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Insights into the production technology of north-Mesopotamian Bronze Age pottery

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ABSTRACT With the aim of contributing to the knowledge of north-Mesopotamian Bronze Age pottery production (3rd millennium BC, early Dynastic and early Akkadian period), the mineralogy of pottery excavated from the site of Tell Beydar (Syria) has been studied in order to make inferences concerning the clay preparation and firing techniques of that period. The fired pottery finds have been classified by archaeologists into three distinct groups on the basis of their aesthetic and visual appearance and their mechanical strength: standard, cooking and "metallic" ware.

SR X-ray powder diffraction data have been collected from 100 individual shards as well as from local clays and one unbaked object, an inscribed tablet. The XRD data is supplemented by SEM-EDS, XRF and polarising microscopy studies of 200 polished thin sections. The synthesis of the results from this extensive study quantifies the basic physical characteristics of the ensemble: the standard ware required no specific clay preparation and firing procedures. The body of the cooking ware contains large inclusions which result in a body texture intended to make them resistant to repeated thermal cycles. Both standard and cooking ware are made of a calcareous clay, typical of pottery from the Middle East. The metallic ware, however, are of a much finer quality with a distinctly different mineralogy than the other two groups.

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1 Introduction

Contrary to other cultural areas in the world, the use of analytical techniques for the study of ceramics is still not very customary in Mesopotamian archaeology. When applied, they provide answers to questions concerning the potteries provenance and/or technology. It is clear, however, that most of the interest so far has gone to decorated and technologically superior ceramics. Several studies for instance have discussed the provenance of painted prehistoric pottery as well as the materials and techniques used for their decoration. In most of these studies, the techniques applied are petrography, neutron activation analysis (NAA), microprobe/scanning electron microscopy (SEM), or a combination of these (e.g. [1-15]). In other studies pottery from historical periods has been analyzed. Typically, Islamic stonepaste ceramics and glazes (e.g. [16-18]), but also pre-Islamic glazes are discussed (e.g. [19, 20]).

In between these two periods of interest lies a virtually unexplored era of Mesopotamian pottery production, ranging from the 4th/3rd millennium BC to the beginning of the Seleuco-Parthian period, spanning some 3000 years. This gap is, we believe, caused by two main factors. During the 4th/3rd millennium BC, there is a sharp decline in the production of decorated pottery. This decline is partly related to the development of mass production, linked with, amongst others, the introduction of the potters wheel. Probably the 'unattractive' appearance of this undecorated, seemingly tedious earthenware, having no striking features, is one of the reasons why Mesopotamian Bronze Age ceramics are hardly ever the subject of technological studies and usually only used by archaeologists as cultural indicators and dating tools. Another, perhaps more limiting factor is the geological homogeneity of large parts of Mesopotamia [21].

As a result of these two main factors – apparent unattractiveness of the pottery and geological homogeneity of the region – little is known of the technological and socioeconomical aspects of 3rd millennium BC north-Mesopotamian pottery production. Nevertheless, valuable studies have been published over the last decade (e.g. [22–31]).

The present study is part of a project set up to study various aspects of the potters craft at a specific time in a specific place, namely 3rd millennium BC at Tell Beydar (Syria). One of our aims is to outline the potteries production sequence. Firing is one of the most crucial stages in the production of pottery, as it is the production step that transforms clay into an imperishable product. The firing technique applied has a direct influence on the mineralogy of the raw materials [32]. Pottery makers and its end users may not have been familiar with the clays or vessels mineralogy in a direct way, but this mineralogy is reflected in several aspects of the pottery that might be of importance to them, such as the colour of the product and its resistance to mechanical or thermal shock. Another part of the production sequence that we have studied by looking at the potteries mineralogy is the selection and preparation of raw materials. This has been done by comparing the clay resources used with those that are (were) locally available.

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This paper combines newly established research results together with some previously published data on specific types of pottery [33–35] with the aim of giving a full overview of the production process of 3rd millennium BC pottery at Tell Beydar.

2 The archaeological site

The site of Tell Beydar, the ancient town of Nabada, is situated in the Syrian Jezireh, some 35 km NW of the modern town Hassake (Fig. 1). The 28 ha settlement is located at the crossroads of two main routes, one connecting the Euphrates river in the west with the Tigris river in the east, the other coming from the more north, the Anatolian plateau, and running further south following the Khabur river. Less then 200 m east of the Tell runs the Wadi 'Awaj, a small seasonal tributary of the Khabur river. To its immediate west lies the Ard es-Sheikh, a plateau formed by quaternary basalt. This plateau is the only disruption in the monotonous landscape of lower Pliocene and upper Miocene sandstones, clayey marls, calcareous clays, sands and siltstones [36].

The site was occupied mainly during the 3rd millennium BC (early dynastic and early Akkadian periods), but more recent remains dating to the 2nd and 1st millennia BC (Mitanni, Neo-Assyrian and Seleuco-Parthian periods) have also been excavated. In this study, only 3rd millennium BC ceramics are considered. During this millennium an urban culture had developed in Mesopotamia. The western part of northern Mesopotamia of that period is characterized by the existence of fortified circular settlements, like Tell Beydar, called Kranzhügeln after their typical concentric shape. Dominating the region from the early dynastic II period (EDII, ca 2700–2550 BC) onwards, these urban centers flourish during EDIII (ca. 2550–2350 BC), a prosperous period for the whole of northern Mesopotamia.

The excavations at Tell Beydar were conducted by a European–Syrian mission under the direction of the European Center for Upper Mesopotamian Studies in Brussels [37].

3 Materials

Apart from local soil samples, clay seal impressions and an object in unbaked clay, all types of 3rd millennium pottery that have been excavated at Tell Beydar are represented in our study. Standard ware (or common ware) is the main group of excavated pottery. This ware was made for every day use and is represented by a variety of sizes and shapes such as jars, bowls, bottles, plates, trays, potstands, etc. standard ware usually has a buff colour (Fig. 2). Most of it is cone wheel made, while large trays and storage jars are handmade using a turntable. A thorough study of forming techniques attested at Tell Beydar is presented by van As and Jacobs [38, 39].

A second group of samples is cooking ware. Tell Beydar cooking ware shows many 'typical' cooking ware attributes [34]. These vessels have almost exclusively rounded, often closed shapes, with a burnished surface (Fig. 3). Their fabrics are characterized by many large limestone and/or basalt inclusions. Although the upper parts of Tell Beydar cooking pots are not particularly thin-walled (typical vessel walls having a thickness of one centimeter or even more), the few larger body shards and more or less complete cooking pots that have been found indicate that they were significantly thinner at the base. An ever-occurring feature of cooking ware from Tell Beydar is the burnished outside surface. Most of the



FIGURE 1 Tell Beydar in NE-Syria



FIGURE 2 Metallic ware jars (indicated by *arrows*) from Tell Beydar, surrounded by contemporary standard ware. Reproduced with permission from Brepols Publishers



FIGURE 3 Cross sections of some typical cooking ware as used in Tell Beydar. After [34]

few open cooking ware shapes that are found also show this feature on the inside.

In addition to these two utilitarian wares, some more luxurious wares are attested. One of them is the so-called metallic ware, typically found at 3rd millennium BC sites in the western part of upper Mesopotamia. It is made from very dense clay, usually showing very few inclusions. It can be grey, reddish brown, or a combination of these, depending on the firing atmosphere (Fig. 2). From a chemical point of view, 3 groups of metallic ware are known at Tell Beydar, mainly depending on their CaO content. One of these groups, the non-calcareous metallic ware, has a non-local origin [33, 35].

4 Experimental

Thin sections of 200 ceramic samples were examined at Southampton University (UK) and Leuven University (Belgium) using polarizing microscopy, enabling us to study the mineral inclusions in the clay matrix as well as the structure of that matrix.

About half of these samples, together with the soil and unbaked clay artefact samples were studied by X-ray diffraction (XRD). XRD is one of the most direct techniques for the identification of the mineral composition of ceramics as it can be used to analyze both inclusions and matrix. Its main disadvantage is that data collection can be quite time-consuming, each data set requiring several hours. It is mainly for this reason that XRD analysis is usually applied only to a small number of samples. The data collection time can be reduced by orders of magnitude using synchrotron X-rays and a fast area detector. An additional advantage of 2D data recording is that an easy distinction can be made between fine-particle phases, such as hercynite in pottery produced under reducing conditions, and overlapping reflections of coarser material such as quartz. In this study 2D diffraction patterns were acquired using a CCD detector at station 9.6 of the synchrotron radiation source (SRS) at Daresbury Laboratory (UK). Small amounts of powder extracted from the ceramics were loaded in 0.5 mm quartz capillaries. The beam footprint was 200-250 microns and the exposure times 3-4 min in single bunch mode (20 mA beam current). Much faster collection times (< 30 s) are easily attained with the SRS operating in normal, multibunch mode (200 mA beam current). The data were polar transformed and azimuth integrated using the ESRF program FIT2D [40]. Macros permit batch processing of several datasets at a time.

A spectrum correlation software utility was used to give quick-glance overviews of degree of similarity of the diffraction patterns, a useful aid in grouping samples according to mineral phase content and relative abundance.

To fully understand the materials mineralogy, however, one should also bear in mind its elemental composition, since mineralogical and chemical compositions are interrelated. Whenever needed, we will refer to results of our chemical analysis, obtained by scanning electron microscopy (SEM-EDS). Analysis were performed with a JEOL JSM-6300 microscope. The investigated shards were first embedded in a resin, whereupon their sections were gradually smoothed using waterproof abrasive paper up to grade 4000. A 20 keV, 1 nA electron beam was used to bombard the samples and signals were accumulated during 100s (live time). For the determination of the approximate bulk composition, several areas of 10-25 mm² were rastered, the exact size of the area and the number of places scanned depending on the size of the sample. Standardless ZAF corrections were used for obtaining quantitative results.

5 Results and discussion

5.1 Soil samples, clay seal impressions and unbaked clay object

In an attempt to characterize the mineralogy of local resources, four local soil samples, 5 clay seal impressions and an object in unbaked clay were analyzed. While we cannot know if the local clay samples represent clay beds that

Sample	Nos	Q	Ca	Dol	Plg	Kfsp	Ill	Kao	Mm	Px	Me	Mu	He	Hem	Mag	Mica
Clay	4	XX	XXX	х	х		х	х	х							
Seal impressions + object	6	XX	XXX	x(x)	х		x+	х	x—							
Cooking ware 1 Cooking ware 2	11	x xxx	XXX XXX	(x-) (x)	$\begin{array}{c} x-\\ x(x) \end{array}$		X X		(x) x	x(+)						
Standard ware	30	XXX	(xx)		x+					XX	$\mathbf{x}(\mathbf{x})$			х		(x-)
Metallic ware 1 Metallic ware 2 Metallic ware 3	30	XXX XXX XXX	(xxx)		x xx(x)	х				xx x(xx)	x (x)	х	х	x	x (x) (xx)	(x-) (x)

xxx – main phase; xx – present in significant quantities; x – present; brackets – only in some samples

TABLE 1Mineral phases of Tell Beydar clay samples, seal impressions and ceramics as revealed by SR-XRD (Nos = Number of samples Q = quartz,
Ca = calcite, Dol = dolomite, Plg = plagioclase, Kfsp = K-feldspar, Ill = illite, Kao = kaolinite, Mm = montmorillonite, Px = pyroxene, Me = melilite,
Mu = mullite, He = hercynite, Hem = hematite, Mag = magnetite)

were exploited in antiquity, we can be sure that the seal impressions and the clay object represent resources that were used during the early bronze age, even though not necessarily for pottery production. The soil samples were taken from different localities near the site; they all have the same mineralogical composition. The composition of the unbaked clay artifacts is very similar yet slightly distinct from that of the soil samples (Table 1). The main constituent of both sets of samples is calcite, followed by quartz. Calcite is not the only carbonate material, since some dolomite is also present. These two calcium-bearing minerals originate from limestone inclusions in the clay. The actual clay minerals are illite, kaolinite and montmorillonite. Finally, plagioclase is present, too.

Between the two groups (soil samples and artifacts), only small differences in illite and montmorillonite are observed. In addition, the relative abundance of dolomite in some of the seal impressions is higher. These differences might be caused by a possible preparation of the raw materials before using them, since these artifacts showed very few mineral inclusions. This preparation (removing sand and limestone inclusions) would have altered the relative abundances of the constituents. However, it is not excluded that also post depositional factors play a role. The latter refers to the issue of whether ceramics are preserved during their burial in the soil. Alteration processes have in the past been investigated by various research teams (e.g. [41–43]).

5.2 Cooking ware

Much of the mentioned mineral components are also present in the cooking ware samples ([34] see cook-

ing ware 1 and cooking ware 2 in Table 1). The presence of carbonates and clay minerals in these samples indicates that cooking ware was fired at a relatively low temperature. Although clay minerals can sometimes hold their structure up to considerable temperatures, the presence of calcite and in many samples even dolomite indicates that a temperature of ca. 800 °C cannot have been exceeded. The exact temperature is difficult to assess from mineralogical data alone, since the exact temperature at which these carbonates decompose also depends on factors like heating rate, firing atmosphere, and grain size. However, in half of the samples no dolomite is left, while in the other half its amount seems to have been already diminished, meaning that the temperature would have been somewhere around 800 °C [44].

Two subgroups can clearly be distinguished when studying the cooking ware samples in thin section, (Fig. 4a and b) [34]. They both contain a rather large amount (< 35%) of large mineral inclusions. One subgroup is characterized by large limestone inclusions of up to 2 mm. In addition, smaller fragments of quartz and plagioclase are also present, and often small black opaque iron oxide inclusions, as well as mica (biotite) are found. In some cases limestone is replaced by angular monocrystalline calcite. The other subgroup is differentiated from the first one by the presence of large basalt inclusions. All other minerals that are present in the samples from the first subgroup can usually also be found in the samples from the second one. This means that in the second subgroup, large limestone inclusions exist next to the basalt inclusions, albeit in smaller amounts. The large size and (sub-)angular shape of the limestone and basalt inclusions indicate that they were added by the potter. Basalt tempered



FIGURE 4 Petrography image (XP) of (a) first subgroup showing clear dominance of badly sorted limestone inclusions (sub-angular to subrounded, occasionally angular); *dark areas* are pores ($80 \times$) and (b) a second subgroup showing a large basalt inclusion (*top* of the image) including pyroxene and plagioclase; most medium sized and smaller inclusions are limestone (*pale colour*) and basalt fragments ($80 \times$). After [34] cooking ware is also known from other sites in the region, e.g. Tell Rad Shaqrah [45], Tell Brak [46], and Tell Bderi [47]. It is clear that these large mineral inclusions in both subgroups were meant to increase the pots resistance to thermal stress. As stated before, all cooking ware samples show an open matrix structure, caused by the presence of elongated (< 1 mm) cracks. Some samples also contain micropores of up to 400 μ m. This open structure would have had the same purpose.

The division of the cooking ware samples in two distinct groups is clearly visible in the SR-XRD results (Table 1). In the first subgroup (cooking ware 1) calcite is the strongest component (hence the lower quartz values), while the second subgroup (cooking ware 2) contains pyroxene and more plagioclase, resulting from the basalt inclusions. Thin section analysis shows these inclusions to be very poor in olivine, consistent with the lack of identifiable olivine reflections in the SR-XRD data.

Besides a difference in added non-plastics, we can not excluded that the firing technology differed between the two cooking ware subgroups, given their differences in dolomite and montmorillonite contents. This could suggest, for the second subgroup (cooking ware 2), a lower temperature, a shorter firing cycle, a different firing atmosphere, or a combination of these factors. However, the lower values for dolomite and montmorillonite in the first subgroup (cooking ware 1) could well be caused by the extremely high value for calcite, its high abundance effectively diluting (lowering) the relative amounts of the other minerals (as it is probably the case for the low quartz values).

Both subgroups show an enormous chemical heterogeneity. Table 2 lists the chemical composition of two shards (one of each type) merely as an indication.

It is not clear why basalt-tempered cooking pottery was made contemporaneously with limestone tempered ware. The shapes of the vessels made with the two different fabrics show no difference, nor do any of their other features. The possible differences in firing technology might indicate that the two kinds of cooking pottery represent different potting traditions or workshops.

5.3 Standard ware

These samples, contrary to the cooking ware samples, show a normal (to dense) matrix structure. Still, they contain 15%–25% of moderate to good size-sorted mineral inclusions (Fig. 5). These are quartz, plagioclase and burnt-



FIGURE 5 Petrography image (XP) of typical standard ware $(80 \times)$

out carbonate material, resulting from limestone decomposition. All samples also contain very small amounts of elongated sub-angular mica inclusions (usually biotite). Because of their very small quantity, these inclusions are not always recognized in the SR-XRD analysis. All inclusions are usually smaller than 200 μ m (occasionally double that size). Quartz and plagioclase inclusions have a sub-rounded to sub-angular shape, the limestone leftovers are always (sub-)rounded. Given the shape and size of the mineral inclusions, we can assume that they were naturally present in the clay. Since all limestone inclusions have decomposed in almost all samples, they are usually absent in the SR-XRD data. However, some samples contain calcite, sometimes in significant amounts. This calcite may originate from incomplete decomposition or from recarbonation [38, 39].

In the standard ware samples all carbonates and clay minerals have decomposed and recombined to form new high temperature phases, indicating a higher firing temperature (Table 1). These new phases are pyroxene (structurally similar to diopside) and melilite (structurally similar to gehlenite). Plagioclase is also formed during firing. The total decomposition of carbonates and clay minerals, together with the existence of these new phases indicate that the firing temperature must have been at least 850 °C. The latter has been determined by investigating the ceramics state of vitrification by means of electron microscopy. The nature of the high temperature minerals also indicates that the calcium content of these samples is high, as confirmed by chemical analysis (15%-25% CaO). The presence of hematite in all samples indicates a prevailing oxidizing firing atmosphere, at least in the final stage of the

Sample	Na ₂ O	MgO	Al_2O_3	SiO_2	K ₂ O	CaO	SO ₃	TiO ₂	Fe ₂ O ₃ t*
Standard ware $(N = 16)$	b.d.l.**	5(1)	13(1)	47(3)	2.4(0.9)	19(5)	4(2)	0.7(0.2)	8(2)
Cooking ware type I (sherd 102)	b.d.l.	4	12	48	3	24	2	0.7	9
Cooking ware type 2 (sherd 201)	b.d.1.	8	16	59	2	14	b.d.1.	0.5	8
Calcereous metallic ware $(N = 14)$	0.8(0.3)	5.0(0.7)	12.2(0.8)	49(3)	2.8(0.6)	21(4)	b.d.1.	0.7(0.2)	8(1)
Non-calcareous metallic ware $(N = 21)$	0.6(0.4)	1.8(0.5)	20(2)	61(4)	4(1)	3(1)	1(1)	1.1(0.2)	7(2)
Intermediate metallic ware $(N = 8)$	b.d.l.	5(1)	15(2)	59(3)	2.6(0.8)	9(5)	b.d.l.	0.9(0.1)	9(1)

* $Fe_2O_3t = total Fe as Fe_2O_3$

** b.d.l. = below detection limits

 TABLE 2
 Average elemental composition of the ceramic groups as revealed by SEM-EDS (SD between brackets)

firing cycle. The buff colour of all samples already indicated a (neutral to) oxidizing atmosphere.

5.4 'Metallic ware'

Investigation of the metallic ware samples using optical and polarizing microscopy, enabled us to distinguish three subgroups, depending on the structure of the matrix



FIGURE 7 SEM image (BSE mode) of non-calcareous metallic ware, showing K-feldspar inclusions (*light grey, dark grey* are quartz inclusions) and on the presence of mineral inclusions, namely calcareous metallic ware (CaO > 15%), non-calcareous metallic ware (CaO < 4%), and an intermediate group (CaO 5%-10%) (Fig. 6) [35]. The latter was separated because the CaO content of this group showed no continuity towards that of the

(CaO < 4%), and an intermediate group (CaO 5%-10%) (Fig. 6) [35]. The latter was separated because the CaO content of this group showed no continuity towards that of the calcareous group, but also because this range of CaO content was not represented in the standard ware. Not only CaO content, but also other elements, including trace elements caused the division. The mineralogical composition of calcareous metallic

and mineralogical composition of calcareous metallic ware resembles that of the standard ware (as does the elemental composition). The only difference is the lack of hematite in the calcareous metallic ware, caused by a reducing atmosphere (Table 1).

SEM-EDS analysis indicate that the non-calcareous metallic ware was made using raw materials that were clearly of non-local origin (Table 2). This is also reflected in the mineralogical composition (Table 1: metallic ware 2). Mullite indicates that kaolinitic clay was used, and that high temperatures (at least 950–1000 °C) were reached. These high temperatures are also indicated by the presence of hercynite, which is formed in a reducing atmosphere. This group is also characterized by small amounts of K-spar, present in the form of inclusions (< 50 µm), as indicated by SEM-EDS analysis (Fig. 7).

The intermediate group differs from the calcareous group and the standard ware, in that it contains less pyroxene, more plagioclase, and no melilite (Table 1: metallic ware 3). This is due to the lower amount of CaO in the intermediate group. Some intermediate samples contain both hematite and magnetite. This is caused by the alternated use of oxidizing and reducing atmospheres, resulting in a combination of red and grey colours on this pottery. The section of these shards usually shows a 'sandwich pattern', which is another indication for the use of an alternating firing cycle. These samples show an even denser matrix than the samples from the calcareous subgroup.

Conclusions

6

FIGURE 6 Petrography image (XP) of (**a**) calcareous metallic ware $(40 \times)$, (**b**) non-calcareous ware $(60 \times)$ and (**c**) the intermediate group $(60 \times)$

The mineralogical analysis of Tell Beydar ceramics by SR-XRD and petrography have given us technological



information on these ceramics, mainly regarding clay preparation and firing techniques.

It is clear that local clays are rich in calcium, mainly caused by the presence of calcite and, to a lesser extent, dolomite. Several artifacts in unbaked clay show virtually the same mineralogical composition as these local clay samples. Slight differences might be caused by clay preparation techniques, in particular the removal of non-plastic inclusions.

Phase analysis of cooking pottery has shown that low temperatures (< $800 \,^{\circ}$ C) were used. Two main types of cooking ware are attested, differing from each other on a mineralogical level. One group is characterized by the presence of large limestone inclusions, added by the potter. The other group also contains limestone inclusions, but here also large basalt inclusions (from local outcrops) are added. The firing cycle for the basalt-rich group might have differed from the firing technology of the limestone-rich group, in that a lower temperature, a shorter firing cycle, a different firing atmosphere, or a combination of these was applied.

Standard ware does not contain obviously added nonplastics. Phase analysis shows that it is clearly fired at a relatively higher temperature (+850 °C), in an oxidizing atmosphere (in the final stage of the firing cycle). The high temperature mineral phases indicate the use of a calcium-rich clay. It is very likely that the local clays were used for production of standard ware (as suggested by chemical analysis).

Metallic ware is clearly to be divided in three subgroups. These three mineralogical groups correspond with the earlier established chemical groups. The first subgroup totally corresponds with the standard ware, except for the firing technology: high temperature phases evidently indicate a reducing atmosphere. A second subgroup of metallic ware clearly has a non-local origin, given the absence of calcium bearing minerals. A kaolinitic clay was fired above 1000 °C in a reducing atmosphere. This pottery shows a very fine matrix with only very small inclusions. A third group differs from the two others from a mineralogical (and chemical) point of view. The values for the high temperature phases differ from the two other subgroups because a less calcium rich clay was used that for the first subgroup, and because a different firing technology was used, namely an alternating oxidizing and reducing atmosphere. The samples in this subgroup have the densest matrix of metallic ware samples. This suggests a special preparation procedure of refining the raw materials.

It is clear that different technologies were used for different kinds of pottery. While standard ware apparently required no specific needs in terms of clay preparation or firing technology, cooking ware and metallic ware did. For cooking ware these specific needs are clearly caused by its utilitarian character. Cooking pots are repeatedly heated and cooled again, and therefore they should contain more than the average thermal stress resistance. Metallic ware was a more luxurious ware. It was probably imported (or made from a non-local non-calcareous clay), and then imitated by local potters using local calcium-rich clays. These imitations started very early, since calcium-rich metallic ware was attested to in the earliest metallic ware levels at Tell Beydar. The meaning of the intermediate group of metallic ware remains unclear. This group might represent another clay origin, or maybe another local potting tradition, where maybe different (local and non-local) clays were blended for the production of metallic ware imitations. Blending of different clays is still a common practice among contemporary traditional potters.

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