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WINE AND OLIVE OIL FROM AN EARLY MINOAN I HILLTOP FORT

Koh, A.J. and Betancourt, P.P.

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Corresponding author: Ppbcourt1@aol.com

ABSTRACT

Aphrodite's Kephali is a small hilltop site in Eastern Crete. Its pottery indicates that it was inhabited during Early Minoan I (EM I), ca. 3200–2700 B.C. The fortified site has a considerable amount of storage, including nine pithoi. The analysis by gas chromatography of sherds from the site indicates that vessels contained olive oil and wine. These results are the earliest documented presence of both of these commodities in Crete. The evidence is important because the domestication of the olive and grape at this early period in Crete has been previously questioned.

KEYWORDS: Early Minoan I, EM I, olive oil, olives, grapes, wine, fortifications, Aphrodite's Kephali, Isthmus of Ierapetra, pithos, pithoi, Hagios Onouphrios, Minoan pottery, gas chromatography, ARCHEM

INTRODUCTION

Aphrodite's Kephali is a small hilltop site in eastern Crete. It is located on the Isthmus of Ierapetra, 7 km north of the southern coast and 5 km south of the northern coast. The location was excavated in 1997 under the direction of Theodore Eliopoulos (Eliopoulos 1998, 312) and in 2003 under the direction of Stavroula Apostolakou. Cleaning and conservation work was conducted on the hilltop in 2006 and 2007 under the direction of Stavroula Apostolakou with the assistance of Philip Betancourt (Betancourt 2008a; 2008b)[1].

The site contains a small building, fragments of other walls that seem to form casements for a fortification enclosing the upper part of the hill, a small open courtyard with an area burned red from fires, and an entrance for a natural cave. The pottery, which is all from EM I (ca. 3200–2700 B.C.), includes vases in the Hagios Onouphrios Style, heavily burnished vessels made of calcite-tempered fabric, and several other classes of pottery. The presence of ceramics made in several different fabrics suggests that the vessels originate from various sources, all of them within Crete. Among the vases are many sherds from pithoi including enough fragments to restore one example.

Organic residues were extracted from several fragments in order to provide information on the commodities contained by the clay vessels. The program of extraction and analysis was accomplished by a collaboration between the following institutions: Archaeochemistry Research in the Eastern Mediterranean (ARCHEM); the 24th Ephorate of Classical and Prehistoric Archaeology; the Foundation for Research and Technology-Hellas (FORTH); the Department of Classics and Archaeology Program at Tufts University, the INSTAP Study Center for East Crete (INSTAP-SCEC) in Pacheia Ammos, Crete; the Museum of Cretan Ethnology Research Centre in Vori, Crete; and the Department of Art History, Temple University, Philadelphia, Pennsylvania. This nondestructive field technique can provide important information to supplement the data recovered by other means[2]. Past studies have helped illuminate the function of both objects and the space in which they were found and the nature of production and consumption (Koh 2008b; Brogan and Koh 2008); in the future, studies will hopefully reveal intricacies related to overarching issues such as economy and trade.

METHODOLOGY

Sherds of pottery for analysis were collected in 2006 under the supervision of Philip Betancourt during the cleaning of the site under the direction of Stavroula Apostolakou (Fig. 1).

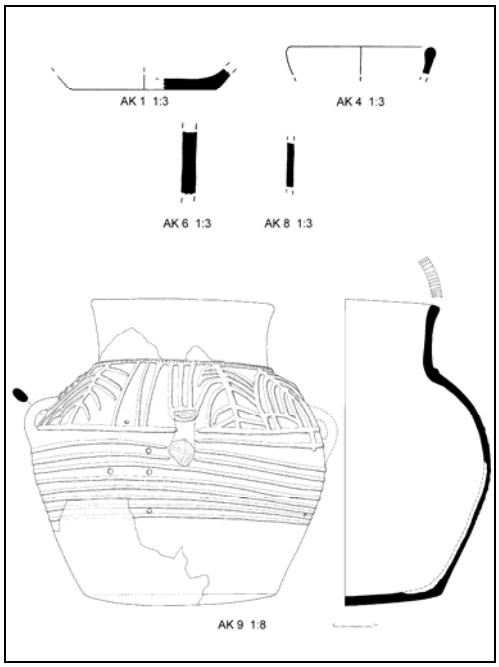

Figure 1. Profile drawings of pottery from Aphrodite's Kephali analyzed for organic residues. Scale as marked.

Sherds were lifted from the soil without being directly touched by anyone, and they were immediately wrapped in aluminum foil in the field and transported to the INSTAP Study Center for extraction of the sample. This swift and careful isolation of sherds likely contributed to the excellent results produced for this present study, which stands in contrast to poor results yielded in the past from objects excavated decades ago and provided by both active excavation projects and museums. Residues for this study were extracted and processed in the program designed by Andrew Koh for ARCHEM (Koh 2008a) on June 25, 2006 with the assistance of Miriam Clinton of the University of Pennsylvania. The residue was extracted by heating the sherd in two different solvents. The process was

performed in the conservation laboratory of the INSTAP Study Center for East Crete, with two samples extracted from each sherd in succession, one using dichloromethane followed immediately by one using ethanol[3]. This process produced twenty-eight samples from fourteen objects. The resulting samples were analyzed at the Museum of Cretan Ethnology Research Centre in Vori using the Centre's GC-MS instrument (Gas Chromatography and Mass Spectrometry)[4]. This instrument couples the separation potential of gas chromatography with the characterization ability of mass spectrometry. It should be noted that a preliminary interpretation of the chromatograms was made without the advance benefit of a formal study of the subsequently cleaned and conserved vessels, minimizing unwarranted influences on interpretation. After a formal study of the vessels by P. Betancourt, the preliminary interpretation of the organic contents was further nuanced by A. Koh, but no significant changes needed to be made. We present below the best interpretations we have to date for the five objects that produced the clearest and strongest chromatograms and are of particular relevance to the study of the site.

As a control on the methodology, a sample of the soil that contained the sherds was collected and processed for extraction of organic compounds. The goal was to discover if any significant traces of organic compounds were included within the natural sediment on this site as a result of leaching from decaying plant matter or from other causes. The soil yielded two samples using dichloromethane and ethanol, and they were processed using the same methodology as that employed for the twenty-eight samples extracted from sherds. The soil samples produced only minute traces of organic residues, indicating the probability that no contamination entered the sherds from casual contact with the soil at the site. In addition to the soil samples, blanks were inserted periodically in each GC-MS run at Vori. Each of these blanks produced no peaks in their chromatograms confirming for us that there were no residual compounds from preceding samples injected into the GC column.

RESULTS OF THE INVESTIGATION (TABLE 1)[5]

AK 1 (ARCHEM 135 [ethanol] and ARCHEM 136 [dichloromethane], Figs. 1 and 2)

Closed vessel, base sherd. D of base ca. 12 cm. A light red fabric (2.5YR 6/6 to 6/8) with angular red inclusions, with the surface missing.

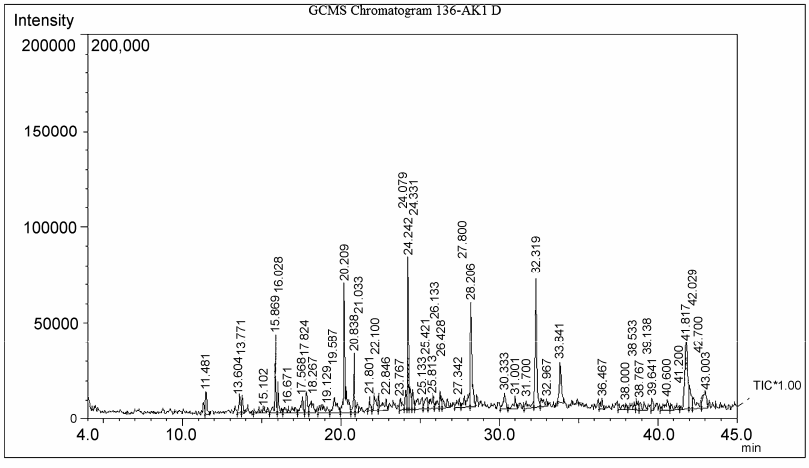

Figure 2. GCMS chromatogram for vessel AK1 (no. 136-AK 1 D).

Comments: The fabric suggests this closed vessel, which is probably a jug or a small jar, originated in eastern Crete. Almost all of the EM I pottery from Kephala Petras, a settlement east of Siteia, is made of this fabric (for the site, see Papadatos 2007).

Analysis Results: The two samples produced nearly identical chromatograms with the strongest peaks suggesting the presence of or-

ganic acids. Most notable were peaks eluting at 11.4 min (tartaric acid), 15.9 min (syringic acid), 20.2 min (stearic acid), 20.8 min (palmitate), 24.2 min (stearate), 28.6 (oleanolic acid), and 32.3 (phthalate).

Conclusions: Tartaric acid indicates the presence of wine, while syringic acid specifies that it is red. Oleanolic acid is a common indicator for the presence of pistacia resin. The vessel probably stored resinated red wine (for short-term use judging by its size).

AK 4 (ARCHEM 139 [DCM] and ARCHEM 140 [E], Fig. 1)

Open vessel, rim sherd. D of rim ca. 12 cm. An unevenly colored, black to dusky red (2.5YR 3/2) calcite-tempered fabric, burnished on interior and exterior. Straight rim.

Comments: The fabric indicates the vessel's production center is different from that of AK 1. This fabric is common on the north coast in the region of the Gulf of Mirabello.

Analysis Results: The E chromatogram produced results that were similar to those of AK 1 but with the absence of oleanolic acid, suggesting that the wine was not resinated. Perhaps this is due to the vessel's closer point of origin and/or its open shape. The DCM chromatogram has strong and distinct palmitic and stearic acid peaks but little otherwise. Why this difference from AK 1? It is difficult to say with certainty, but it is possible that the sherd's location on the rim (versus the base for AK 1) played a role in how the residues were absorbed and preserved on the different vessels.

Conclusions: The vessel held red wine, which was likely not resinated. The sherd was a rim fragment, which often produces quantitatively weaker and qualitatively distinct results in comparison with samples from bases and body sherds.

AK 6 (ARCHEM 143 [DCM] and ARCHEM 144 [E], Fig. 1)

Jar, body sherd. Max dim 5.6 cm. An unevenly colored, mostly black fabric with a reddish yellow (5YR 6/6) surface, with angular red inclusions, wiped on exterior and rough on the interior.

Comments: The rough interior indicates that this was a closed vessel. The fabric is similar to that of AK 1.

Analysis Results: This sample has a chromatogram that is both qualitatively (multitude of fatty acid peaks) and quantitatively (limits of the y-axis for intensity had to be tripled to accommodate unusually strong peaks) different from the previous samples.[4] Most importantly, the peak for oleic acid at 24.2 min is especially strong, strongly supporting the presence of olive oil.

Conclusions: Vessel AK 6 was probably a storage jar for olive oil.

AK 8 (ARCHEM 147 [ethanol] and ARCHEM 148 [DMC], Figs. 1 and 3)

Closed vessel, body sherd. Max dim 4.8 cm. A reddish yellow fabric (5YR 7/8), with rounded to subrounded inclusions of many different stones (South Coast Fabric), wiped on exterior.

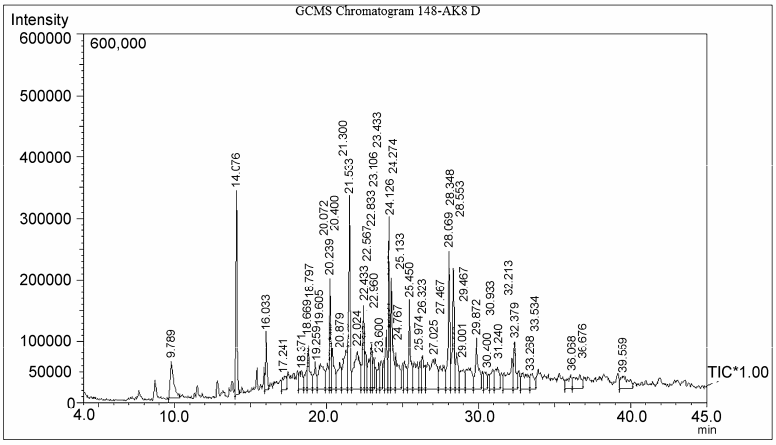

Figure 3. GCMS chromatogram for vessel AK8 (no. 148-AK 8 D).

Comments: This fabric is from a different source from the other vessels discussed in this study. The vessel had a closed shape (it was probably a jug or a jar).

Analysis Results: The two chromatograms are quite similar to those of AK 6, down to the unusually strong peaks produced by the DCM extraction.

Conclusions: Strong oleic acid peaks suggest the storage of olive oil.

AK 9 (ARCHEM 149 [DCM] and 150 [ethanol], Figs. 1 and 4)

Pithos, complete profile. Ht 74.6, ht of collar 14.5 to 15, d of rim 42, d of body 65, d of base 42 cm, capacity 150 liters filled to the bottom of the collar, 165 liters filled to the rim. Fabric with angular red inclusions, mostly light red (2.5YR 6/8), wiped on exterior. Flat rim with short incised lines, above a plain collar; body with rounded profile, with four vertical handles with thick oval sections; slightly rounded base. Decorated with raised moldings: band with vertical incisions at base of collar; large herringbone motifs, placed vertically, on shoulder, with sets of three vertical bands above the handles; nine horizontal bands on body. Dark slip (varying from red to black) on exterior.

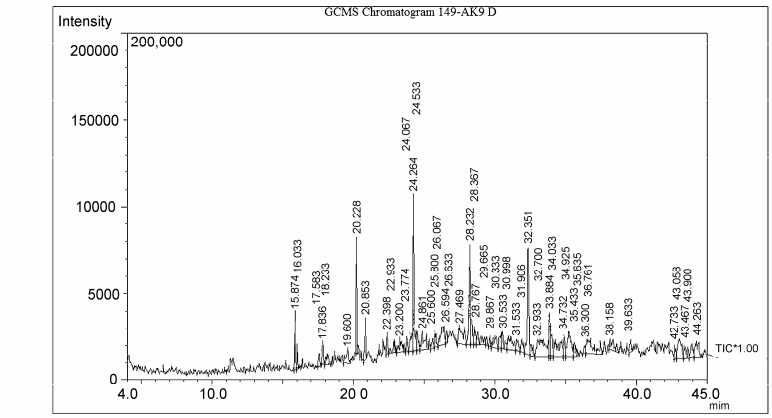

Figure 4. GCMS chromatogram for vessel AK9 (no. 149-AK 9 D).

Comments: The fabric indicates that the vessel comes from East Crete (see AK 1). Sherds from similar pithoi come from several other EM I contexts in Crete, including from Knossos (Wilson and Day 2000, 48–50), Debla (Warren and Tzedhakis 1974, 323), Kalo Chorio (Haggis 1996, 674 and fig. 30, no. KT 87), and Kephala-Petras (Papadatos personal communication; for the site, see Papadatos 2007).

Analysis Results: The results are somewhat similar to those for AK 4, but with some notable differences. The DCM chromatogram is devoid of any of the organic acid peaks that typically occur early in the chromatogram up to 15 min. The ethanol chromatogram produced weak peaks for the presence of wine with no evidence for resination.

Conclusions: This pithos shows weak evidence for red wine, possibly non-resinated. The large vessel had a long enough life for it to crack and then be repaired by drilling to allow a cord to be laced across the crack. Because storage of liquid would have been impossible after the crack appeared, the wine must have been stored in the vessel at an early period in its history of use. This conclusion is supported by the relatively weak chromatographic evidence for wine noticed from the outset during the preliminary interpretation before cleaning and conservation. Perhaps the pithos' attractive appearance provided a second life for a vessel originally used to store wine.

DISCUSSION

Authors studying early agriculture have remarked on the absence of data to support the production of wine and olive oil during the Early Minoan period (for olive oil, see Hansen 1988, 44–46, 51–52; for wine, see Halstead 1988, 529). The evidence from gas chromatography for the presence of both commodities is important for an understanding of the agricultural

production of the early stages of Minoan culture. In fact, the evidence provides much more than the information that these two products were present. Sherds from the pithos no. AK 9 survived in enough fragments to allow its restoration (Fig. 1), and tests with filling the large jar demonstrated that it would hold 150 kg if filled to the bottom of the collar and ca. 165 kg if filled to near the brim. This represents the storage of a large quantity of wine. A minimum of nine pithoi could be recognized from the large number of pithos sherds at the site, which indicates that the site was engaged in bulk storage of commodities in large amounts. That dry products were stored as well is proved by the continued use of the pithos after it cracked and was repaired. Wine from several different places was also present in smaller vessels, and olive oil was being stored at this site as well. Only red wine was recognized from these analyses, but some of it was resinated. The presence of resinated wine is not a surprise, because this class of beverage has already been recognized by gas chromatography of Minoan vessels from the end of the Early Bronze Age (Beeston et al. 2006, 420–423, 426) and even earlier by liquid chromatography in Levantine vessels found in the Protodynastic tomb of Scorpion I at Abydos, Egypt (McGovern 2003, 85-106).

Though the sample set is small, the possibility exists that there is a correlation between the resination of wines and their point of origin, though this can be difficult to ascertain when there is a quantitative difference in extant organics and additional variables inherent to vessels from disparate origins. Besides the evidence for pistacia resin, there were no obvious signs of flavors added to the wine at Aphrodite's Kephali, though the possibility exists that they did not survive or they are still waiting to be discovered in the hundreds of minute peaks yet to be identified in each chromatogram. However, judging by the uncluttered simplicity of the chromatograms (based on the relatively few, but strong peaks present) and the nature of the site itself, chances are that the vast majority of commodities were used as basic sustenance in a setting surrounded by few luxurious adornments.

The remarkable preservation of wine and olive oil at such an early site in Crete presumably has much also to do with the nature of the site and its associated vessels. As a relatively short-lived hilltop fort overlooking a strategic route across the Isthmus of Ierapetra, Aphrodite's Kephali, in fact, provides an ideal situation for organic residue analysis. Its size and function predicates that the storage of basic commodities will likely be emphasized in its archaeological record with less likelihood for a preponderance of luxury or specialized vessels complicating the task of residue interpretation, arguably the most difficult part of the analytical process since scientific instruments only identify compounds, not the original commodities from which they were derived. Therefore, final interpretations in the end must rely on a holistic knowledge of a site and the characteristics of its natural landscape, from which the commodities derive, in order to deduce the most likely identification of the commodities in question. Naturally, interpretations must periodically be reassessed, further nuanced, and even altered when new information arises. ARCHEM studies in the past have shown that simpler contexts such as the one at Aphrodite's Kephali, or, conversely, extraordinarily specialized spaces, such as perfumed oil workshops, provide easier targets for immediate interpretation than typical, daily, domestic spaces, where vessels were often reused in antiquity to hold different organics, like today's kitchen (cf. Brogan and Koh 2008, 128-129).

Finally, it should be noted that one methodological advantage of using a double-solvent extraction technique, as shown through this study, is the hint it provides as to the nature of commodities originally contained in the vessel. This results from comparing the two resultant chromatograms from the same object with each other and comparing these chromatograms, in turn, with those produced by other objects from the same context. In the case of Aphrodite's Kephali, the vessels used to hold olive oil, and its various fatty acids, were separated more discriminately by the two-solvent process, especially when compared to vessels used to hold wine and its diagnostic organic acids. While this two step extraction

process is not absolutely necessary and constraints related to resources and time often make it impractical, this study shows that double-solvent extractions can be an invaluable tool for the characterization of ancient residues in the right situation and can be selectively utilized when certain compound are expected (e.g. fatty acids).

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Table 1. Objects and Contents

OBJECT	SHAPE	FABRIC	PLACE OF ORIGIN	CONTENTS
AK 1	closed jug or jar	light red with angular red inclusions	East Crete	resinated red wine
AK 4	open vessel	black to dusky red	Gulf of Mirabello	red wine
AK 6	closed jar	black with reddish yellow surface and angular red inclusions	East Crete	olive oil
AK 8	closed jug or jar	reddish yellow with rounded/subrounded inclusions	south coast	olive oil
AK 9	pithos	light red with angular red inclusions	East Crete	originally red wine but later only dry goods

NOTES

1. Thanks are expressed to Stavroula Apostolakou and Theodore Eliopoulos for permission to publish this site. Research was supported by Temple University and the Institute for Aegean Prehistory. Koh would like to thank his co-author for inviting him to Aphrodite's Kephali and introducing him to Aegean Prehistory those many years ago. This study would not have been possible without the capable staff of the Institute for Aegean Prehistory in both Philadelphia and Pacheia Ammos. Christophe and Despina Vallianos opened up their office and home in Vori to support this research; the Museum of Cretan Ethnology Research Centre has been an invaluable resource for the study of ancient commodities. Finally, it is safe to say that both measurable (Figs. 2-4 layout) and immeasurable contributions by Laura Labriola Koh were vital to this study's completion.

2. In addition to the insight gained into the efficacy of this technique from this present and past studies, research is now being conducted at Tel Kabri with the support of co-directors Assaf Yasur-Landau and Eric H. Cline to directly compare this technique with destructive techniques commonly used today (cf. Beeston et al., pp. 413-414, for a good description of this destructive extraction technique and an excellent overview of organic residue analysis in general; cf. Gerhardt et al., p. 42, and Biers et al, p. 19 for studies showing the efficacy of non-destructive extraction techniques). At Tel Kabri, adjacent sherds from vessels were collected by ARCHEM and Kristine Merriman of Oxford University with the intent of comparing results in the near future. The ARCHEM sherds were processed using the non-destructive extraction technique outlined in this study while Merriman will use the destructive extraction technique favored by chemists. Combined with petrographic thin-section analysis by David Ben Shlomo of the Hebrew University, our ultimate goal, however, is to illuminate patterns in both local consumption and trade not possible without the contribution of these analytical techniques.

3. Sherds were placed in a Buchi Polyevaporator with ~200 ml of solvent for ~20 min at 60°C under vacuum and then gravity filtered to produce 20 ml master samples.

4. For GC-MS analysis, subsamples were taken from the master samples by measuring 300 μl of solution into glass inserts placed into 2 ml vials, which were then centrifugally evaporated and refilled with dichloromethane for injection into a Shimadzu 5050A GC-MS at the Museum of Cretan Ethnology Research Centre.

The splitless injector and interface on the GC were both set to 250°C with a 1 minute interface time. The carrier gas (helium) had a column inlet pressure of 60 kPa with a linear velocity of 53.8 cm/sec. The initial oven temperature was set to 130°C and held for 2 minutes before reaching 250°C at a rate of 5°C/min, at which time it was held for an additional 19 minutes, giving a total program

time of 45 min/sample. A fused silica SPB-5 GC capillary column (30m x 0.32 mm, 0.50 μm) was used in the GC and directly inserted into a methane chemical ionization source set at 0.85 kV with a 50-450 m/z scan.

5. The chromatogram produced from the non-polar DCM solvent had significantly stronger peaks overall but certain compounds were especially pronounced compared to the E chromatogram. This not only points to differences in the vessel's contents when compared to other vessels at the site, but also possibly hints at the nature of the vessels use in antiquity and its state of preservation over the millennia since.