IDENTIFYING MATERIALS, RECIPES AND CHOICES:
SOME SUGGESTIONS FOR THE STUDY OF ARCHAEOLOGICAL CUPELS

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ABSTRACT

Used cupels are increasingly identified in archaeological assemblages related to coin minting, alchemy, assaying and goldsmithing across the world. However, notwithstanding some valuable studies, the informative potential of cupellation remains is not always being exploited in full. Here we present a review of past and ongoing research on cupels, involving analytical studies, experiments and historical enquiry, and suggest some strategies for more productive future work. The archaeological case studies discussed are medieval and later assemblages from France (Pymont and Montbéliard) and Austria (Oberstockstall and Kapfenberg), which have been analysed using optical microscopy, SEM-EDS, ED-XRF, WD-EPMA and ICP-AES.

Using suitable analytical and data processing methodologies, it is possible to obtain an insight into the metallurgical processes carried out in cupels, and the knowledge and skill of the craftspeople involved. Furthermore, we can also discern the specific raw materials used for manufacturing the cupels themselves, including varying mixtures of bone and wood ash. The variety of cupel-making recipes raises questions as to the versatility of craftspeople and the material properties and performance of different cupels. Can we assess the efficiency of different cupels? Are these variations the results of different technological traditions, saving needs or peculiar perceptions of matter?

KEYWORDS

Lead, silver, cupellation, fire assay, technological choice, bone ash, wood ash

INTRODUCTION

Cupellation is a high-temperature oxidising reaction aimed at refining noble metals. Usually, it involves mixing the impure gold or silver with an excess of lead, and placing the metal on a porous substrate under a highly oxidising fire. As the lead oxidises, it triggers the oxidation of any other base metal presents, and these metal oxides are absorbed by the cupellation base. The pure noble metals, more resistant to oxidation and sustained by their higher surface tension, settle on top.

Silver cupellation has been carried out on a relatively large scale since the Bronze Age at least, as documented by many extant lead oxide-rich cupellation hearth bottoms [1, 2]. In later periods, cupellation was also routinely performed on a small scale, used as an analytical technique for the assay of ores, for quality control in coin mints, for recycling of debased metal in jewellery, or as part of experiments conducted in al/chemical laboratories [3, 4].

Since the Middle Ages, the basic tool for small-scale or analytical cupellation is the so-called cupel, a small vessel shaped as an inverted, truncated cone, with a shallow cavity on the top and a thick body made of ashes: during the high-temperature reaction, the thick body absorbs the lead and other base metal oxides by capillary action, while the refined noble metals settle on the top surface (Fig. 1). The reason why ashes were preferred instead of conventional ceramics is the fact that, unlike ashes, siliceous materials such as ceramics readily react with lead oxide to form a viscous slag that hinders the separation of the noble metals.

Fig. 1. Schematic representation of the cupellation process (drawing N. Thomas).

The standardisation in the shape of cupels throughout the medieval and post-medieval periods is startling. Given that they could only be used once, they often appear in large numbers. Furthermore, given their diagnostic appearance and specialised use, there is hardly any doubt in attributing these archaeological finds to the refining of noble metals. However, when suitable analytical methods are used, it is possible to go beyond the mere ascription of cupels to cupellation practice, and to infer further technological issues of broader archaeological relevance. This paper presents some examples, mostly taken from ongoing research, which illustrate how cupels may be employed as sources of information about past skill, versatility, efficiency and cultural traditions, and outlines the future potential of a methodologically standardised, comparative approach to cupellation remains.
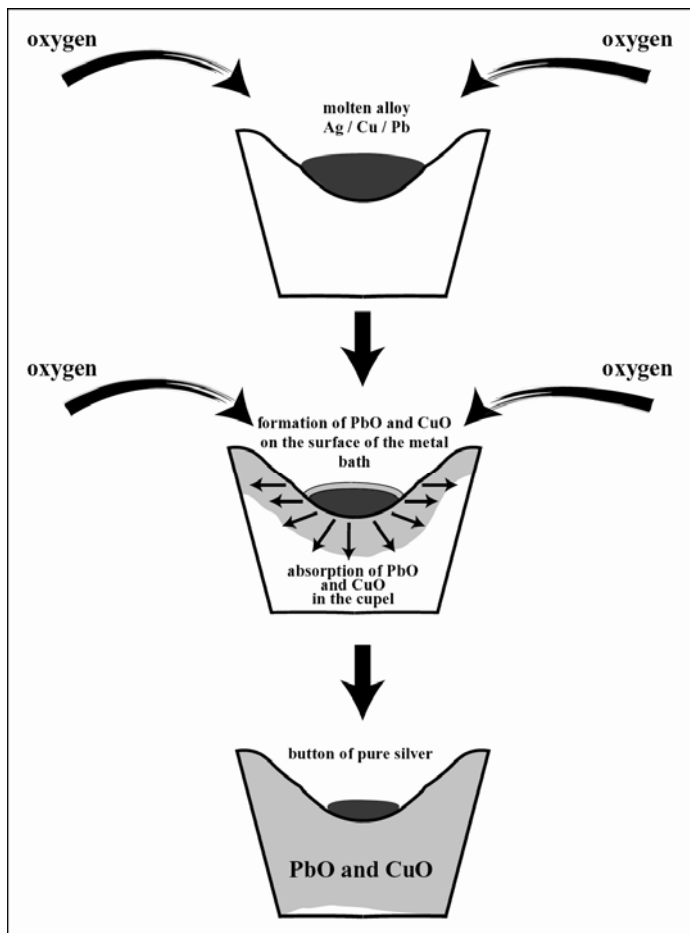
ARCHAEOLOGICAL ASSEMBLAGES

We will not provide much detail on the specifics of each archaeological site, given that our purpose here is to show the methods and potentials of our approach, rather than contributing to individual site reconstructions. Further details of each particular site have appeared or will appear in separate publications. Even though our case studies concentrate on medieval and early modern small-scale cupellation sites, it is hoped that some of our observations should be of use for those analysing cupellation remains of other nature or chronology.

1. The castle of Pymont (France) is a fortified structure dating from the 13th to the 15th century. Among many other finds, about twenty cupels were recovered, which were related to the activities of a 14th century illicit mint historically documented at the site [5, 6].

2. The cupel from Montbéliard (France) was found in a secondary pit, together with two possible alembic fragments, in rescue excavations at the medieval quarter of the town, a neighbourhood known to have been inhabited by several miners and assayers in the late 16th century [7, 8].

3. The assemblage from Oberstockstall (Austria) comes from a late 16th-century laboratory excavated in the sacristy of a church attached to a manor house. It comprises hundreds of al/chemical laboratory instruments, including nearly one hundred cupels [9-12].

4. The cupels from Kapfenberg (Austria) were found with crucibles and distillation equipment recovered next to a small furnace,

Fig. 2. Selection of cupels recovered in Kapfenberg, Austria.

located in a concealed gallery under the fortification wall of a castle [13]. The likely deposition date is estimated to have been in the 17th-18th century. The analytical study of this assemblage is ongoing (Fig. 2).

UTILISATION AND EFFICIENCY

As noted above, cupels are indeed diagnostic of cupellation, but cupellation can be used for different purposes (mineral assaying, recycling, minting…), with different social and technological implications. The analytical study of cupels may shed some light on this issue. Under the microscope, there is relatively little difference between cupels: typically, it is just possible to identify a few grains of burnt bone, recognisable by their spongy texture, and a matrix of large lead oxide crystals. Chemical analyses are usually more informative of the cupel utilisation.

| | | Oxides related to cupel manufacturing materials | | | | | | | | |
| | | Na_2O | MgO | Al_2O_3 | SiO_2 | P_2O_5 | Cl | K_2O | CaO | MnO | FeO |
	Technique	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
Pymont	ICP-AES	0.2	0.6	0.2	1.9	10.6	-	0.1	11.4	0.1	0.2
Kapfenberg (#102)	SEM-EDS	-	1.7	1.4	6.1	5.9	-	0.1	23.2	0.2	0.2
Oberstockstall (#918b)	ICP-AES	0.1	1.4	0.9	4.8	4.2	-	0.1	12.0	0.0	0.5
Montbeliard	WD-EPMA	-	0.7	0.7	2.1	13.4	0.6	0.1	19.7	0.3	0.4

| | | Oxides related to contamination through use | | | | | | | | |
| | | PbO | CuO | NiO | ZnO | As_2O_5 | Ag_2O | SnO_2 | Sb_2O_5 | Bi_2O_5 |
		wt%	wt%	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Pymont	(cont.)	72.2	2.5	-	-	392	-	125	662	-
Kapfenberg (#102)	(cont.)	57.4	1.3	5000	-	-	1400	-	21000	-
Oberstockstall (#918b)	(cont.)	72.2	3.7	76	454	704	189	314	859	3560
Montbeliard	(cont.)	60.8	0.6	-	-	-	740	320	1870	-

Table 1. Bulk chemical composition of four cupels. Results normalised to 100%.

Table 1 shows the bulk chemical compositions of four cupels, each one as typical of their corresponding site. The top half of the table shows oxides related to the material constituents of the cupel itself, and will be discussed later. Heavy metal oxides (bottom half) are clearly related to contamination through use, and therefore more indicative of the possible use of the cupels. It is

immediately obvious that lead oxide is the main contaminant, making up over 50% of the current weight of the cupel in all cases. This is not surprising, considering that an excess of lead was always required for cupellation, and this would always be added if required. In much lower concentrations, the next significant component is copper oxide (and in one case Sb_2O_5), which indicates that copper (and possibly lead) was the main contaminant of the silver. Coming to the oxides in lower concentrations, some differences emerge. The cupel from Pymont shows relatively low levels of impurities, rarely exceeding 500 ppm. On the contrary, all the other cupels contain concentrations of Ni, Zn, As, Sb and Bi, in different combinations but always reaching together levels above 1000 ppm. This observation could suggest that the cupel from Pymont was used for processing pure grade metals obtained from the market, whereas in the other three cupels some more impure metals would have been processed, probably derived from the processing of ores, which would carry impurities into the cupel.

In fact, the historical and archaeological interpretations support the analytical data, in that the suggested archaeological context for Pymont is a mint (hence the use of pure metals) [5] whereas in Montbéliard [7, 8], Oberstockstall [9] and Kapfenberg (unpublished results) the evidence indicates that complex sulphidic minerals were processed in crucibles, before refining the resulting metals with lead in cupels. In this sense, it could be claimed that the trace elements in a cupel may give us a hint as to the specific utilisation of the vessels, at least to accept or refute previous hypotheses. However, it remains true that the best source of information to this end is the broader archaeological context, which will normally provide indications as to the overall function of the workshop. In fact, particularly for earlier periods, the impurities in circulating metals reach significant levels, which might mislead the interpretation of analytical results.
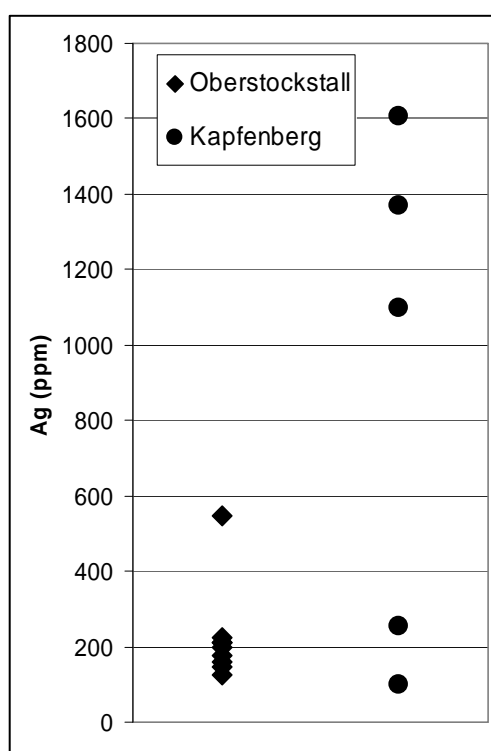

Fig. 3. Silver losses in cupels from Oberstockstall (by ICP-AES data, one analysis per cupel) and Kapfenberg (by SEM-EDS; each plot represents average from several area analyses, taking values below detection limit (~0.3%) as equal to zero).

The last column of interest in Table 1 is that of silver. The traces of silver confirm that the cupels would have been used for refining silver, but they also show that some valuable metal was lost into the cupels. Any silver left in a cupel would be a loss, and these losses would be particularly important when conducting assays, e.g. when the silver content in small ore samples was used to estimate the silver richness of an ore body. We should not take the presence/absence of silver in a cupel as a direct indication of the (in)efficiency of the process, given that we cannot know how much silver there was (if any) in the metal processed. However, when silver is detected in a range of cupels, we can make inferences as to the reproducibility of the analyses in a given archaeological laboratory.

Figure 3, for example, compares the silver contents in cupels from Oberstockstall and Kapfenberg. In Oberstockstall, all but one of the silver losses cluster around the 200 ppm level. As the analysis of crucibles and other residues have confirmed that relatively rich silver ores were processed in this workshop [9], these results show that the artisans working here had very good analytical protocols, ensuring minimum losses and the reproducibility of their experiments. On the contrary, the few cupels from Kapfenberg so far analysed show a much broader scatter of silver losses,

several of them reaching relatively high levels. This is, in turn, suggestive of a less standardised practice, perhaps due to the more haphazard nature of the reactions, or to the lack of experience or skill on the part of the artisan. This would be in agreement with the context of this laboratory, clearly concealed in a secluded gallery, where everything suggests that the activity was illicit, and possibly carried out by a non-specialist [13].

There are several reasons why silver may be lost into the cupel, and not all of them are accessible archaeologically. One can be the lack of skill of the craftsperson using the cupel, who may conduct the reaction without enough care, or use incorrect temperatures. An argument sometimes suggested to explain silver losses into cupellation material is an insufficient Pb/Cu ratio [2]. If there is not enough lead in the cupel, some free Cu_2O will form; and this, unlike PbO or $PbO \cdot Cu_2O$, can dissolve some silver and carry it down into the cupel. This, however, was not the problem in any of the sites discussed here, where Pb/Cu ratios are very high.

Finally, assuming comparable operators and operating conditions, the higher or lower losses may be related to the varying qualities of the cupels. This is an area which has received very little attention, and one on which the rest of this paper will concentrate.

RECONSTRUCTING CUPEL RAW MATERIALS

How can we investigate the manufacture and material properties of a cupel? Unused cupels would be ideal for this purpose, but their fragility means that they are hardly durable in archaeological deposits, and findings of unused cupels are exceptional. As noted above, the microstructure of a used cupel is usually blurred by the sheer amounts of lead oxide soaked up in the matrix, which is matched by the elevated PbO levels routinely detected in chemical analyses.

There is, however, a way around this. Assuming that all of the elements heavier than nickel come from contamination through cupel use, we can neglect these and re-normalise the results to 100%. Even though any analytical error will be exacerbated, the resulting figures can be taken as indicative of the composition of the cupel prior to use.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	Cl	K_2O	CaO	MnO	FeO
Pymont	0.9	2.4	0.8	7.4	41.8	-	0.4	44.9	0.5	1.0
Kapfenberg (#102)	-	4.3	3.5	15.8	15.1	-	0.2	60.2	0.4	0.6
Oberstockstall (#918b)	0.3	6.0	3.7	20.0	17.7	-	0.3	49.8	0.1	2.0
Montbeliard	-	1.9	1.8	5.6	35.3	1.6	0.2	51.7	0.7	1.1

Table 2. Reconstructed "unused composition" of the cupels shown in Table 1, after re-normalisation.

Table 2 shows the compositions of the same cupels presented in Table 1, after processing the data as described above. Thus we can see more clearly the compounds present in the cupel raw materials and their relative proportions. In all cases, CaO seems the major constituent, together with variable concentrations of P_2O_5, SiO_2 and other oxides. There is, however, some variation from cupel to cupel.

Microscopic examination allowed us to identify bone fragments in all of the cupels. However, were all the cupels made of pure bone ash? The inorganic component of bone is known to be composed primarily of the mineral hydroxylapatite [$Ca_5(PO_4)_3(OH)$] (~85%), with small amounts of calcium carbonate (~10%) and compounds of silicon, magnesium, sodium, fluorine and other elements supplying a minor fraction (~5%). Therefore, when the main constituents of the cupel material are the oxides of calcium and phosphorus, one can reasonably assume that the cupel was made of pure

bone ash. This is the case of the cupel from Pymont, where CaO and P_2O_5 together account for almost 90 wt% of the original material of the cupel, and the CaO/P_2O_5 ratio is 1.1 (Table 2, and see discussion below). The situation is more complicated for the other cupels, where this ratio is higher, and oxides other than CaO and P_2O_5 appear in significant levels. Here, bone was clearly mixed with something else, which for the time being we can term "additive". In these cases, a rough estimate of the additive composition can be attempted by calculating the weight ratio of calcium oxide to phosphate, and presuming that all of the phosphate in the cupel originates from bone. These calculations, however, will be based on two assumptions: firstly, that the additive would not have contained any major amounts of phosphorus – which is generally the case for calcite, clay and plant ashes –; secondly, that the CaO/P_2O_5 ratio in bone is relatively stable and predictable.

Regarding the second assumption, it has to be acknowledged that the bone CaO/P_2O_5 ratio does vary: reported ratios for different human bones are around 1.3 [14, 15]; in rats, these vary from 1.1 to 1.3, depending on the part of the body; known ratios for rabbit bones are 0.8 and 1.1, and the ratio for lamb is 0.8 [16]. In archaeological examples, analyses of medieval bone-ash lined cooking pots have yielded a ratio of 1.15 [17], while this value for the Pymont cupel was around 1.1 (see above). Our unpublished analyses of an experimental cupel made with industrial bone ash and used for silver refining yielded values ranging from 1.4 to 1.6.

Further technical limitations can be mentioned: on the one hand, calcium and phosphorus are precisely some of the elements more susceptible of post-depositional alteration in archaeological materials [18, 19] – which highlights the convenience of combining chemical with microscopic information; on the other hand, ZAF correction procedures in standard analytical equipment may not be calibrated for such high-lead matrices as the used cupels. Significantly, in our SEM-EDS analyses of an experimental bone-ash cupel, higher CaO/P_2O_5 ratios were systematically detected in those regions with higher PbO concentrations, and analytical totals reached up to 140% prior to normalisation. Most likely, these are analytical problems to be clarified.

Still, whilst bearing in mind that our estimates cannot be fully quantitative, some calculations can be performed. We begin by accepting a nominal CaO/P_2O_5 ratio in bone of 1.2. Subsequently, by multiplying the P_2O_5 weight in the cupels by 1.2, we obtain an indication of the relative amount of CaO that would come with the bone ash. We can now label those components as "bone", and isolate them in the compositional data. The remainder will be the "additive".

	Na$_2$O	MgO	Al$_2$O$_3$	SiO$_2$	P2O5	Cl	K$_2$O	CaO	TiO$_2$	MnO	FeO
Kapfenberg (#102)	-	6.4	5.3	23.6	-	-	0.2	63.0	-	0.6	0.9
Oberstockstall (#918b)	0.6	9.8	6.1	32.7	-	-	0.6	46.8	-	0.2	3.2
Montbeliard	-	8.7	8.2	25.0	-	7.3	1.1	41.9	-	3.3	4.7
Beech ash (leached)	0.4	10.3	8.0	30.0	5.6	0.1	2.9	38.2	0.5	0.2	3.2
Birch ash (leached)	0.0	10.8	1.4	11.5	7.4	0.1	3.5	56.1	0.2	5.3	1.4

Table 3. Reconstructed "additive" composition of three cupels, and compositions of leached wood ashes for comparison. Data for wood ash after [20].

In Table 3, we have performed such calculations with the cupels from Montbéliard, Oberstockstall and Kapfenberg. In the Montbéliard cupel, for example, we start with a P_2O_5 value of 35.3% (Table 2) which, multiplied by 1.2, renders 42.3. Thus we proceed to neglect all of the P_2O_5, and substract 42.3 from the CaO value (*i.e.* 51.7 – 42.3 = 9.4% residual CaO content). The resulting figures are then re-normalised to 100% to facilitate comparison. The table thus shows the estimated composition of this "additive" which is dominated by CaO and SiO$_2$. We can now ascertain that this additive is, in fact, the ashes of wood that had been leached to remove potash and impurities. The

bottom rows of the table show, for reference, two compositional analyses of leached wood ash obtained by Stern and Gerber [20]. Despite the wide variability of wood ash compositions across the world, and the likely magnified error of the cupel data following re-normalisation, the data processed in this way thus allow us to determine that the three cupels were made of mixtures of bone ash and washed wood ash.

Now it is also possible to calculate the ratios between bone and wood ashes for each cupel, using the data from Table 2 and following these simple equations:

(1) Bone ash % = $P_2O_5 + (P_2O_5 \times 1.2)$ %

(2) Additive % = $\sum$ normalised cupel raw composition (100%) – bone ash %

For the Oberstockstall[1] and Montbéliard cupels, the "bone ash" value is typically around 40-60%, indicating that these cupels where made with a mixture of wood and bone ash in equal proportions. In Kapfenberg, this value ranges from 20 to 33%, suggesting a more wood-rich recipe. In the cases addressed here, bone and wood ash suffice to account for the total composition of the cupels. However, larger cupellation hearths and historical sources document the use of crushed limestone, clay, shell, calcite and other materials as cupellation matrices [2, 7]. Owing to this, microscopic studies will always be essential as a first step, and these calculations may not always be possible.
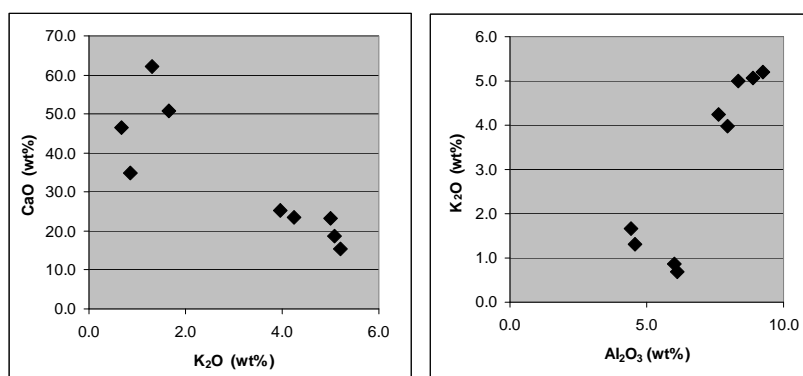

Fig. 4. Comparison of selected oxide ratios from the reconstructed "additive" composition in cupels from Oberstockstall, demarcating two compositional groups.

If sufficient numbers of cupels are analysed, we can study standardisation in cupel-making, and also compare the residual compositions of the wood ashes. As an example, Figure 4 compares the ratios of several oxides as present in the reconstructed wood ash compositions of Oberstock-stall cupels. The data clearly indicates that two types of wood are present. This suggests that two slightly different types of wood were used, perhaps in different batches.

Finally, when carrying out compositional analyses of cupels mounted as polished cross-sections, it is advisable to systematically raster several areas from the top surface to the base. In some cases, we have found that the residues are very unevenly distributed, which suggests changing parameters in the course of the reaction. Furthermore, this strategy may provide further information about the raw materials and manufacture of the cupel. Figure 5 shows a plot of selected elements scanning the Montbéliard cupel from top to bottom. While the P_2O_5 content (indicative of bone) decreases sharply from a depth of about 5 mm, other oxides such as MgO, Al_2O_3 and MnO increase. In fact, when the above calculations are performed separately for the top surface and the body of the cupel,

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[1] In previous publications [21, 22], similar estimations were performed for the Oberstockstall data but, although the analytical results were correct, a miscalculation was introduced in the data processing. The results reported here thus overrule previous interpretations.

it becomes apparent that, while the bulk of the cupel was made of a 50:50 mixture of bone and wood ash, a top facing was applied of 100% pure bone ash [7].
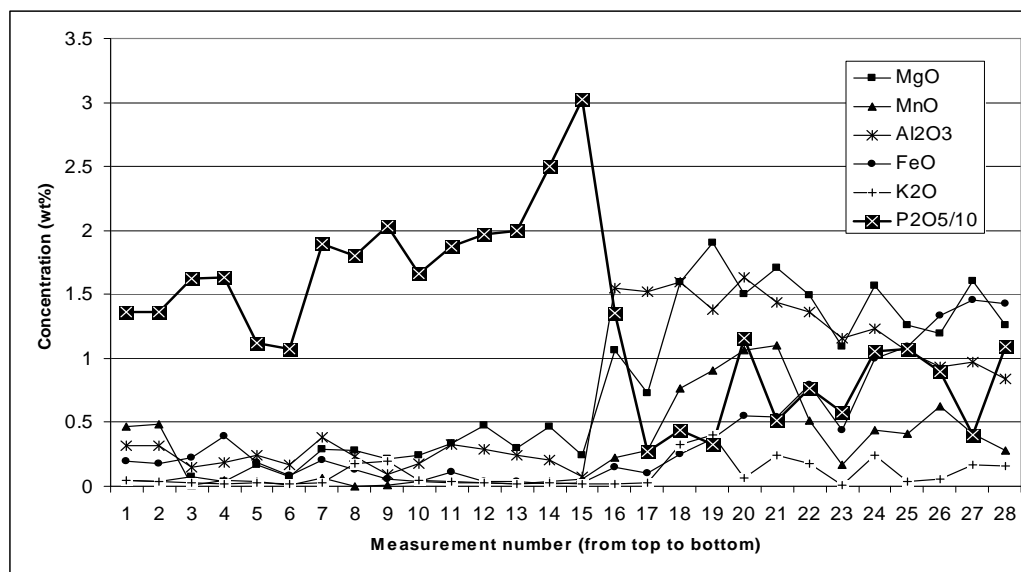

Fig. 5. Compositional profile of selected oxides in the Montbéliard cupel, scanning areas from top to bottom using EPMA.

TRADITIONS, VERSATILITY AND PERFORMANCE: EXPLAINING VARIATION

The variety of cupel-making recipes shown above demands and explanation. One of the possible reasons behind this could be the existence of different technological traditions. It is known that cupels were generally made by the users, given their high degree of specialisation and their fragility for transport. Thus, the different recipes could be the result of the individual artisans having learnt how to make cupels from different masters. In a review of relevant historical sources, we have found a variety of cupel-making recipes, going to the extent of suggesting specific bone or wood species, and different mixtures of them. All of the recipes identified archaeologically have a historical counterpart, with Ercker [23] mentioning the importance of a pure bone top facing (as in Montbéliard), and describing the mixture of two parts of wood ash to one part of bone ash (as in Kapfenberg), whilst Biringuccio [24] mentions pure bone (as in Pymont), and Agricola [25] emphasises the need to thoroughly wash the wood ashes.

A related aspect affecting cupel manufacture may be the availability of materials or an assessment of costs and benefits. Our own experiments have taught us that preparing fine and clean bone ash is an extremely tedious task with a very small yield, and this might be the reason why some craftspeople would choose to "bulk up" their bone by adding wood ash. In a different context, late 19th-century coin minters in Paris used cupels made of a mixture of bone and wood ash, admittedly due to economic reasons and whilst acknowledging that pure bone ash worked better [26].

We cannot assume that ancient people would have made identical choices for identical reasons, though, and indeed not all Renaissance authors agree that pure bone ash is the ideal material. In fact, all of the cupellation materials used since ancient times seem to share just one perceptive feature: their white colour – and perhaps this factor helped their identification and led to early experiments. From a present-day analytical perspective, it appears that wood-containing cupels have a slightly lower capacity to absorb lead oxide than their pure bone counterparts. This is due to the fact that the presence of silica from the wood ash triggers the formation of calcium-silica-

phosphates, which are impervious to the absorption of metal oxides [21]. However, if noticed, this limitation could have been overcome by making larger cupels. If a top layer of pure bone was used, as in Montbéliard, the separation of the noble metal bead from the cupel might be achieved just as easily.

We cannot elaborate here on all the possible reasons explaining this variability. The choices made by past cupel-makers may have oscillated between "effectiveness" (*i.e.* a cupel that performs its task), and "efficiency" (*i.e.* one which does so with the minimum expenditure of time and effort) [7]. Different understandings of the properties of matter, learning traditions, or availability of materials may have also played a role, and there may well be other factors beyond our modern and self-complacent "common sense". Clearly, a convincing explanation can only be approached with reference to the archaeological and cultural contexts of reference. Whatever the case, a systematic analytical approach such as the one illustrated here may facilitate the identification of these choices, as a necessary step to then discuss the reasons behind them.

METHODOLOGICAL CONSIDERATIONS

The cupel assemblages discussed above have been analysed over a number of years, end employed a variety of analytical techniques - not all of them with equal success, and making comparisons more difficult. We are now in a position to suggest some analytical protocols that should allow the observations described above and facilitate comparative studies.

Cupels should be first studied under the microscope, ideally mounted as polished sections, in order to assess the texture and identify particles of the raw materials such as bone fragments, and remaining prills of noble metals. For compositional analyses, the ideal techniques are SEM-EDS or WD-EPMA, which allow the scanning of relatively large areas from top to bottom and enable compositional profiling. After experimenting with several protocols, we have found that relatively large analytical areas (~1.5 x 2 mm) provide more conclusive results, as they balance out the internal heterogeneity of the cupel and facilitate the identification of significant compositional patterns. Ideally, the analytical study should be completed with an instrument with lower detection limits, such as ICP or XRF, in order to quantify the metal impurities and noble metal losses at the trace elemental level.

CONCLUSION

This paper has suggested some analytical approaches and data processing strategies that allow for a fuller exploitation of the informative potential of archaeological cupels. Although exemplified here with studies of individual cupels, the background work has involved the analyses of many more samples. The consistency of the patterns identified within and between sites indicates that, despite the necessary assumptions and gross numerical normalisations, this is a valid approach.

A systematic, comparative study of archaeological cupels opens a new path to identify variation in the choices made by early craftspeople, and a useful background to address cultural traditions, efficiency, cost-effectiveness and performance. Clearly, these aspects are relative to their cultural framework, and further focused experimental work is needed to understand the technical parameters related to the manufacture and performance of different cupels. Although such an approach requires some commitment in terms of methodological standardisation, the results of ongoing work are encouraging - and we hope that they will encourage others.

ACKNOWLEDGEMENTS

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