Initial experiments on silver refining: how did a cupellation furnace work in the 16th century?

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ABSTRACT: Cupellation was for long the only known method to retrieve silver from argentiferous lead. The operation took place in a cupellation furnace, a structure made of a wall supporting a sole (the base for the hearth) and topped with a dome. The hearth, where the alloy was placed, was made from clay and ash. There is very little archaeological evidence of this operation. However, since the 16th century, silver refining has been well documented in detailed and illustrated metallurgical treatises, such as Book X in Georgius Agricola's De re metallica (1556). Conducting experimentations is therefore necessary to understand the technological choices of ancient metallurgists. Eight operations have been carried out over the past two years. They are entirely based on Agricola's record. The whole process is taken into account in this study: temperature and atmosphere conditions, hearth composition, drawing out of molten litharge and structure of litharge cakes.

Introduction

Cupellation is a refining process for noble metals such as gold and silver. Still used in assaying, it was used as well on a larger scale in the metallurgical operating sequence associated with argentiferous lead ores such as galena (PbS) or grey copper ores (tetrahedrite-tennantite series (Cu,Fe)12(As,Sb)4S13). This paper deals with this latter aspect. We will not discuss here the case where lead has to be added to separate silver from copper, and will consider a simple silver-lead alloy like that produced by smelting galena. To retrieve silver, the alloy is placed on an ash hearth. This operation, known as cupellation, requires an oxidizing atmosphere to oxidise the molten lead. At over 890°C, lead oxide (litharge, PbO) is liquid and floats on the molten alloy. Part of the litharge is absorbed by the ash, but most of it has to be gathered and drawn out of the furnace. In this way the alloy becomes enriched in silver. A temperature above 960°C is necessary to keep the noble metal molten until the end of the operation.

Cupellation gives the possibility of getting total purity. Yet, in general, large-scale operations aimed at an incomplete refining. The alloy was brought up to the desired fineness during a subsequent operation. This second cupellation was carried out on smaller amounts of metal already at a high fineness, and in another kind of furnace This last process is not a part of these experiments.

Known since antiquity (Wagner and Weisgerber 1985) cupellation was for long the only known method for retrieving silver from argentiferous lead. Its use began to decline with the appearance of Pattinson's enrichment process in 1833 and the later Parkes process for the recovery of silver. Yet very little archaeological evidence of this operation remains from medieval times and the early modern period, mostly because all by-products were recycled, including litharge cakes, ie the ash hearths impregnated by lead oxide. However, since the 16th century silver refining has been well documented in detailed and illustrated metallurgical treaties such as Vanoccio Biringuccio's *De la pirotechnia* (Smith and Gnudi 1990, Bk III, Ch 7), and particularly Georgius Agricola's *De re metallica* (Hoover and Hoover 1986, 464–83). At this period, the cupellation furnace was described as a structure made of a wall supporting a sole (the base for the hearth) and topped with a dome. The hearth, where the alloy was placed, was made from clay and ash.

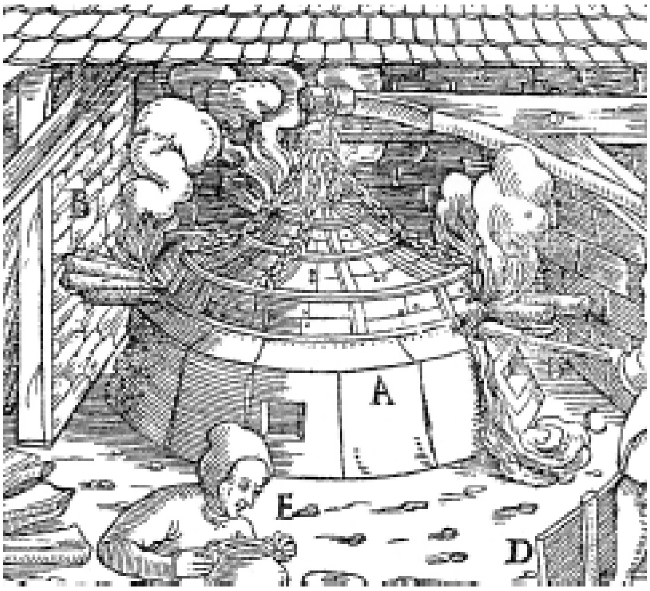


Figure 1: One of the furnaces for large-scale cupellation illustrated in De re metallica.


Figure 2: Experimental furnace during a firing.

Which features of 16th-century furnaces determine the necessary conditions for the operation? Since historical sources are incomplete and biased, conducting experi-ments appears necessary to understand the technological choices of ancient metallurgists. What should be the shares of natural and mechanical air draughts? How can temperature be comprehended and controlled in this kind of furnace? Which qualities are required for an ash hearth? How to manage the litharge?

The experiments were carried out at the *Plate-forme d'Archéologie Expérimentale des Arts du Feu* in Melle (Deux-Sèvres, France), dedicated to research on ancient metallurgical operations. The experimental platform was designed to study and explain to the public three essen-tial stages of silver-lead metallurgy: ore smelting, large scale cupellation of argentiferous lead, and fire-assay. Large scale cupellation experiments are entirely based on Agricola's book, from the construction of the furnace to the conduct of the process. Eight firings were con-ducted between April 2007 and July 2009. They form a firm basis on which future operations will develop.

Furnace structure

In *De re metallica*, the cupellation furnace is made of a circular stone wall supporting a round stone sole for the hearth and topped with a dome which can be moved aside by a crane (Hoover and Hoover 1986, 467; Fig 1). The hearth is made out of clay at the bottom and ash on top. A pair of bellows is used for forced air draught. The furnace runs on wood. The experimental furnace built in Melle (Fig 2) is based on Agricola's structure. For a comparison of dimensions and construction materials, see Table 1. Main material differences aim at a greater flexibility for the experiments, needed to compensate for the loss of technical knowledge of industrial large-scale cupellation.

The cupola

The experimental dome is a composite structure, made of ceramic inside and metal outside. The clay body is made out of powdered clay, grog and hemp tow, the last to give mechanical strength to the dome before firing. The one-piece clay cupola is about 30mm thick. The first firing took place 18 days after its construction. A metal cupola, made of steel plates screwed onto a metal structure, covers the ceramic cupola, with a common base but no other contact. In *De re metallica*, the cupola is a ceramic-metal composite as well but it is built with a different technique. The clay hangs in direct contact with the metallic dome, due to pierced iron plates fixed inside it.

The experimental cupola has four openings: one on top, with a lid, and three doors. The bellows' tuyere is aligned with the rear door, for forced air draught. One of the doors in front is used to work inside the furnace and draw litharge out. Fuel can be loaded through all three doors. The lid, also composite, can be lifted up separate from the dome, in order to regulate air draught.

The dome weighs 300kg and is removable with a crane. Each time the hearth is remade, ie before each firing, the cupola is lifted up and placed on trestles. Besides its refractory function during firings, the ceramic structure also has to withstand the unavoidable jolts of transport.

Table 1: Construction data for large-scale cupellation furnaces

		De re metallica - Agricola (text)	Experiments
base	material	~ 14 stones	bricks
	diameter	14 x stone's length ÷ π = 1.65m	1.65m
	thickness	1 foot (290mm)	130mm
	height	4 feet and 3 palms (1.40m)	0.85m
	in the ground	1 foot and 1 palm (370mm)	no
	air vents	6	4
	support the hearth	inner brick walls and circular stone wall	no
hearth	base	stone sole	steel table
	diameter	base diameter - (2 x thickness) + (2 x interior cut) = 1.20m	1.40m possible, 1.10m used
	clay	lute mixed with straw	clay, chamotte and hay
	ash	ashes from which lye has been made	wood ash
	tools	different rammers, feet, iron plate and hands	earth rammer and light mallet
	clay thickness	unknown	70mm
	ash thickness	unknown	50mm
	depth of hollow for the alloy	2 palms and 2 digits (180mm)	40mm
dome	exterior	iron bands and bars	steel sheets
	interior	lute	clay, chamotte and hemp tow
	height under dome	unknown	0. 50m
	openings	4 doors and 1 lid	3 doors and 1 lid
ventilation	type	water-wheel driving 2 bellows	electric blower

Since experiments are spaced in time, the movements of the dome are multiplied, but it is still working perfectly after eight experiments (Fig 3).

The hearth

The hearth requires a good mechanical strength to bear the weight of the metal and the wood that are loaded on it, refractory properties to withstand heat, and a chemical composition that does not react with lead, hence the ash layer on top.

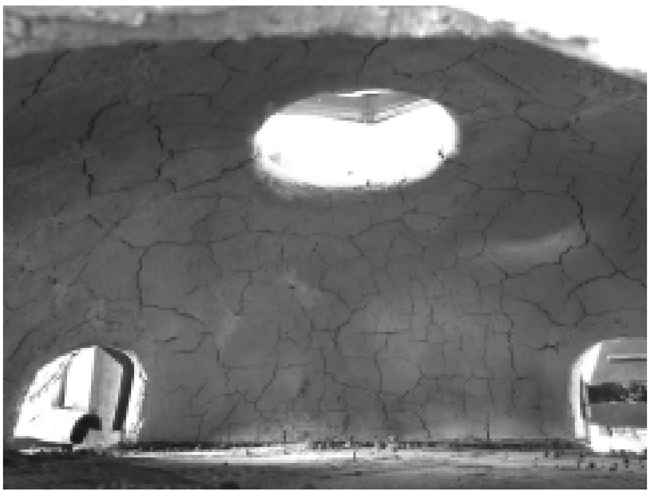


Figure 3: The ceramic cupola is still perfectly useable after eight firings.

The circular base of the experimental furnace is built out of bricks, covered with clay, and has four air vents. Height is variable, as two or three rows of bricks can easily be added. This wall surrounds a round steel table

Figure 4: Hearth making illustrated in De re metallica; ash is tamped with a rammer.

Figure 5: Experimental hearth making; the technique shown in Figure 4 is used.

with adjustable height as well. The hearth is built on this table, and is partly or completely renewed before each firing. This is the so-called working space.

Making the hearth is a decisive phase for successful cupellation. The hearth is composite with the lower layer, lying directly on the steel table, made out of clay. Before each operation, this part is repaired if necessary rather than remade. On top, the surface which will be in contact with both fuel and metal is made out of ash, and is single-use, at least for the part that gets impreg-nated with litharge. The experimental furnace is 1.4m in diameter, but the hearth is only 1.1m wide as it is enclosed by a row of bricks and clay connected to the outside wall (Fig 5).

First a moistened mixture of powdered clay, grog and hay (red fescue) is rammed down on the hearth surface. As in *De re metallica*'s explanations and illustrations, the lower clay layer is tamped by a man standing on the hearth with an earth rammer (Hoover and Hoover 1986, 469–71; Figs 4 and 5). However, in the experi-men-tal furnace, the vibrations of the steel table lessened the efficiency of the compaction, and the circular brick wall is not strong enough to stand the pressure of the mixture being compacted. The operation was therefore completed with a light wooden mallet, hitting on a board. To improve its resistance, the top of the wall was hooped in July 2009. In the end, the clay layer is about 70mm thick. Its total weight is around 200kg.

Over this clay base lies the ash layer. Either bone ash or wood ash can be used, as for assay cupels. For large-scale cupellation, Agricola only mentions ashes from which lye has been made ie washed wood ash (Hoover and Hoover 1986, 469). In the experiments, wood ash was used, but since it was not washed, once humidified it is very alkaline.

The ash layer is laid when the clay base is almost dry. Ash compaction begins as for the clay mixture, and is completed by a direct tamping of the ash with the small wooden mallet. Because of the small quantities of argentiferous lead used so far, a final thickness of 50mm is sufficient to ensure that no litharge will reach the clay beneath the ash.

Drying

The hearth has to be dried before it is used. Agricola insists on heating the hearth with charcoal for several hours, with great care, to properly dry the whole surface (Hoover and Hoover 1986, 471). He also comments on the six air-holes in the circular wall, through which moisture escapes when the furnace is heated, created so that the ash surface would not be damaged (Hoover and Hoover 1986, 467). In the experimental furnace, the steel table prevents such drying as water would remain against it. Yet, thanks to the four air vents in the circular brick wall, it is possible to dry the furnace by lighting a fire on the floor, underneath the hearth.

The clay shrinkage is about 20mm out of the initial 1.1m diameter. Since clay shrinks more than ash, link-ing these two materials could cause cracks in the ash. When formed over two different days, the layers have no adhesion. The ash surface forms a hard crust. During drying, vapour escapes at the junction between the two layers and through the edge of the hearth. This way, the surface is kept almost intact. Yet if a crack appears in the clay, it spreads into the ash layer as well, and has to be repaired before adding metal and starting cupellation. The surface is otherwise left untouched, whereas in De re metallica, after drying, the ash layer is entirely resur-faced and at least repaired, and sometimes a facing is added (Hoover and Hoover 1986, 472).

Drying is necessary. Besides the cracking problems, a damp furnace cannot work properly. Even when only the ash layer of the hearth is remade, which does not involve much water, a thorough drying is necessary. Furthermore, the furnace is sheltered but nevertheless outdoors. The base clay layer, even if old, must not be considered

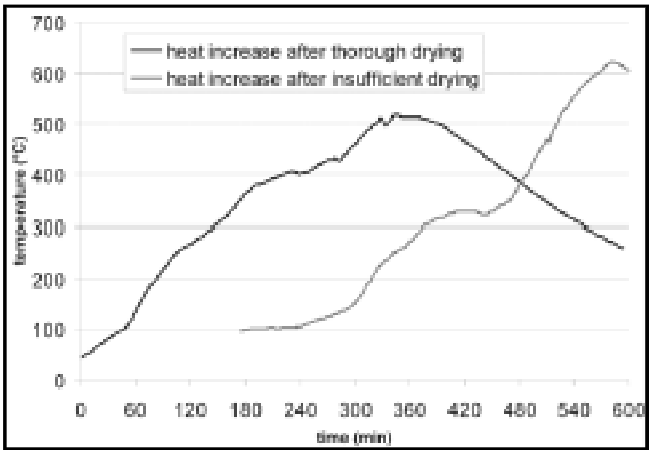


Figure 6: The effect of hearth drying on the operation of the furnace. Temperatures were measured in the clay layer at the centre of the furnace. The measurements start with the firing. The maximum temperatures correspond to the operation stopping.

totally dry. Figure 6 shows the temperature measured by a thermocouple placed in the clay layer at the centre of the furnace. When the furnace has been dried, the hearth accumulates heat in the same way as the ceramic cupola and the temperature rises steadily. But when drying is incomplete, the temperature stays at 100°C until all moisture is gone. Meanwhile, cracks may appear, leading to the loss of metal inside the hearth, and to the failure of the operation. Thoroughly drying the hearth before starting the operation must therefore be systematic.

Temperature and atmosphere conditions

Atmosphere: ventilation

The experimental furnace has a pair of manual bellows for mechanical air draught. Yet so far, an electric blower has been used, allowing reproducible conditions (Fig 7). The metal tuyere emerging in the furnace has been

Figure 7: The ventilation system. For now the electric blower replaces the bellows.

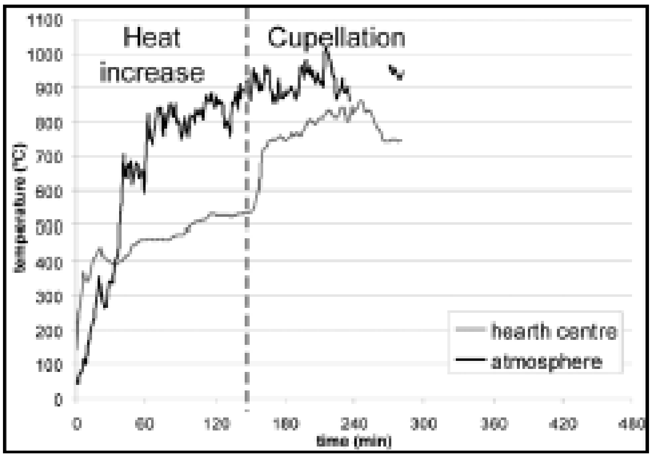


Figure 8: First firing regime where the cupellation stage immediately follows the rise in temperature.

covered with clay to maintain its airtightness. Draught speed ranges between 1.6 and 3.0m/s.

Forced air draught does not really affect temperature. However, it is crucial regarding the redox conditions as a strongly oxidizing atmosphere has to be produced deep inside the furnace. Moreover, the air flow pushes aside the molten oxides floating on top of the alloy, ac-cumulating and directing them towards the doors of the cupola where the litharge is drawn out.

Agricola indicates that the stone sole has to fit the exterior stone wall, and that any crack must be filled (Hoover and Hoover 1986, 467). The steel table of the experimental furnace replaces the stone, but the principle remains the same. The hearth is entirely connected to the circular wall with lute. In this kind of furnace, natural ventilation must not prevail, being in contradiction with heat accumulation in the dome. This principle will lead later on to the modification of the cupellation furnace into a reverberatory furnace, where the alloy being processed is separate from the fuel.

Temperature: firing stages

Two different ways of conducting the operation were compared during the experiments. Two steps are sufficient (Fig 8). After the temperature has risen, the so-called cupellation stage can follow immediately, with the opening of the dome lid and the running of the electric blower. But this ignores the potential of the ceramic dome to accumulate heat. Adding an intermediate phase, during which the cupola, and even the hearth, store up heat, is more efficient (Fig 9). As all openings are closed, additional wood consumption is limited and the ac-cumulated heat will be beneficial during cupellation. The temperatures reached in the second case are not higher, but the acquired thermal inertia allows the opening of

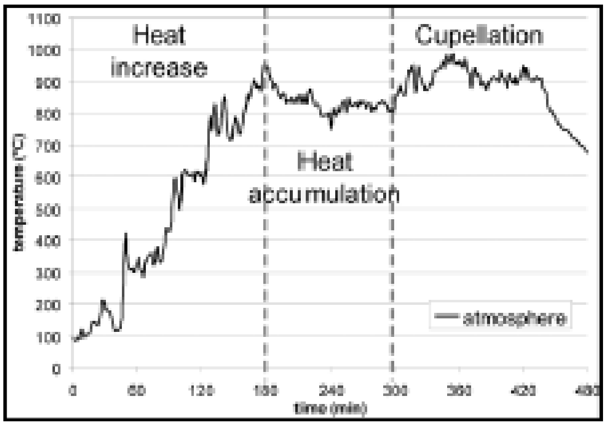


Figure 9: Second firing regime, with an intermediate stage of heat accumulation inside the ceramic cupola.

some doors, to permit work inside the furnace, without losing too much heat.

According to measurements, in both cases 890°C is quite easily and consistently reached in the atmosphere. Yet the temperature only intermittently reaches 960°C. But these data only reflect local conditions which can not be applied to the entire volume of the furnace, including the area where the alloy is placed.

Tests with assay cupels

The thermocouples used for measuring temperatures are handy but insufficient devices. They only give relative and, most important, very localized information, thus not permitting the assessment of the real temperature gradient inside the furnace. Furthermore, cupellation also relies on an oxidizing atmosphere. To test the

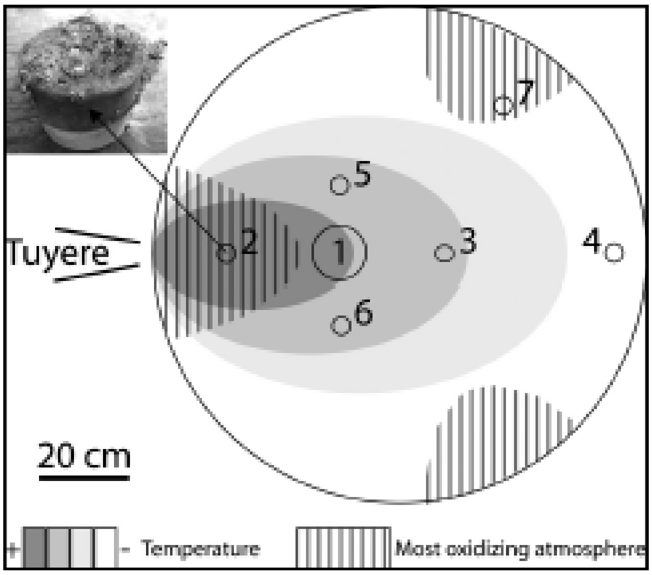


Figure 10: First arrangement of assay cupels on the hearth and the firing conditions model. Darker shades represent higher temperatures and the striped areas the most oxidizing atmosphere. Only assay No2 was successful.

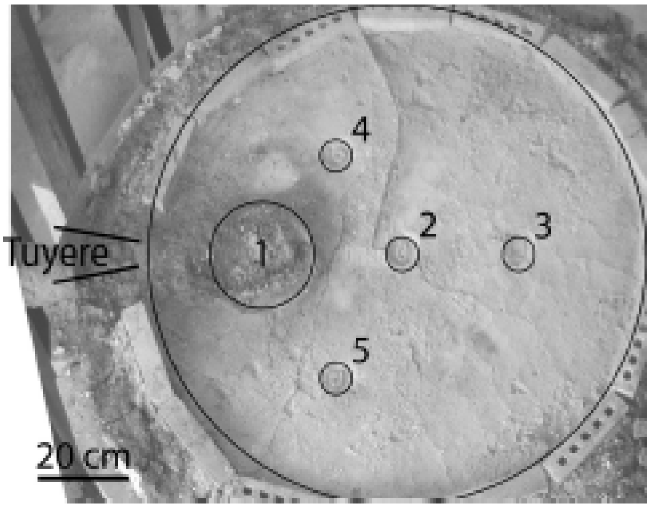


Figure 11: Second arrangement of assay cupels on the hearth (cleaned up after the firing).

cupellation possibility all over the hearth, small assay cupels containing small amounts of metal can be inserted at different locations. Thus if one of these individual refinings succeeds, ie if silver is separated from lead in the end, it means all conditions necessary for cupellation were present at its position, at least for a short period.

For the first test, seven assay cupels each containing about 15g of lead containing 10% silver were inserted in a loose clay hearth, around a central ash hole filled with 1.05kg of non-argentiferous lead. The chosen locations were distributed along the axis of the tuyere, around the main position where the hottest part of the hearth was expected, and in front of one of the cupola doors (No7 in Figure 10). Only assay No2 was successful.

From these results, a model of the temperature gradient inside the furnace is suggested (Fig 10). The maximum temperatures are reached in front of the tuyere door, and along its axis. It may seem unlikely for the hottest place to be situated right under the tuyere, since it blows cool air, and it is the only door in the cupola that is never totally closed. However, this opening is the most protected by piled fuel. The atmosphere is more oxidizing in front of the three doors in the dome, one of them corresponding to the mechanical air draught. The location of cupel No2 is the only one which has both the high temperature and the oxidizing atmosphere required.

From this model, a different scheme of arrangement of test cupels was used during the next firing (Fig 11). This time, all holes were directly dug in the ash surface of the hearth. The largest amount of alloy, 860g of lead with 7.5% silver, was placed right in front of the tuyere (No1).

Other 30g assay samples, with the same composition, were distributed around it. Only the main location was successful, which validates the temperature model. It shows most of the surface of the experimental furnace is not optimized for cupellation. The efficient area must therefore be broadened.

Working inside the furnace: managing the metal

Fuel

Loading this kind of furnace is delicate since alloy and fuel occupy a single space. In the experiments, the fire on the hearth is started with embers and small wood pieces, at the centre of the furnace. The fuel consists in a combination of chopped woods, oak, hornbeam and ash, 500mm long. The fire is spread to properly heat the cupola. Fuel is loaded through all doors, with three sticks per door every ten minutes or so during the temperature increase phase. Wood is always positioned leaning on previous sticks so as to maximise air circulation and thus oxidising conditions. During the heat storage phase, wood consumption is much lower. When reaching the cupellation stage, all the embers floating on the liquid metal are pushed aside, so that the entire alloy surface oxidises more easily, and wood is loaded only along the rim of the cupola. As time passes, fuel loadings get smaller, but more frequent. At the very end, only the smallest pieces of wood are used.

Agricola indicates that the fire is started with embers and small pieces of wood but the main fuel consists of a total of 40 to 60 pieces, sticks of wood several meters long (Hoover and Hoover 1986, 473–474). Their use requires a complicated set-up with trestles, weights and iron bars, so smaller sticks were used so far in the experiments.

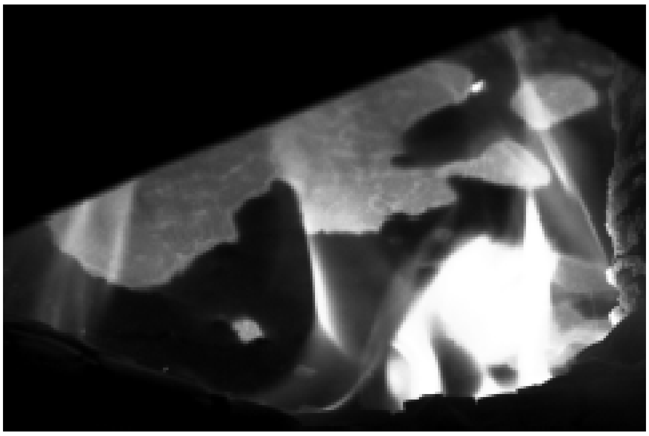


Figure 12: During cupellation litharge (mid grey) forms on the surface of the molten lead (lighter grey). The liquid litharge breaks up and moves down round the lead, to get into contact with the ash layer.

For the drying stage, a little charcoal was used at the beginning, but the fuel is mostly wood. The total amount of fuel used during a firing is the same with or without a drying phase. In *De re metallica* only charcoal is used for drying, but as mentioned above this phase had to be adapted.

Cupellation phase

During cupellation, litharge forms darker areas on the metallic lead (Fig 12). Some of this oxide is absorbed in the ash, but most of it has to be extracted from the furnace. In assay cupellation, all the litharge is allowed to impregnate the ash. In refining, this is neither poss-ible nor desirable, for technical and commercial reasons. First, the quantities of ashes required for each operation would be excessive. Second, the temperature gradient in the hearth would prevent litharge from penetrating deep into the hearth. In an assay furnace, the whole cupel reaches high temperatures since it is heated from all directions, whereas in the present case, the hearth is only heated from the top, and ashes are very refractory. Agricola indicates that the impregnation is often around 70mm deep, for a single operation where several tons of argentiferous lead are processed (Hoover and Hoover 1986, 472 and 475). Finally, even if the primary aim of cupellation is to obtain silver, litharge certainly is not a waste material. Both litharge and the lead obtained by its reduction were marketed, with the exception of litharge from early stages of cupellation, which con-tains too many impurities, and that from the end of the process which is too rich in silver. The latter were processed once again. Litharge cakes were used as flux for ore smelting.

Figure 13: Drawing out of the litharge (C) illustrated in De re metallica.

Figure 14: In the experiments, litharge is directed towards a cupola door by a channel dug in the ash and a long scraper.

To draw litharge out of the furnace, a channel leading to one of the cupola doors is dug in the ash, and a bar is used to control the flow (Hoover and Hoover 1986, 475; Figs 13 and 14). Forced air draught helps direct the molten oxide. During the experiments, yellow crystallized foliate litharge was obtained. Litharge can also be accumulated on a tool like a poker, a method that Agricola already associates with ancient times (Hoover and Hoover 1986, 475; Fig 15). One has to be careful not to remove metallic lead with the litharge, since it would mean the loss of some silver. Both the drawing out of the litharge and its absorption by the hearth result in the reduction and fragmentation of the remaining melted alloy.

This operation is very delicate and not yet mastered. Doors must be opened to work inside the furnace, yet

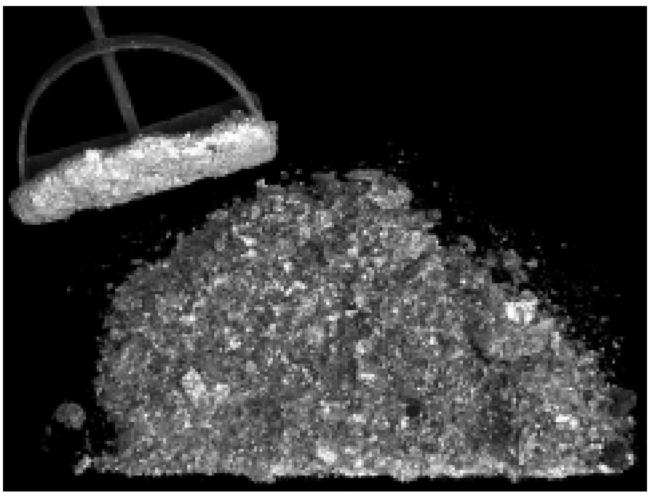
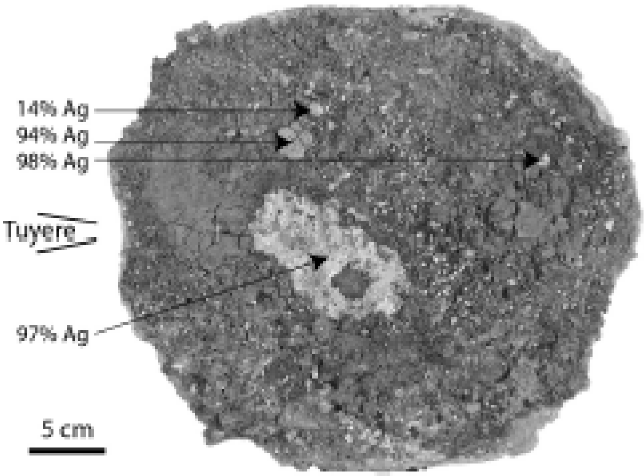


Figure 15: The experiments produced yellow crystalline foliate litharge, some of which accumulated on the scraper.



igure 16: Experimental litharge cake with a large mass and prills of refined silver of varying fineness; the initial metal was 7.5% silver.

heat loss has to be limited. Accumulating enough molten litharge to be able to make it flow out of the furnace is difficult given the small quantities of alloy that were used in each experiment. With 26kg of lead processed at once as the maximum, all the oxide produced could easily be absorbed by the hearth. Eventually, an experi-ment with 300kg of alloy is planned.

Litharge cakes and silver

At the end of the firing, the refined silver has to be separ-ated from the lead-impregnated hearth or litharge cake, ie ash impregnated with lead oxide. In the experiments, the metal does not completely gather together. As well as the main silver mass, the fragmentation of the molten alloy creates many metallic prills distributed over the whole surface of the hearth that was in contact with the alloy from the beginning; they are concentrated es-pecially at the periphery of the impregnated part of the hearth, opposite the tuyere. The ash surface might either be insufficiently smooth or sloping. The ratio between the quantity of alloy and the surface area of the hearth might also play a part.

The litharge cake and metal shown in Figure 16 are representative of those obtained in the experiments. In this case, the initial metal weighted 5kg and contained 7.5% silver. Its cupellation gave metal prills varying from poorly-refined to almost pure silver (14–98%) as well as a main well-refined metallic mass (220g; 97% silver; analyses by SEM-EDX). The silver has to be gathered for a second refining process to get it to the desired fineness.

The litharge cake was easily removed from the furnace. Its bottom is very even, since the penetration depth of

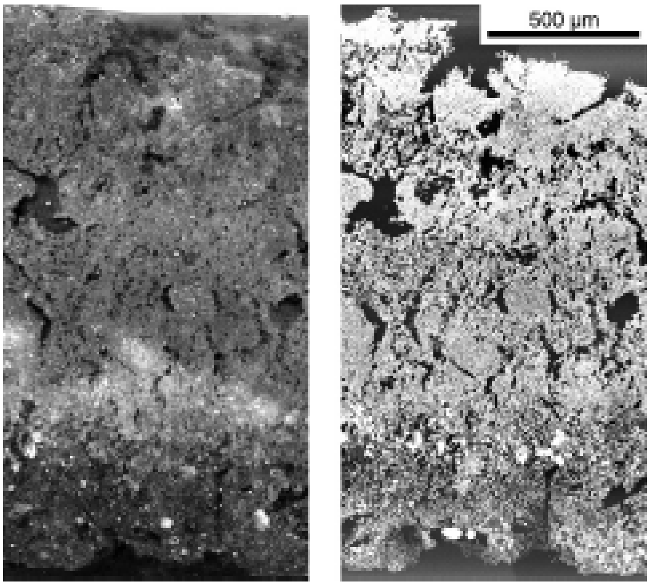


Figure 17: Vertical section of an experimental litharge cake. Optical (left) and SEM image (right). Below the litharge concentrations (bright spots) the bottom quarter is poorer in litharge; the non-impregnated ash has already been lost.

the oxides mostly depends on the temperature gradient inside the hearth. The centre is a little thinner as the oxides formed on the surface of the molten alloy float on it and impregnate the ash only at its edge. So far, the maximum thickness for the litharge cake has been 20mm. The inner structure is composed of several horizontal layers with varied litharge contents (Fig 17).

The ash untouched by either the alloy or the oxides remains intact (Hoover and Hoover 1986, 476). All non-impregnated ash is reused for the following firing, sieved and mixed with fresh ash. In general, the clay layer is not affected by the operation and can also be reused; if cracks are present, they are filled. If some metal reaches the clay surface through cracks in the ash layer, a green glaze forms at the interface and the impregnated ash sticks strongly to the clay, preventing its reuse.

Conclusions

Cupellation has been carried out successfully, and the furnace structure is validated. These experiments confirm the importance of ventilation. Forced air draught is used in order to create a strong oxidizing atmosphere and does not prevent heat accumulation in the cupola. Experiments in such a large structure are time consuming. We are not professional metallurgists, and even though assay cupellation still exists, the technical knowledge for large-scale cupellation is now lost. Each question must be tackled step by step, during several firings

This study continues. Our next operations will focus on two main limiting parameters: quantity of alloy and ventilation. The mass of argentiferous lead processed-has to be increased to reach key stages such as the management of the litharge. Then all the litharge will not impregnate the ash, and enough will accumulate to be drawn out of the furnace through the channel in the ash. The electric blower is not an accurate substitute for bellows. In the present conditions of use the air draught is insufficient. In addition, the blower provides an air flux but bellows apply draught pressure. This has to be adjusted since ventilation is such an important parameter, even if difficult to quantify.

These experiments not only aim at better understanding the ancient metallurgists' choices regarding the structure of the furnace and the conduct of the operation, but are also necessary to create analogues that can be compared with archaeological finds, especially litharge cakes. Their compositions can be extremely diverse, from bone-ash litharge cakes from the 17th-century royal mint of La Rochelle, France (Guirado and Téreygeol in press), to wood-ash furnace bottom from Santa Isabel, Bolivia (Téreygeol and Cruz in press). Therefore, reference material will be produced by testing different ash compositions for the hearth.

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