

Tracing fineware production in the Neo-Assyrian empire: Neutron activation analysis of common
and Palace Ware in the Upper Tigris River Valley, Turkey

Short title:

Neutron activation analysis of common and Palace Ware in the Upper Tigris River Valley, Turkey

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Abstract (<300 words. 300)

In the Iron Age, the Neo-Assyrian empire (c. 900-600 BC) conquered territory across southwest Asia and established regional capitals along its borders to secure its gains. Governors at these centers oversaw resource extraction and craft production for shipment to the imperial heartland in modern-day northern Iraq. Metals and textiles were the crafts most carefully managed by the administration. We know less about centralized control over ceramic production but hypothesize that fineware production and distribution would have been of interest to imperial administrators. A fineware type known as Palace Ware has been found throughout the empire and is considered an indicator of elite Assyrian dining traditions. Excavations at one regional capital, Ziyaret Tepe (ancient Tušhan) produced pottery of various skill levels used by residents. In this study neutron activation analysis (NAA) was used to characterize and compare the fabrics used to make Palace Ware vessels with more common wares to see if the former vessels were imported from the imperial heartland. Palace Ware is macroscopically distinct, but this does not always indicate an import. Chemical composition of the samples fell into four main groups, and both Palace and common ware were found to have similar compositions. Comparison of these data with those from contemporary sites showed that the two main Ziyaret groups matched the chemical composition of pottery from the Assyrian capitals of Nimrud and Nineveh. Our conclusions show that there is considerable homogeneity in the clays of the upper Tigris river valley in Turkey and the lower Tigris in northern Iraq. Given this similarity, it is possible that Palace Ware at Tušhan was produced locally, imported, or both. If it was manufactured locally, as has been shown at the urban center

of Tell Sheikh Hamad, potters in the imperial peripheries may have produced fineware pottery independent of direct imperial control.

Introduction

Pottery is often used in archaeology as an indicator of craft production or trade in raw materials, as well as for its basic chronological use in charting stylistic change over time. In the context of Iron Age greater Mesopotamia, we know from textual records and finds of raw materials like metal ores outside their source area that significant trade took place. Craft production can be more difficult to trace because workshops are rare finds on large sites as modern excavations often sample only a small proportion of the site area. Contemporary cuneiform texts, while primarily economic, are more concerned with government control of valuable items, usually metals, textiles, or basic foodstuffs (especially grain and flocks). When ceramics are mentioned at all, it is often to discuss the edible contents of ceramic jars and not jars and bowls themselves. Assyrian military expansion and conquest often involved a re-organization of political control and economic production, the latter including standardization of specialist craft production [1]. It is unknown to what extent pottery production was controlled by the imperial administration [2].

In past decades, archaeologists hypothesized where pottery production took place based on macroscopic clues such as style, form, and decoration. These features can be misleading especially in the case of local imitations of foreign-made wares. Ethnographic research has

shown that potters usually use the clay source closest to them for production, so clay composition would be another way to distinguish production locales [3]. Provenience studies in pottery are possible because clay composition differs more significantly between geological regions than within a single geological source. Low-power microscopic analysis of the petrography of the clay body is one way to characterize the geologic differences in clays. Modern chemical provenience studies such as Neutron Activation Analysis (NAA) can characterize pottery accurately by detecting its elemental composition down to the parts per million. During its construction, other materials ('temper') are usually added to the clay body of a vessel to make the clay easier to shape and improve its firing characteristics. A study of its chemical composition, both major and trace elements, will detect characteristics of its clay as well as the additional temper added by the potter.

From 1997 to 2014, the Ziyaret Tepe Archaeological Project investigated an ancient mounded site in southeastern Turkey [4]. The site has a series of occupations extending back to the Early Bronze (c. 3000 BC), Middle Bronze, and Late Bronze Ages, reaching a maximum extent as a provincial capital of the Neo-Assyrian empire in the Iron Age (900 to 600 BC) when the urban site (32 ha) was called Tušhan. Tušhan was located approximately 270 km northwest (although 425 km by river transport) from the Assyrian homeland in northern Iraq (Figure 1). Texts from King Ashurnasirpal II (ruled 883-859 BC) describe three regional capitals being established as the northern frontier along the Tigris River: Tušhan, Sinabu, and Tidu. The Assyrians installed a governor at Tušhan and built a palace for him, garrisoned troops there, and built an encircling city wall. Cuneiform texts found in its palace and administrative buildings detail the military,

economic activities such as food and textile production, and governance of the site [5, 6].
Smaller sites nearby specialized in grain production and work groups stationed at Tušhan
supplied the empire with materials from this region, especially timbers from the nearby
mountains that were floated down the Tigris [7], and likely funneling metal ores from mountain
sources to the Assyrian heartland. Later occupational levels at Ziyaret Tepe dated to the Late
Iron Age, Medieval, and Ottoman periods.

Fig 1. Map showing Ziyaret Tepe, Nineveh, Nimrud, Tell Sheikh Hammad, Tell Jemmeh, Khirbet
Khatuniyeh, Khirbet Qasrij, and Qasrij Cliff.

Several different types of pottery wares are found across the excavated buildings and burials at
Tušhan, differing in skill level, appearance, and function. The most common ware is called Plain
Simple Ware (LA01) in our recording system. It is medium in coarseness with multiple different
kinds of temper and was fired to a light reddish-brown to buff color. It is found in a variety of jar
and bowl shapes. Other wares include two cooking wares (LA03 and LA04) used to create large
globular pots, as well as finewares such as Palace Ware (LA05), a Near Palace Ware (LA06), and
a rarer Neo-Assyrian glazed ware (LA10). All necessary permits were obtained from the Turkish
Ministry of Culture and Tourism for exporting the potsherds for the described study, which
complied with all relevant regulations.

113 Palace Ware is thin, fine-grained, and made of well-levigated clay; its manufacture is a highly
114 skilled technique. Palace Ware vessels come in a set of standard shapes like a dining set [8, 9].
115 This sophisticated type was found in low quantities in most domestic Neo-Assyrian contexts at
116 Tušhan and visually mirrors pottery used in Assyrian capitals. It is absent in the preceding Early
117 Iron Age contexts when a small part of the high mound at Ziyaret Tepe was used by indigenous
118 groups between 1050 BC and the Neo-Assyrian conquest in the early 9th century BC. Palace
119 Ware was first defined in the 1950s after numerous examples were found at the Assyrian
120 capitals [10, 11] and it is only found in a narrow time period (late 9th century BC through the
121 end of the 7th century BC). Wherever Palace Ware appears, whether it is in Syria, Iraq, Israel, or
122 Turkey, it is regarded as an indicator of Neo-Assyrian influence [12; 2]. Three basic shapes of
123 Palace Ware have been defined using measurements of vessels from the capitals of the Neo-
124 Assyrian empire (Nineveh and Nimrud), and all are drinking-related: bowls, cups, and small jars
125 [9]. We have found examples of all three of these forms at Tušhan and they are evenly spread
126 between houses of different statuses across the site [13]. Their overall frequency is a small
127 percentage of the pottery assemblage (1-7% depending on the context), but even in small
128 quantities it is significant as an indicator of a “foreign” dining tradition in a provincial context.

129 ~~The pieces were, we assume, relatively expensive, but likely not the most elite dining vessels.~~
130 ~~Perhaps we should broaden our terms beyond materials and propose that the truly rare and~~
131 ~~“fine” wares were not ceramic at all, but were metal or glass vessels, which would have been~~
132 ~~even more valuable and restricted in manufacture.~~

133

In this study, we analyzed Plain Simple Ware and Palace Ware at a chemical level to characterize their similarities and differences. With the naked eye, it is easy to distinguish Palace Ware from the vast majority of other contemporary pottery, as it is clearly different in color, thickness, texture, and temper. However, such macroscopic distinguishing features do not necessarily mean the Palace Ware and more common pottery are made from different clay sources, or in far-flung workshops. Macroscopic variations could be due to the skill level of the potter, preparation processes for the clay, manufacturing (hand versus wheel), and firing techniques and conditions. Chemical studies of the clay body should show whether or not these two wares were made with different clays and tempers, thereby indicating multiple, geographically distinct clay sources and workshops.

One key contribution of our analysis is a chemical characterization of the clays of the upper Tigris River valley in southeastern Turkey. As discussed below, chemical studies have been conducted on clays in pottery from the Assyrian imperial capitals in northern Iraq and a few sites within 50 km of the imperial capitals along the Tigris. Other scholars have chemically analyzed clays at the western frontier of the Neo-Assyrian empire in the Levant. However, few have sampled the upper Tigris valley on the northern frontier of the empire, with the exception of Kibaroğlu [14].

Our hypothesis was that, given the fragility of Palace Ware for travel, we expected to find that potters were producing both Palace Ware and common wares using local Upper Tigridian clay sources in workshops located at, or in the immediate vicinity of, Ziyaret Tepe. This NAA study

cannot directly address the possibility that the potters themselves were resources and could have been moved into the region from the imperial homeland, either to produce or to train other potters in elite, Palace Ware production techniques.

Materials and Methods

At other sites, it has proven difficult to distinguish Palace Ware that is imported from local imitations. Some local potters copied the type if they were skilled enough to do so because it was a luxury good, presumably of greater value. Imitation pieces have been found in Palestine, Transjordan, and Syria [15, 16, 2]. In attempting to source Palace Ware from sites on the edges of the empire, techniques such as ceramic petrography have been used but, by itself, this method was not always successful in distinguishing between clays at sites, and chemical methods have proven more useful. For example, Hunt was able to distinguish clays along the Euphrates River from those along the Tigris using NAA due to their slightly different clay minerals [2].

One significant issue addressed in this study is the relative homogeneity of geological formations along the Tigris River, discussed below. As a result, in some cases it has not been possible to distinguish clay fabrics between nearby sites on the Tigris, e.g., when comparing pottery from Arpachiyeh and Tell Gawra to that found at Khirbet Qasrij and Qasrij Cliff 25 km to the south [17]. Similarly within 50 km of the Neo-Assyrian capital, a study of 60 potsherds from Khirbet Khatuniyeh overlapped in chemical composition with clays used at nearby Qasrij Cliff and Khirbet Qasrij [18]. Since Tušhan is 425 km upstream from the capital, we hoped to find a

distinctive clay chemical signature for common ware at the site that would contrast with the known signature for Palace Ware as already defined in the imperial heartland in the studies cited above.

Several studies have used NAA to compare the chemical characteristics of pottery from Tell Sheikh Hamad (ancient Dur-Katlimmu), Tell Jemmeh (in Israel), and from two Neo-Assyrian capitals [19, 2]. Tell Sheikh Hamad was contemporary with Tušhan and also functioned as a regional Neo-Assyrian provincial capital. It is located about 230 km to the southwest of the Assyrian heartland on the lower Khabur River, a tributary of the Euphrates. Based on Hunt's petrographic and chemical analysis of the pottery, she concluded that potters at Tell Sheikh Hamad made their own version of Palace Ware using local clays that looked very similar to examples made in the capital cities [2]. Hunt used geologic methods and chemical methods such as NAA to characterize Palace Ware at the capitals of Nimrud, Nineveh, and Aššur as her baseline for comparison [9, Hunt and Sterba 2013]. Her results are discussed below in relation to our own chemical analyses on the Ziyaret Tepe samples.

A total of 50 pieces were selected for analysis from the exported sherds taken from the Ziyaret Tepe excavations (see Table 1 below). These included 40 samples of probable local pottery. We used a standard concept called the 'criterion of abundance technique' to characterize the local pottery samples from the site [20]. Simply stated, we can safely assume that Plain Simple Ware is local because it makes up the majority (81% in primary contexts) of Iron Age pottery at the site and there would be no need to trade or import ordinary pottery or cooking wares from

elsewhere. Also as noted above, potters typically use clay found near their workshop location. Plain Simple Ware provides one indicator of local clay chemical signatures. Another source of information on the clays used are the discarded mistakes from pottery kilns at the site, overfired pieces called ‘wasters’.

Some contexts from which our samples are drawn date from other time periods than the Iron Age but are assumed to have used the same local Upper Tigridian clays in their production as did the potters of the Neo-Assyrian period. These include: one Middle Bronze Age cooking ware sherd, four Early Iron Age Plain Simple Ware sherds, and eleven Medieval cooking ware sherds. The one piece of Cilician Ware, stylistically a clear foreign import found in a single primary context in the palace at Tušhan, was included. Painted pottery is rare in the Neo-Assyrian period and comparanda suggest that this painted piece may be an import from Cilicia, 500 km to the west of Ziyaret.

Table 1: List of samples by ware, quantity, and period

Ware type	Ware name	No. of samples	Period
LA01	Plain Simple	10	Neo-Assyrian
LA03 and LA04	Cooking	10	Neo-Assyrian
LA05	Palace	6	Neo-Assyrian
LA06	Near Palace	3	Neo-Assyrian
ER01	Plain Simple	4	Early Iron
ME03	Cooking	11	Medieval
MB03	Cooking	1	Middle Bronze
Waster	unknown, vitrified	3	Medieval
Waster	unknown, vitrified	1	no date
XX	Cilician ware?	1	Iron Age?

230 We did not collect modern clay samples from the region during the project. Our permit for the
231 Ziyaret Tepe project was limited to on-site mapping and excavation and did not include survey
232 of the surrounding area so we did not map nearby clay sources while in the field. The
233 composition of the local clay, however, can be hypothesized from the geological context.
234 Geologically, the upper Tigris River flows through Lower Miocene and Upper Miocene-Pliocene
235 rock formations [21, 22]. The Lower Miocene limestone and sandstone formations contain
236 abundant quartz, feldspar and silt and the Upper Miocene-Pliocene formations are
237 conglomerates, clay, and silt [21]. At its upper elevations the Tigris also flows through pre-
238 Neogene limestones composed mostly of calcium carbonate in the form of calcite and ophiolitic
239 mélanges containing sedimentary and igneous rocks [21]. Given this geologic signature of the
240 region, we expected NAA to show high amounts of calcium, silicon, and likely iron and sodium
241 and/or potassium from the feldspars in the local clays.

242

243 Since pottery contains temper added by the potter, we also expected to find some elements
244 deriving from the mineral or organic inclusions visible in cross-sections of the sherds. One
245 common temper is grain chaff, and other types frequently seen in the clay macroscopically are
246 white quartz grains, mica, and black, white, or red mineral inclusions. From a macroscopic
247 perspective, it is not possible to identify these minerals, except that we may hypothesize
248 feldspar, quartz, or crushed conglomerate from the nearby river deposits. Previous petrographic
249 and chemical (X-ray fluorescence) study of common ware pottery from the Upper Tigris region
250 has indicated it often contains quartz and muscovite inclusions and that the local clays are iron-
251 rich [14].

NAA Methods

NAA was conducted by the Archaeometry Laboratory at the University of Missouri Research Reactor (MURR) using the standard methods and parameters at that laboratory. These methods are described in detail elsewhere [23, 24, 25]. To briefly summarize, a fragment of roughly 1 cm² was removed from each sherd. Because NAA is a bulk analytical technique, all surfaces were removed by burring using a silicon-carbide grinding tool to account for any compositionally distinct surface treatments, like clay slips or pigments applied as decoration. This also accounts for any post-depositional contamination from taphonomic processes. After burring was completed, samples were rinsed in deionized water and allowed to dry. Samples were then homogenized into a fine powder through grinding with an agate mortar and pestle and placed in a drying oven to remove any remaining moisture in the samples for a minimum of 24 hours at 105°C. Once completely dry, aliquots were measured into two vials: 100 mg of powder was measured into a high-density polyethylene vial, and 200 mg of powder measured into a high-purity quartz vial and sealed under vacuum. Masses were recorded to the nearest 0.01 mg, and all values were within ± 2 mg of the target mass.

Two at a time, the aliquots in the polyethylene vials were loaded into a larger polyethylene container called a 'rabbit' and transported to the reactor via a pneumatic tube system for an irradiation of five seconds by a neutron flux of $8 \times 10^{13} \text{ n cm}^{-2} \text{ s}^{-1}$. During this process, three samples of standards of certified reference material from NIST of SRM1633c Coal Fly Ash and SRM688 Basalt Rock, and an in-house quality control of New Ohio Red Clay were also irradiated

under the same parameters. After being allowed to decay for 25 minutes, samples were counted for a period of 12 minutes by high-purity germanium detectors, yielding values in parts per million for nine elements: Al, Ba, Ca, Dy, K, Mn, Na, Ti, and V.

Aliquots in quartz vials were bundled into groups of 50 samples along with four samples of standard SRM1633c, and quality controls of SRM679 Brick Clay and New Ohio Red Clay. These bundles were irradiated for a period of 24 hours in a neutron flux of $6 \times 10^{13} \text{ n cm}^{-2} \text{ s}^{-1}$. After an initial decay of seven days, these samples were washed and detected by high-purity germanium detectors for a period of 30 minutes each, yielding counts for As, La, Lu, Nd, Sm, U, and Yb. Samples were then allowed to decay for an additional two weeks before a second detection period of 2.5 hours, yielding counts for Ce, Co, Cr, Cs, Eu, Fe, Hf, Ni, Rb, Sb, Sc, Sr, Ta, Tb, Th, Zn, and Zr.

After all three periods of detection were complete, datasets were assembled and evaluated using a suite of multivariate statistical routines that are commonly applied to compositional data of archaeological ceramics and other materials [26, 27, 28, 29, 30]. This began with a calculation of a total variation matrix (TVM) [31, 32, 33], a table composed of log-transformed data where each element is expressed as a ratio of all other elements in the dataset. Total variation (vt) is the sum of all variances in the variation matrix divided by twice the number of elements in the matrix [33]. This value provides a metric to evaluate variability in a chemical dataset which is compatible with both variances and Euclidean distances. This value is significant to the evaluation of ceramic composition studies as it is an indicator of what is

referred to as a monogenic or polygenic datasets. A high value indicates a polygenic dataset. For a study of ceramic composition, this translates to multiple compositional groups made from chemically discrete raw materials.

Groups were next identified using a combination of different statistical methods which are commonly used in the interpretation of compositional data of archaeological ceramics [20], including principal components analysis (PCA), hierarchical cluster analysis, and total variation matrix. ~~Data was next subjected to principal components analysis (PCA). This test~~PCA demonstrated that greater than 95% of the cumulative variance can be explained by the first eight principal components. ~~Using the results of the PCA, along with hierarchical cluster analysis, s~~Sherds were assigned into four distinct compositional groups, with one outlier. After group assignments were made, group membership was evaluated and refined through the calculation of Mahalanobis distances. After group assignments were made, group members were examined across different attributes, including ware and time period.

The Archaeometry Laboratory at MURR maintains a database of compositional data of archaeological objects and source materials, including over 300,000 archaeological ceramics. Additionally, the Archaeometry Laboratory curates data from other reactors, some of which are no longer operational and others that no longer use NAA on archaeological materials. NAA data from this research was compared to relevant datasets from these databases. To compare to data from Hunt and Sterba [18] analyzed at the Technische Universität Wien, it was necessary to calculate a new PCA, removing values from elements that were not detected in common

between the two reactors (aluminum, calcium, dysprosium, manganese, titanium, vanadium, and tungsten).

Results

The chemical composition of the 50 sherds was detected by element. The University of Missouri Research Reactor (MURR) then described these patterns with various statistical techniques. In the calculation of the total variation matrix, chromium (Cr) showed highest amount of variation while dysprosium (Dy) showed the least. The TVM of the samples has a total variation (vt) value of 4.405. Often the integer is equivalent to the amount of groups present in a single dataset, so a vt value of 4.405 suggests that this data is polygenic and is made up of at least four compositionally discrete groups.

The chemical compositions of the samples cluster statistically into four main groups and four outliers in the principal components analysis. Figure 2 shows this pattern using the first two principal components, and accounts for 72.3% of the variation in the data. Groups 1 and 2 contain the majority of the pottery and are therefore assumed to represent the chemical signature for local clays. They are somewhat distinct from each other though they vary more from Group 4 (purple) and Group 3 (green).

Fig 2. Main compositional groupings of the Ziyaret Tepe samples. The scatterplot shows the sample distribution using the first and second principal components representing 72.3% of the total variance. The ellipses are drawn at 90% confidence intervals.

Another view of this data can be seen in Figure 3 below, where the elemental vectors in the principal components analysis are included. Chromium, as the element contributing the most difference, has a noticeably long vector compared to most other elements, with nickel being the second longest.

Fig 3. Biplot showing the distribution of samples using the first and second principal components with elemental vectors added. Ellipses are drawn at 90% confidence intervals.

Figure 4 graphs the compositional groups again, showing the four wasters as purple dots, showing that all wasters fall within either Group 1 or 2. Three wasters are from Medieval contexts and one from an undated context. The one waster seen in Group 1 is medieval in date.

Fig 4. Graph of primary and secondary principal components, showing wasters plotted as purple dots.

357 Though the scatterplots above are useful for describing the local clay signature near Ziyaret,
358 they do not take the time periods or wares of the samples into account. When mapped by our
359 defined wares for the site, the Iron Age wares fall into Groups 1, 2, and 3 but not 4. Figure 5
360 shows the same composition groups as oval border lines and in this case the symbols indicate
361 the Iron Age samples only. The possible import from Cilicia is coded as "Import?" in this plot.

362

363 Fig 5. Graph of the primary and secondary principal components showing compositional groups
364 as ovals with symbols for the Iron Age ware types. The ellipses indicate 90% confidence
365 intervals.

366

367 Nearly all the Iron Age samples fit into or fall near Groups 1 and 2. One pattern visible in Figure
368 5 is that all the samples within Group 3 are cooking wares (LA03 and LA04), represented by
369 green squares. Other samples of cooking wares are also present in or near Group 1. MURR
370 determined that the key distinguishing element separating Group 3 from Groups 1 and 2 was
371 calcium. Group 3 pots contained 18-21% Ca compared to Groups 1 and 2, where Ca levels were
372 between 5 and 10%. When the additional Ca was corrected for and the principal components
373 analysis run again, those three samples then fell within Group 1. Therefore, these cooking pots
374 were made using the same clay as other pottery at the site, but with a higher level of calcium.

375

376 With Figure 5, we can see where the Palace Ware (LA05 and LA06) fits as compared to the local
377 chemical signature indicated by Groups 1 and 2. Most of the Palace Ware pieces sampled match
378 the chemical composition of the local pottery as represented by the Plain Simple and cooking

379 wares from Ziyaret composing Groups 1 and 2. ~~In this plot~~Compositionally, the fine wares
380 cannot be distinguished from the chemical signature of the local pottery through the statistical
381 analyses that were applied.

382
383 The chemical composition of pieces from Ziyaret was compared with other regional capitals and
384 the imperial capitals of Nineveh and Nimrud in the heartland of the empire using the data of
385 Hunt and Sterba [18]. The other two regional capitals are relatively nearby, ancient Dur-
386 Katlimmu (modern Tell Sheikh Hamad in Syria) and on the outskirts of the empire, Tell Jemmeh
387 in modern Israel [18]. A PCA calculated with the 25 elements detected in common between the
388 Missouri and Vienna reactors demonstrated that greater than 95% of the cumulative variance
389 can be explained by the first nine principal components. Figure 6 shows the PCA when all these
390 samples are combined, graphed by the resulting first and second principal components.

391
392 Fig 6. Scatterplot of principal components 1 and 2, representing 66.2% of the total variance in
393 the data. Individual samples from Ziyaret are shown as plus signs while the samples from the
394 other sites analyzed at the Vienna lab are other symbols.

395
396 Much of these data overlap in Figure 6 except the Tell Jemmeh (blue squares) samples which
397 are more distinct from the others. Ziyaret Group 1 as outlined in red contains a broad area in
398 this scatterplot and overlaps with most of the samples from Dur-Katlimmu (purple dots).
399 Nineveh and Nimrud are closer to each other and further away from Tell Jemmeh than Ziyaret
400 Group 1. Given that Tell Jemmeh is in Israel, the chemical signature of its clay is quite different

than the other sites. Dur-Katlimmu is on a tributary leading into the Euphrates and so may be expected to be significantly different from the others along the Tigris, but in fact falls within Group 1 from Ziyaret. Group 2 from Ziyaret is a small area in this plot and does not contain many samples from other sites and in particular does not contain any of the signatures of samples from Nineveh and Nimrud.

To detect further differences between the Ziyaret samples and those from the capitals and Dur-Katlimmu (Tell Sheikh Hamad), we noted that Hunt found chromium (Cr) and hafnium (Hf) distinguished some of her samples [2]. MURR then took these elements into consideration. Figure 7 below shows the same compositional ovals as above for Ziyaret but graphs the concentration of Hf versus Cr in each sample. It includes all Ziyaret samples (shown as plus signs) and highlights Ziyaret fine wares and fine wares from Nimrud and Nineveh as reported by Hunt [9]. The most obvious pattern is the overlap between the Ziyaret types and samples from the Assyrian heartland. Nimrud samples are green triangles and those from Nineveh are pink diamonds, and they either fall into Group 1 or 2, or just outside them with slightly less Hf. The Nimrud samples fall into two clusters, one with lower Cr and slightly lower Hf, and one with higher values of both. All of the samples from Nineveh fall completely within Group 1 or 2.

Fig 7. Scatterplot of Cr and Hf showing ellipses and samples from Ziyaret Tepe, along with samples from Nimrud and Nineveh. Ziyaret Tepe Palace Ware (LA05) samples are represented as dark blue triangles, and Near Palace Ware (LA06) samples are teal diamonds. Ellipses are drawn at 90% confidence intervals.

423

424 Two samples from Ziyaret (ZT 48352/1 and ZT 5738/3) plot closely to several samples from
425 Nineveh and are also ones that were identified on other charts as near the Nineveh samples
426 through PCA and Euclidean Distance analyses. Several other sherds of Palace or Near Palace
427 Ware also plot closely to Nineveh in Figure 7. Their closeness was confirmed by MURR with a
428 Euclidean distance search using the 25 elements analyzed in common for the samples between
429 MURR and the Vienna lab. Overall, four Ziyaret samples (ZT 48352/1, ZT 503/8, ZT 503/2, and ZT
430 5738/3; see Appendix A) show the greatest similarity with the Nineveh samples from Hunt's
431 analysis. Since the Vienna lab did not measure Ca, a major component of the Ziyaret pottery,
432 and the clays up and down the Tigris apparently exhibit very little variation in their major
433 components, interpretation of these results are made with caution.

434

435 **Discussion/Conclusion**

436 We are now able to define chemically the local clays used in the Neo-Assyrian period in the
437 Upper Tigris river valley. ~~Chemical~~Statistical analyses of the chemical characterization places
438 most of the Plain Simple Ware as well as the cooking pots into Groups 1 and 2 ~~by PCA~~, and
439 additionally the presence of all the wasters in the same groups confirms that those groups
440 represent clays local to Ziyaret.

441

442 An unforeseen result of the chemical analysis was the discovery of extra Ca in some of the Neo-
443 Assyrian cooking pots. The MURR lab suggests that a production method involving slightly
444 varied clay preparation would cause this chemical pattern of higher Ca, probably related to a

need to create pots that could withstand thermal stress. This result is promising and provides information on pottery production methods that is difficult to detect otherwise since we did not find Neo-Assyrian pottery workshops at Ziyaret.

Regarding finewares, it is likely that Palace Ware at Ziyaret Tepe was produced locally, imported, or both. There are few significant chemical differences between the clays of the upper Tigris river valley near Ziyaret and the Tigris river near the Assyrian capitals. It is therefore difficult to clearly separate imports from local products through NAA. If any are imported, Nineveh represents the most likely source among those discussed here, given the close proximity between its samples and several from Ziyaret in Figure 7.

Our initial hypothesis was that the finewares used at Neo-Assyrian Tušhan during the imperial period were made using local clay. The results of the NAA study undertaken to test this hypothesis do not provide any clear evidence to reject or revise this hypothesis. As noted earlier, if Palace Ware was made at Ziyaret, the larger significance would be that there are very highly skilled potters operating at a regional capital, as appears was the case at another regional capital, Tell Sheikh Hamad in Syria. A second confirmed case of highly skilled local potters indicates that the production of such an elite type of pottery was not restricted to workshops in the imperial heartland. Unlike other crafts such as metalworking and textile production, the Assyrian bureaucrats did not closely track the movements of finished ceramic vessels and, based on evidence presented here, appear to have allowed regional production either by highly skilled craftspeople brought in from the imperial heartland, or local imitators who followed the

467 form and fashions set there, or both. Potters at Tušhan, Dur-Katlimmu and in other imperial
468 peripheries therefore likely produced fineware pottery independent of direct government
469 control.

470

471 In the future, sampling a greater variety of pottery from sites up and down the Tigris may make
472 it possible to distinguish slight differences in the local compositions of clay used for pottery.

473 Other researchers should take note of the proportion of rare earth elements such as chromium
474 and hafnium, which may vary more significantly over the landscape than other more common
475 elements. In our own ongoing research, we submitted NAA samples this year from two small
476 farmstead sites in the Erbil Plain within the Assyrian heartland as part of the Sebittu Project. The
477 samples were Plain Simple Ware as well as a few pieces of Palace Ware, to see how these vary
478 chemically from the others already studied. Unlike a regional capital such as Tušhan or Dur-
479 Katlimmu, we do not expect potters at such small sites to have produced fineware pottery
480 themselves. We are likewise expanding our NAA study to include glazed wares from Tušhan in
481 order to characterize this fineware chemically and see if it contrasts with Palace Ware. Broadly,
482 glazed wares are even more rare than Palace Ware as their production required careful control
483 of glaze preparation and precise temperature regulation as the vessels cooled after firing, in
484 addition to other skilled manufacturing techniques.

485

486 **Acknowledgments**

487

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490 to BH. JD performed the statistical analyses and wrote the report summarizing the results from
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493

494 **Author Contributions**

495 Conceptualization: BH, TM

496 Funding acquisition: BH

497 Data Curation: JO

498 Formal Analysis: JO

499 Resources: TM, JO

500 Writing – original draft: BH

501 Writing – review and editing: TM, JO, BH

502

503 **Supplemental Information**

504 **Appendix A:** Raw NAA data. List of sample numbers by sample number and their elemental
505 compositions, with additional tabs for PCA, total variation matrix calculation, and Mahalanobis
506 distances.

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509 **References**

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511 1. D’Agostino A. The Upper Khabur and the Upper Tigris valleys during the Late Bronze Age:
512 settlements and ceramic horizons, in Bonatz, D., ed. The archaeology of political spaces: The
513 Upper Mesopotamian piedmont in the second millennium BCE. Berlin, Boston, De Gruyter;
514 2014. p. 169-199.

515

516 2. Hunt A. Palace Ware Across the Neo-Assyrian imperial landscape: Social value and semiotic
517 meaning. Leiden, Brill; 2015.

3. Arnold, D. Ceramic Theory and cultural process. Cambridge, Cambridge University Press; 1985.
4. Matney T, J MacGinnis, D Wicke, and K Köroğlu. Ziyaret Tepe: Exploring the Anatolian frontier of the Assyrian empire. Edinburgh: Caique Publishing Ltd; 2017.
5. Parpola S. Cuneiform texts from Ziyaret Tepe (Tušhan), 2002-2003, State Archives of Assyria Bulletin. 2008;17: 1-113.
6. MacGinnis J, Monroe MW. Recent texts from Ziyaret Tepe, State Archives of Assyria Bulletin 2015;20: 47-56.
7. Lanfranchi G, Parpola S. The correspondence of Sargon II, Part II: Letters from the Northern and Northeastern provinces. 1990;State Archives of Assyria 5
8. Anastasio S. Atlas of the Assyrian pottery of the Iron Age. Subartu 24. Turnhout, Belgium, Brepols; 2010.
9. Hunt A. Assyrian palace ware definition and *chaîne opératoire*: Preliminary results from Nineveh, Nimrud, and Aššur, in M Martínón-Torres (Ed.), Craft and science: International perspectives on archaeological ceramics. Doha, Qatar: Bloomsbury Qatar Foundation; 2014 <http://dx.doi.org/10.5339/uclq.2014.cas.ch15>
10. Oates J. Late Assyrian pottery from Fort Shalmaneser, Iraq 1954;21(2): 130-146.
11. Rawson PS. Palace Ware from Nimrud: Technical observations, Iraq. 1954;16: 168-172.
12. Parker B. The mechanics of empire: The northern frontier of Assyria as a case study in imperial dynamics. Helsinki, Vammalan Kirjapaino Oy; 2001.
13. Matney T, Rainville L. Archaeological investigations at Ziyaret Tepe: 2003-2004, Anatolica. 2005;31:19-68.
14. Kibaroglu M. Archaeometric analysis of Early Bronze Age dark rimmed orange bowl ware (DROB ware) from the Upper Khabur (NE-Syria) and the Upper Tigris valley (SE-Anatolia), Arkeoloji Dergisi. 2021;26(4):91-106.
15. Courtois LC, Doray AM. Technologie et ceramiques Levantines au temps de la domination Assyrienne. Comptes Rendus du 108e Congrès National des Sociétés Savantes, Grenoble: Ministère de L'éducation Nationale Comité des travaux historiques et scientifiques. Paris: CTHS; 1983. p. 125–136.
16. Engstrom C. The Neo-Assyrians at Tell el-Hesi: A petrographic study of I=imitation Assyrian

- Palace Ware, Bulletin of the American Schools of Oriental Research. 2004;333: 69–81.
17. Freestone IC, Hughes MJ. Appendix III: Examination of ceramics from Qasrij Cliff and Khirbet Qasrij. In: J. Curtis (ed.) Excavations at Qasrij Cliff and Khirbet Qasrij. London: British Museum Publications Ltd., 1989. p. 61–75.
18. Hughes MJ, Freestone IC, Humphrey MS. Neutron activation and petrographic analysis of pottery, in Curtis, J. and A. Green, Excavations at Khirbet Khatuniyeh. Saddam Dam Report 11, Ministry of Culture and Information, Iraq. London, British Museum Press; 1997. p. 95-101.
19. Hunt A, Sterba JH. Chemical composition by Neutron Activation Analysis (INAA) of Neo-Assyrian Palace Ware from Iraq, Syria and Israel, Journal of Open Archaeology Data. 2013; 2:e10, DOI: <http://dx.doi.org/10.5334/joad.aa>
20. Bishop R, Neff H. Compositional data analysis in archaeology. In: Allen R, editor. Archaeological Chemistry IV, Advances in Chemistry Series 220. American Chemical Society; Washington, DC; 1989. p. 576–586.
21. Doğan U. Holocene fluvial development of the Upper Tigris Valley (Southeastern Turkey) as documented by archaeological data, Quaternary International. 2005;129: 75-86.
22. Nicoll K. Landscape development within a young collision zone: Implications for post-Tethyan Evolution of the Upper Tigris River system in Southeastern Turkey, International Geology Review. 2010;52(4-6): 404-422.
23. Glascock MD. Characterization of archaeological ceramics at MURR by neutron activation analysis and multivariate statistics. In: Neff H, editor. Characterization of Ceramic Pastes in Archaeology. Madison: Prehistory Press; 1992. p. 11–26.
24. Glascock MD. Compositional analysis of archaeological ceramics. In: Glascock MD, Neff H, Vaughn KJ, editors. Ceramics of the Indigenous Cultures of South America: Studies of Production and Exchange through Compositional Analysis. Albuquerque: University of New Mexico Press; 2019.
25. Glascock MD. Instrumental neutron activation analysis and its application to cultural heritage materials. In: D’Amico S, Venuti V, editors. Handbook of Cultural Heritage Analysis. Cham: Springer International Publishing; 2022. pp. 69–94. doi:[10.1007/978-3-030-60016-7_5](https://doi.org/10.1007/978-3-030-60016-7_5)
26. Baxter MJ. Archaeological use of the Biplot--a neglected technique? In: Lock G, Moffett J, editors. Computer Applications and Quantitative Methods in Archaeology, 1991. Oxford: Tempvs Reparatum, Archaeological and Historical Associates; 1992. pp. 141–148.

27. Baxter MJ, Buck CE. Data handling and statistical analysis. In: Ciliberto E, Spoto G, editors. Modern Analytical Methods in Art and Archaeology. New York: John Wiley & Sons, Inc.; 2000. p. 681–746.
28. Neff H. RQ-Mode principal components analysis of ceramic compositional data. Archaeometry. 1994;36: 115–130. doi:[10.1111/j.1475-4754.1994.tb01068.x](https://doi.org/10.1111/j.1475-4754.1994.tb01068.x)
29. Neff H. Neutron activation analysis for provenance determination in archaeology. In: Ciliberto E, Spoto G, editors. Modern Analytical Methods in Art and Archaeology. New York: John Wiley & Sons, Inc.; 2000. pp. 81–134.
30. Neff H. Quantitative techniques for analyzing ceramic compositional data. In: Glowacki DM, Neff H, editors. Ceramic Production and Circulation in the Greater Southwest: Source Determination by INAA and Complementary Mineralogical Investigations. Los Angeles: Cotsen Institute of Archaeology; 2002. pp. 15–36.
31. Aitchison J. The statistical analysis of compositional data. Dordrecht: Springer Netherlands; 1986.
32. Buxeda i Garrigós J. Alteration and contamination of archaeological ceramics: The perturbation problem. Journal of Archaeological Science. 1999;26: 295–313. doi:[10.1006/jasc.1998.0390](https://doi.org/10.1006/jasc.1998.0390)
33. Buxeda i Garrigós J, Kilikoglou V. Total variation as a measurement of variability in chemical data sets. In: van Zelst L, editor. Patterns and Process: A Festschrift in Honor of Dr Edward V Sayre. Suitland: Smithsonian Center for Materials Research and Education; 2003. p. 185–198.

Captions: NEW Figure 1: Map showing sites mentioned in the text, and the Assyrian heartland.