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A study with field and museum work using portable analysis pXRF

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Cover: Completely preserved crucible from Augst/CH, Roman town of Augusta Raurica. Covered on the outside with a hard-wearing and protective layer of clay ("lutum"). After Furger 2018, pl. 15, cat.-no. T475; photo Alex R. Furger

Back cover; "Traders hoard" with new, unused Roman crucibles from a deposit in Insula 19; clay group 1, originating from Châtelat in the Swiss Jura (photo Ruth Steiger, Roman Museum Augusta Raurica).

The origin of the clays of the Roman crucibles from Augusta Raurica (Augst, Switzerland). A study with field and museum work using portable analysis pXRF

by Alex R. Furger[1], and Markus Helfert[2]

Keywords: clay reference groups, crucibles, metallurgy, Roman crafts, surface analysis

Abstract

The project involved analysing 893 Roman crucibles for non-ferrous alloys from Augusta Raurica (Switzerland) using laboratory and portable devices. This resulted in the identification of 5 clay groups, which were compared to 60 reference groups from the surrounding area. Most of the crucible clays came from 2 clay deposits, one of which was located nearby, while the other was situated 50 km away in the Jura region.

The metals casted show a large variety of bronze- and brass-alloys, due to scrap-recycling. Brass making by cementation could be proved in many crucibles.

The crucibles were composed of two layers: a wheel made core of fired ceramic and a *lutum* layer made of local materials applied to the exterior (and sometimes a thin engobe applied to the interior). In the casting process, the mechanically stabilising and insulating *lutum* swelled up and vitrified considerably.

1. Introduction

1.1. The site of Augusta Raurica and the evidence of its bronzeworking

Augusta Raurica was a Roman town on the Upper Rhine near Basel in Switzerland (Fig. 1). Founded ca. 15 BC, the *colonia* gradually evolved into the present-day village of Kaiseraugst from around AD 400 onwards (Berger 2012; Furger 1995). At the height of its boom around AD 200 the town had a population of some 16,000 inhabitants. Approximately one quarter of the ancient area of the town has to date been excavated. 1.7 m finds and a vast amount of excavation records are available to scientists for their active ongoing research into the town.

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Augusta Raurica was an important trading hub and crafts centre located in the area where the River Rhine ceased to be navigable. Bronze foundries have been discovered and excavated in several places (Martin 1978; Furger 1998). The body of evidence pertaining to the casting of non-ferrous metals contains hundreds of artefacts which have been analysed in the last five years (Furger 2018). The crucibles alone account for 893 catalogue entries.

1.2. Material analysed, sample selection and questions dealt with in this paper

Many amorphous finds, for the most part slag-like objects, cannot be interpreted by means of a visual examination and cannot be attributed to any particular craft. The same can be said for the crucible fragments, which often exhibit green discolouration pointing to the presence of copper alloys. Other remains and metalworking tools, including clay and stone moulds, mis-cast and semi-finished objects, possible touchstones (Furger 2017), slag in various colours, as well as unidentifiable and even dubious objects, barely allow us to interpret them unless they are analysed with regard to their metal content.
Many of these objects connected to bronzeworking were analysed on site at the Museum Augusta Raurica, in a non-destructive manner, without sample preparation and in a timely fashion by using portable XRF analysis. In addition to the investigations dealt with here, this method will allow us to analyse casting moulds, soil samples, slag and utensils, and to identify hundreds of alloys (in uncorroded borings taken from bronze objects found in workshop contexts).

This paper deals with the analysis of a big number of Roman crucible fragments, the chemical characterisation of five clay groups and their correlation with clay samples from various sites in the area of the ancient *Colonia Raurica* (Fig. 1). The questions asked are:

- Is it possible based on geochemical analyses to identify different clay types that correspond to different clay deposits and thus different origins of the crucible clays?
- We took clay samples from approximately 60 deposits within a 50-km radius around Augusta Raurica and analysed them (in addition: Eramo 2006). Can any of the deposits be correlated with the crucible clay types defined?
- Is it possible in the case of clays from outside the locality to ascertain whether they were brought to Augusta Raurica in order to be made into crucibles there or whether

the crucibles themselves were sold to the consumers in Augusta Raurica as finished products?

- Which methods are best suited to analysing the metal residue (green staining, coloured “glazing”, metal drops and spills) mainly visible in crucibles?
- Which alloys were melted in the crucibles in Augusta Raurica and what is the difference in the evidence of metallic traces in the clays, the green spots, the glazings and the metal prills?
- Is there any evidence for brassmaking by cementation of zinc ore (e.g. calamine) and copper in crucibles?

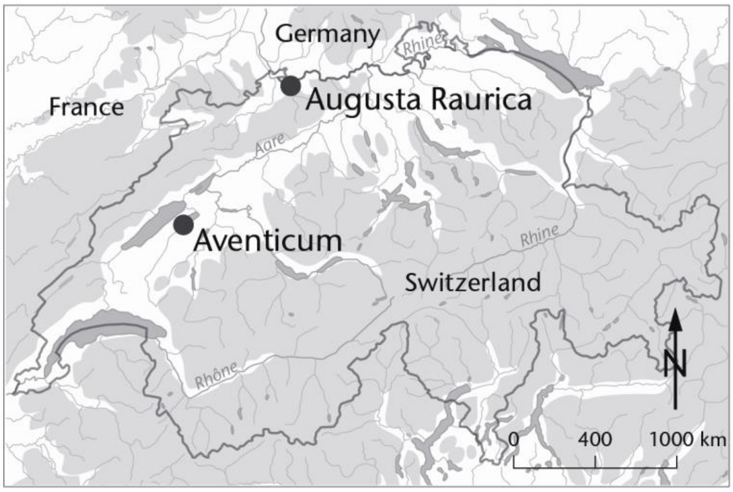

Figure 1: Map of *Switzerland with the Roman sites of Augusta Raurica (Augst and Kaiseraugst) and Aventicum (Avenches).*

Our investigations are based on various earlier studies, particularly on crucibles from other Roman sites (Rehren 1997; Bayley et al. 2001; Bayley and Rehren 2007). The main emphasis of this study was on the crucible *clay* and the identification of its origins (all results in Furger 2018).

1.3. Shape, size and dating of the crucibles

The vast majority of the 893 crucibles (98% of catalogue entries) used for processing non-ferrous alloys in Augusta Raurica are wheel made and egg-shaped (Fig. 2). Their rims are slightly inward-curving and thickened on the inside, the bases are usually rounded or even pointed. The crucibles measure approximately 4.5–18 cm in height and have a capacity to hold between 50 g and more than 4 kg of bronze.

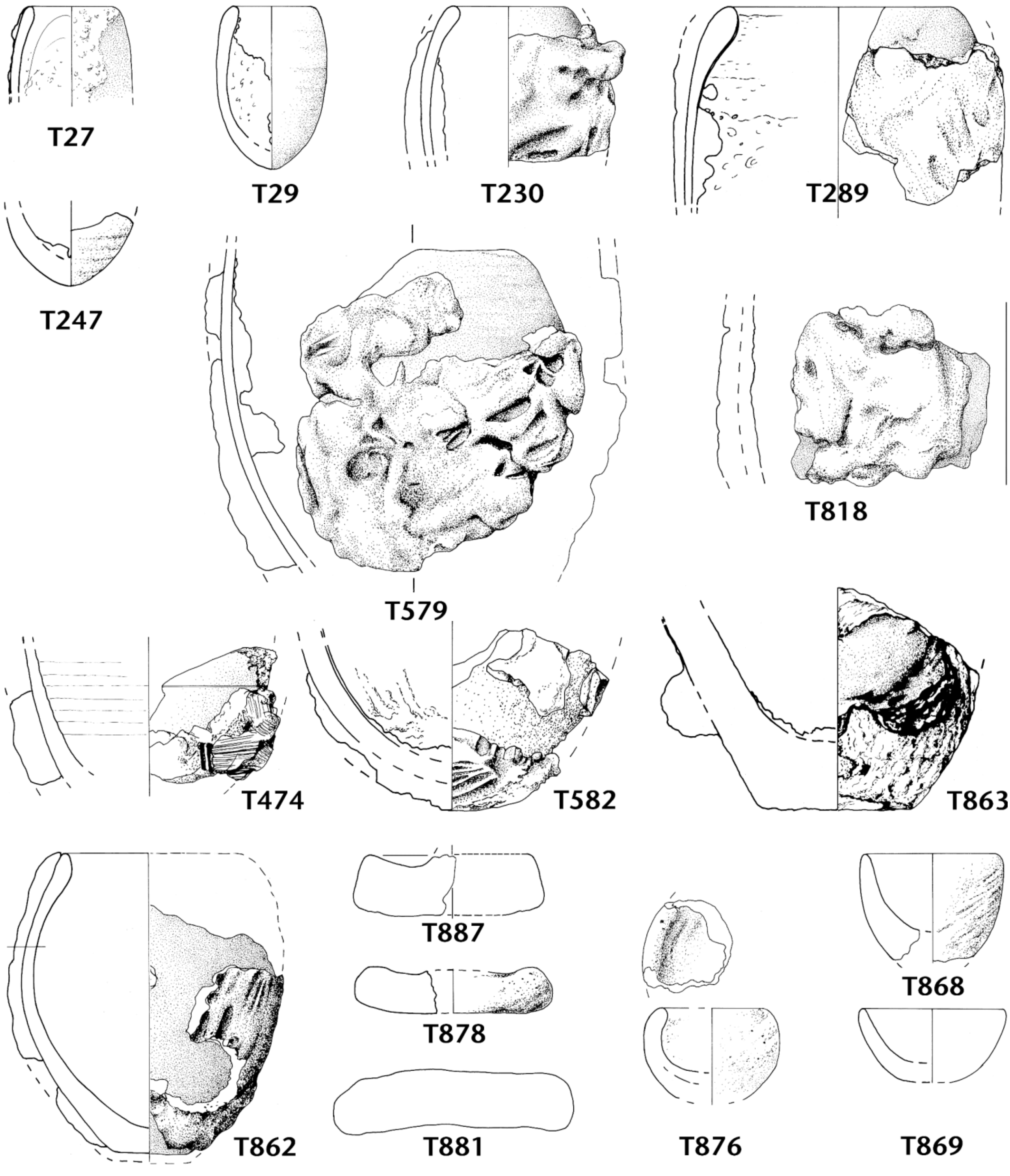

Figure 2: *Crucibles and lids from Augusta Raurica (Augst, Switzerland) made of clays 1–5. All date from the 1st to the 3rd centuries AD; a more detailed typological or technological chronology could not be established. Shapes T27–T582, wheel made and usually encased in a layer of lutum, were the predominant shapes within the range (98%); small bowl-shaped crucibles (T867–T869) were very rare, generally handmade using local clay 2. Scale ca. 1: 2½.*

***T27** (Inv. 1961.6526): clay 1?, Cu++, Sn+, Zn, Pb+++, As++, Ag++; **T29** (Inv. 1967.29586): clay 1?, Cu+++, Sn+, Ag++, Au++; **T230** (Inv. 1978.24280): clay 1, Cu+, Sn+, Zn+++; **T289** (Inv. 1979.18596): clay 1, Zn+++; **T247** (Inv. 1969.13094): clay 1?, Cu+, Sn++, Zn+++, Pb, Au, Hg; **T579** (Inv. 1967.3543): clay 1, Cu++, Sn, Zn+++, Pb+; **T818** (Inv. 1978.10113): clay 1, Cu++, Zn+++, Ag; **T474** (Inv. 1978.24295): clay 2, Zn+++; **T582** (Inv. 1913.453): clay 2, Cu++, Zn+++, Hg; **T863** (Inv. 1913.452): clay 2, Cu++, Zn+++, Hg; **T862** (Inv. 1968.6215): clay 5 (inner lining clay 3?), Cu, Zn; **T887** (Inv. 1978.22766A): lid, clay ?, Cu++, Zn+++; **T878** (Inv. 1978.24302): lid, clay 2 (poss. 3?), Zn++; **T881** (Inv. 1969.13809): lid, clay 2?, Zn; **T867** (Inv. 1977.2214): clay ?, Ag, Au+, Hg; **T868** (Inv. 1978.783): clay 2?, Pb; **T869** (Inv. 1984.3005): clay ?, Pb+++, As++, Ag.*

Despite the sound archaeological basis consisting of numerous dated finds assemblages, it was not possible to identify a change in the crucible *shapes*, either for typological or technological reasons. The only detectable change over the course of the 300-year history of the town was in the size of the crucibles used by the different workshops and the use of five different types of clay (clays 1–5, see section 3.2) in their manufacture.

Smaller crucibles were particularly common in the early 1st and – even more so – in the late 2nd and 3rd centuries, while large and very large examples appeared relatively late and only became more frequently used in the advanced 2nd century, possibly due to a rationalisation of the craft of casting.

Crucibles made of fine, light-grey clay 1 (section 3.2) were in use from the mid-1st to the third quarter of the 3rd centuries with an emphasis on the first half of the 3rd century. The crucibles made of clays 3–5 and particularly those made of the local clay 2, on the other hand, were in regular use from the second quarter of the 1st to the mid-3rd centuries, with an emphasis on the second half of the 2nd century. The overlap between the periods, both with regard to the crucible sizes and the clays used, shows that all crucibles were used more or less at the same time.

2. Method

A total of 188 crucible fragments from Augusta Raurica were selected for portable energy-dispersive X-Ray fluorescence analysis (p-XRF) and 485 measurements were taken on the archaeological site in the museum in Augst (Switzerland). Since the procedure has been discussed in detail elsewhere, only the main features shall be outlined here (Helfert and Böhme 2010; Daszkiewicz and Schneider 2011, Goren et al. 2011; Helfert et al. 2011; Helfert 2013). P-XRF has been used for a number of years as a qualitative and quantitative method of identifying multiple chemical elements in inorganic materials using a wide range of applications (cf. Potts and West 2008; Helfert and Ramminger 2012; Shackley 2012; Shugar and Mass 2012). The portable instruments allow us to quickly carry out the measurements on site in museum storage depots and excavations in a non-destructive or minimally-invasive manner on object surfaces or prepared areas and outcrops. The crucibles were measured using the analyser owned by the Institute of Archaeological Sciences at the Goethe University in Frankfurt/Main. It is an X-Ray fluorescence spectrometer XL3t 900SHe GOLDD (Geometrical Optimized

Large Area Drift Detector) made by Thermo Fischer Scientific Niton which uses the latest detector technology and software.
While measurements can be carried out using the industrial calibration based on an international set of standards, an additional empiric fine calibration is recommended. This is necessary because measurements are not usually carried out on homogenous, plane powder samples but, as in the case of archaeological ceramics, on fresh fractures, resulting in more pronounced matrix effects. Therefore, the spectrometer was calibrated in the “mining Cu/Zn” mode prior to its use by measuring 140 samples of various types of fine and coarse pottery (from different places of production), where were previously measured by WD-XRF and by comparing the reference and actual values. This process allowed us to correct the systematic discrepancies in the measurements between the different types of analysis. Without this fine calibration, the measurements generated would not have been sufficiently comparable to other sample series that were created using other methods. As it stands, the configuration of the spectrometer allows us to precisely identify nine major and thirteen trace elements which can be used in the study (Helfert 2013).
The analyses were carried out in a confined space at a room temperature of between 18 and 25°C and a relative humidity of approximately 50%. Prior to being measured the crucible fragments had been air-dried and stored in plastic bags. All measurements were carried out on fresh fractures. This was done to avoid measuring contaminants from the deposition in the ground or from the use of the crucibles. It was sometimes difficult to find a suitable location to measure used crucibles because they often bore *lutum* on the outside (see section 3.1) as well as casting and slag residue on the inside. Because the ceramic fragments were sometimes only a few millimetres thick, it was not always possible to exclude the presence of residual contamination on the samples in the measurement areas, which had diameters of 8 mm.
The measurements were all carried out on the interior surfaces and in some cases also on the exterior surfaces in order to identify the metal remnants on the crucibles. Because the metal residues were very small in some cases, the quantitative results cannot be viewed as representative of the alloys melted in the Roman-period crucibles, so that the study explored this particular question only from a qualitative point of view. Each artefact took 360 seconds to measure. The fragments were generally measured once. In cases where a fresh fracture was long enough, several measurements were carried out. It was hoped that more precise results could be obtained by creating average values, particularly for crucible clays tempered with coarse grains of quartz.

3. Results and discussion

3.1. Requirements and types of crucible clay

The *clays* used to make crucibles had to be easy to procure, yet at the same time meet serious challenges with regard to heat resistance, thermal shock resistance and insulating behaviour. The performance of the less heat-resistant clays could be somewhat improved by adding substantial amounts of quartz temper for high-temperature use (Bayley and Rehren 2007; Martinón-Torres et al. 2008). The *lutum*, which was added to the outside of most of the crucibles, on the other hand, had to adhere well to the crucible surfaces, blister and swell up in the heat and thus provide insulation; it was supposed to be viscous when fired to protect the crucible, which was being heated to its limit. The *lutum* was thus supposed to prevent breaks in the crucibles and keep them intact, and one of the casters' tasks was to apply the *lutum* and touch it up where necessary (our analysis, experiments and reconstructions of *lutum* is not part of this paper; see Furger 2018).

Of the 893 crucibles investigated archaeologically, 201 were geochemically analysed with regard to their origins by portable XRF.

With the exception of 17 new, unused Roman crucibles from a deposit in Insula 19 (see back cover), the problem with the others was that they were contaminated to varying degrees by the components of the melted alloys, so that the elements Cu, Sn, Zn, Pb, Ni, and Cr could not be used for the characterisation of the crucibles or for the identification of their origins. 131 of the crucibles could be examined thanks to fresh fractures or saw cuts without interference due to crusts or slag on the interiors or exteriors. These samples formed the basis of the statistical analysis.

A total of five crucible clay groups could be identified among the finds analysed. They were clearly distinguishable, particularly due to the elements rubidium and strontium (Fig. 3).

Clay group 1 was the biggest group and contained 72 of the crucibles analysed. It was separated from the other four groups particularly by its very high silicon content of approximately 75 per cent by weight. The Rb concentration lay between 7 and 50 ppm, that of Sr between 31 and 66 ppm. The group is characterised by low Fe_2O_3 content with an average of 2.2 per cent by weight on one hand, and low CaO and K_2O concentrations of 1.7 and 0.7 per cent by weight on the other.

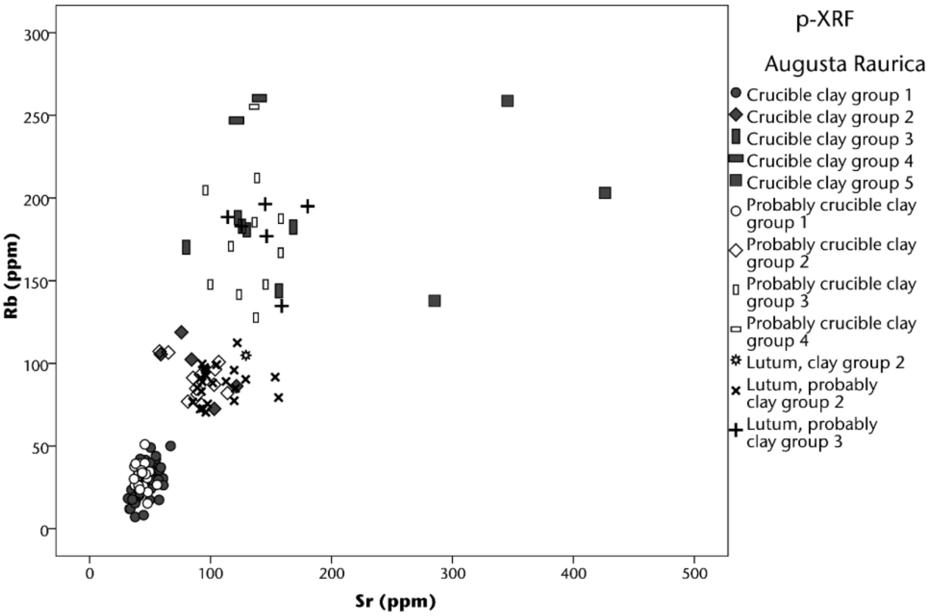

Figure 3 *Scatterplot Rb vs. Sr (in parts per million) of the geochemical groups of crucible and lutum samples from Augusta Raurica.*

Groups 2 to 5 yielded Fe_2O_3 concentrations of approximately 5.2 per cent by weight and, based on the low CaO concentrations of between 2 and 3 per cent by weight, can be classed as calcium-poor (Maggetti and Galetti 1982).

Clay group 2 contained 12 crucibles with a Rb content of between 72 and 118 ppm and a Sr content of between 58 and 129 ppm. 25 *lutum* samples, some of which had been taken from group 1 crucibles, also belong to this group. The group also contained 4 lids that had been used for covering crucibles (Fig. 2, T878–T887).

Clay group 3, characterised by even higher concentrations of Rb (143 to 188 ppm) and a slightly higher Sr content (80 to 168 ppm), contained twelve crucibles, six *lutum* samples and one lid.

Clay groups 4 and 5 contained three crucibles each. While group 4 was quite homogenous (Rb =247 to 260 ppm, Sr =121 to 140 ppm), the samples from group 5 with Rb values of between 138 and 259 ppm and a Sr content of between 285 and 426 ppm varied greatly. Since clay groups 2 to 5 are characterised by dense sand temper, we cannot exclude the possibility that groups 4 and 5 actually belonged to either group 2 or 3, and were just tempered to varying degrees using different types of sand.

A remarkable result obtained from the high-volume sampling of Roman crucibles from Augusta Raurica, which dated from three centuries, was that 71% of the analysed crucibles were made using the same type of clay, which probably even came from the same deposit. This allowed us to conclude that the clay, which was particularly well suited to producing fire-resistant pottery, was either already known at the beginning of the Roman occupation, or was discovered by systematically surveying the area and extracted over a long period of time because of its special properties.

3.2. Origins of the crucible clays

Reference data available for the discussion of the crucibles from Augusta Raurica included a series of analyses carried out by the Archaeometry Research Group at the University of Fribourg with regard to the pottery and tile production in Augst. They included 280 individual samples from reference groups and local clay deposits (Jornet 1982; Jornet and Maggetti 1986; Maggetti and Galette 1993; Schmid et al. 1999; Jornet and Maggetti 2003). Also used were analysis results on fire-resistant clays from the Swiss Jura region, so-called *hupper* earth, which were systematically studied by Eramo (2006). Moreover, in order to ascertain the origins of the crucible clays, raw clay samples were taken from 60 deposits within a 50 km radius around Augusta Raurica, analysed by p-XRF and used for comparison (Fig. 4). All the sites have been known at least since post-medieval times to yield normal and fire-resistant clays. They have been used since then for various purposes and some are still in use today (e.g. in Lengnau/AG).

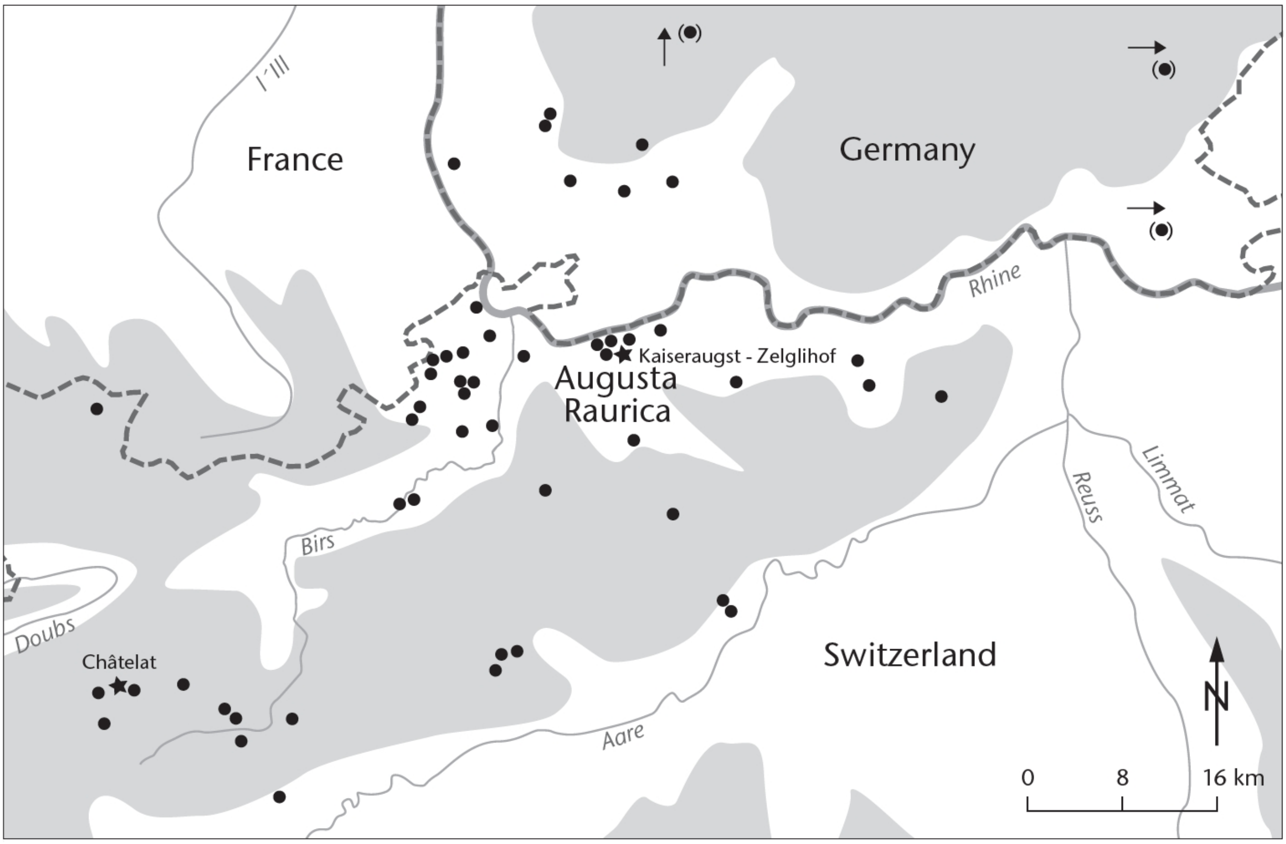

Figure 4 *Distribution of the deposits of the clays analysed for this paper in north-western Switzerland and southern Germany. Most of the deposits (●) are located in ancient Colonia Raurica territory. ✶ indicate the deposits "47 Châtelat" and "3 Kaiseraugst-Zelglihof", clays from which were proved to have been used for groups 1 and 2.*

Because bronzeworking always requires crucibles, the hypothesis was formulated that all or at least the majority of them had been made in Augusta Raurica itself using local clay. However, no immediate connections could be identified between clay group 1 and the local clay deposits in Augst and Kaiseraugst. On the contrary, by studying the geochemical element profiles of the 60 clay deposits, a process of elimination initially led to a selection of six potential deposits: Aedermannsdorf/SO, Châtelat/BE, Court/BE, Matzendorf/BE, Souboz/BE and Witterswil/SO. Strikingly, all potential clays were *hupper* sands ("Huppererde" in German) with a high silicon content.

As the study progressed, Aedermannsdorf, Matzendorf, Souboz and Witterswil were excluded as potential places of origin due to the iron content being either too high or too low and due to varying trace element concentrations. The closest similarities with the crucibles in clay group 1 were identified in the Eocene siderolite clay samples from Châtelat, 50 km from Augusta Raurica as the crow flies, published by Eramo (2006, 191 Tab. 2 Nos. 249, 250). During fieldwork in the summer of 2013 various clays from different bands of clay in Châtelat were sampled and analysed using p-XRF. From a geochemical point of view, sample 364 and the clay variants extracted from it by means of wet and dry-sieving are identical to the crucibles of group 1 at Augusta Raurica (Fig. 5). We may therefore assume that the deposits associated with this type of clay in the Swiss Jura region (cf. Eramo 2006, 188 Fig. 1, siderolite pockets, Eocene) were in all likelihood known in Roman times and deliberately targeted for the large-scale production of crucibles.

Analyses carried out on the crucibles from Aventicum (Fig. 1) revealed that the clays used there were very similar to the clay groups at Augst/Kaiseraugst (König 2014). We may therefore assume that the knowledge of the fire-resistant clays from the Jura region led to similar, if not even the same clay deposits, being used for the crucible production in Aventicum.

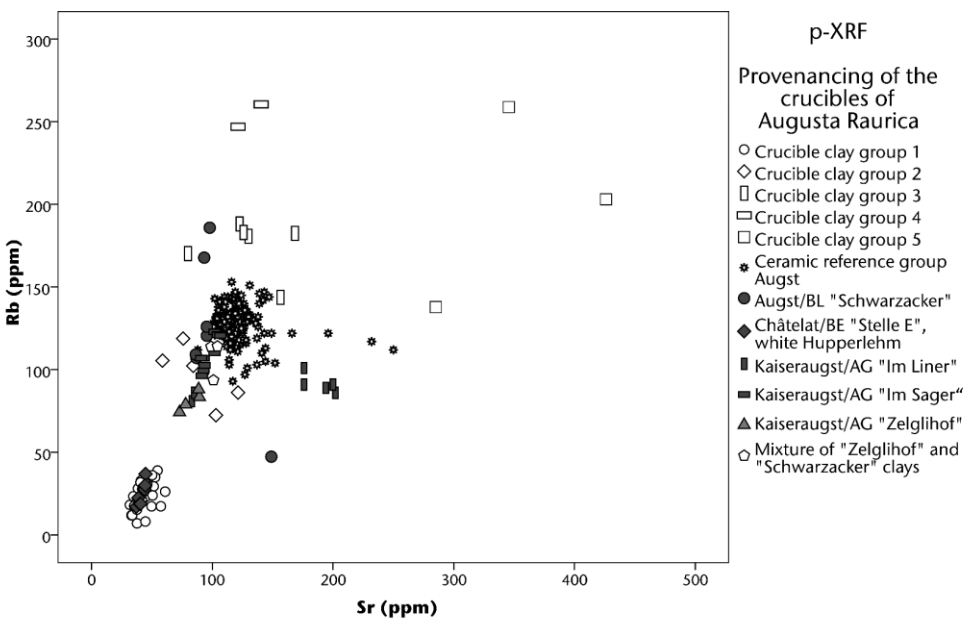

Figure 5 *Scatterplot Rb vs. Sr (in parts per million) of the geochemical groups of crucible and lutum samples from Augusta Raurica including different clay and ceramic reference groups.*

The determination of the origins of the crucible and *lutum* samples of clay group 2 was of particular interest. Once again it was assumed that this clay type was local because *lutum* suffered much from wear and tear and was often replaced several times, so that one would expect the raw material to have been available locally. From a geochemical point of view, however, clay group 2 does not match the ceramic products made in Augusta Raurica (Fig. 5). The attention therefore turned to the clay raw material analyses carried out by Maggetti and Galetti (1993) and the clay samples recently taken in and around Augusta Raurica. While, from a geochemical point of view, the deposits in the "Schwarzacker" area in Augst/BL and the "Im Liner" and "Im Sager" areas on the lower terrace of the River Rhine in Kaiseraugst/AG only partially match, the clays from the "Zelglihof" area (samples 60–63), classified as loess loams, correlate quite closely. An experiment was subsequently conducted where the loess loam from the "Zelglihof" area was mixed with the "Schwarzacker" lower terrace loam. The resulting clay bore the closest similarity to the *lutum* and crucible samples from clay group 2. We may therefore conclude that material was extracted in Roman times from the transition zone between loess loam and lower terrace loam to produce crucibles and that the same material was also used to make *lutum* (and even casting moulds).
No correlations could be detected among the available data that would have matched the smaller crucible clay groups 3 to 5 at Augusta Raurica. It must remain an open question, therefore, whether these were local or imported clays. As mentioned earlier, it is possible that the geochemical variability was caused by varying temper components.

3.3. Crucibles in metallurgical melting processes

The analysis of metal traces in the crucible ceramics and of metallic drops and reguli in the crucibles revealed the entire range of non-ferrous alloys commonly found at Roman sites: copper, tin bronze, brass, tin brass, tin lead-brass, speculum metal, silver-bearing lead bronze and lead-copper alloys. Realistic, quantitative results of melted alloys could only be provided by analysing metal drilling chips from droplets in the inner crucible surfaces.

The melting processes, which were often a form of recycling old metals by using scrap metal usually took place in various-sized crucibles in the standard egg shape made of the light-coloured clay group 1 (Fig. 2, T27–T582). However, we realised that brass also tended to be made in crucibles made of dark-grey clay with dense quartz temper and fired in a reducing atmosphere. These crucibles often had flat bottoms and profiled rims and were usually made of the local clay group 2 (Fig. 2, T863).

It was possible for the first time to identify *brass production* using the cementation method in the Roman province of Germania superior. The calamine deposit in Wiesloch (Germany), which was mentioned by Pliny (*hist. nat.* 34, 2) and was only 208 km north-east of Augusta Raurica, was probably used to this end. The cementation was carried out using crucibles of medium size loosely covered with lids (Fig. 2, T878–T887). Both the crucible and lid ceramics yielded very high values of zinc; this had previously been seen in Autun and Lyons (France) and also in Xanten (Germany) (König and Serneels 2013, König 2014).
The seven small bowl-shaped crucibles (0.8% of the entire crucible assemblage) were all made from dark, probably local clays of clay groups 2 and 3 and usually contained quite dense temper (Fig. 2, T868–T876). They bore a variety of metal traces, which can be associated with various processes: silver and gold alloys on one hand and mixtures with a lot of lead as well as traces of mercury and arsenic on the other.

4. Conclusion

The interdisciplinary project on bronzeworking in the Roman town of Augusta Raurica (Switzerland) revolved around 893 crucibles. With a few exceptions (seven in total) they were all of a uniform shape (Fig. 2), although their heights varied considerably (4.5–18 cm). 188 crucibles were analysed by means of 485 p-XRF measurements.
The large amount of data available allowed us to identify the crucible ceramics and the melting and cementation processes which took place in them.

1. By conducting a chemical trace analysis, the crucible ceramics could be divided into five clay groups.
2. The two most frequently used groups could be correlated with some of the 60 clay deposits that we sampled and analysed: Châtelat in the Bernese Jura region (clay group 1) and Kaiseraugst-Zelglihof (clay group 2) in close proximity to Augusta Raurica (both in Switzerland). Clay groups 3–5 were only used in a small number of cases and their origins have not yet been pinpointed.
3. While the crucibles of clay group 1 were brought to Augusta Raurica as finished products (“traders hoard” with unused clay group 1 crucibles in Insula 19), those of clay group 2 were produced by local potters on site. The *lutum*, also often made of clay group 2, was probably applied to the crucibles by the casters themselves (in

some cases repairs were carried out repeatedly). The metalworkers evidently used the same local clay to make their moulds.

4. Traces of the entire range of “bronzes” commonly used during the Roman period were identified on the ceramics and also in metal stains and drips. They included tin bronzes, brass, lead bronzes and mixtures of all three. A few small crucibles also yielded marked traces of silver. Based on crucible lids and crucibles with very high zinc concentrations, it was possible to identify cementation of brass with copper and zinc ore (calamine) for the first time in the Roman province of Germania superior.
5. With the exception of the new and unused crucibles, they all bore traces of metals. The crucible ceramic (and the outer *lutum* as well) always contained the volatile element zinc and often also higher concentrations of lead. Copper and tin were always underrepresented and present only in slightly higher traces. More representative results with regard to the alloys created by the craftsmen were obtained by taking surface measurements on green bronze stains and the often colourless remnants of glaze on the crucible fragments. However, as in corrosion and patina layers on bronze objects, the metallic elements in these stains often significantly changed so that we may only speak of qualitative surface data. The most secure method of achieving quantitative results is to carry out p-XRF measurements on borings from large bronze drops in the crucibles (Furger 2018, fig. 85).
6. Visual examinations and geochemical analyses of crucibles used in Roman-period non-ferrous crafts found in Augusta Raurica revealed that some of them were created at a central location with local clays. The majority of the crucibles had been produced elsewhere with a highly refractory clay from the Jura some 50 kms from Augst and would then have been brought to the individual metal-processing workshops via the usual trade routes.

In summary we can state that obtaining representative series of measurements as well as identifying metallic remnants, portable XRF is ideal.

5. Acknowledgements

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