Provenance Studies and Beyond

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Abstract: Provenancing of ceramics, i.e. the establishment of a ceramics physical origin, greatly enhances our understanding of ancient civilizations, their cultural development, exchange and movement patterns. Basing provenance studies on objectively measureable and reproducible data like the chemical composition of the material offers important insights in this respect. Various multivariate statistical methods are in use to analyse the large datasets produced by analytical methods like Neutron Activation Analysis (NAA). Of those statistical methods, a well established method using a modified Mahalanobis Distance that takes measurement errors as well as dilution effects (best relative fit factors) into account has repeatedly proven its usefulness specifically for the provenancing of ancient ceramics. Three examples from recent studies, two from East Asia and one from Northeast Africa show what the combination of NAA and this statistical approach can provide beyond simple establishment of a ceramic sample's origin.

Keywords: Neutron Activation Analysis; Best relative fit; Provenancing; Ancient ceramics; Statistical analysis

5.1. Introduction

Provenancing of ceramics is important for understanding ancient civilizations, their cultural development and their exchange and movement patterns. Provenancing by archeological means alone presents difficulties because typologies, changes in form or pattern, and even production techniques can be exchanged between sites. Basic raw materials like clay, however, are very rarely exchanged over significant distances. Objectively measurable and reproducible data, like the chemical composition of the material, provide additional information on the raw materials used in production which can lead to a much clearer understanding of provenance and cultural exchange (e.g. Jones 1986).

Provenancing, i.e. the assignment of a sherd to its production site based on chemical composition, relies on three basic premises:

- 1. The object under investigation is chemically homogeneous.
- 2. All objects within one group or provenance preserve and share their chemical composition.
- 3. Objects from a different origin have a clearly distinguishable chemical composition.

Reliance on those three premises is a challenge and provides an opportunity to go beyond the mere localization of ceramics. An important part of the first premise, the homogeneity within one sample, is that

the sample size under investigation needs to be large enough to be representative of the sample as a whole and represent the bulk chemistry while ignoring surface contaminations. The second premise refers to the fact that different production techniques, or recipes that require e.g. mixtures or cleaning of raw materials, can potentially lead to different chemical compositions, allowing for more than one group or chemical fingerprint within a single production site.

Differentiation of changes to the chemical fingerprint due to changes in production techniques are usually small but systematic (e.g. Garcea et al. 2020; D'Ercole et al. 2017). Thus, recognizing such changes and correctly assigning analyzed ceramics not only to a production site, but to a specific production technique or period within the production site, requires statistical analysis of the compositional data specifically suited to detecting those minute changes. A ceramic-specific multivariate statistical filter that is able to reduce the variation introduced into the dataset by inconsistencies in the paste production was developed in Bonn in the 1980s (Mommsen, Kreuser, and Weber 1988; Beier and Mommsen 1994). Provenancing based on chemical composition as described above puts several restrictions on an appropriate analytical method to measure the chemical composition. The method needs to be able to measure a multitude of elements, especially many of the minor and trace elements, to be able to "see" such minute changes in the composition as would be expected from e.g. differing mixtures or the introduction of a coloring agent.

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Furthermore, the precision and reproducibility of the measurement itself needs to be high. This is due to the fact that the comparison of measurement values needs to take measurement errors into account. It is obvious that two values of 4 ± 1 and 5 ± 1 are indistinguishable, whereas 4 ± 0.1 and 5 ± 0.1 are very clearly different.

Finally, the analytical method needs to be able to report bulk chemical composition, i.e. penetrate beyond the surface of the object and be relatively insensitive to minimal inhomogeneities.

All of the requirements on the analytical method mentioned above are easily fulfilled by Neutron Activation Analysis (NAA) (Greenberg, Bode, and De Nadai Fernandes 2011).

5.2. Materials and methods

5.2.1. Neutron activation analysis

Neutron Activation Analysis (NAA) is a radiochemical method for elemental analysis. To perform NAA, the sample under investigation is irradiated with neutrons, thereby producing radioisotopes from stable isotopes contained in the sample (activation). After activation, the radioactivity of the sample is measured by gamma spectroscopy. Interpretation of this gamma spectrum leads to the identification of the radioisotopes produced in the sample by activation as well as to the determination of their abundance (activity). Since the radioisotopes can, in most cases, only be produced from a specific stable isotope, their existence is indicative of the stable isotope being contained in the sample. The abundance or activity of the radioisotope is directly related to the abundance of the stable isotope before activation. By irradiation and measurement of reference samples of known composition, comparison of the activities measured leads directly to the elemental concentrations in the sample.

Due to the nature of neutrons and their interaction with atomic nuclei, no information on the oxidation state of the elements measured can be obtained. However, since the penetration depth of neutrons in regular matter is on the order of several centimeters, the elemental concentration values obtained represent the bulk chemistry of the sample. Main elements like Hydrogen (H), Oxygen (O), Nitrogen (N), Carbon (C) and Silicon (Si) are hardly activated or don't produce an easily measurable radioisotope, thus those elements could be described as being "invisible" to the method. In the case of the analysis of biological and geological material, this is an asset since the matrix (i.e. $SiO₂$ in the case of geological material) does not interfere with the measurement. Other elements, specifically many of the rare-earth elements that are usually only contained in minute traces, activate quite easily, resulting in very low detection limits for those elements on the order of ng/g. This is another asset for the application of NAA to provenancing, since the trace elements contained in a sample are especially highly characteristic of its composition.

As described above, NAA directly relates elemental concentrations in a sample to a measurement of radioactivity. Since even low amounts of radioactivity can easily be measured, the total sample mass needed for this type of analysis is usually on the order of 50–150 mg. However, during sampling, great care has to be taken that the sample is representative of the full object under investigation, which can make it necessary to take larger sample amounts of up to 500 mg.

In summary, to perform NAA on a ceramic object, a small sample of 50–500 mg needs to be taken off the object. This sample is then irradiated in the neutron flux of a reactor and subsequently measured on a gamma spectrometer. Usually, at least two subsequent measurements are done to be able to gain precise data on short- and long-lived radioisotopes. This results in a total time requirement of three to five weeks from irradiation to results. For this reason, NAA is usually performed in batches of 30–100 samples. The elemental concentrations measurable are Na, Mg, Al, Cl, K, Ca, Sc, Ti, Cr, Mn, Fe, Co, Ni, Zn, As, Rb, Sr, Zr, Sb, Cs, Ba, La, Ce, Nd, Sm, Eu, Tb, Dy, Yb, Lu, Hf, Ta, W, Th and U. Not all of those elements are of equal importance for provenancing, so usually a smaller selection of about 25–28 elements is chosen for analysis. For detailed descriptions and more information see e.g. (Minc and Sterba 2017; Sterba 2018).

5.2.2. Statistical analysis

Once the chemical composition of a sample, often called its chemical fingerprint, has been established, it is necessary to compare the sample's fingerprint to other chemical fingerprints, either of other unknown samples or of already established groups. To compare two chemical fingerprints, all elemental concentration values are compared to each other, ideally taking the associated measurement error into consideration. If all elemental concentrations of two samples are sufficiently similar, the two samples are considered to be made from the same material, and thus from the same origin or provenance.

Since the number of measured samples as well as the number of potential candidates for comparison is usually very large, a comparison by hand is, if not impossible, at least extremely inefficient. Thus, different multivariate statistical methods are usually applied to process a large number of samples more quickly.

Bivariate plots

A very simple exploratory method is to use bivariate plots of datasets. In this method, two to four (if using ratios) elements are selected from the dataset and each sample is plotted onto an area spanned by those elements (see Fig. 5.1). Careful selection of the elements (or ratios) used can lead to visible grouping of the points representing samples.

Figure 5.1. Comparison of bivariate plots of two datasets of ceramics, previously grouped by NAA and subsequently by statistical analysis. Lacking this analysis, no grouping information would be available. While, in the top figure, a visible separation into three groups occurs, the same set of elements leads to no visible groups in the second plot.

While this strategy is sometimes useful for specific datasets and sets of elements, it lacks several important features:

- 1. It uses only a small subset of the information available (i.e. at most four elements out of a total of more than 20).
- 2. It ignores measurement errors, potentially showing stronger separations than the data allows.
- 3. It is usually possible to find some combination of elements to plot that show some grouping, sometimes finding several groupings that cannot be correlated with each other.
- 4. It is an exploratory method only; decisions on the exclusion or inclusion of samples at the border of visible groups can hardly be founded on statistical measures.

Principal component analysis

A method related to bivariate plots is Principal Component Analysis (PCA), a statistical method that finds the linear components in a dataset that express the most variation, resulting in a reduction of variables necessary to describe the dataset. After PCA calculation is done, the first few principal components are then used to produce similar plots

Figure 5.2. Principal Component Analysis (PCA) of the same dataset as in Fig. 5.1 (top part). The grouping is visible, however, especially along the borders of the groups, it is not clear if samples belong to a group or not.

to bivariate plots, again sometimes showing groupings. Fig. 5.2 shows a PCA of the dataset also used in Fig. 5.1 (top plot). The grouping that was apparent in the bivariate plot is still clearly visible. However, it is not obvious how the singles should be handled (specifically the one single "inside" of group 02), and there seems to be a separation between the samples belonging to group 01. Without the different shapes of the data points in the figure, it would not be clear how to group them.

Depending on the dataset and the number of outliers or singles therein, a PCA can lead to usable groupings. However, similarly to bivariate plots, no information about measurement errors is used. Furthermore, PCA is again an exploratory method; handling of samples along the borders of visible groups is not straightforward.

Cluster analysis

Cluster analysis is a statistical method that uses distance measures (e.g. the euclidian distance) to calculate closeness or similarity between samples. In an iterative algorithm, the samples are then grouped by their respective distances. This leads to a visualization that is called a dendrogram, where the dataset is represented in a tree-like structure. The samples forming the leaves and their closeness along the stem represent their distance (Fig. 5.3).

To use a dendrogram for grouping, a cut-off distance is defined and all samples linked below that distance are grouped together. In the example of Fig. 5.3, a cut-off distance of 15,000 leads to two large groups and one single sample, whereas a cut-off distance of 5000 results in five groups and several singles. Thus, the grouping produced by cluster analysis relies on the distance measure, the linking method used to connect samples, and the cutoff distance applied. While there are some general best practices on how to select those three components, no definitive rules exist. This makes it possible to reach different groupings by trial and error. Furthermore, as in both methods mentioned above, no information on the measurement error is included in cluster analysis.

Figure 5.3. Cluster analysis and subsequent dendrogram of the dataset used in Fig. 5.2 and Fig. 5.1. Distances are calculated as euclidian distances, and clustering is done by the complete linkage method. Instead of sample names, group names are used to make comparison of the grouping methods more visible. The grouping done by NAA and statistical analysis (group01, group02 and group03) agrees with the archeological grouping of this dataset; the grouping that would follow from the dendrogram cannot be related to archeological groups.

A ceramic-specific approach

From the examples above it is clear that it would be preferable to have a statistically valid method for grouping ceramics with respect to their chemical composition, potentially allowing statistical testing or prediction. This method needs to 1) include measurement errors in the analysis, and 2) find a way to overcome additional spread introduced into the dataset because of dilution effects during paste production (Mommsen and Sjöberg 2007).

Dilution in this case means that some component is added to (or removed from) the raw clay that does not contribute to the elemental composition. For example, the addition of quartz sand during tempering would increase the total mass of the sample; however, since both silicon and oxygen (Si and O) are usually not measured, the relative concentration values of all other elements wouldn't change. Similarly, the use of an organic compound would only result in a dilution. Such a dilution results in additional spread that is introduced into the dataset, for the simple reason that between batches of paste produced by the potters, slight differences are highly likely. Fortunately, such a dilution can be easily corrected for by introducing a "dilution factor," i.e. a single number by which all elemental concentration values of one sample are multiplied, to reduce the apparent difference to another sample.

To illustrate this process, Fig. 5.4 shows the comparison of a dataset before (left) and after (right) dilution correction. It is the same dataset used in Fig. 5.1. As can be seen, just by correcting for the dilution factor, the group members come much closer to each other. Samples classified as "single" don't change their position, because dilution correction can only be applied against another sample or group mean. In Fig. 5.4, dilution correction is done for all samples belonging to one group against their common mean.

Figure 5.4. Comparison of a dilution corrected dataset before and after correction.

Interestingly, such a correction of the raw data can easily be done mathematically and, from a statistical point of view, leads only to the reduction of the number of degrees of freedom by one. This means that the corrected data can still be analyzed by all multivariate statistical methods, as long as the number of degrees of freedom is reduced by one.

However, it would be preferable if the similarity between two samples could be described numerically. Ideally, this numerical value would take into consideration the dilution factor mentioned above as well as the measurement errors. Such a measure was developed in the 1980s in Bonn (Mommsen, Kreuser, and Weber 1988; Beier and Mommsen 1994; Sterba et al. 2009). It used a modified version of the so-called Mahalanobis distance, which in its original form could be described as calculating the distance between two vectors in units of their standard deviation. By replacing the standard deviations with the measurement errors and minimizing the distance with respect to the dilution factor, this modified Mahalanobis distance provides a statistically

valid means of defining the similarity of two chemical fingerprints.

With the calculation of the modified Mahalanobis distance between each possible pair of samples in a given dataset, it is possible to reach a first grouping of a subset of samples with small distances. From this, a group mean can be calculated as well as the modified Mahalanobis distance from all samples in the dataset to this group mean. From this calculation, more samples can be added to the group, if they are sufficiently close to the mean, leading to a modified group and a modified group mean. By iteratively repeating this process until no more samples come close to the group mean, groups can be formed.

Fig. 5.5 shows the modified Mahalanobis distances between the group means and all samples in the dataset already used repeatedly. It is clear to see that between the last sample belonging to a group and the first sample not belonging, a gap exists. This is usually a good indication of a complete group.

Figure 5.5. Histograms of the modified Mahalanobis distances between the group mean and all other samples for three groups in the dataset. It is important to note that only the distance from 0 is relevant; interpretation of the distances between samples marked in the histogram ignore the multidimensional nature of the Mahalanobis distance.

It is important to note two properties of this grouping process: Firstly, once a group is defined in the way described above, new samples can always be compared to this group and, if suitable, added, changing the group mean. Once a group reaches a large enough size (several tens of samples), the group mean stabilizes. Secondly, because of the properties of the Mahalanobis distance, the modified Mahalanobis distances are distributed according to the X^2 -distribution (if the reduction in numbers of degrees of freedom is taken into account), making predictions or statistical tests possible.

5.2.3. Beyond provenancing

The process described above, i.e. the combination of NAA and a ceramic-specific statistical approach to grouping, offers a reproducible and statistically sound way of comparing and grouping ceramic material. Due to the dilution factor, a large component of the spread usually visible in such datasets is removed, allowing an

unobstructed view of the similarities and dissimilarities of the samples. With this, it becomes possible to associate samples not only with a region of origin but with a specific recipe in use in a spatially and temporarily delimited region. This takes the usual approach of provenancing beyond its traditional borders by providing information on temporal shifts in recipes or transport routes, as well as on changes in the recipe connected to technological or cultural choices.

5.3. Case studies

The potential of the approach to provenancing described in the previous section is best demonstrated in case studies. Three significantly different outcomes from systematic research based on NAA are introduced, and while two of these case studies from East Asia are presented in this volume as well – the Nakadake Sanroku kiln site center and the Kamuiyaki kiln site center, the third, complementary case study is taken from Northeast Africa.

5.3.1. Kamuiyaki Kiln Site Cluster

The Japanese National Historical Site "Kamuiyaki kiln site center" is located in the south of Tokunoshima Island and is understood to have been the production center of a specific pottery type that resembles traditional Sue ware. Its discovery (Hirose 1933) has been dated to the Gusuku period of the Ryūkyū islands, mostly equivalent to the Japanese Middle Ages (National Museum of Japanese History 1997). Pottery from the Kamuiyaki kiln site center is widely distributed throughout the Ryūkyū islands. Within the site, two chronologically significant typological groups can be distinguished (Satō 1970, Shinzato 2018).

In a preliminary study (Sterba et al. 2020), 20 vessels from a specific context were selected for NAA and subsequent data analysis. Of the 20 samples, 10 vessels each were selected from the aforementioned typological groups A (older) and B (younger). All vessels but one were found within one ash heap associated with several kilns, the two groups found in two different layers with a distinct separation layer without any finds. Typological and stratigraphical assessments agree.

NAA was performed on the 20 samples at the TRIGA Center Atominstitut of TU Wien, applying standard procedures (Sterba 2018) and subsequent statistical data analysis. Application of dilution correction and grouping of the samples with respect to their chemical fingerprint resulted in two groups of ten and eight samples, respectively, as well as two singles. The two groups could clearly be separated according to their chemical differences, mainly in the metals and metalloids Sc, Cr, Fe, Co, As and Sb. Furthermore, the two groups clearly correlate to the two chronological/typological groups already established, even though no *a priori* information was used in the chemical grouping. Every vessel from the older group A was chemically grouped into one group, whereas all eight samples from the second chemical group belong to the younger typological group B. The two singles (samples that cannot be chemically assigned to a group) also belong to the older group.

The most obvious conclusion from this result is that the typological and chronological grouping can be confirmed by chemical analysis. Thus, future finds of pottery fragments that have no clear stratigraphy or that show no distinguishable typological features can still be assigned to one of the two groups by chemical analysis. With this, NAA and statistical analysis not only yield information on provenance but also chronological information within the production site.

More importantly, however, this result indicates that the two periods of production at this subcluster of the Kamuiyaki kiln site center applied different recipes to the production, either by selection of different raw materials or through different preparation of the paste. Further investigations of other subclusters could potentially yield either a better definition of the two chemical fingerprints or additional chemical fingerprints corresponding to more recipes.

5.3.2. Nakadake Sanroku – a complex kiln cluster

The kiln cluster at Nakadake Sanroku, close to Minamisatsuma City in Kagoshima Prefecture, southern Japan, was a production center for Sue ware during the ninth and tenth centuries AD. It contains more than 50 Sue kilns and covers an area of over 6 km² (Nakamura and Shinoto 2015; Shinoto and Nakamura 2016).

Sue ware was widely distributed throughout Japan, including the southern islands. Hence, more than 200 samples of Sue ware found at the kiln cluster were analyzed by NAA with the prospect of defining a chemical fingerprint for the production center (Sterba 2015). The samples were collected from several different contexts; more than 60 samples are clearly from kilns or associated wasters (haibaras), while most of the remaining sherds were collected along the brooks within the kiln cluster. The kilns are distributed along the side of Mt. Nakadake on steep slopes. In ongoing studies, the find locations of the samples are tentatively associated with three major drainage basins, denoted A, B and C. Within the basins, geographical sublocations along watersheds can be defined, resulting in sublocations Ap02, Bp01 etc.

Statistical analysis of the chemical fingerprints of the samples yielded 23 chemical groups, which are often chemically similar but clearly distinct, as well as a large number of chemical singles that probably indicate numerous other small groups where only a single sample was found.

Considering the geographical situation of the kiln cluster, associations of the chemical groups with the sublocations or at least with the main drainage basins would be expected. However, a comparison of the find locations and the chemical groups, as can be seen in Fig. 5.6, show a much more complex situation.

As can be seen in Fig. 5.6, the two chemical groups NG03 and NG10 cover almost all geographical sublocations, while chemical groups NG16, NG17 and NG18 can only be found in the sublocation Bp03. Some sublocations yield only one or two chemical groups (i.e. Cp01, Cp02 and Bp01), while others contain a multitude of chemical groups (i.e. Bp03 or Bp08).

Compared to the situation described above (Kamuiyaki kiln site center), a clear assignment of external samples to Nakadake Sanroku seems much more complicated or even impossible. However, it is important to note that for two of the chemical groups (NG03 and NG06), corresponding samples were found on the islands Tanegashima and Kikaijima, both to the south of Nakadake Sanroku (Sterba 2015). On Tanegashima, the much closer island, only samples from the chemical group NG06 were found; on Kikaijima, only samples from the chemical group NG03

Nakadake Sanroku Chemical groups and find location

Figure 5.6. Comparison of the chemical groups found in the *Sue***-ware corpus of Nakadake Sanroku with the geographical find locations, based on watershed divides. A, B and C denote the major drainage basins with sublocations along minor watersheds.**

were found. Chemical group NG06 only has representative samples in sublocation Bp07 at Nakadake Sanroku, whereas samples from chemical group NG03 can be found in most sublocations.

While the total sample number in this specific case is very small (only two samples each on the two islands), the distribution might hint at complex distribution patterns of the products from Nakadake Sanroku. It is also important to note that, due to the archeological context, the chronological development and typology in Nakadake Sanroku is mostly unclear. This means that the distribution of the samples over the different geographical locations lacks the chronological information that could help to explain the distribution of the visible chemical groups.

5.3.3. The cooking pots of Nile clay

In the course of the European Research Council Across-Borders project (Budka and Auenmüller 2018), over

300 ceramic samples from Sai Island in northern Sudan were investigated by Neutron Activation Analysis (NAA) (D'Ercole and Sterba 2018). Sai Island, a border settlement between Upper Nubia and Egypt, was repeatedly under different political influence, which included the establishment of an Egyptian town during the New Kingdom (ca. 1539–1077 BC). Preliminary investigation of the chemical composition of the ceramic samples quickly resulted in a large group (more than 100 pieces) of ceramics made from Nile clay. Since Nile clay as a raw material is chemically very homogeneous, and thus is not necessarily local to Sai Island but could be from any point along the Nile River, additional and more careful statistical analysis of the compositional group was performed. Typologically, this group could be separated into "Egyptian-Local," "Nubian-Local" and "Egyptian-Import" wares, i.e. ceramics made in Egyptian style but most probably locally made, ceramics made in Nubian styles but most probably locally made, and material in Egyptian style but most probably not produced locally.

Dilution factor separation - All Data

Figure 5.7. Boxplot of the dilution factors for the three different typological categories found within the ceramics made from Nile clay. While the distribution of the imported samples has a large range of dilution factors, the range for the other two typologies is much smaller, indicating a more standardized production.

A comparison of the dilution factors of the three different typological categories (Fig. 5.7) yielded some interesting information: The distribution of the dilution factors within the three groups is different. While in the two groups that were most probably locally made from Nile clay found close to or on Sai Island, the dilution factors vary only in a small range, in the samples most probably from somewhere else along the Nile, the dilution factor distribution covers a much larger range. This can be explained by the locally produced ceramics having a much smaller range of different amounts of additives (organic material or sand) because they all were made within a community and within a fairly small area. The ceramics produced elsewhere show a much larger range of amounts of additives used, since they came from many different workshops along the Nile.

Taking this approach one step further, a closer look at the dilution factors of samples typologically categorized as Egyptian Imports is shown in Fig. 5.8: Some of the samples could easily be recognized as cooking pots. Their dilution factors are, in general, larger (closer to 1) while the dilution factors of all other samples are much smaller (around 0.8). This indicates a difference in the amount of temper, be it sand or organic matter, that was added to the two groups, although both used the same raw materials. Considering the technological demands on cooking pots, this is a clear indication of a conscious decision of the potters to use different mixtures of the same raw materials to achieve different technological properties.

5.4. Conclusion

Moving from purely archeological provenancing to archeometric analysis to support typological data with measurable chemical data allows the application of statistical methods, and thus leads to a more reproducible approach to the establishment of the provenance of ceramic material. Applying chemical analysis, specifically Neutron Activation Analysis (NAA) to provenancing

Dilution factor separation - Egyptian Imports only

Figure 5.8. Boxplot of the dilution factors for the samples made from Nile clay and classified as Egyptian Imports. The samples that could clearly be identified as cooking pots are shown separately, showing a significantly different average dilution factor from all other samples. This strongly indicates a conscious choice to reach necessary technological properties.

of ceramics is well established and has been producing meaningful results and support for archeology since the 1970s (Perlman and Asaro 1969; Yellin et al. 1977; Jones 1986; Mommsen et al. 1995; Hein, Mommsen, and Maran 1999; Mommsen and Maran 2001; Sterba et al. 2009; Zuckerman et al. 2010; Moreno Megías et al. 2020).

Taking into consideration the case studies provided above, it can be seen that, with careful statistical analysis of the chemical data and in combination with archeological information, traditional provenancing can be pushed beyond the establishment of a single source of production. It becomes possible to establish different recipes employed within a single production center. In cases where reliable dating information is available, not only can the physical location of origin be established, but the time frame or sequence of production also comes within the method's reach. Changes in recipes become visible and provide information on changes in production techniques or use of raw materials.

In comparison to the macroscopic viewpoint provided by traditional methods of provenancing, NAA and subsequent statistical analysis offer a microscopic scale on which to interpret and contextualize archeological findings.

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Part II

Advances of Kiln Manufacture in China