From calves' sculls to finished bone-ash cupels and testing of precious metals: An investigation of the cupellation practised in the Mint in the Archbishop's Palace, Trondheim, in medieval times

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Abstract

The paper discusses three topics: (1) the production process of bone-ash cupels from bones in calves' sculls to finished cupels, (2) cupellation of a silver and a gold alloy with known compositions in replica cupels and (3) how lead oxide is distributed in the cupel after cupellation. Following the precept of Ercker in his treatise published in 1574, the authors' production of cupels is described in detail, from boiling the calves' heads to the finished cupels, each with a fine-grained surface layer. The microstructure of bone-ash particles and the flow of molten lead oxide into the cupel in a replica cupel and a cupel found during the archaeological excavations in the Archbishop's Palace, Trondheim, in 1991–1995 are compared. The experience gained from making cupels and later use of the cupels for cupellation led the authors to the conclusion that the cupels found during the archaeological excavations on the premises of the 16th century Mint in the Archbishop's Palace were probably produced locally at the Mint. By using his own cupels, the contemporary moneyer might have measured the fineness of precious metals with an acceptable degree of precision.

Introduction

Following a fire in 1983 that ravaged the wooden buildings forming the eastern and southern wings of the Archbishop's Palace of Nidaros (present-day Trondheim), archaeological excavations took place between 1991 and 1995 (McLees, 1996; Saunders 2001; Nordeide 2003). The excavations identified three successive mint complexes, one above the other, in the northern part of the eastern wing. All of them were near the perimeter wall of the Palace. Their structure and layout, with workbenches, tiled floors and hearths, clearly indicate the purpose of the buildings as workshops of medieval moneyers, corresponding to illustrations and instructions of minting in contemporary treatises. The interpretation of the buildings as mints was further corroborated by archaeological evidence from the excavations, with important finds of metalworking debris from coin production, such as bone-ash cupels, crucible fragments, hammered rods, blanks, and coins. The finds are mainly from the site's archaeological period 6, corresponding to AD 1500–1532, and period 7, corresponding to AD 1532–1590. The majority of the cupels relate either to destruction layers associated with the second and third workshop or to waste layers. Judging from dendrochronological measurements of timber in the

walls, the oldest mint complex was established about AD 1500 (Olsson, 2000) and is today exhibited in-situ in the museum part of the Archbishop's Palace, Trondheim.

During the excavations, 143 bone-ash cupels and 31 cupel fragments were unearthed (Saunders, 2001, pp. 27–33).[1] The total has since been increased from 174 to 186 cupels and cupel fragments (Risvaag et al., 2021).

The use of bone-ash cupels was a standard technique in the assaying of precious metals in the 16th century. The cupels have an inherent capacity to enable the separation of precious metals from other metals, thereby permitting calculations of the fineness (i.e. purity) of a sample. Unused cupels have a low weight. The weights of the excavated cupels indicated that all of them had been used. As the archbishops in medieval Trondheim did not have any silver mines, bone-ash cupels were used to measure the fineness of precious metals being paid to the archbishops and to establish the correct alloy composition when making bullion for use in coin production.

Guidelines on how to make cupels are given in several contemporary treatises, including those by Biringuccio (1966 [1540]), Agricola (1950 [1556]) and Ercker (1951 [1574]). For our project in which we attempted to make bone-ash cupels, we followed the recommendations provided by Ercker. As pointed out by Bartels (2010), even though Ercker was as a skilled university-educated theorist, his knowledge relied as much on hands-on experience as would an apprentice. Thus, knowledge of procedure in practice was as important as theory, which in turn meant there were some gaps in his treatise. Although Erker was quite meticulous in his description, both of the making of cupels and the cupellation process, some degree of interpretation is left to the modern reader. What he regarded as a hands-on procedure remained unsaid, possibly to avoid stating the obvious. Ryle's classical distinction of 'knowledge-how' and 'knowledge-that' springs to mind (Ryle, 1949 (1990)).

Modern investigations of 16th century cupels from the Tower of London (White, 2010) have shown that they were made from bone-ash, while cupels from Oberstockstall, Austria (Rehren, 1998; Martinon-Torres and Rehren, 2005) were made of about two-thirds bone-ash and one-third clay mixed in as a binding agent. Mid-14th century bone-ash cupels from Pymont, France (Rehren and Eckstein, 1998) contained c.10% by weight of other materials.

Furthermore, investigations have provided detailed information about testing methods (e.g. Bayley, 1991; Sieblist, 2006). A modern description of how to assay precious metals is available in *ASTM E1335 - 08(2017): Standard Test Methods for Determination of Gold in Bullion by Fire Assay Cupellation Analysis* (ASTM International).

However, few papers deal with the microstructure of cupels and the resulting beads after cupellation in general (Téreygeol and Thomas, 2003). This gap in knowledge was addressed by an investigation of bone-ash cupels from the Mint in the Archbishop's Palace in Trondheim (Ulseth et al., 2015). However, one question remained open: Were the cupels imported, similar to the crucibles that were imported from Germany,[2] or were the cupels made at the Mint in Nidaros (present-day Trondheim)? In an attempt to answer this question we

[1] Se also: Bergstøl, S., & S. W. Nordeide. 1993. "Analyses of crucibles from the late medieval mint in the Archbishop's Palace, Trondheim,". Unpublished.

[2] Lohne, O., Ulseth, P. and Risvaag, J.A., 2015. Undersøkelse av digelfragmenter fra funn i Erkebispegården. En statusrapport. 24 pp. [Unpublished report in Norwegian]

decided to produce cupels from scratch and to conduct some experiments in the cupellation of precious metals with known fineness. We anticipated that if we managed to make cupels that allowed for acceptable results in terms of fineness of the samples after cupellation, it would be likely that a skilled moneyer appointed by the Archbishop of Nidaros would have managed to produce good cupels (Lohne and Ulseth, 2021, this volume).[3]

As it is possible to make metallographically prepared specimens from used coupels, we compared a cross-section of a used replica cupel with a cross-section of a cupel (N124860) found during archaeological excavations in 1991–1995.

Experiments

Preparation of bone-ash cupels

From bones to bone-ash powder: Agricola (1950 [1556]) and Ercker (1951 [1574]) proposed both that calves' sculls were the best material for bone-ash cupels. Our investigations of cupels unearthed in the archbishops' mints showed that they were made from animal bones (Ulseth et al., 2015). When replicating the manufacture of the cupels, rather than using commercial bone-ash as described from experiments by Téreygeol and Thomas, (2003), we went to a local slaughterhouse, where we were given two calves' heads. The heads were boiled to remove the flesh (Fig. 1) and the bones subsequently cleaned by brushing.

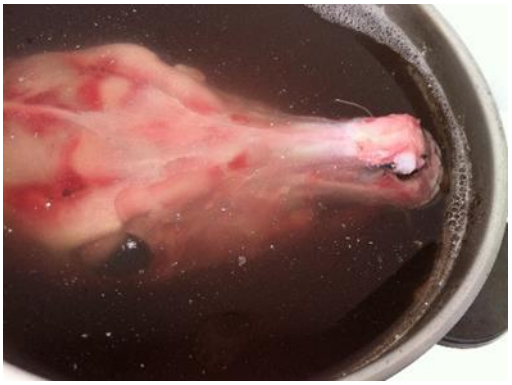


Figure 1. A calf's head during boiling.

The different bones that made up a calf scull were separated and then calcinated by putting them in a wood-fired oven. After 20 minutes the bones were taken out and cleaned to remove the ashes (Fig. 2). Bones from the forehead were broken into pieces, ground in a mortar and sieved (Fig. 3).

[3] See also: Lohne, O. and Ulseth, P., 2016. *Framstilling av beinaskekupeller fra grunnen av og erfaringer med kupellering*. Notat. 15 pages in Norwegian.

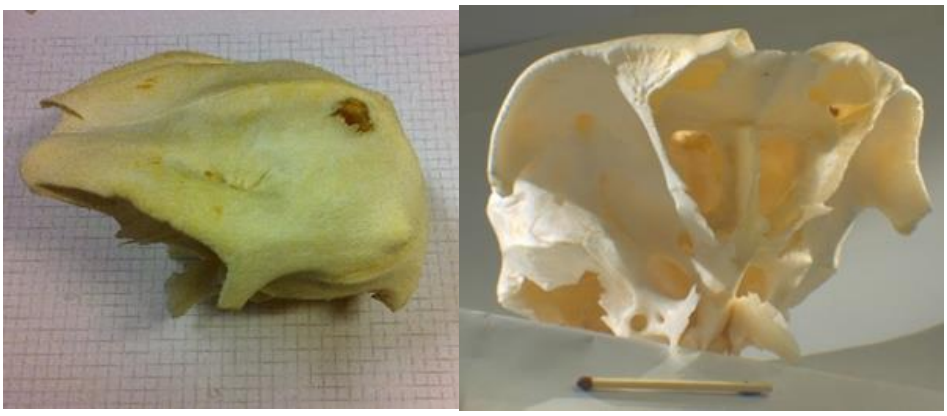


Figure 2. A cleaned calf's scull viewed from two directions. Dimensions of paper squares: 5 × 5 mm.

Figure 3. The forehead bones ground in a mortar and then sieved. Sieve mesh aperture: 1 × 1 mm.

Coarser particles were removed and then ground and sieved once again. The bone-ash powder of 173 g from one scull had a volume of 200 ml in a beaker.

Washing and drying the powder: Warm water was added to the beaker while stirring. The volume of powder + water was 600 ml. Coarse particles settled, while fine particles remained as a muddy liquid. The liquid had a pH of 9–10.

After 30 minutes the water was still muddy, but a thin layer of foam had appeared on the surface of the water. The foam was skimmed off and 370 ml of the muddy water was poured into another beaker. Thereafter, 370 ml warm water was added to the beaker with the remaining coarse particles while stirring. After standing for 16 hours at room temperature, the water appeared clean in both beakers, with settled powders at the bottom (Fig. 4).

Figure 4. The beakers and their contents after standing for 16 hours at room temperature. The water appears clean in both beakers, with coarse powder (left) and fine powder (right) at the bottom.

The water in both beakers was poured off and the powders taken out with a spoon. The fine powder was placed in a separate glass dish and the coarse powder places in six heaps in a further two glass dishes (three heaps in each dish) (Fig. 5). All three dishes were placed in a drying cabinet heated at 50°C for 24 hours until the powders were dry.

Figure 5. Wet powder before the drying process was started.

a) b)

Figure 6. Powders after drying and being ground and sieved once again. a) Coarse powder, b) Fine powder.

After drying, both the coarse and fine powders are ground separately and sieved (Fig.6). The fine powder was later used for the surface of the cupel (i.e. for the 'facing'), while the coarse powders were ground and sieved once again before mixing. The latter material was later used in the to make the main body of the cupels.

Shaping and drying the cupels: A special tool for making cupels has been locally turned from brass (Fig. 7). It consists of a conical ring, 20 mm high and with an inner top diameter of 34.6 mm and an inner bottom diameter of 27.4 mm, together with a stamp, the 'monk', which fits into the top of the ring. The size of the tool corresponds to the most frequent dimension (35 mm) of cupels found during the excavations of the Archbishop's Palace in 1991–1995 (Risvaag et al., 2021, this volume).

Ercker (1951 [1574], p. 29) explained the process of shaping cupels as follows:

> Moisten the powder with strong beer. Do not use too much of the liquid. When you press some in your hand the lump should just hold together. Then pack the cupel mold full of ashes, scrape or wipe off excess, and with one to four blows of the mallet drive the monk into the mold. Wipe the monk clean and with a little wooden spoon sprinkle good facing ashes on the cupel while it is still in the mold. Spread the facing ashes with your fingers. Then put the monk back in again, fit it on straight, and with two or three straight blows, whichever the case may require, beat down the facing in the cupel so that it adheres very evenly. Then lift out the monk and press out the cupel from the mold.

Figure 7. The cupel tool, ring (left) and the 'monk' (right).

We used the following process:

1. We used strong ale from the brewery Nøgne Ø. The ale had a 10% ABV (alcohol by volume) and was unfiltered. We did not test the use of water instead of ale.
2. It was important that the ashes were not too wet. When liquid was observed seeping out between the ring and the monk it indicated that the ashes were too wet.
3. Two ways of drying the cupels were tested: (1) 24 hours in a drying cabinet at 50°C, and (2) two weeks at room temperature. Drying at room temperature for two weeks gave the strongest cupels.

a) b) c)

Figure 8. a) Filling the mould. b) Driving the 'monk' into the mould. c) Cupels with fine grained facing on the bowl surface. Dimensions of paper squares: 5 × 5 mm.

Cupellation of a silver alloy and a gold alloy

Two alloys were tested: a silver alloy with a fineness of 925 ‰ silver (Ag) (the rest was mainly copper (Cu) and zinc (Zn)), and a gold alloy with 585 ‰ gold (Au) and 198 ‰ Ag. When testing the 925 silver alloy, a piece of thin lead plate weighing 1.40 g was folded around the piece of silver weighing 0.15 g and put into a pre-heated cupel lying in a vessel and placed in a furnace operated at a temperature of 900°C. We were able to follow the process by looking through a quartz glass window. The specimen melted quickly and turned into a yellowish white sphere (Fig. 9).

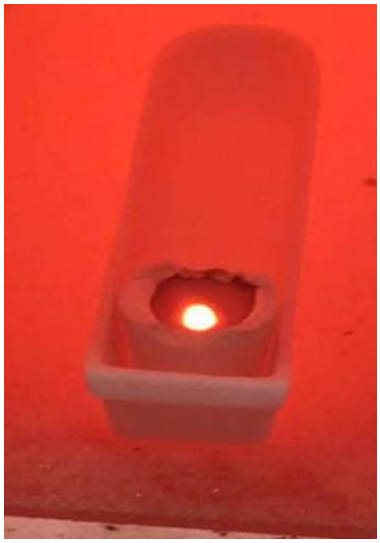
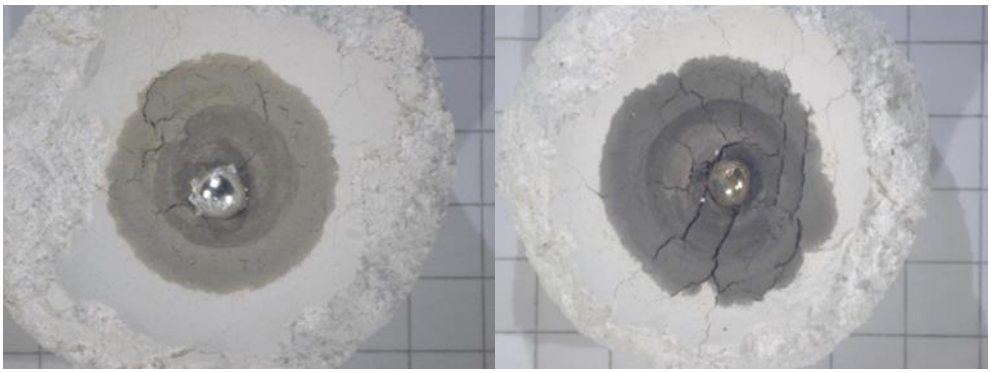


Figure 9. The yellowish white melted metal sphere in the cupel placed in a vessel in a furnace operated at a temperature of 900°C.

During annealing some evaporation above the melt button was observed and a ‘flash’ was seen. After five minutes, the vessel containing the cupel was taken out of the furnace, placed on a table at room temperature and left to cool. The surface of the bead from the silver alloy was smooth and shiny in some places and rough elsewhere (Fig. 10a, and Fig. 10c). Investigations using a scanning electron microscope (SEM) showed that the smooth area was almost pure silver, and the erupted material was silver and lead oxide with a small amount of copper and traces of calcium and phosphorous (from the bone-ash). The weight of the bead after cupellation of a silver alloy was 0.14 g.



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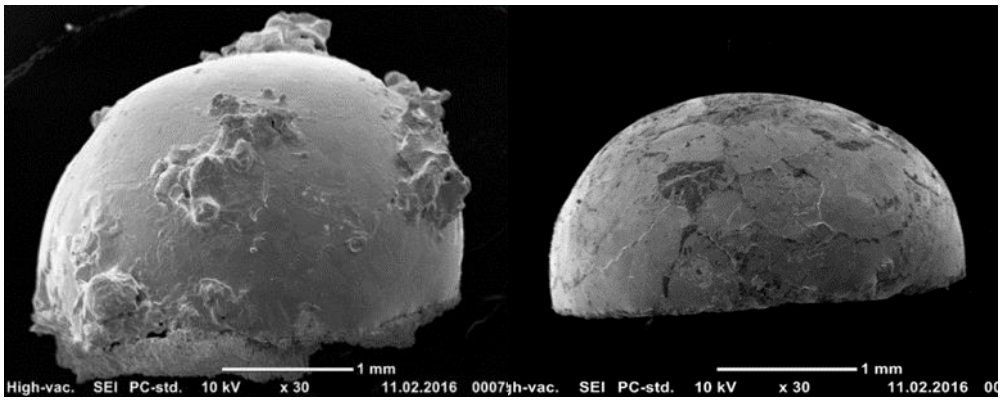


c) d)

Figure 10. Cupels with a bead after cupellation of a) 925 ‰ silver alloy and b) 585 ‰ gold alloy. Dimensions of paper squares: 5 × 5 mm. In images c) and d) the beads have been removed from the cupels and investigated using SEM (side view). Note the rough surface in c), which shows that the silver bead was 'spitting'. No spitting is visible on the gold bead in image d).

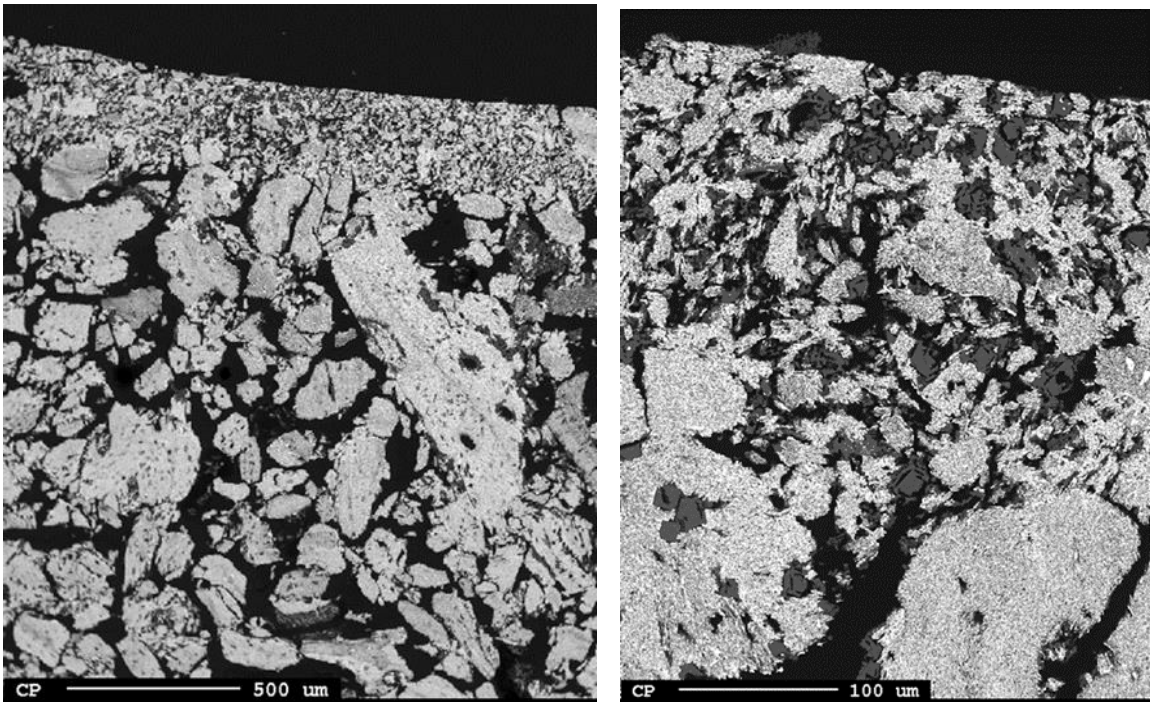
The ASTM - E1335-08 standard recommends that the assaying procedure should be repeated if 'spitting' occurs (ASTM International). Later experiments conducted using the silver alloy and with longer annealing time at 900°C and slower cooling did not result in spitting but there was a small shrinkage cavity on top of the bead and 'islands' of thin lead oxide flakes on the surface (Lohne and Ulseth, 2021, this volume).

The same procedure was used when testing the gold alloy (Fig. 10b). However, no spitting was observed. Instead, some surface grains were displaced, resulting in a rough surface (Fig. 10d).

Investigations of the surface layer of cupels after cupellation

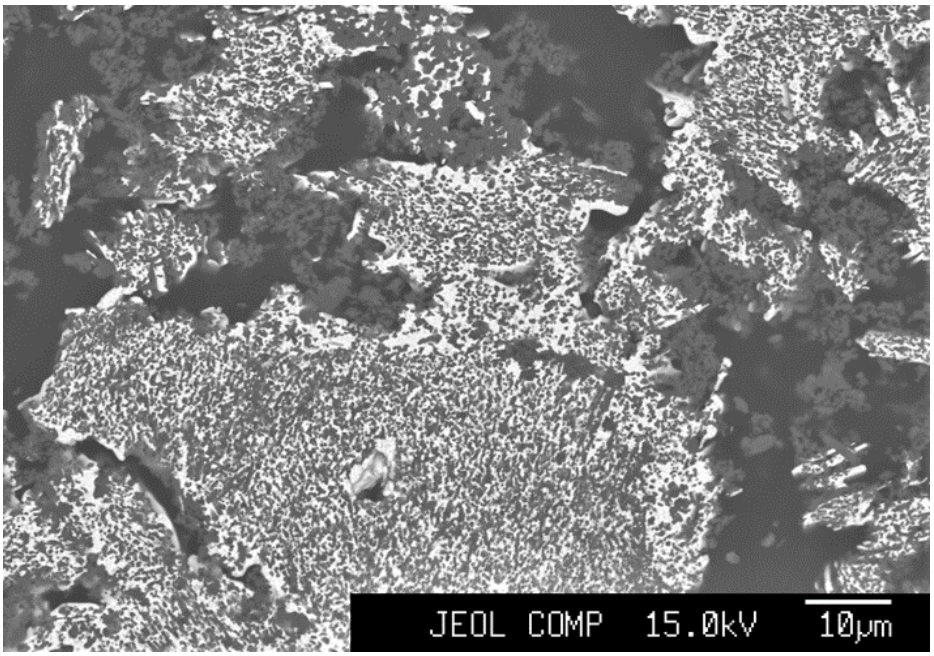
Used replica cupels: A piece of the surface layer of a cupel after cupellation was cut out for further investigation. The piece was cast in resin (EpoFix) and then the cast was cut to show a cross-section of an area, where subsequently we observed that the lead oxide had been sucked into the cupel. After grinding on SiC (silicon carbide) grinding paper and diamond polishing,

the specimen was carbon coated and investigated by using an electron probe microanalyser (EPMA) equipped with an energy dispersive x-ray spectrometer (EDS) and wavelength dispersive x-ray spectrometers (WDS) to measure the chemical compositions of microscopic areas (Figs. 11–13).



a) b)

Figure 11. Microprobe images at different magnifications of a cross-section of a used replica cupel taken near to the bowl surface. a) Microstructures near the surface, with a fine-grained bone-ash layer with a thickness of c.200 μm on top. Black areas are pores filled with resin. Bone-ash particles (grey) are filled with lead oxide (white) (see Figure 12). b) The fine-grained surface layer at higher magnification.



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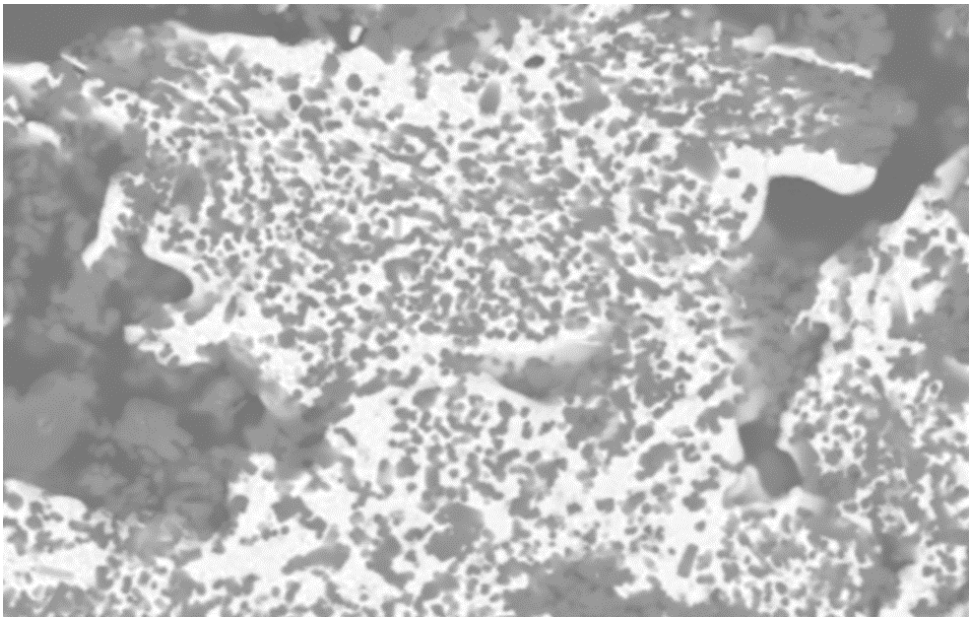
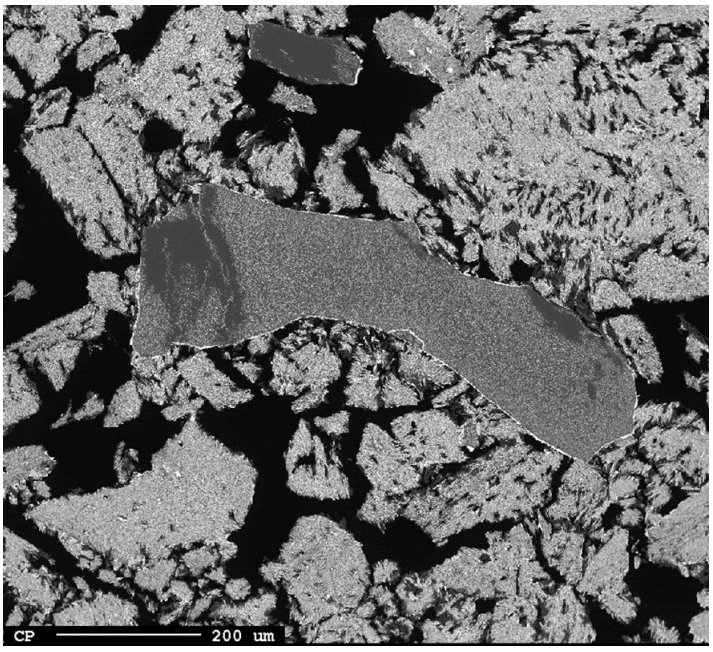


b)

Figure 12. Microprobe images. a) Coarse particles from just below the bowl surface layer. Dark areas are cavities, grey are bone-ash and white are lead oxide. The porous bone-ash particles are filled with lead oxide. b) A central grain seen in a) is magnified 3 ×. Note that the lead oxide is both lying on the surface of the bone-ash particles and filling the fine bone-ash canals. The cavities between the coarse particles (see Figure 11), are not filled with lead oxide.



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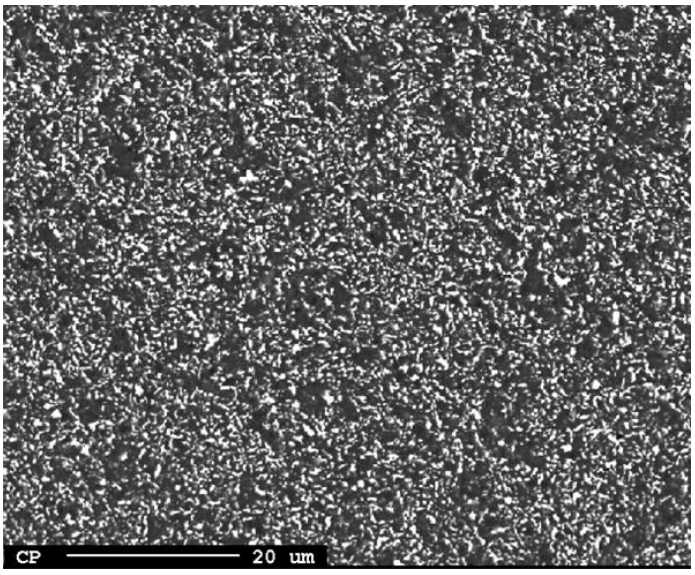


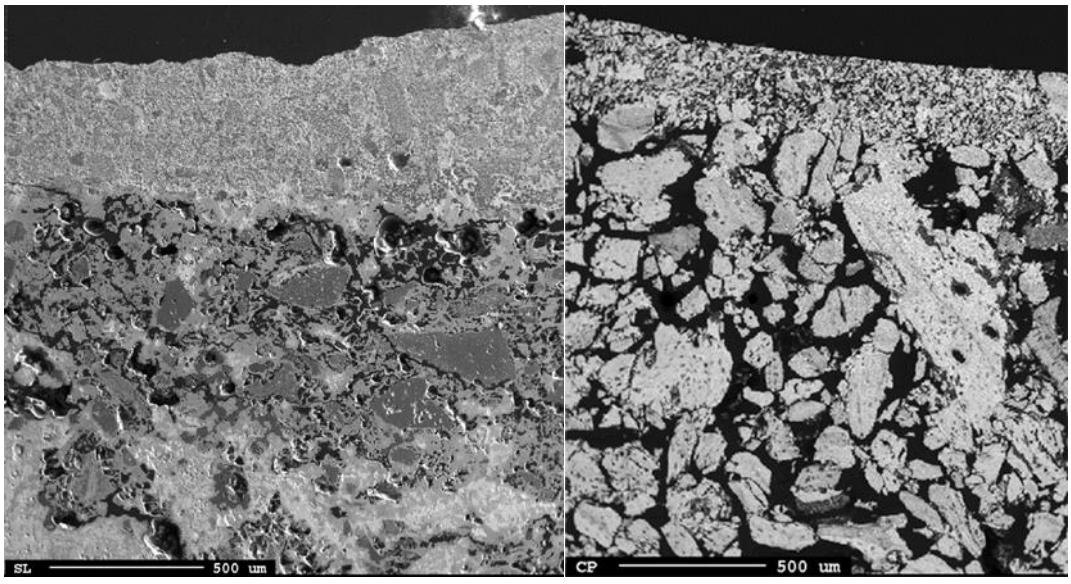
b)

Figure 13. Microprobe images. a) Bone-ash particles appear in different shades of grey according to the size of the lead oxide filled canals (white). Note the white rim of the dark central particle. b) Part of the central dark particle in a) shown at higher magnification. White lead oxide patterns are clearly visible. Molten lead oxide has filled even the finest canals.

Comparison of a used replica cupel and the excavated cupel N124860

Cross sections of cupels are shown in Figs. 14-16:

Figure 14. A cross-section of the excavated cupel N124860. The upper, outer rim diameter is 35 mm and the height 23 mm. Note the fine-grained surface layer (i.e. the facing) at the bottom of the bowl. The two dark areas at the bottom of the cupel show where lead oxide has filled the cavities between the bone-ash particles, as well as the interior of the particles (see Figure 16). Photo: Åge Hojem, NTNU University Museum.



a) b)

Figure 15. Microprobe images from material near the bowl surface of the cupel. a) N124860 and b) used replica cupel. The area fraction of cavities (black) is higher in the replica cupel than in the excavated cupel. Additionally, the facing is thicker and denser in N124860 than in the replica cupel.

Discussion

No tools for making cupels were found during the archaeological excavations in 1991–1995. This raises the question of how and where the cupels were produced. To address this question the main aim of our experiments was to generate some background information. We wanted to determine whether the excavated bone-ash cupels were produced locally in the Mint at the Archbishop's Palace or whether they were imported, as were the crucibles that had production marks pointing to the Hessian town of Großalmerode (Stephan, 1995).[4]

Microstructure of cupels

Unused bone-ash cupels are brittle. By contrast, the used cupels filled with lead oxide are strong and gave the opportunity to produce, by metallographic techniques, specimens that could be used to compare the microstructures of the replica cupels with cupels found during the archaeological excavations.

In Fig. 15 two main differences are seen:

1) The fine-grained facing layer in N124860 is denser and at least 50 % thicker than in our replica cupel. There will of course be variations, and no statistical variations are available, but the tendency may be clear. Except from one or two cupels all excavated cupels had a facing layer (Risvaag et al., 2021).
2) In N124860 there is a greater variation in the size of bone-ash particles in bulk than in our replica cupel, and the porosity is less. The same structure could have been obtained in our replica cupel if some fine-grained bone-ash powder had been mixed in, as proposed by Ercker, and that the compaction of the wet lump of ashes had been better.

Although there may be a greater variation in bone quality in N124860 than in the replica cupels, the structure of N124860 may be better suited for cupellation than our replica cupels.

The flow of liquid lead oxide into the cupel

Figures 11–16 show what happens inside the cupel during cupellation. Molten lead oxide wets the bone-ash, flows over the surface of the particles, and through capillary action is sucked into the canals (whether fine or coarse) in the bone-ash. The bone-ash particles are in contact with each other and the molten lead oxide may flow from one particle to the next without filling the cavities between the particles. When the lead oxide has solidified, the filled bone-ash particles become stronger, and are glued together. Volumes in which the particles have not been hardened this way will, after the cupellation process, continue to be brittle and easily crumble away. This may account for why most of the excavated cupels (83%) lack part of their original bottom part and upper rim (Fig. 3) (Risvaag et al. 2021, this volume).

[4] See also: Lohne, O., Ulseth, P. and Risvaag, J.A., 2015. Undersøkelse av digelfragmenter fra funn i Erkebispegården. En statusrapport. 24 pp. [Unpublished report in Norwegian]

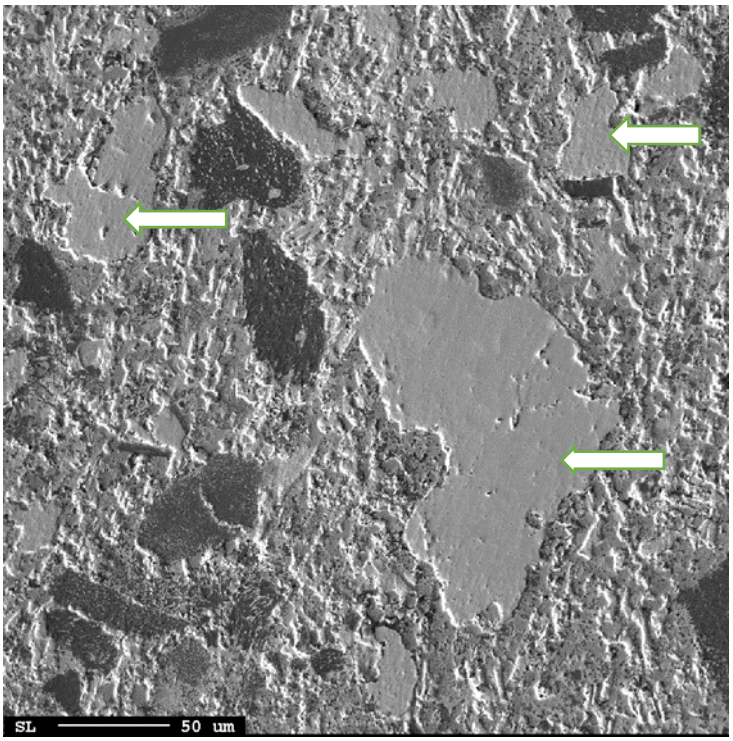


Figure 16. Microprobe images of dark areas from N124860 (see Figure 14). 'Lakes' of lead oxide (pale grey areas, indicted by arrows) have filled the cavities between bone-ash particles. Filled bone-ash particles appear in different shades of grey.

On the other hand, re-use of bone-ash from parts of the cupels that had not been impregnated with molten lead oxide has been tested in other new replicas, with good results. Thus, it is possible that the moneyer in Nidaros reused bone-ash from such materials without problems.

If there is still some molten lead oxide left around the silver bead in the cupel when all bone-ash particles in the cupel have been filled, the melt will start to fill the cavities between particles from the bottom (see the dark areas in Fig. 14 and details in Fig. 16). Therefore, it would have been necessary for the money to select the size of the cupels according to the expected amount of lead oxide produced during the cupellation process and the capacity of the cupel to absorb molten lead oxide. Among the 186 cupels of five size classes, the majority (83%) lacked part of their bottom (Risvaag et al., 2021, this volume). This indicates that the assayer normally selected cupels of the right size when he performed his cupellation measurements.

To summarize, molten lead oxide wets the bone-ash particles, covers the surface of the particles, and is sucked into the particles by capillary action (Fig. 12). The molten lead oxide does not fill the pores between the particles until all bone-ash particles have been filled. When all particles have been filled, the pores between the bone-ash particles start to fill, from the bottom up (Figs. 14 and 16).

Estimation of fineness

At Kongsberg, Norway, the mining of silver started in AD 1625. The Royal Mint Museum, Kongsberg, has some working precepts that inform how the fineness is calculated in a specimen after cupellation:

> The silver bead is picked out of the cupel with a pair of tweezers and carefully cleaned. The bead thus produced, which consists of pure silver, is then weighed on the specified pair of balances. The weight of the silver bead compared with the weight of the starting specimen gives the percentage of silver in the material from which the specimen has been taken.[5]

The silver bead shown in Fig. 10c had a weight of 0.14 g, indicating a content of 930 ‰ Ag in the starting piece of 0.15 g. This is close to the fineness given by the metal producer (K.A. Rasmussen, Hamar), 925 ‰ However, we know that some copper would have been dissolved in the silver and possibly some lead and/or lead oxide and copper and/or copper oxide in between the grains in the bead (Lohne and Ulseth, 2021, this volume). Fig. 10c shows a small piece of facing of low weight sticking to the bottom of the bead. These factors might have resulted in a higher weight than the weight of the pure silver in the bead. However, some silver might have been lost to the lead oxide and either evaporated or sucked into the cupel. The silver content of the lead plate was measured as c.0.5%. This would have added to the weight of the bead. Consequently, we should have measured weight of the beads with a balance scale with a higher precision than the one used in the experiments. Although the result is slightly uncertain, a more accurate result could have been obtained by repeating the process with longer holding times at 900°C, slower cooling, better cleaning of the bead, and more precise weighing.

The cupellation procedure for the gold alloy was the same as for the silver alloy. However, the cupellation process does not distinguish between silver and gold; it only gives the sum of both. Without cleaning the gold bead and correcting for copper dissolved in the alloy, the result was 778 ‰ Au+Ag compared with 753 ‰ (Au + Ag), as given by the producer of the gold (K.A. Rasmussen, Hamar). The same arguments about uncertainty for the silver alloy apply also to the gold alloy.

We may conclude that the replica bone-ash cupels gave fairly good results when silver and gold alloys with known fineness were tested. However, a skilled assayer might have done better.

The phenomenon of spitting

Spitting is a well-known effect when pure silver solidifies. At melting point, in liquid form pure silver contains 0.32 wt % (2.1 at %) oxygen and solid silver contains 0.006 wt % oxygen (at equilibrium) (see phase diagram in Massalski 1986, p. 49). This means that 0.314 wt % oxygen must leave the melt during solidification. If the melt in the button is sufficiently clean after annealing for 5 minutes at 900°C that it behaves like pure silver, and if the silver bead is cooled quickly after the annealing, the metal may freeze on the surface before all internal melt

[5] From: Katalog for Sølvverksmuseet, Kongsberg, 1942. Handwritten, possibly by a secretary under dictation of the foreman of miners Bjarne Sanness. Our translation. Ref. Per Øyvind Østensen, 2019. Cupellation is found under “Probering” in ch. “Oppredning”.

has solidified. Then, internal gas pressure may build up when melt inside the bead solidifies, causing an eruption from within and through the solid surface of melt. If the solidification is more complete and the cooling slower, the oxygen may leave through the surface in canals of melt and not cause any eruption.

It is known that by adding gold to silver the melt will contain less oxygen. In our case, the gold alloy contained more gold than silver and the surface did not show any eruptions (Fig. 10d). Therefore, in the tested gold alloy, the solubility of oxygen in the melt was low and we did not expect to observe eruptions at the surface under cooling conditions.

Conclusions

Following the guidelines given by Ercker in his book *Treatise on Ores and Assaying* from 1574 (Ercker 1951 [1574]), the procedure for making bone-ash cupels is, in our experience, a straightforward process. Although the book was printed many years later than the operative time of the Mint in the Archbishop's Palace, the knowledge of cupellation is likely to have been common among skilled people.

Unused bone-ash cupels are brittle and not suitable for transportation. However, the moneyer in Nidaros in the period AD 1500–1537 would have had access to the necessary ingredients for making bone-ash, tools, and furnaces on his premises. It is therefore reasonable to assume that the bone-ash cupels found during the archaeological excavations in 1991–1995 in the Archbishop's Palace were made locally at the Mint.

The excavated cupel investigated, N124860, might have been of better quality than our replica cupels, which gave acceptable results when used for testing two alloys with known chemical composition. Thus, the moneyer in Nidaraos might have produced cupels of a high standard and measured the fineness of precious metals with an acceptable degree of precision.

Acknowledgements

We thank Goldsmith Helge Karlgård for the providing of silver and gold alloys and for valuable discussions, the organizers of workshop for making the cupel tools in the Department of Physics, NTNU, Birgitte Karlsen, SINTEF, for metallographic preparation of specimens, and Morten Raanes, NTNU, for the microprobe analysis.

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