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► **To cite this version:**

Alessandra Pecci, Ruth Smadar Gabrieli, Fernanda Inserra, Miguel Cau-Ontiveros, S.Y. Waksman. Preliminary results of the organic residue analysis of 13th century cooking wares from a household in Frankish Paphos (Cyprus). *STAR: Science & Technology of Archaeological Research*, 2015, 1 (2), pp.99-105. 10.1080/20548923.2016.1183960 . hal-01381374

HAL Id: hal-01381374

<https://hal.science/hal-01381374v1>

Submitted on 14 Oct 2016

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To cite this article: Alessandra Pecci, Ruth Smadar Gabrieli, Fernanda Inserra, Miguel Angel Cau & Sylvie Yona Waksman (2016): Preliminary results of the organic residue analysis of 13th century cooking wares from a household in Frankish Paphos (Cyprus), STAR: Science & Technology of Archaeological Research, DOI: [10.1080/20548923.2016.1183960](https://doi.org/10.1080/20548923.2016.1183960)

To link to this article: <http://dx.doi.org/10.1080/20548923.2016.1183960>



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Published online: 01 Jun 2016.



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Preliminary results of the organic residue analysis of 13th century cooking wares from a household in Frankish Paphos (Cyprus)

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Abstract Organic residue analysis was carried out on kitchen wares from a Medieval household at Paphos (Cyprus) in the framework of the POMEDOR Project, which aims to gain insight into food practices in the Eastern Mediterranean during the Medieval period. The samples were selected from a household assemblage which included nearly two hundred table, cooking and storage vessels, and provided a rare insight into everyday life in Frankish-period Paphos. Both glazed and unglazed vessels were chosen for this first step towards the study of foodways at the site: 4 glazed vessels (3 pans/baking dishes and 1 cooking pot) and 7 unglazed vessels (3 cooking pots, 1 jug and 3 pans/baking dishes). Samples were analysed using gas chromatography – mass spectrometry, following different extraction methods to identify the residues preserved. The preliminary study confirmed that glazed ceramics absorb residues. Animal products were identified in the unglazed pots and in glazed pans/baking dishes. Only one unglazed pan shows residues of possible wine or its derivatives, that may have been used to flavor the foodstuff cooked.

Statement of significance Ceramic vessels can be studied in order to recover information about their original content through specific chemical analyses.

This study is part of the first step of a broader project, which aims to gain insights into food practices in the Eastern Mediterranean during the Medieval period. The project incorporates historical, archaeological and archaeometric research. The study provides information on the use of a range of vessels, such as pots, pans/baking dishes, and jugs, as well as on the food cooked in them.

An important aspect of this study is that it confirms that glazed ceramics absorb and preserve organic residues, yielding information about the original content of the vessels.

Keywords Medieval Cyprus; Foodways; Glazed ceramics; Cooking wares; Organic residues; Gas chromatography-mass spectrometry

Received 30 June 2015; **accepted** 10 March 2016

1. Introduction


The analysis of organic residues preserved in archaeological ceramics started in the 1970s, when Condamin and Formenti published their first articles on amphorae analysis (Condamin et al. 1976; Condamin and Formenti 1976). Since then, this field of study has progressed, and in recent years residue analysis of ceramics has become widespread and is being

carried out by many laboratories (Barnard and Eerkens 2007; Colombini and Modugno 2009; Evershed 1993, 2008; Garnier 2007; Guash Jane et al. 2004; Kimpe et al. 2004; Nigra et al. 2015; Pecci 2009, 2014; Regert 2011; Romanus et al. 2009). However, the common opinion has been that obtaining information about the organic residues preserved in glazed ceramics and thereby about their content was

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10.1080/20548923.2016.1183960

STAR201620548923.2016.1183960

probably not possible. This led to a lack of studies of glazed ceramic vessels, which are an important part of the cooking wares in Medieval and post-Medieval ceramic assemblages.

Recent research has, however, shown that the analysis of glazed ceramics can yield good results (Pecci et al. 2012; *in press*) and encouraged us to carry out analysis of samples from glazed and unglazed cooking vessels found in Paphos, Cyprus. The study was undertaken within the framework of the POMEDOR Project (People, Pottery and Food in the Medieval Eastern Mediterranean - www.pomedor.mom.fr). The aim of the POMEDOR project is to gain insight into food practices in the Eastern Mediterranean during the Medieval period. The objective of this paper is to provide a first insight into the use of some ceramic vessels through the identification of the organic residues that were preserved in them.

The vessels in this study came from a Roman tomb that was excavated by Dr. Raptou (Department of Antiquities of Cyprus) at Odos Ikarou (Icarus Street, Paphos, Cyprus) in 2001. Raptou suggests a probable date for the tomb at the end of the Hellenistic-beginning of the Roman periods (Raptou 2006:328). The tomb was re-used as a household during the 13th century AD (Raptou 2006). A large proportion of the pottery in the tomb proved to belong to its re-use in the Medieval period. The assemblage included the complete content of a household, with nearly two hundred table, cooking and storage vessels, and provided a rare insight into everyday life in Frankish Paphos. The table wares date the assemblage to the second half of the 13th century. In addition to the local Paphos-Lemba wares they include imports from the Levantine coast, Italy and Spain (Cook, pers. comm.).

The coarse wares in the tomb were mostly hand-made and unglazed, manufactured in a dark, reddish-brown fabric local to the Troodos area according to petrographic analysis (Gordon-Smith in Gabrieli 2006). As a comprehensive content of a household, the material from Odos Ikarou has a potential to contribute to a study of food and foodways of Medieval Cyprus, and of production of cooking and storage vessels. The unglazed cooking vessels are mostly pots, in three approximately standard sizes (small, medium, large). Other types include jugs with a pinched spout and one handle, sometimes bearing soot marks that indicate they were used on the fire; two-handled jars that were probably used as liquid containers; and very few pans/baking dishes (Gabrieli 2008) (Minimum Number of Vessels (Min. No.) = 45 pots, 9 jugs, 5 pans/baking dishes, 3 jars; the Minimum number of Vessels was calculated according to Orton 1989: 94). We use the dual term pans/baking-dishes because this shallow, open shape is usually referred to as pan, but the soot marks on these vessels, and the absence of handles on the unglazed ones, as well as the small handles that hug the wall of most of the glazed ones, suggest that they were mainly

used for baking various types of food in the oven, rather than over direct fire.

The glazed vessels are pots and pans/baking dishes of Levantine types, well known from the Crusader principalities (Avisar and Stern 2005:91, Type II.2.1.2; 96–97, Type II.2.3.2), and a large centre of their production was identified in Beirut (Waksman 2002; Waksman et al. 2008). The open vessels are predominant in the Odos Ikarou glazed ware assemblage (Min. No. 11 pans/baking dishes; 3 pots). For a comprehensive study of the assemblage, see Gabrieli (2008). The origin of these wares is currently under investigation by X-ray fluorescence and petrography in Lyon (Gabrieli et al., forthcoming).

2. Materials and methods

A total of 11 samples were selected for this paper (Table 1): 4 glazed vessels (3 pans/baking dishes and 1 pot) and 7 unglazed vessels (3 pots, 1 jug and 3 pans/baking dishes). The samples were analysed using gas chromatography – mass spectrometry to identify the residues preserved.

The ceramics were carefully cleaned with a scalpel and the glaze was removed before subsampling. Each sample was pulverized and subjected to three extractions: a. Total lipid extraction, following the protocol proposed by Mottram et al. (1999) was carried out. 2 g of ground sample was extracted twice with CHCl₃/MeOH (2:1 v/v, 3 mL) in a sonication bath for 40 min at 70°C. The liquid fraction was recovered after centrifugation and dried using a gentle stream of nitrogen; b) Hydrolysis of the total lipid extract: 3 ml of KOH in MeOH (1 M) were added to half of the total lipid extract and left overnight. 3 ml of ultrapure water were added and solution was acidified with HCl. Finally 3 ml of CHCl₃ were added and mixed with a vortex and the supernatant evaporated (Pecci, Cau and Garnier, 2013a). c) Extraction for the identification of wine markers was carried out on 0.5 g samples following the protocol proposed by Pecci et al. (2013b): 0.5 g of sample was extracted with 3 ml of KOH in H₂O (1M) in a sonicated bath at 70 °C for 90 min. After acidification 3 ml of ethyl acetate were added to the extract and mixed with a vortex. After centrifugation the supernatant was evaporated with a gentle stream of nitrogen (Pecci et al., 2013b). 5 µl of dotriacontane (1 mg/mL) was added as internal standard. The solvents were of gas chromatography analysis quality and of organic trace analysis quality.

All the extracts were derivatized by adding 25 µL of *N, O*-bis(trimethylsilyl) trifluoroacetamide (BSTFA, Sigma-Aldrich) and heated to 70 °C for 1 hour. After cooling, 75 µL of hexane were added. 1 µL of the obtained extract was injected for analysis by GC-MS.

All samples were analysed using a Thermo Scientific TraceGC ultra chromatograph equipped with a column TRB-5MS Teknokroma 5% phenyl 95% methyl polysiloxane, 30 m long, 0.25 mm internal diameter, 0.25 µm film thick and coupled with a

Table 1 Samples analysed.

archaeological id.	Lyon lab. id.	Barcelona lab. id.	surface	Form	part sampled
OI98	BZY614	213	glazed	Pan/baking dish	bottom
OI99	BZY615	214	glazed	Pan/baking dish	bottom
OI100	BZY617	215	glazed	Pan/baking dish	bottom
OI107	BZY620	216	glazed	Pot	bottom
OI90	BZY622	217	unglazed	Pan/baking dish	bottom connected with wall
OI91	BZY623	218	unglazed	Pan/baking dish	bottom connected with wall
OI92	BZY624	219	unglazed	Pan/baking dish	bottom connected with wall
OI151	BZY628	220	unglazed	Jug	Wall
OI172	BZY634	221	unglazed	pot-small	Wall
OI143	BZY630	222	unglazed	pot-large	Wall
OI121	BZY631	223	unglazed	pot-medium	Wall

Table 2. Synthesis of the results obtained with organic residues analyses

archaeological id.	Lyon lab. id.	Barcelona lab. id.	surface	Form	residues identified
OI98	BZY614	213	glazed	Pan/baking dish	animal products
OI99	BZY615	214	glazed	Pan/baking dish	animal + vegetal products
OI100	BZY617	215	glazed	Pan/baking dish	animal + vegetal products
OI107	BZY620	216	glazed	Pot	animal products
OI90	BZY622	217	unglazed	Pan/baking dish	
OI91	BZY623	218	unglazed	Pan/baking dish	
OI92	BZY624	219	unglazed	Pan/baking dish	Wine
OI151	BZY628	220	unglazed	Jug	animal products
OI172	BZY634	221	unglazed	Pot-small	animal products
OI143	BZY630	222	unglazed	Pot-large	animal products
OI121	BZY631	223	unglazed	Pot-medium	animal products

Thermo Scientific ITQ900 mass spectrometer operated in the electron ionization mode (70 eV). A liner LNR TQ CE 3 mm ID Single tap (105 mm long) splitless type was used. The injection temperature was 270 °C and the total flow of the injector was 30 ml/min and the carrier gas flow (helium) was 1.00 ml/min. The purge valve time was 1.00 min of splitless time.

The mass range was scanned in the range of 40–650 m/z. The GC oven temperature was held for all samples at 50 °C for 1 min, then increased 5 °C/min up to 300 °C and held isothermally for 10 min. The analyses were carried out at the Centres Científics i Tecnològics de la Universitat de Barcelona. Peak assignment

was made by comparison with the NIST library and standards.

3. Results

The results of the analyses confirm that in spite of their supposedly waterproof coating, glazed ceramics absorb residues, as was previously suggested (Pecci et al. 2012, *in press*). This is an important issue in Medieval archaeology, as Medieval ceramics were often coated with glaze. The absorption could be attributed to cracks that developed due to a poor match between

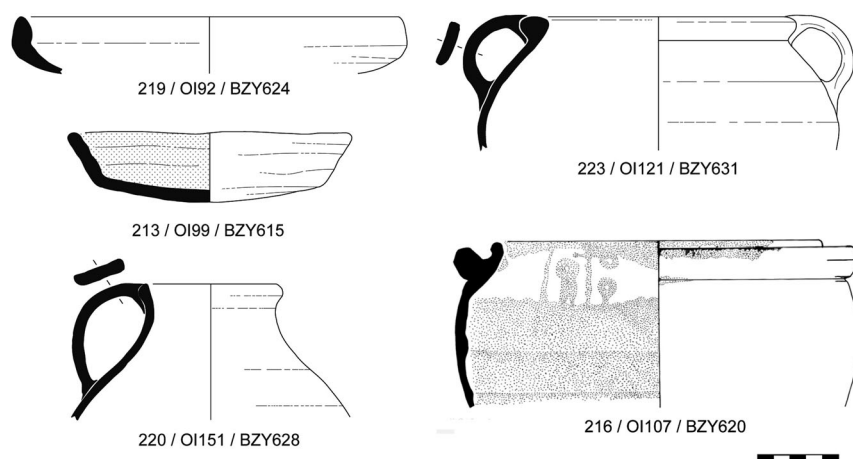


Figure 1 Representative examples of the different types of cooking wares analysed: 219- OI92: unglazed pan; 213- OI99: glazed pan; 220- OI151: unglazed jug; 223-OI121: unglazed pot; 216- OI107: glazed pot.

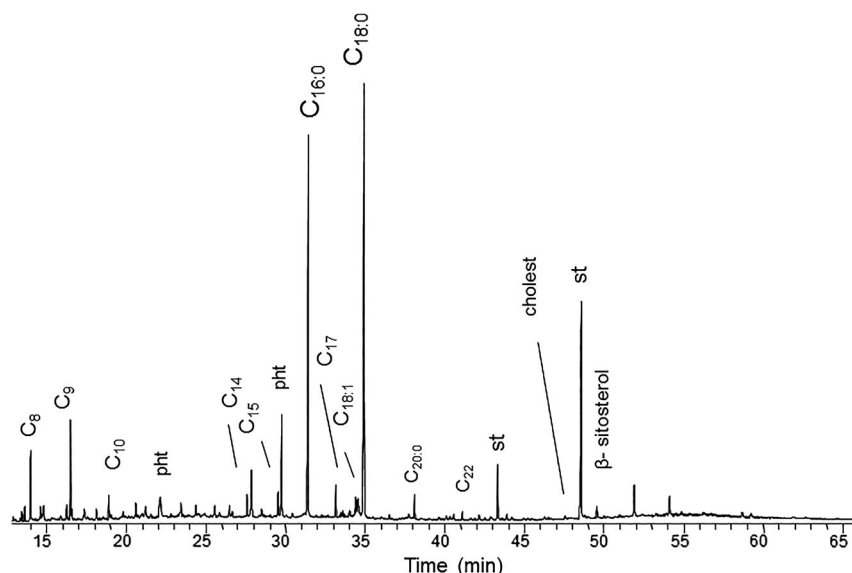


Figure 2 Mass chromatogram of the total lipid extract of sample 214 (OI99).

the thermal expansion coefficients of the glaze and the body (or the slip), or to use wear.

In the glazed vessels the presence of cholesterol and abundant stearic acid ($C_{18:0}$) (which could be attributed to the breaking of the TAGs and DAGs with stearic acid) in all the samples indicates that animal products came in contact with all the vessels. Residues are present in the pans/baking dishes (samples 213/OI98, 214/OI99 and 215/OI100), and in the pot (sample 216/OI107). This is probably related to the cooking of animal meat, but could also be due to animal fats being used as condiments.

In glazed pans/baking dishes 214 and 215 (OI99 and OI100) there are plant oils, indicated by the presence of β -sitosterol in the extraction obtained by procedure (c), together with abundant azelaic acid (which is the most abundant among the dicarboxylic acids) in

sample 214; and abundant C_9 (which is the most abundant among the short chain fatty acids) in the total lipid extract of sample 215 and of azelaic acid in extraction (c). This distribution of fatty acids is usually found when plant oils, are cooked (Pecci 2005; Pecci et al. *in press*), and suggests that plant oils, possibly olive oil, were used for cooking in the pans /baking dishes.

As for the unglazed pottery, residues are relatively scarce in the three pans/baking dishes that were analysed (samples 217/OI90, 218/OI91, 219/OI92). This could be attributed to these vessels having been used to bake dishes that left no detectable residues, such as legumes or grains, or for parching (e.g. dry-roasting of peas, beans etc.). In sample 219 (extract c) tartaric acid was present together with succinic, malic and isocitric acids. Tartaric acid is usually

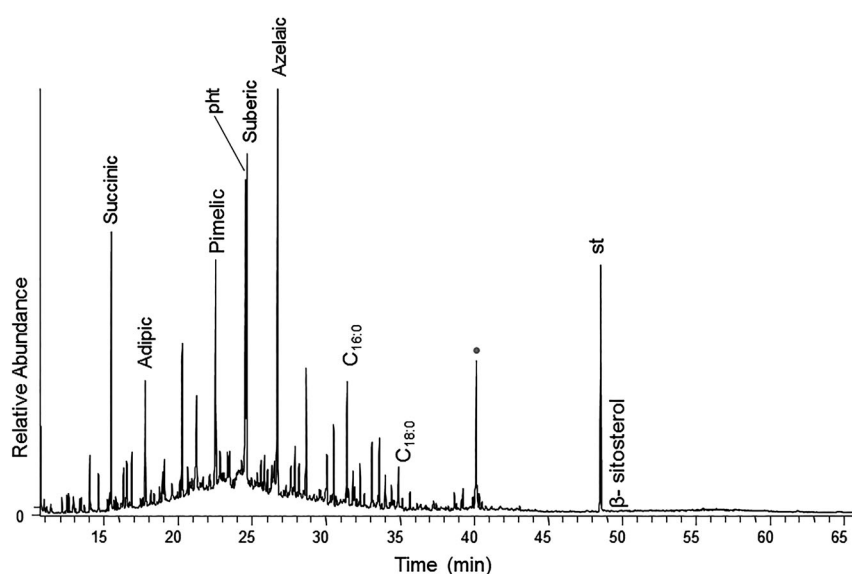


Figure 3 Mass chromatogram of the extract (c) of sample 215 (OI100), where β sitosterol is present and azelaic acid is the highest among dicarboxylic acids. The spot indicates a compound that is often present when experimental cooking or storing of oil is carried out.

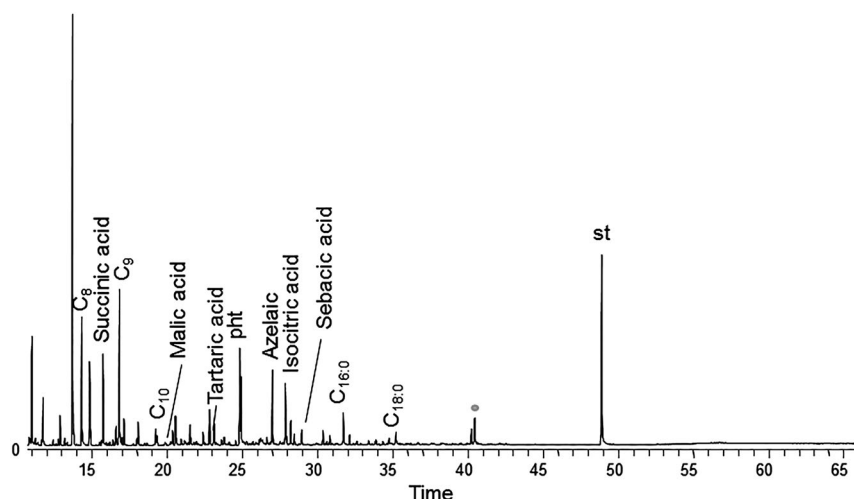


Figure 4 Mass chromatogram of the extract (c) of sample 219 (OI121), where tartaric acid, together with succinic, malic and isocitric acids (markers of wine or its derivatives) are present.

considered to be the marker of wine (Guash Jané et al. 2004; Garnier 2007; McGovern 2003; Pecci et al. 2013a).

Although other fruits contain this acid (Barnard et al. 2011), due to the diffusion of wine, vinegar and wine sauces in the Medieval diet, it is possible that such substances were used as condiment for the food that was cooked in the pan. We can also not exclude the possibility that a sauce made of fermented tamarind, which is also rich in tartaric acid (Barnard et al. 2011) and was traded in the Eastern Mediterranean (e.g. Goldberg 2012), could be used as a condiment. However, experiments should be carried out to verify this hypothesis.

The soot deposit on this pan is considerably denser than on the other unglazed ones, suggesting a different mode of cooking for this vessel.

Unglazed pots, on the other hand, show relatively abundant residues, regardless of their dimensions (small, medium or large). Here, the presence of cholesterol in all the samples indicates that animal-origin products were cooked in the vessels, probably as broths and soups. In these broths and soups also other products, such as grains that cannot be identified with the analyses performed, were probably cooked.

In sample 223 (OI121) there are traces of β -sitosol in the (c) extraction, together with abundant C_9 which is the most abundant among the short chain fatty acids, as well as azelaic acid, which is the most abundant among the dicarboxylic acids, a combination that suggests the cooking of plant oils, possibly olive oil, in the pot. Also in samples 222 (OI143) and 221 (OI172) there are abundant C_9 and azelaic acids.

No traces of wine residues are present in the jug (sample 220/OI151), as no tartaric acid is present in the extraction (c) of the sample. Residues are relatively scarce in this ceramic vessel, however there are traces of cholesterol, which taken in conjunction with the soot marks on its walls, suggest that it was used for heating animal-origin broths.

4. Conclusions

The preliminary study that was carried out on cooking wares from Paphos confirmed that glazed ceramics absorb residues and can be studied in order to recover information of their content.

In general, animal fats were identified in glazed pans/baking dishes. Residues in the glazed pans/baking dishes seem to be more abundant than in the unglazed ones. Residues can vary within the same vessel depending on the part sampled (Charters et al. 1993), but our samples were all taken from the bottom of the vessels, and we can therefore suggest that different dishes or at least a different mode of cooking could be related to the two types of vessels. Just one unglazed pan shows residues of possible wine from grapes or its derivatives, or eventually from another fruit such as tamarind, which was possibly used to flavor the foodstuff cooked. Similar traces have been previously identified in Medieval pots from Tuscany (in Piombino and Florence; Pecci 2015; Salvini et al. 2008), but never in pans.

Unglazed pots were also rich in residues, mainly of animal origin, suggesting the cooking of broths and soups. Plant oils were also part of the diet and were in this case probably used as a condiment or for cooking. However, we should remember that probably also other foods such as grains were cooked in the pots that cannot be identified with the analyses performed.

No evident differences could be identified in the residues from the three unglazed pots with the different dimensions (small/medium/large), therefore the choice of the pot to be used is probably related only to the difference in their dimensions; using similar ingredients to cook different quantities of food may have been related to the number of people eating. It is also possible that the choice of the pot to be used was related to the type of food — as for example main course (e.g. broth), as opposed to side dishes accompanying staples.

The unglazed jug contained residues, and was possibly used to heat broths or soups. The same pattern has been observed in unglazed jugs previously analysed from Late Antique and Medieval sites in the Mediterranean area (Pecci 2009, 2015; Inserra et al. 2015).

Future studies should focus on narrowing down the identification of the animals that were cooked, using isotopic analysis and on broadening the samples to be studied in order to obtain a better understanding of diet and foodways in Medieval Paphos and more in general the Eastern Mediterranean.

Acknowledgements

This study was funded by the French National Research Agency (ANR) through the POMEDOR project (People, Pottery and Food in the Medieval Eastern Mediterranean, www.pomedor.mom.fr), directed by Waksman, and we acknowledge the support of the ANR under reference ANR-12-CULT-0008.

It is also part of the activities of the ERAAUB, Consolidated Group (2014 SGR 845) funded by the Comissionat per a Universitats i Recerca del DIUE of the Generalitat de Catalunya and of the Ramon y Cajal contract (RYC 2013–13369) founded by the Ministerio de Economía y Competitividad of A. Pecci. The samples were analysed at the Centres Científics i Tecnològics of the Universitat de Barcelona (CCiTUB).

We are grateful to the Department of Antiquities of Cyprus, and in particular to Dr Eustathios Raptou, for giving us access to the material and to the team of the Paphos Museum for facilitating our work; to Holly Cook, from the Paphos Theatre Excavation Project of the University of Sydney, for the information regarding the table wares from the assemblage; to Céline Brun and Jacques Burlot (CNRS Lyon) and to the Paphos Theatre Excavation project of the University of Sydney for the drawings of the samples.

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