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## Unveiling the function of long-spouted ceramics at Sialk, Iran: Insights from organic residue analysis

Emmanuelle Casanova<sup>a,b,\*</sup>, Julien Cuny<sup>c</sup>, Antoine Zazzo<sup>a</sup>, Marjan Mashkour<sup>a</sup>

<sup>a</sup> UMR7209 Archéozoologie, Archéobotanique - Sociétés, Pratiques et Environnements, Muséum National d'Histoire naturelle/CNRS, Paris, France

<sup>b</sup> Laboratoire des Sciences du Climat et de l'Environnement, UMR 8212 CEA CNRS UVSQ, Université Paris Saclay, Gif-sur-Yvette, France

<sup>c</sup> Département des Antiquités Orientales, Musée du Louvre, Paris, France

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### ABSTRACT

The site of Sialk, with its long settlement history and large necropolises offers one of the most important relative chronology of Iran. During the Iron Age, particular pottery vessels with a long spout and elaborate decorations appeared at the site. While these ceramics had been the driven factor behind excavations in the area of Sialk, their function remains the object of numerous hypotheses. Here their function was investigated through organic residues analysis. Results show ruminant dairy fats and ruminant carcass fats were processed in the ceramics. Considering the pottery's shape these products would have been in a liquid state to be poured through the spout, thus milk-based and, blood-based or tallow-based mixtures. These results provide insight into the function of spouted vessel for pouring liquids of various origin during Iron Age funerary rituals.

### 1. Introduction: Sialk a protohistoric site in the Kashan Plain, Iran

The site of Sialk (or Tepe Sialk) in the Kashan Plain of Iran (Fig. 1a), is one of the major sites in Iranian archaeology (Nokandeh et al., 2019). It exhibits a long occupation sequence spanning six phases from the late Neolithic (beginning of the 6th millennium BCE) up to the eve of the Achaemenid period (around 550 BCE) (Fahimi, 2013; Fazeli Nashli et al., 2023; Ghirshman, 1938; Nokandeh, 2010; Pollard et al., 2013; Shahmirzadi, 2004). The site was absolutely dated by the radiocarbon method and by optically stimulated luminescence with a particular focus for the domestic and oldest occupation of the site compared to the Iron Age occupations and cemeteries (Fazeli Nashli et al., 2023; Pollard et al., 2013). Pottery typology has allowed for the establishment of a relative chronology that is commonly referred to for comparison with the relative chronology of other regions of Iran.

The site of Sialk was first excavated by an expedition of the Louvre Museum (Paris, France) led by Roman Ghirshman between 1933 and 1937. It comprises two large mounds, the North mound presenting the Neolithic settlements and the South mound presenting an occupation from the Chalcolithic period to the Iron Age with an 800-year hiatus during the 2nd millennium BC (Pollard et al., 2013). The Iron Age is mainly represented by two necropolises: Necropolis A from the Early

Iron Age (phase V), located south of the South Mound, and Necropolis B from the Iron Age (Phase VI), located West of the South Mound (Ghirshman, 1939, 1938, 1935).

The interest in carrying out excavation in the Kashan region during the early 20th century arose from the arrival of particular painted ceramics with long-spout on the local art market, from illegal digging in the Kashan plain (Ghirshman, 1935). These vessels exhibited refined red-on-cream decorations (example of typology shown in Fig. 1b). Excavations were conducted at Sialk in late autumn of 1933, and it was during the second excavation season in 1934 that the Necropolis B, at the origin of such pottery typology, was discovered. During the 1934 and 1937 seasons, a total of 218 graves were identified in Necropolis B, some of which suffered from pillage before their discovery, although not all were excavated (Ghirshman, 1939, 1938). The graves typically contained only one body, except in a few cases where graves were reused in a later period. In the graves from Necropolis B, several objects were recovered alongside the dead such as ceramic, bronze and lead vessels, bronze and iron weapons, silver and iron jewels, or bronze figurines with variable quality across the tombs allowing to distinguish between the tombs of higher and lower status (Ghirshman, 1935). More excavations were carried out in the 2000s with the "Sialk reconsideration project" but these focused on habitation parts of the site (Helwing, 2005; Soltysiak and Fazeli Nashli, 2010). As per the law in force at the time of

\* Corresponding author at: Laboratoire des Sciences du Climat et de l'Environnement, UMR 8212 CEA CNRS UVSQ, Université Paris Saclay, Gif-sur-Yvette, France  
E-mail address: [emmanuelle.casanova@lscce.ipsl.fr](mailto:emmanuelle.casanova@lscce.ipsl.fr) (E. Casanova).

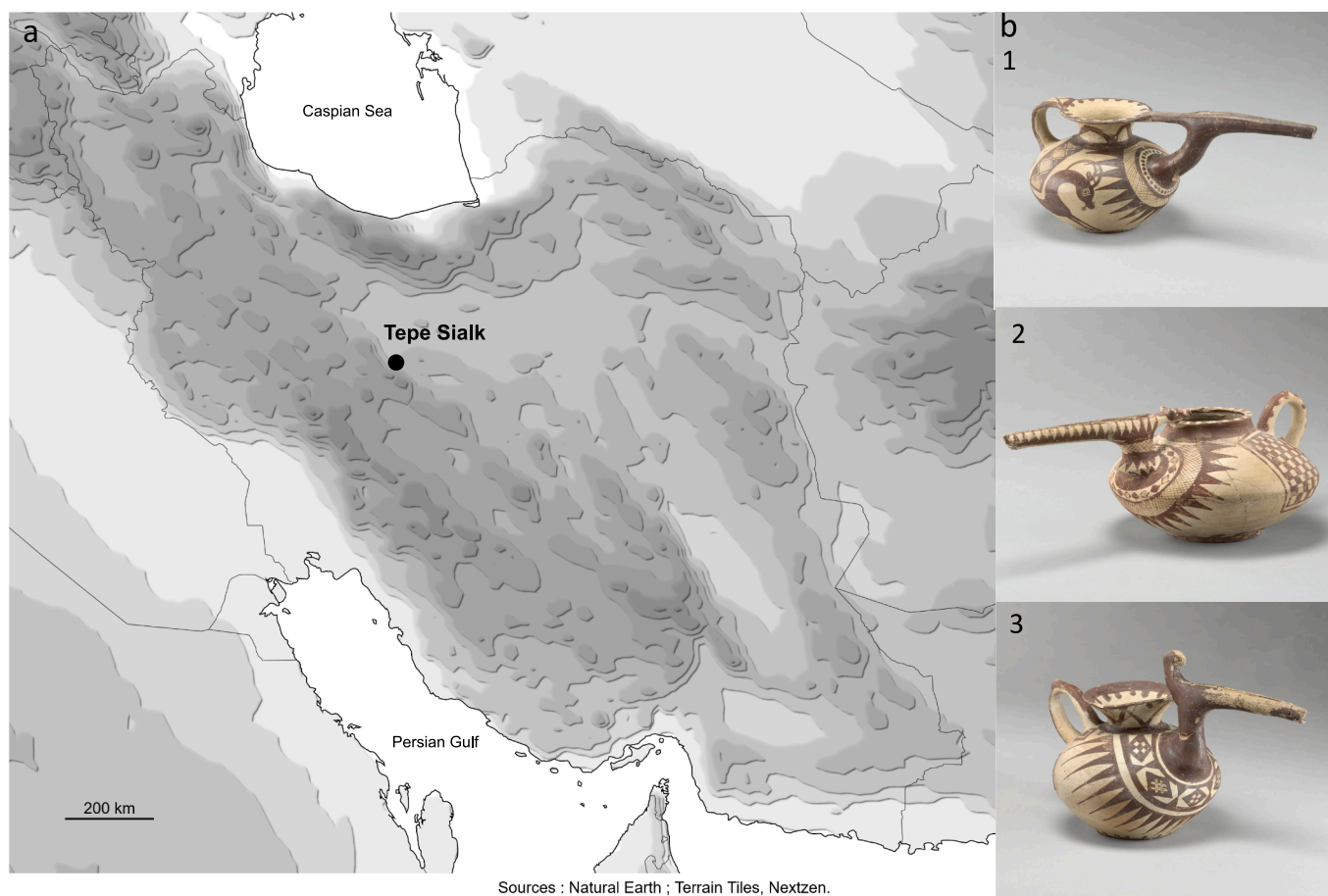
Ghirshman's excavations, the finds were divided equally between the excavators and the Iranian government. The collection of artefacts discovered in the 1930s excavations at Sialk brought to France, including 430 pottery vessels (full shape or fragmented), was granted to the Department of Near Eastern Antiquities of the Louvre Museum which partially funded the excavations (Bridey and Cuny, 2019). About 200 items from Sialk (all materials considered) from the Neolithic, Chalcolithic, proto-Elamite and Iron Age periods are now on display in the Louvre (Fig. 1b).

The pottery assemblage associated with the graves of Necropolis B presents various typologies. It includes the particular long-spouted vessels that originally revealed the importance of conducting excavations in the Kashan plain. These ceramic vessels were divided into four categories based on fabric and surface treatment: painted, grey, red, and common buff ware (Ghirshman, 1935) with some variants: those with a flange joining the spout to the body (bridged-spout, e.g. Fig. 1b 1, 2) and those without (e.g. Fig. 1b,3). Spouted shapes are common in Northern Iran during the Iron Age but the painted and refined specimens are only known from Sialk.

In Iron Age Sialk as in other contemporary sites, most of these spouted vessels were found in graves (Danti, 2013). At Sialk long-spouted vessels were exclusively recovered in graves, at the exception of one misfired pottery spout found in a domestic context (Helwing, 2005). It is uncertain whether these ceramics correspond to items the dead and their relatives used during their lifetime or if they were created only for burial and ritual purposes. Their fragile shape and the fine quality of fabric and decoration led some scholars to posit a ritual function limited to the funerals (Fazeli Nashli and Nokandeh,

2019). Indeed, their shape is thought to be suitable for libation, which is a pouring ritual of liquid or grains. Another hypothesis based on the shape, suggests that the long spout was used to dispense alcoholic beverages (Ghirshman, 1939). Although no detailed discussion on the use of such vessels have been published, their function has been a continuous source questioning among experts of the Iranian archaeology. Their absence in domestic contexts is in favor of a function highly related to rituals rather than domestic use. Nonetheless, it remains uncertain if those were used to contain any goods that would have been poured in rituals or remained empty and used only to accompany the dead in the afterlife.

One way to get insight into the long-spouted vessel function is to analyze them for their lipid residue. This technique is widely used on pottery vessels from domestic contexts to reconstruct past diets and, to a lesser extent, on ceramic vessels found in graves to understand funerary foodways (Outram et al., 2011; Šoberl, 2011; Oras et al., 2018; Vykukal et al., 2021) or the function of specialized pottery typologies (Dunne et al., 2019). Recently a study from the Iron Age Ghalekuti Necropolis (Dailaman) in Northern Iran using organic residue analysis provided evidence that all the vessels recovered in the graves were used before deposition for both animal and botanical products with some levels of specialization based on typology (Casanova et al., 2024). Herein, long-spout pottery vessels from Necropolis B at Sialk were studied here for their lipid residue to gain the first insights into their possible function in funerary rituals performed during the Iron Age at Sialk.



**Fig. 1.** a. Map of Iran with the location of Tepe Sialk. b. Examples of long-spout vessels with red-on-cream decorations from Sialk Necropolis B (1 AO 17719; 2 AO 19477; 3 AO 19478) on display at the Louvre Museum. ©2010 Musée du Louvre / Raphaël Chipault. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

**Table 1**  
Description, lipid preservation and source assignment of the three long-spout vessels studied.

Lab code	Louvre inventory number	Grave	Description	Sampling location	Lipid concentration ( $\mu\text{g.g}^{-1}$ )	$\delta^{13}\text{C}_{16:0}$ (‰)	$\delta^{13}\text{C}_{18:0}$ (‰)	$\Delta^{13}\text{C}$ (‰)	Assignment
SIA-0444a	S 1564	87	Refitted, grey clay, no flange	spout	130	-25,5	-27,6	-2,0	Ruminant carcass fats
SIA-0444b	AO 17722	15	Full vessel, cream clay, flange	body	42	-26,8	-28,2	-1,4	idem
SIA-0467	S 1421	112	Refitted, red on cream decoration, flange	spout	88	nd	nd	nd	Animal fats
SIA-0468				spout	24	-24,9	-29,1	-4,2	Ruminant milk fats

## 2. Materials and methods

Long-spout vessels from Sialk Necropolis B, curated in the Louvre Museum, were studied for organic residue analysis (ORA) to gain insight into their function. To address the question of use, especially for pouring liquid, the spout at the junction with the body was defined as a strategic sampling location. Lipid residue analyses are destructive by nature. Only archaeological ceramic vessels with a long-spout that were not on display and were fragmented (all of which were restored) were authorized to be sampled. The very refined and decorated specimens were considered too precious by the curator to undergo destructive analyses. A number of specimens presented ancient restoration work in strategical sampling areas, meaning these were not suited for ORA either leaving only a handful of specimens suitable for ORA. Therefore, the work conducted here is on uniquely suited long-spout specimens from museum collections and the data available from them is extremely valuable. The three sampled vessels are all originating from Necropolis B graves. They correspond to three different ceramic groups (grey, buff and painted red-on-cream wares) (Table 1; Fig. 2.1a, 2.2a, 2.3a). Vessel S 1564 is a monochrome grey spouted vase without flange from grave 87 AO 17722 is a buff undecorated vase with a bridged spout from grave 15, and S 798 is a bridged-spouted red painted buff ware jug with triangle decoration from grave 112. Sampling was performed inside the spout, just after the junction of the spout to the rest of the pottery vessel (Fig. 2.1b, 2.2b, 2.3b). Vessel S 1564, lacking some pieces in the body area, was further sampled from the body for comparison. To comply with conservation constraints, we minimized sampling from 1 to 2 g to ~50 mg of clay. Cleaning the surface of the pottery for exogenous contaminants was performed with a modelling drill, then about 50 mg of clay was drilled out and collected in aluminum foil for analysis. The power was transferred to culture tube alongside 10  $\mu\text{g}$  of internal standard (*n*-tetratriacontane) for extraction.

Lipids were extracted using a methanolic solution of sulphuric acid (10 % v/v, 5 mL, 70 °C, 1 h), followed by a liquid-liquid extraction with *n*-hexane (4 × 3 mL) to obtain the total lipid extract (TLE) (Correa-Ascencio and Evershed, 2014). TLEs were aliquoted, and 20  $\mu\text{L}$  of BSTFA was added to create trimethylsilyl derivatives. An analytical blank followed the entire procedure as control of contamination. This method was selected as it reaches higher lipid extraction yields compared to solvent based extraction, which was necessary considering the small samples taken from these vessels.

Lipids extracts were then injected (1  $\mu\text{L}$ , 280 °C) onto a 7890B Agilent Technology GC (gas chromatography) - FID (flame ionization detector) instrument fitted with an HP5 column (5 % phenyl-dimethylpolysiloxane, 30 m, 3.2 mm id, 0.25  $\mu\text{m}$  film thickness) using He as carrier gas for lipid quantification. The temperature program started at 50 °C for 2 min then increased to 300 °C at a rate of 10 °C/min followed by 10 min holding time. TLEs were also injected (1  $\mu\text{L}$ , 280 °C) in a Trace 1300 GC coupled with an ISQ GD mass spectrometer (MS) fitted with a BP5 X5 column (5 % phenyl-dimethylpolysiloxane, 30 m, 3.2 mm id, 0.25  $\mu\text{m}$  film thickness) using the same temperature program as the GC-FID instrument for compound identification. The MS function in electronic impact operating at 70 eV, and compounds analyzed in total ion current with full scan mode over the range *m/z* 50–650 and SIM mode (including analyses on a more polar column) for the detection of aquatic biomarkers (Cramp and Evershed 2014). TLEs containing fatty acids (FA) as methyl esters derivatives (FAME) were also injected in a 7890B Agilent Technologies GC instrument linked to an Elementar 5 combustion unit and a VisION Isoprime MS for lipid source identification. Measurements of  $\delta^{13}\text{C}$  values of  $\text{C}_{16:0}$  and  $\text{C}_{18:0}$  FAMES in the TLEs were performed in duplicates. The measured  $\delta^{13}\text{C}$  values were calibrated against a homemade standard mix of  $\text{C}_{14:0}$ ,  $\text{C}_{16:0}$  and  $\text{C}_{18:0}$  FAMES which values was first calibrated against the F8–3 international mixture, then corrected for the derivative methyl function following a simple mass balance to obtain the  $\delta^{13}\text{C}$  values of FAs. The average and standard deviation of FAs  $\delta^{13}\text{C}$  are reported as the accepted



**Fig. 2.** 1,2,3 a. Long-spout ceramic vessels from Sialk Necropolis B studied for lipid residue analysis, S 1564, AO 17722 and S 1421, respectively (©2011 Musée du Louvre/Antiquités Orientales). b. Sampling zones defined by the red circle for the same ceramic vessels. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

value. Column and temperature programs are similar to the GC-FID instrument and combustion reactor operated at 450 °C.

### 3. Results

The TLEs of the three vessels present lipid concentrations exceeding 5 µg per gram of clay (Table 1) with a maximum of 130 µg.g<sup>-1</sup>. While this is a relatively low lipid concentration it falls in the range of lipid concentrations found in ceramic vessels from other settlements in the Iranian plateau (Casanova et al., 2023). For TLEs of vessel S 1564, the concentration was higher in the spout with 130 µg.g<sup>-1</sup> than in the body with 42 µg.g<sup>-1</sup>. Inhomogeneous partitioning of lipids within the pottery vessel is common and varies with vessel typology and use (Charters et al., 1993; Šoberl et al., 2014; Casanova et al., 2020; Drieu et al., 2022).

Palmitic (C<sub>16:0</sub>) and stearic (C<sub>18:0</sub>) acids characteristic of degraded animal fats dominate the TLEs (Fig. 3). In the TLEs of vessel S 1564 (both body and neck samples) and S 1421, the fatty acids (FAs) were fully methylated as fatty acids methyl esters upon simultaneous extraction and methylation (Correa-Ascencio and Evershed, 2014). The TLE of vessel AO 17722 showed unmethylated fatty acid, with TMS derivatives created during the derivatization step with BSTFA. This suggests the reaction did not go to completion and a potential explanation could be that the concentration of the acid used in the extraction may have been diminished due to the presence of carbonates in the clay. Exogenous contamination introduced during the procedure was excluded here based on the quality of analytical blanks. No further GC-C-IRMS analysis to determine the source of animal fats could be performed on this particular vessel, nor was the extraction repeated as all the sampled clay was used. Vessel S 1421 TLE is dominated by palmitic and stearic acids characteristic of animal fats. In addition, a plasticizer, and a small bump around 20 min is a cluster of even and odd long chain *n*-alkane suggesting derived petroleum products which would likely originate from the proteinaceous glue fabrication process used in the restoration of the fragmented spout (Fig. 2.3b; Brandis, 1990). The vessels are directly stored on shelves, without use of plastic bags or other containers that

could have contributed to the contamination source. Even though, care was taken to sample away from restored areas, trace amounts of glues could have been deposited elsewhere on the vessels during restoration work that was likely carried out shortly after excavations, or diffused by capillarity from the glues area. No other compounds namely long chain fatty acids, long chain ketones, dihydroxy fatty acids, *ω*-(*o*-alkylphenyl) alkanolic acids or isoprenoid acids were detected in the TLEs.

Both samples of vessels S 1564, taken from the body and the spout, presented lipid distribution with δ<sup>13</sup>C<sub>16:0</sub> and δ<sup>13</sup>C<sub>18:0</sub> values characteristic of ruminant adipose fats (0.3 ‰ > Δ<sup>13</sup>C > -3.1 ‰; Table 1; Fig. 4). This demonstrates homogeneity in the contents despite the inhomogeneous lipid partitioning in the vessel. TLE of vessel S 1421 display δ<sup>13</sup>C<sub>16:0</sub> and δ<sup>13</sup>C<sub>18:0</sub> values characteristic of ruminant dairy fats with Δ<sup>13</sup>C (=δ<sup>13</sup>C<sub>18:0</sub> - δ<sup>13</sup>C<sub>16:0</sub>) < -3.1 ‰ (Table 1; Fig. 4), and fits perfectly in the range of the modern local reference values for dairy fats (Casanova et al., 2023).

### 4. Discussion

The detection of lipids in the spout suggests that these vessels were used and had a function before its deposition in the grave, and did not just serve as empty accompanying objects for the dead. Considering the pottery typologies of the studied ceramic, contents would have been poured through the spout meaning the commodities identified should have clearly been under a liquid state. The same conclusions could be evidenced for the Iron Age Ghalekuti Necropolis that studied wide range of vessels with spouts, tubular spouts, narrow neck jars that only allow pouring of liquid commodities (Casanova et al., 2024).

Lipid residues identified in vessel S 1421 likely originate from milk, as other dairy products with a solid state (e.g., butter, cream, yoghurt, etc.) would not be suitable to be poured through this type of pottery. Milk would have been obtained from domesticated herds of caprine (*Ovis aries* and *Capra hircus*) that dominate the faunal remains (between 60 and 70 %), or even domesticated herds of cattle (*Bos taurus*) that were also present in a lower number (10 to 15 %), (Mashkour, 2004; Mashkour and Bon, 2019). Ruminant milk could have been processed in the

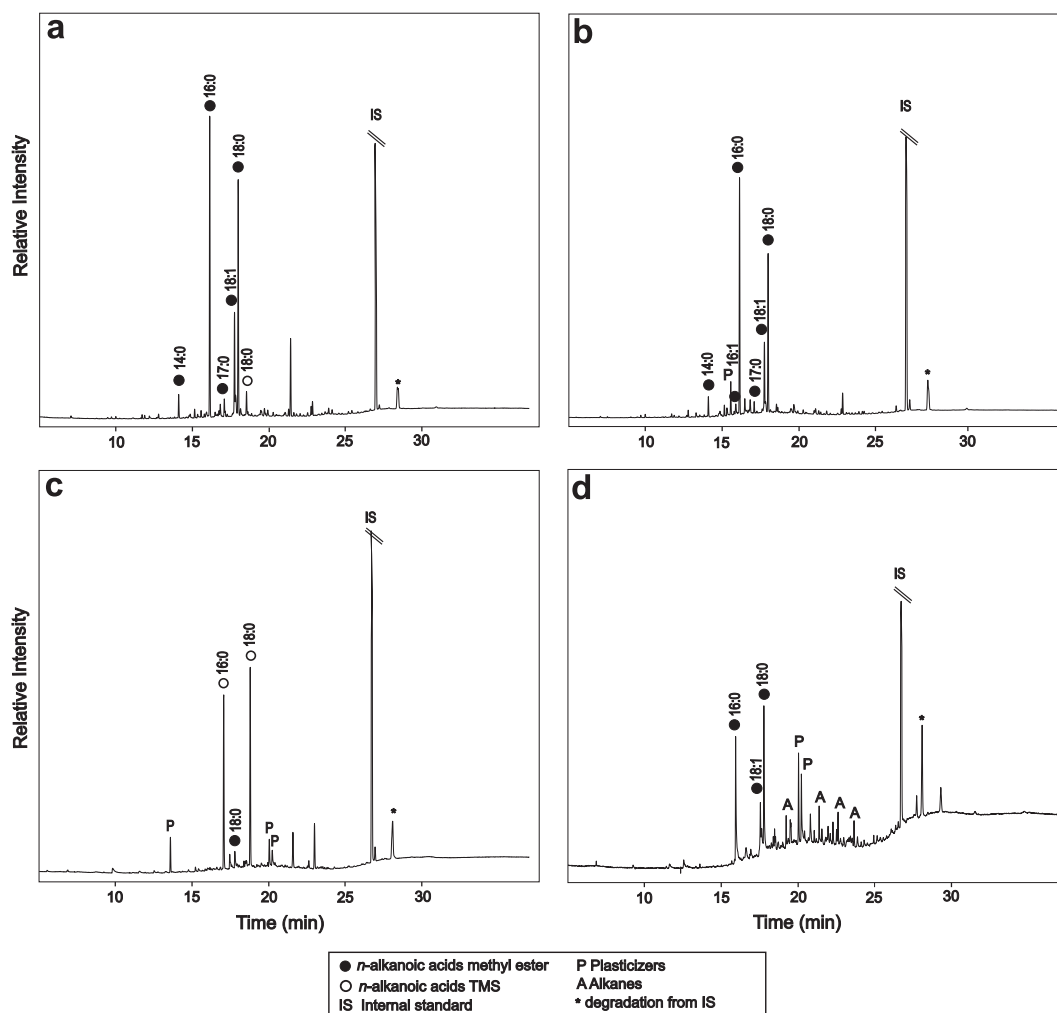


Fig. 3. Partial chromatogram extracted from vessels a. S 1564 spout, b. S 1564 body, c. AO 17722 spout and d. S 1421 spout. The numbers “x:y” correspond to the carbon chain length and number of unsaturation, respectively. Parallel bars mean peaks of the IS is not display to its full relative height.

vessels as fresh or fermented but these sub-products cannot be distinguished based on their lipid distribution (Rao and Reddy, 1984). We do not exclude the milk could have been mixed with other commodities but the lack of other compounds with archaeological origin identified in the TLE does not allow to further comment on this possibility.

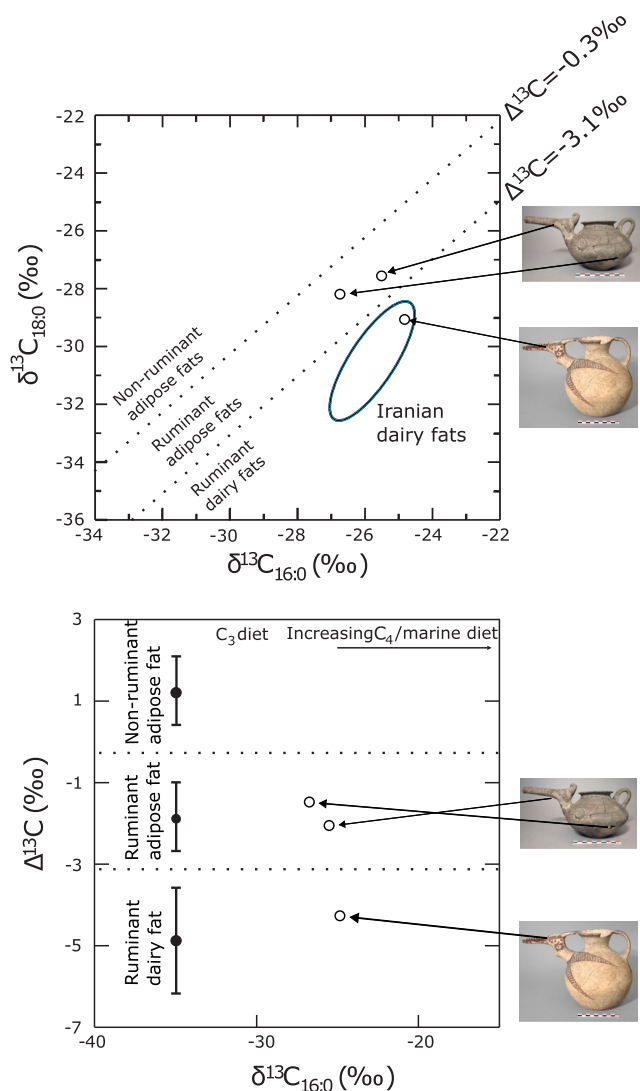
Lipid residues in vessel S 1564 originate from ruminant carcass fats products which should be in a liquid state to fit with pottery typologies. It should be noted that animal fats are solid at ambient temperatures compared to oils (Woodgate and van der Veen, 2014). Therefore, one hypothesis is that the animal fats would have been heated inside the vessel to reach a liquid state. Animal fats, like tallow<sup>1</sup> have a range of melting point based on their precise lipid composition but these would generally be above 35 °C (Woodgate and van der Veen, 2014). However, no evidence suggesting heating on an open fire, such as burn marks on the exterior of the ceramic or the detection of biomarkers forming at high temperatures (e.g. long chain ketones) (Evershed et al., 1995), has been identified in the TLEs. It should be noted no burn marks have been recognized on the overall long-spout ceramic collection in the Louvre. Another hypothesis if the contents were tallow-like carcass fats, these could have been heated in other containers then transferred, even filtered, directly to the long-spout ceramics to have a liquid to use in

<sup>1</sup> Animals species identified at the site being majority ruminant species (caprine 60–10 %, cattle 10–15 %, gazelles 3–4 %) (Mashkour, 2004) the term tallow is more accurate here rather than lard that is used for porcine products.

funerary rituals.

Another possibility would be that lipids in vessel S 1564 originated from a blood-based product of ruminant animals. Blood lipids are dominated by fatty acids, triglycerides (broken down to fatty acids during extraction) and cholesterol, with a relative proportion of about 80 % and 10 %, respectively (Bauchart, 1993; Krebs, 1950). To corroborate the hypothesis of ruminant animal blood being processed in the vessel GCMS spectra were further investigated for the presence of cholesterol. The latter was detected in trace amounts. The survival of cholesterol in low concentration compared to fatty acids could relate to their uneven natural abundances but also some preferential degradation of cholesterol. While cholesterol is resistant to microbial degradation it degrades induced by the presence of clay and fatty acids when heated to a relatively low temperature (under 100 °C) (Hammann et al., 2018). Considering extraction of lipids is performed at 70 °C during this laboratory experiment, any cholesterol surviving in the pottery vessel could have degraded at this stage. It should be noted that blood coagulates to open air in a few minutes (Lee and White, 1913). This means long-spout ceramics would have been used rather rapidly after collecting the animal blood. Other carcass and blood based liquid products such as meat juice or broth, is a possibility. The absence of compounds in the TLEs other than palmitic and stearic acids does not allow to further comment on potential mixtures.

The presence of a spout means that the pottery typology is only suitable for pouring a liquid. This provides an argument in favor of blood, meat or tallow-based mixtures for the residue identified as



**Fig. 4.** Stable carbon  $\delta^{13}\text{C}_{16:0}$  values plotted against the a.  $\delta^{13}\text{C}_{18:0}$  and b.  $\Delta^{13}\text{C}$  ( $\delta^{13}\text{C}_{18:0} - \delta^{13}\text{C}_{16:0}$ ) for the studied ceramic vessels. Ranges denotes modern reference values (Casanova et al., 2023; Copley et al., 2003).

ruminant adipose fats. Without further evidence it is difficult to firmly identify one over the other. If it was indeed ruminant blood fats, this would reinforce the hypothesis that long-spout ceramic vessels were purposely made for funerary rituals rather than domestic use, as animal blood would have been uncommon for daily consumption.

Interestingly, two different animal products have been identified in the long-spout vessels suggesting they were not dedicated to only one commodity or purpose. The animal products identified in the long-spout vessels are available by milking, and perhaps bloodletting. It means animals were not necessarily slaughtered for funerary rituals. One can wonder if the animal products used for funerary rituals originated only from one ruminant animal species or several. It is worth noting that while lipids of animal origin were recovered in funerary vessels, animal offerings (bones) are virtually absent from the graves (no mention of it in excavations reports). Insights from slaughtering patterns and butchery marks from faunal remains on the various ruminant species at the domestic contexts of the site show the presence of consumption marks on all the ruminant species. Therefore, we cannot discriminate with certainty from which ruminant specie (caprine, cattle, gazelle) the products found in funerary vessels originate. All ruminant species have the potential to have played a role in the funerary ceramics. Only the milk

products, that is only available from domesticated animals, could be narrowed down to the caprine, or cattle.

Long-spout vessels are sometimes hypothesized to have a relation with alcoholic beverages without necessarily supporting evidence other than pottery typologies (Ghirshman, 1938, 1939). Our results, do not provide either evidence suggesting the use of alcoholic beverages. The lipids distributions show a dominance of animal fats with no other abundant compound that would suggest their mixing with other commodities. In the case of the vessel were ruminant milk was identified, if it was fermented it could perhaps have been used in the preparation of alcoholic beverage comparable to kumis a traditional milk alcohol from central Asia (Akuzawa et al., 2011), and this hypothesis cannot be corroborated based on the molecular data. Our results however are in favor of the use of long-spout ceramics, not known from any domestic contexts, in pouring rituals involving liquids of various origins from ruminant animals.

## 5. Conclusion

The function of long-spout vessels was investigated through their past content residues. Sampling of vessels directly in the spout support these vessels were used to pour liquid products from domesticated ruminant animals, in one case milk and the second case likely a blood related or tallow related product. This suggests long-spout vessels would indeed have a role in rituals such as libation as such vessels are absent from the domestic contexts. With this study the distinctive long-spout ceramics from the Sialk B necropolis started unravelling their purpose in Iron Age funerary rituals in the Iranian plateau.

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## CRedit authorship contribution statement

**Emmanuelle Casanova:** Writing – original draft, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Julien Cuny:** Writing – review & editing, Investigation, Data curation. **Antoine Zazzo:** Writing – review & editing, Validation, Supervision. **Marjan Mashkour:** Writing – review & editing, Supervision, Investigation, Conceptualization.

## Declaration of competing interest

The authors declare no conflict of interest.

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