in Lower Austria (Soukup and Mayer, 1997; von Osten, 1998; Martinón-Torres et al., 2003). Here, we will concentrate on the well-known, yet little-understood triangular crucibles, but will also detail some aspects of the scorifiers and the cupels as a counterpoint. By studying the technical standard of the different specialised ceramics, we will argue that the remarkable development of fire assay in the Renaissance was only possible because of these ceramics, especially designed and adapted to fit their respective functions. In addition, we will discuss the black colour of the crucibles, a technological choice that cannot be explained in the light of modern technical requirements, and compels an investigation of the socio-cultural context of the production and use of these ceramics.

2. The triangular crucibles

Not long ago, Cotter (1992, p. 256) outlined the "mystery" of the post-medieval triangular crucibles and regretted that "remarkably little attention" had been paid to an artefact type that seems to have been traded in the millions during that period. This type of vessel would become an indispensable tool for the fire assay, but also an icon of the goldmaker's workshop, as depicted in innumerable paintings. They would be used in copper and brass metallurgy, for gold and silver working and in all sorts of assays. Metallurgical assayers

needed them, but also chymists of all kinds, apothecaries, physicians, coin minters, glass-workers and even potters for glaze preparation (Cotter, 1992, p. 265). This wide range of possible functions stresses the need for well-preserved archaeological contexts and analytical studies in order to identify specific activities.

Lacking analyses it is difficult to assess to what extent the renowned crucibles from Hesse, Bavaria, Bohemia and Vienna were traded in post-medieval Europe, or whether just the much standardised design was progressively adopted and imitated across the continent. Whatever the case, the presence of triangular crucibles during the Renaissance is attested in historical sources such as the treatises written by Biringuccio (Smith and Gnudi, 1990), Agricola (Hoover and Hoover, 1950) and Ercker (Sisco and Smith, 1951), as well as in archaeological remains from a very wide region, from Britain and Scandinavia to Italy, from Portugal to Austria, and even including colonial north America (e.g. Cotter, 1992; Eckstein et al., 1994; Rehren, 1996; Straube and Luccketti, 1996; Lopes et al., 2000; Martinón-Torres and Rehren, 2002; Bayley, 2003; Salter, 2003).

The triangular or "laboratory" crucible was made by simply pushing inwards the upper parts of the walls of a (truncated) conical vessel, in order to create a triangular mouth with three very convenient spouts which allowed handling and pouring with tongs from any direction (Fig. 1). In some of the Oberstockstall samples, it can be seen how one of the pouring spouts has been used to decant slag, and another one for pouring molten metal. Their height, ranging from as little as 16 mm up to 185 mm, is normally equal to the width of the mouth. The crucibles had a flat base and stood in a chamber within the assay furnace on top of the fire box. This indirect heating mode prevented direct contact between the fuel and the vessel. Almost three hundred such vessels were recovered in Oberstockstall, many of which are stamped with one or two "T's" on the base.

The triangular crucibles were normally used in reducing operations, such as fluxing reactions in order to process ores, resulting in the primary concentration of the noble metals in a lead pool. The relatively closed shape of these vessels would facilitate the control of redox conditions. In addition, the crucibles could be closed by lidding the top using either lute, a recycled sherd, or an upturned scorifer; all three possibilities are documented in Oberstockstall. In many cases, the ceramic walls of the crucibles appear orange or brown on the outer surface, and grey towards the inside, showing that although the atmosphere in the furnace chamber was oxidising, the reaction inside the crucibles took place in reducing conditions.

Another noteworthy technical feature of these crucibles is the surface smoothness. As it is apparent in some of the vessels — particularly in those *not* tempered with graphite — the crucibles were dipped in liquid, well-refined clay, which was then wiped onto their surfaces with a brush or cloth, thus making the crucible surfaces as smooth as possible. This is interpreted as an attempt to prevent prills of molten metal from being trapped in a rough surface. Fire assays were based on quantitative reactions on small samples, normally containing noble metals, the results of which led to potentially significant economic conclusions about the metallic richness or chemical behaviour of the original ore deposit mined or metal traded. Therefore, it was crucial to avoid any loss of metal, which would significantly harm the overall inferences.

A further essential requirement for the triangular crucibles was thermal and chemical refractoriness. The ability to withstand high temperatures without melting or bloating is primarily a factor of the chemical composition of the ceramic material. In the case of Oberstockstall, if we compare the average composition of the crucible fabrics to that of another assaying instrument such as a receiver (used for collecting distilled liquids, hence not subjected to particularly high temperatures), then the refractory composition of the former type stands out, especially the significantly higher alumina content — above 30 wt% — and the lower

potash concentration (Table 1). Although the iron oxide concentration in the crucibles is relatively high — above 5 wt% — it can be assumed that this would not necessarily affect the performance of the vessels, given that the reactions in the crucibles were normally conducted under reducing conditions, and some of the iron oxide could be reduced to the metallic state.

TABLE 1

Average composition by SEM-EDX of the ceramic body of five unused triangular crucibles (top rows), followed by the composition of a receiver (bottom).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	FeO
OB288	0,2	0,7	31,8	57,9	0,2	1,4	0,8	1,4	5,4
OB290	0,3	0,6	33,5	56,0	0,2	1,8	0,6	1,4	5,6
OB395	0,1	0,5	34,2	55,5	0,2	2,0	0,3	1,3	5,7
OB394	0,1	0,8	30,9	58,5	0,2	2,3	0,6	1,3	5,3
OB560	0,2	0,7	30,8	56,9	0,2	2,8	0,8	1,6	6,1
OBn002	0,5	0,4	23,0	66,7	0,2	5,2	0,4	1,1	2,4

Figures are averages normalised to 100wt% of 5-10 measurements of different areas ~70μm by ~50μm in mounted cross-sections of the crucibles.

The most important feature, making most of these crucibles especially fit for their function, is the nature and abundance of temper. Normally, these vessels are tempered with about 30 vol% of graphite (Fig. 2). The inclusion of graphite in the ceramic paste enhances most of the technical requirements of the crucibles. Being one of the most stable minerals under high temperatures — it does not melt but sublimates only at ca. 3500°C —, graphite temper would contribute to the crucibles' thermal refractoriness. In addition, its chemical inertness would mean that the graphite-tempered crucible would remain significantly less attacked by the highly reactive hot metal oxides. If reacting — as seen in some used crucibles, where part of the graphite temper has burned off —, since graphite is pure carbon, it would contribute to inhibit the oxidation of the charge. Although the mechanical behaviour of graphite as ceramic temper remains uninvestigated, we may assume that its platy shape and flaky fracture could make it ideal for preventing the expansion of potentially fatal cracks across the body of the fabric, as is the case of mica (Tite et al., 2001), which has a very similar crystallographic structure. This would increase both the toughness and the thermal shock resistance of these crucibles, which often had to be quickly removed from the furnace while hot and holding considerably heavy loads. Graphitic crucibles are traditionally reported as extremely resistant to thermal shock and capable to withstand repeated runs (Percy, 1875, p. 122). Finally, graphite being an excellent heat conductor, it would improve the thermal conductivity of the vessel and thus allow saving on time and fuel.

In summary, the use of remarkably refractory clays, the shape and surface finish of the vessels, and especially the graphite temper render these triangular crucibles particularly suitable for their functions. Without them, fire assay practices could not have been developed to the extent they did, and this may be the reason why this crucible type was so highly esteemed in Renaissance and later assaying laboratories across Europe and beyond, from adventurous gold makers through coin minters and up to metallurgists and other analytical chymists.

The Oberstockstall crucibles, however, also exhibit some technical limitations. The ceramic paste contains about 20 vol% of silicate minerals, normally below 0,5 mm in diameter. Although these show moderate degrees of angularity, comparison with local clay indicates that they are natural inclusions rather than crushed minerals used as temper. These
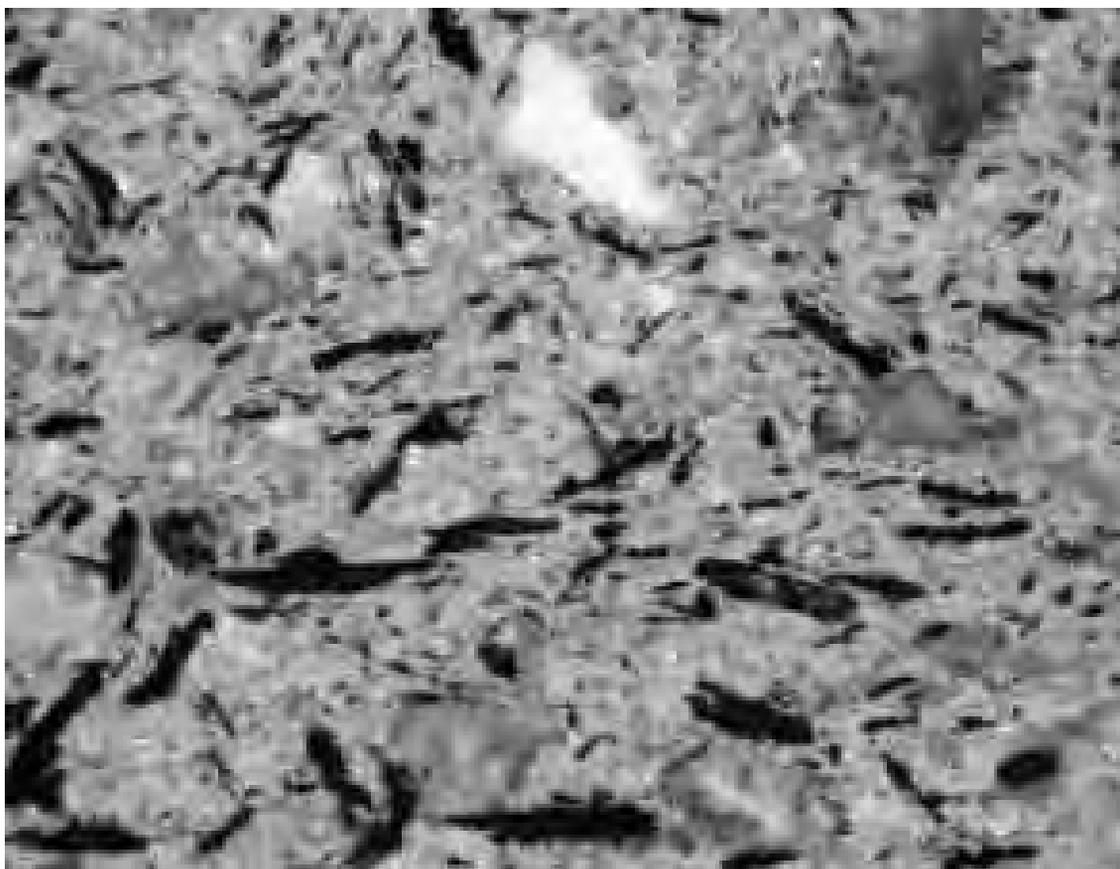
FIG. 2 – Section through black crucible, where abundant graphite temper can be noticed (black phases), together with silicate minerals (translucent phases). Photomicrograph under cross-polarised light, 50x magnification. Width of the image is ca. 2 mm. (Specimen OB286si)

inclusions would generally enhance the toughness and thermal shock resistance of the vessel (Tite et al., 2001). However, while quartz would remain unaltered to any significant extent, other minerals present such as potassium feldspar and iron silicates melted at the high temperature to which they were exposed during the use of the vessels, fluxing the ceramic surrounding them and hence leaving some softer, weaker areas in the crucible wall. Whilst this is seen as a potential flaw, it should be noted that none of the crucibles in the assemblage appeared catastrophically bloated or distorted; therefore this feature may have been irrelevant for the crucibles' performance and the expectations of Renaissance assayers.

Finally, there is one technological choice documented in the triangular crucibles from Oberstockstall that cannot be explained in the light of the technical requirements. As can be seen in the unused vessels, these were deliberately fired in a very smoky kiln that left a lustrous, black soot on their surface. At first sight, one could argue that this was simply a side effect of originally firing the crucibles in a reducing atmosphere, in order not to burn out the graphite temper. However, this explanation would not suffice to explain why a few crucibles of the assemblage, which microscopic analysis revealed not to contain any graphite, had been reduced fired as well (Fig. 3). SEM-EDX analyses indicate that the clay originally used to make both types of crucibles was taken from the same source – only the temper was different. Of the triangular crucibles whose compositions are shown in table 1, only the first three (OB288, OB290, OB395) are graphitic, while the other two (OB394 and OB560) are non-graphitic. The slight difference in alumina and potash probably reflects higher feldspar content in the latter crucibles, rather than different clay sources.
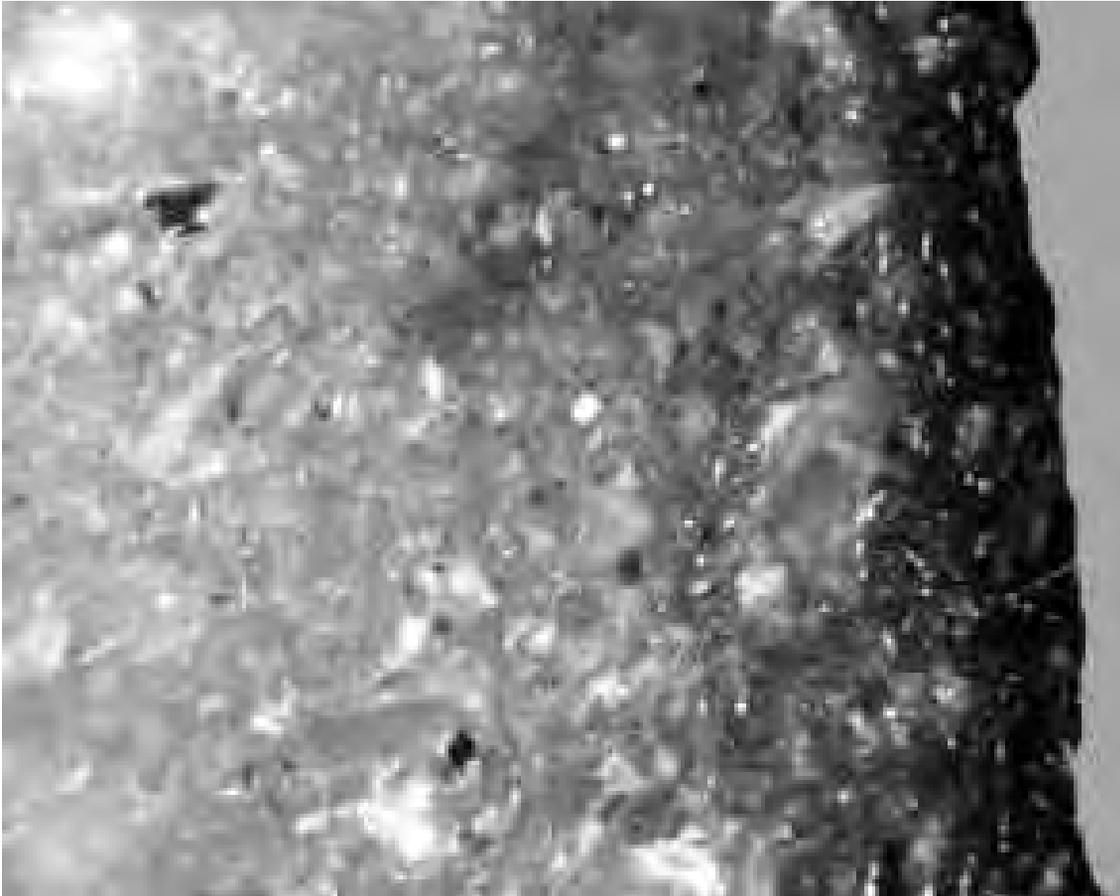
FIG. 3 – Section through another black crucible, where abundant translucent silicate minerals can be seen, but there is no graphite at all. The surface (right), however, appears deliberately blackened as in the graphitic crucibles. Photomicrograph under cross-polarised light, 50x magnification. Width of image is ca. 2 mm. (Specimen OB394SI)

Strikingly, it seems that the non-graphitic crucibles were deliberately made to look like the graphitic ones. If we were to use modern commercial concepts to explain this deliberate blackening of the crucibles, the idea of a potter trying to deceive their consumer by mixing some cheap, non-graphitic crucibles within the stock would appear tempting. If, instead, we consider the Aristotelian theory of matter, still widely current in the Renaissance, then the black colour could indeed be seen as a technical feature. According to Aristotle, matter was one and only, a product of the four elements, although it appeared showing different qualities, depending mostly on the ranges wetness-dryness, heat-coldness (Holmyard, 1968). Since what really counted was the quality of matter and not matter itself, perhaps these people were making crucibles that looked alike in the belief that they would perform alike. This hypothesis is, however, challenged by the fact that some ordinary ceramics of the same region and period show a very similar black lustre, even though they would not have been subjected to the thermal, chemical and mechanical stresses usual for the triangular crucibles. Possibly the black colour was simply a stylistic signature of the pottery makers of the area, which might have been originally constrained to graphite-tempered ceramics but then extended to other types of pottery. In any case, it is important to document this technological choice for black crucibles in Renaissance Oberstockstall, which appears to originate from outside modern technical concepts and expectations (cf. Sillar and Tite, 2000).

3. The scorifiers

The scorifier is another versatile vessel type frequent in many sorts of laboratories and workshops; however — contrary to the crucibles — its use seems to be constrained to fire assay practices, as reported in contemporary written sources and documented in several European sites (e.g. Pittioni, 1975, 1978, 1985; Bayley, 1988, 1992; Eckstein et al., 1994; Rehren, 1996; Sperl, 1996; Bayley and Eckstein, 1997; Kamber et al., 1998; Rehren, 2002; Bayley, 2003). Formally, the scorifier is just a relatively shallow ceramic dish. As the name indicates, these were used for scorification processes, i.e. to transform the unwanted components of a sample into slag, in a typically oxidising reaction. These operations could be, for example, the roasting of a mineral, the primary melting of an ore or metal sample with some flux, or the oxidation of part of the lead bullion in order to concentrate the noble metals prior to cupellation.

The approximately fifty scorifiers found in Oberstockstall range in diameter from 5 to 13 cm. Those analysed so far exhibit a relatively wide variability as regards the original ceramic paste. Table 2 shows the chemical composition of five scorifiers. The first three samples seem to originate from the same clay deposit (their slightly varying silica, alumina and potash concentrations probably reflect variability in their feldspar and quartz content). Their average composition is very similar to that of the clay used for the crucibles (see table 1), although with lower titania and iron oxide concentrations. In contrast, the composition of the scorifiers' fabrics shown in the two bottom rows of table 2 clearly indicate a different origin for each. As also seen in their varying compositions, the fabrics are generally but not always refractory. Their only notable inclusions are about 30-40 vol% silicate minerals and, occasionally, grog.

TABLE 2

Average composition by SEM-EDX of the ceramic body of five scorifiers.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	FeO
OB268	0,2	0,7	33,2	58,9	0,3	1,8	0,6	0,6	3,6
OB281	0,3	0,5	31,3	60,4	0,3	1,9	0,6	0,6	4,2
OB282	0,3	0,5	29,5	62,0	0,3	2,2	0,6	0,6	4
OB279	1,0	0,3	32,4	54,5	0,3	6,0	0,5	1,1	3,9
OB269	0,4	1,0	26,0	62,8	0,3	1,8	2,2	0,8	4,3

Figures are averages normalised to 100 wt% of 5-10 measurements of different areas ~70µm by ~50µm in mounted cross-sections of the scorifiers.

The wider variability in paste compositions seems to indicate a less standardised, more opportunistic approach to the ceramic materials used for scorification. Furthermore, from a present-day scientific perspective it seems surprising that siliceous ceramics were routinely used for reactions involving the oxidation of considerable amounts of lead, since liquid lead oxide is highly reactive with silica and tends to form a lead silicate glass and often badly penetrates into the scorifier. However, we believe that the use of these plates meant a maximisation of resources in economic terms. Since scorification was an intermediate step, perhaps it was worthwhile using these possibly cheaper scorifiers for as long as they could hold it, for a primary oxidation of the lead bullion. Subsequently, the smaller metal button, with a much higher concentration of noble metals, would be processed in the more specialised and expensive bone-ash cupels for the final refining.

Significantly, as detected in the SEM-EDX analyses, the only scorifier hitherto found to be graphite-tempered (OB279, table 2) was used for processing a sample that contained copper which, having a higher melting point than the usual lead-rich alloys, would have demanded a more refractory scorifier. Again, this seems to emphasise the dexterity of the specialists working in Oberstockstall, using the ceramic materials to the limit of their capabilities, and reserving the better ones for the most demanding operations.

4. The cupels

The third and most specialised vessel type related to fire assay is the cupel, used to liberate any noble metals previously collected in the lead bullion in an oxidising process. Cupellation, the burning of argentiferous lead metal to lead oxide and metallic silver on a porous substrate, goes back to the Early Bronze Age (Pernicka et al., 1998). Only during the Renaissance, however, do we see cupellation in a quantitative analytical context rather than as part of a production or recycling process (Bayley and Eckstein, 1997). The cupels had to withstand the attack by liquid lead oxide at temperatures around 1000°C, absorbing the liquid without reacting with it. This was achieved by using finely crushed bone ash as the main component to form the cupels, and by giving them a very thick body. An initial investigation of a sample of the approximately sixty Oberstockstall cupels (Rehren, 1998) showed them to be made of about two thirds bone ash and one third calcareous or dolomitic clay mixed in as a binding agent. Among nine cupels analysed so far, this clay component indicates the use of two different types; one being richer in magnesia and lime than the other, which in turn is higher in potash and silica (Table 3).

TABLE 3
Reconstructing the raw materials of cupels from Oberstockstall, and their ability to absorb lead oxide.

	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO*	FeO	clay	bone	Bi+PbO
OB936	2,1	4,6	10,9	68,6	6,6	2,4	5,1	11,2	15,4	64,2
OB876h	1,9	5,3	11,3	68,3	7,2	3,5	4,3	12,2	18,0	60,5
OB943	2,7	6,2	10,5	66,8	6,4	2,4	5,5	13,1	18,9	60,0
OB917	2,6	8,8	10,7	70,3	4,5	2,8	2,2	8,2	19,1	65,1
OB932	1,9	10,0	10,6	68,5	4,9	2,4	3,0	7,8	15,6	70,3
OB928	2,6	11,6	12,0	64,0	3,5	7,7	5,6	4,7	11,8	69,8
OB876d	1,5	16,8	8,7	70,0	1,5	3,2	1,4	5,8	11,1	74,3
OB919a	2,1	17,6	8,9	62,0	3,0	6,0	3,9	5,6	8,8	74,9
OB918b	1,0	18,4	11,3	60,9	1,2	5,9	6,0	6,7	7,9	72,4

The first seven columns detail the theoretical clay composition (columns Na_2O to FeO), the original combined weight percentage of these oxides in the used cupels (column clay), the percentage of bone ash in them (column bone), and the sum of bismuth and lead oxide found in the used cupels (last column). The theoretical clay composition was obtained by first reducing the total CaO content by 2.2 times the weight of P_2O_5 to account for bone ash, and normalising the weight percent of the light oxides including any remaing CaO to 100%. The sum of any P_2O_5 and 2.2 times this weight in CaO was defined as the likely bone ash / apatite component. ICP-AES analyses by W. Steger, Deutsches Bergbau-Museum, Bochum.

The relatively high amount of clay relative to bone ash exceeds clearly what 16th-century authors such as Agricola (Hoover and Hoover, 1950, p. 228-230), Biringuccio (Smith and Gnudi, 1990, p. 137-139) and Ercker (Sisco and Smith, 1951, p. 26-33) suggest; they recommend using pure bone ash. Microscopic investigation of the used cupels demonstrates that the bone apatite becomes unstable in the presence of silica, reacting with the lead oxide to dissolve and recrystallise as complex calcium silica phosphates (Rehren, 1998), and in doing so clogging up the porosity needed to mechanically absorb the liquid lead oxide. This is probably reflected in the significantly lower concentration of lead oxide in the first group of cupels (on average 64 wt%), which is richer in silica, compared to the second group (c. 73 wt% PbO), which is richer in magnesia and lime.

5. Conclusions

The analytical study of the laboratory remains from Oberstockstall shows the importance of the ceramic materials used for fire assays in Renaissance Europe. The development of fire assay practices was dependent on the availability of different specialised ceramics carefully designed to perform their specific functions. In addition, the specific design, use and optimisation of technical ceramics provides information regarding the degree of expertise and understanding of the natural world of Renaissance assayers, but also hints at the culturally influenced parameters of their technical knowledge.

The technical standard of the graphite-tempered triangular crucibles can explain the widespread distribution of this artefact type across Europe during this period, when fire assays were routinely conducted for various purposes. However, the fact that the crucibles were deliberately made black even when not containing graphite demonstrates that some technological choices in the design of the ceramics were dictated by socio-cultural factors, rather than by what we would identify today as technical requirements and experimental observations. This stresses the need to avoid present-day assumptions when looking at past technologies.

It has been demonstrated that the production of these crucibles is part of a specialised ceramic tradition, based in Obernzell in southern Germany, producing a range of technical as well as domestic vessels. The relationship of Obernzell crucibles to other such vessels, particularly the well-known Hessian ware, and their respective role in the Europe-wide trade during the Renaissance, is part of our ongoing research.

Acknowledgements

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3   The terms *chymistry* and *chymists* are used here to designate inclusively what we nowadays call either "alchemy" or "chemistry", depending on their context and connotations. It should be emphasised that such a distinction did *not* exist in the Renaissance (see Newman and Principe,1998; Martinón-Torres and Rehren, forthcoming *a* and forthcoming *b*).

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