THE CONTENT OF AFRICAN KEAY 25 / AFRICANA 3 AMPHORAE: INITIAL RESULTS OF THE *CORONAM* PROJECT

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ABSTRACT

This paper presents the first results of the CORONAM (COntent of ROmaN AMphorae) project, an informal international research network on amphorae and the history of Roman trade. Samples of several Keay 25 (Africana 3) subtypes were analysed in an attempt to determine vessel content. Visible residues from the interior of vessels as well as ceramic samples of the vessel walls were analysed by Gas Chromatography/Mass Spectrometry (GC/MS). Results indicated that the vessels' interiors were coated with Pine pitch and some samples indicated a content of wine.

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INTRODUCTION

Did African amphorae carry wine? Such a question might surprise people unaware of the importance placed by historiography on the olive oil production of Roman Africa since the beginning of the 1970s (Carandini, 1970). Indeed, even more than grain production, olive growing has been considered as the motor of the economic development of the province from the second century AD onwards (Mattingly, 1988a; 1988b; 1993; Mattingly & Hitchner, 1991). It was obvious that the numerous African amphorae discovered in consumption sites all around the Mediterranean reflected trade in African olive oil. Nevertheless, this emphasis on the export of African olive oil has probably excessively marginalized the other African foodstuffs that were likely to have been shipped in amphorae: fish products (salted fish and fish sauces), and wine.

As a matter of fact, Africa did produce wine. Robert Lequément was the first to recall this obvious fact by listing a series of literary and epigraphic sources (Lequément, 1980), to which Jean-Pierre Brun added a revision of the archaeological evidence: many of the Roman presses surveyed in the African countryside were devoted to the production not of olive oil, but of wine (Brun, 2003). The local distribution of this production would not be surprising, especially considering that the use of oil skins and even barrels is well attested in Roman Africa (Marlière & Torres Costa, 2007). But to what extent was this African wine ever exported, or did it serve a purely local market? And if it was exported, can we recognise the amphorae in which it would have been shipped?

Seeking out some possible African wine amphora types, beside some neo-Punic amphorae and African imitations of Italian and Gaulish types, a hypothesis was recently proposed that the medium-sized Keay 25 amphorae (or Africana 3) were intended for the export of African wine in the fourth century (Bonifay, 2004; 2007). Indeed, these containers, almost always showing traces of an internal pitch lining, are widely distributed throughout the Mediterranean and beyond, even in places where African amphorae are normally rare, and even in regions with a massive local production of olive oil. The appearance of this new type of African container seems to coincide with the beginning of the *canon uinarius* established within the *annona system* at the end of the third century or the beginning of the fourth century. Nevertheless, while attractive, this hypothesis had not until now been confirmed by chemical analyses (except for one attempt: Formenti & Joncheray, 1995).

The question of the principal contents of the Keay 25 was therefore chosen as one of the first targets for the *CORONAM* project, an informal research network on the *COntent of ROmaN AMphorae*, initiated in 2006 as an international collaboration between experts in amphorae (Dario Bernal, Michel Bonifay, Simon Keay, Florence Richez), archaeometry (Dirk De Vos, Nicolas Garnier, Alessandra Pecci, Mark Pollard, Marshall Woodworth) and the archaeology and history of Roman trade (Jeroen Poblome, André Tchernia, Andrew Wilson). Kerlijne Romanus produced a preliminary report concerning the characterization of pitch biomarkers (Romanus 2008). The project aims to identify the contents of a number of common amphora types whose principal contents are either unknown or unclear, via a programme of laboratory analysis of a large enough series of samples of each type to have statistical validity.

The established view that form has a close relationship to contents had recently been challenged; a question to be addressed was therefore whether some types may have been polyvalent containers. Nevertheless, the presumption was that contradictions in the evidence, such as different samples of the same type yielding biomarkers for both fish and wine, may most naturally be explained by re-use, which might be either domestic, or for regional trade, or perhaps for long-distance trade. Wreck assemblages should present less chance of producing reused material. For each type, the aim was to sample amphorae from a range of depositional environments including both marine and terrestrial environments.

KEAY 25 / AFRICANA 3 AMPHORAE

The Keay 25 amphora class consists of 30 variants, the most common of which, variants A–V, Keay grouped into three main subtypes (Keay, 1984: 184-212; cf. Bonifay, 2004: 119-122). The Keay 25.1, also now called the Africana 3A, may have first appeared around the end of the third century AD, but the main production belongs to the fourth century. It was produced in several coastal regions of Tunisia, both in northern Tunisia (Zeugitana), at Nabeul and at el-Ariana near Carthage (Ghalia *et al.*, 2005; Panella, 1982), and in coastal central/southern Tunisia (Byzacena), at Leptiminus, Sullecthum, Thaenae and Oued el-Akarit (Peacock *et al.*, 1989; Bonifay, 2004). There may also have been some production in Algeria (Bonifay, 2004). Its capacity averaged 25–30 litres. This amphora type was widely exported around the western Mediterranean, where it is one of the most widely distributed African forms: it is common at Ostia and Rome, and in Catalonia and southern France; it was also shipped to the Eastern Mediterranean as abundant examples from Beirut show (Manacorda, 1977; Keay, 1984; Remolà, 2000). Cargoes of Keay 25.1/Africana 3A amphorae are found on wrecks off the coast of southern Gaul: Pampelonne (Lequément, 1976), Héliopolis 1 (Joncheray, 1997), Pointe de la Luque B (Dovis-Vicente, 2001).

A second sub-type, the Keay 25.3 or Africana 3B, has a thickened and bent rim, with a conical and elongated neck and tall handles, and a slightly smaller average capacity: 20–25 litres. It was produced at Nabeul and probably also elsewhere in Tunisia (Bonifay, 2004), and has a western Mediterranean distribution, with examples at Carthage (Panella, 1982; Freed, 1995) and in Catalonia, and the south of France and Italy. Also a fourth-century form, its production in part overlapped chronologically with the Keay 25.1, since both types occur together on the Pointe de la Luque B wreck (Dovis-Vicente, 2001).

The Keay 25.2 (= Africana 3C) is a later variant, characterised by a pronounced everted lip on the rim, and smaller still, with a capacity of 15–23 litres. It was produced from the end of the fourth to around the middle of the fifth century AD, and appears to have succeeded the Keay 25.1 in many of the same production centres, e.g. Nabeul, Sullecthum, Thaenae, and Oued El-Akarit (Peacock *et al.*, 1989; Bonifay, 2004; Ghalia *et al.*, 2005). It too was widely distributed in the western Mediterranean, in Spain, Italy, and the south of France (Panella 1982; 2002; Bonifay 2004), including the Dramont E (Santamaria, 1995) wreck off the French coast, and it also reached the eastern Mediterranean.

SAMPLES ANALYSED

The project analysed a total of 32 samples across all three variants of Keay 25: 10 samples of 25.1, 1 sample of a Keay 25.1/2, 2 samples of 25.2, 4 samples of 25.3, 2 samples of a transitional type between Keay 25.3 and 35B as well as 13 samples of visible residue from the interiors of vessels.

A series of 28 samples comes from underwater contexts, including French Mediterranean coastline wrecks and stray finds, and excavations in the river Rhône at Arles (Figs. 1 and 2, Table 1). Most of the samples (Aix/40 to 79) were provided by the Département des Recherches Archéologiques Subaquatiques et Sous-Marines (DRASSM), and selected by Michel Bonifay (CNRS, CCJ) and Florence Richez (DRASSM). Three other samples (Aix/80 to 86) have been taken from the collections of the Musée Départemental Arles Antique (MDAA), with the help of Jean Piton and David Djaoui (MDAA). Some of the samples are pitch samples and some of them are ceramic samples from the amphorae (Table 1).

Four important wrecks with 'African' cargoes are represented among these samples:

1) Six samples, Aix/40 to 45 (Table 1), come from the Heliopolis 1 wreck. Excavated in

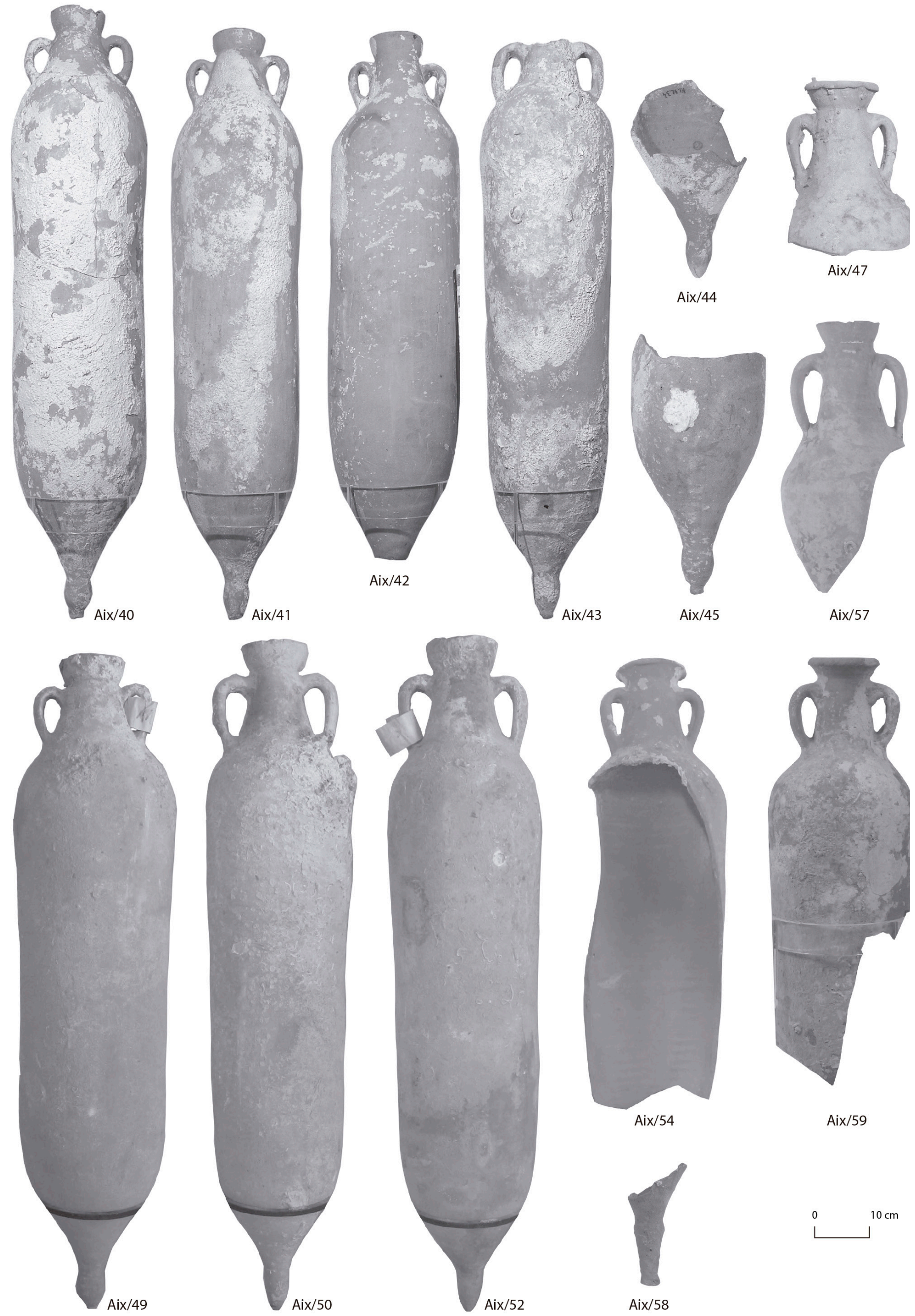

Figure 1 - Keay 25 amphora samples from French Mediterranean underwater contexts. Photographs from DRASSM database (Fl. Richez).

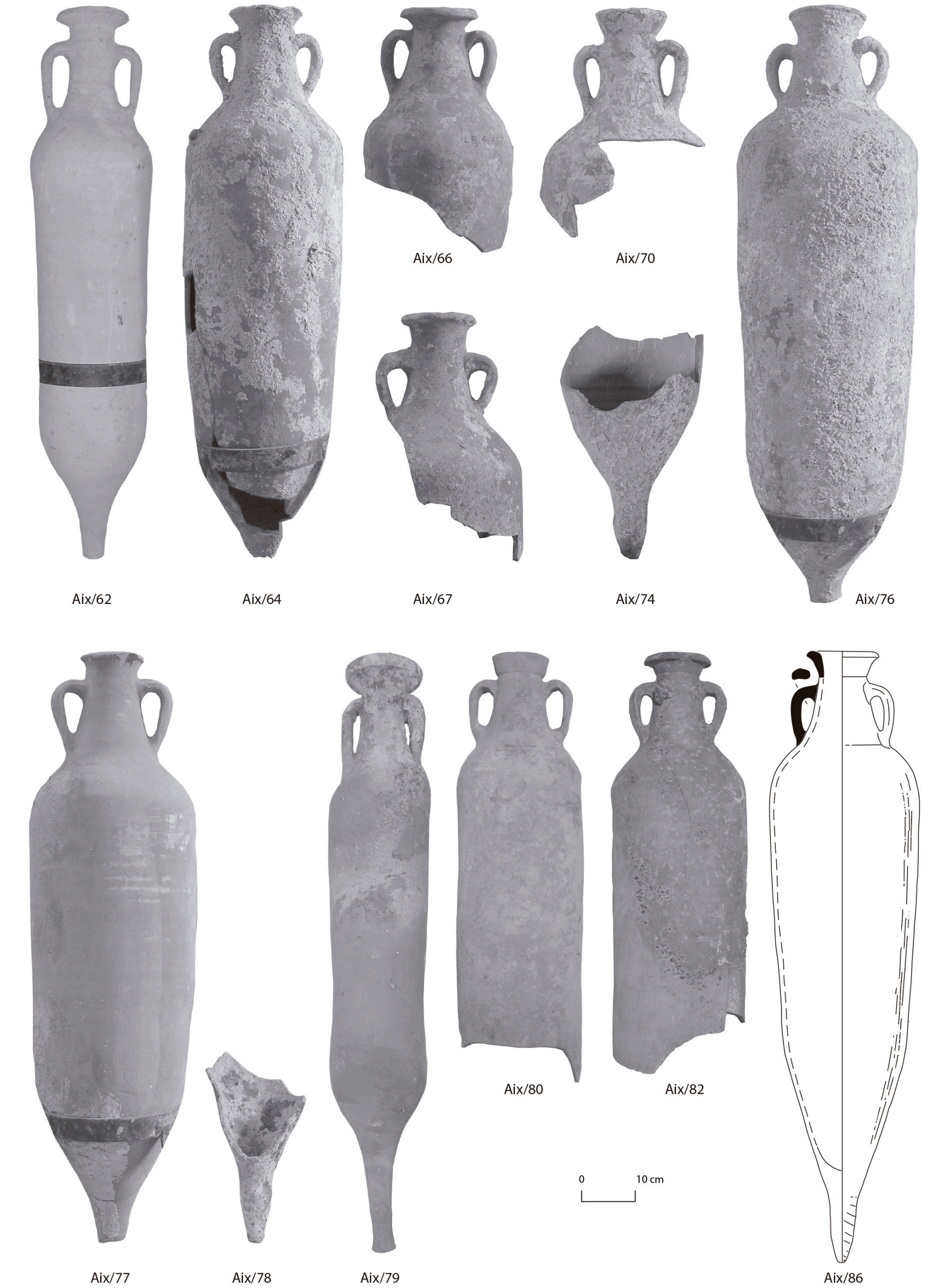

Figure 2 – Keay 25 amphora samples from French Mediterranean underwater contexts. Photographs from DRASSM database (Fl. Richez), except Aix/80 and 82 (photograph M. Bonifay), and Aix/86 (Bonifay and Piton 2008, fig. 2.10).

the 1990s by Jean-Pierre Joncheray off the Levant Island at Hyères (Joncheray, 1997), this wreck, dating back to the beginning of the fourth century AD, contained a cargo of early variants of Keay 25 amphorae originating from the city of *Sullecthum*/Salakta in Byzacena (Bonifay, Capelli & Long, 2002). Initial chemical analysis performed on one of these containers had already revealed traces of tartaric acid (Formenti & Joncheray, 1995), suggesting that the contents were wine.

2) Three samples, Aix/49 to 52 (Table 1), come from the Pampelonne wreck. Surveyed in the 1970s by Robert Lequément, this wreck is located in the Saint-Tropez bay and more or less contemporaneous with the previous one. The cargo included late variants of Africana II C and early variants of Keay 25 amphorae (Lequément, 1976), probably originating from the city of *Neapolis*/Nabeul on Cape Bon (Bonifay, 2009).

3) Seven samples, Aix/66 to 77, come from the Pointe de la Luque B wreck. Fully excavated in the 1970s off the Frioul archipelago at Marseilles, this wreck, dating back to the mid fourth century, contained a cargo of Keay 25.1 and 25.3 amphorae, along with a secondary cargo of lamps produced in the workshops of Cherchell or Tipasa in *Mauretania Caesariensis* (Dovis, 1999), perhaps the departure port of the ship itself (Bonifay, 2009).

4) One sample, Aix/79 (a pitch sample, Table 1), comes from Dramont E wreck. Fully excavated by Claude Santamaria off the Dramont cape at Saint-Raphaël, this wreck of the first half of the fifth century yielded a cargo of Keay 35A and B large containers completed by a series of Keay 25.2 and *spatheion* 1 amphorae, and a secondary cargo of African Red Slip ware (Santamaria, 1995). The whole amphora cargo originates from *Neapolis*/Nabeul (Bonifay, Capelli & Long 2002), while olive pits have been found inside of most of the Keay 25.2 and *spatheion* 1 amphorae (Santamaria, 1995, 123).

Other samples from isolated and sometimes unknown find spots along the French Mediterranean coastline have been selected due to their typological interest (Samples Aix/47, 54, 57, 58, 59, 62, 64, 78, and 86, Table 1). Lastly, two samples were selected from among the amphorae from the Rhône river excavations conducted at Arles by Luc Long (DRASSM) (Samples Aix/80 and 82, Table 1).

In addition to samples from maritime contexts, a second set of four samples from a terrestrial excavation were also examined (Figure 3 and Table 1). *Iulia Traducta* (modern Algeciras) was a Roman colony founded by Augustus in the Bay of Algeciras, opposite Gibraltar, in order to offset the importance of *Colonia Latina Libertinorum Carteia* on the other site of the Straits of Gibraltar. In the last two decades different rescue excavations carried out in the so called "Villa Vieja" have confirmed the close involvement of the city's inhabitants with the exploitation of marine resources between the first and fourth centuries AD. Some of the streets of the ancient Roman city have been unearthed, and between them an important area of the industrial quarter devoted to the fish-salting industry (Jiménez-Camino & Bernal, 2007). To date, five fish-salting plants have been excavated (called "Conjunto Industrial I, II, A, B & C") around modern San Nicolas street, under study by an interdisciplinary team of the University of Cádiz, whose results remain still mainly unpublished, with preliminary works published so far (Bernal *et al.*, 2003; Bernal & Expósito, 2006). Most of the vats were abandoned in the late fifth or early sixth century AD, well dated thanks to the large quantities of imported wares (mostly African) and coins recovered. Four samples were selected for this study (samples SN02, 07, 15, 16, Table 1). They come from the layers filling vats numbered 3 and 11, and from the sediment covering the floor of the H-100 room pavement of the Conjunto Industrial I, including late variants of Keay 25.2 (SN/02 and 07) and a transitional type between Keay 25.3 and 35B (SN/15-16), already known in Alexandria (Bonifay & Leffy, 2002, 50 and fig. 6.48).

METHODS

Sample extraction

All the samples were analysed at the Research Laboratory for Archaeology and the History of Art in Oxford. Ceramic samples were analysed to identify amphorae contents and organic coatings, using techniques designed to identify biomarkers for wine or its derivatives, and lipid extraction to test for lipids of either plant or animal origin that might suggest lipid-rich commodities such as olive oil or garum which may have been transported in the vessels. The lipid extraction technique was also used to characterize the visible pitch samples.

Sherds were first surface-cleaned before the ceramic sample for analysis was drilled. Powdered samples were each extracted using two techniques in order to access lipid and pitch constituents as well as possible biomarkers of wine products. All reagents and analytical standards were acquired from Sigma Aldrich; water used for aqueous extractions was obtained from an in-house MilliQ water system. An electric modelling drill with tungsten carbide drill bits was used. Drill bits were washed three times with chloroform/methanol (2:1 v/v) before and between uses. For each sample 500 mg of ceramic powder was extracted; however, for the analyses of samples AIX/47 and SN03 350 mg was extracted due to the small size of the sherds.

Sample No.	Typology	Site	Nature of the site	Date of excavation	Chronology	Description	Pitch	Type and Location of the sample	Inventory No.
Aix/40	Keay 25.1 early	Heliopolis 1	Wreck (30–38 m deep)	1993	Early 4th c.	Complete	Yes	Pitch sample (bottom)	DRASSM 10807
Aix/41	Keay 25.1 early	Heliopolis 1	Wreck (30–38 m deep)	1993	Early 4th c.	Complete	Yes	Pitch sample (bottom)	DRASSM 10795
Aix/42	Keay 25.1 early	Heliopolis 1	Wreck (30–38 m deep)	1993	Early 4th c.	Nearly complete	Yes	Pitch sample (bottom)	DRASSM 10791
Aix/43	Keay 25.1 early	Heliopolis 1	Wreck (30–38 m deep)	1993	Early 4th c.	Nearly complete	Yes	Pitch sample (bottom)	DRASSM 10785
Aix/44	Keay 25.1 early	Heliopolis 1	Wreck (30–38 m deep)	1993	Early 4th c.	Base	Yes?	Wall sherd (lower part)	DRASSM 10810
Aix/45	Keay 25.1 early	Heliopolis 1	Wreck (30–38 m deep)	1993	Early 4th c.	Base	Not visible	Wall sherd (lower part)	DRASSM 10809
Aix/47	Keay 25.1/2	Unknown	Underwater stray find	1988	4th c.	Neck	Not visible	Shoulder sherd	DRASSM 9644
Aix/49	Keay 25.1 early	Pampelonne	Wreck (65 m deep)	1975	Early 4th c.	Complete	Yes	Pitch sample (bottom)	DRASSM 7931
Aix/50	Keay 25.1 early	Pampelonne	Wreck (65 m deep)	1975	Early 4th c.	Complete	Yes	Pitch sample from the bottom	DRASSM 7932
Aix/52	Keay 25.1	Pampelonne	Wreck (65 m deep)	1975	Early 4th c.	Complete	Yes	Pitch sample (wall and bottom)	DRASSM 7934
Aix/54	Keay 25.3	Unknown	Underwater stray find	Unknown	4th c.	Nearly complete	Not visible	Wall sherd (lower part)	DRASSM GE 7529
Aix/57	Keay 25.1 variant	Unknown	Underwater stray find?	1960 +	4th c.	Neck	Not visible	Wall sherd (medium part)	DRASSM 6713
Aix/58	Keay 25.1?	Unknown	Underwater stray find?	Unknown	4th c.	Base	Yes	Pitch sample (bottom)	DRASSM 6670
Aix/59	Keay 25.3	Island of Jarre (Marseille)	Underwater stray find	1967	4th c.	Nearly complete	Yes	Wall sherd (lower part)	DRASSM 968
Aix/62	Keay 25.2	Bonifacio (Corse)	Underwater stray find	Unknown	5th c.	Complete	Yes	Pitch sample (bottom)	DRASSM 2701
Aix/64	Keay 25.1	Unknown	Underwater stray find	Unknown	4th c.	Nearly complete	Yes	Base sherd	DRASSM 5909
Aix/66	Keay 25.3	La Luque B	Wreck	1970s	4th c.	Neck	Yes	Wall sherd (medium part)	DRASSM 6164
Aix/67	Keay 25.3	La Luque B	Wreck (30–36 m deep)	1970s	4th c.	Neck	Not visible	Wall sherd (medium part)	DRASSM 2085
Aix/68	Keay 25.1	La Luque B	Wreck (30–36 m deep)	1970s	4th c.	Neck (not illustrated)	Not visible	Wall sherd (medium part)	DRASSM 1085
Aix/70	Keay 25.1	La Luque B	Wreck (30–36 m deep)	1970s	4th c.	Neck	Not visible	Shoulder sherd	DRASSM 2094
Aix/74	Keay 25.1?	La Luque B	Wreck (30–36 m deep)	1970s	4th c.	Base	Yes	Wall sherd (lower part, with pitch)	DRASSM 1814
Aix/76	Keay 25.1	La Luque B	Wreck (30–36 m deep)	1970s	4th c.	Nearly complete	Yes	Pitch sample (bottom)	DRASSM 1808
Aix/77	Keay 25.3	La Luque B	Wreck (30–36 m deep)	1970s	4th c.	Nearly complete	Yes	Pitch sample (bottom)	DRASSM 1813
Aix/78	Keay 25.1?	Unknown	Underwater stray find	Unknown	4th c.	Base	Yes	Base sherd	DRASSM 6393a
Aix/79	Keay 25.2 late	Dramont E	Wreck	1970s	5th c.	Complete	Yes	Pitch sample (bottom)	DRASSM 11062
Aix/80	Keay 25.1 early	Arles-Rhône	Underwater Dump (4–8 m deep)	1980s	4th c.	Nearly complete	Yes	Wall sherd (lower part, with pitch)	no inventory
Aix/82	Keay 25.1	Arles-Rhône	Underwater Dump (4–8 m deep)	2005	4th c.	Nearly complete	Yes	Wall sherd (lower part)	RH.05.SN. 86.1186
Aix/86	Keay 25.1	Unknown	Underwater stray find	1992–93	4th c.	Complete	Yes	Pitch sample (bottom)	MDAA FAN 92.00.3599
SN/02	Keay 25.2 late	Conjunto Industrial I (San Nicolas street, no. 3–5)	Fish salting plant Layer inside fish tank no. 3	2001	Late 5th c.	Nearly complete	Not visible	Middle part of the neck	UE 1416/40 – 2005/46
SN/07	Keay 25.2 or *spatheion* 1	Conjunto Industrial I (San Nicolas street, no. 3–5)	Fish salting plant Layer inside fish tank no. 11	2001	Late 5th c.	Rim-neck	Not visible	Middle part of the neck	UE 1422/126
SN/15	Keay 25/35	Conjunto Industrial I (San Nicolas street, no. 3–5)	Fish salting plant Layers over the floor of the H-100 room pavement	2001	Late 5th c.	Rim-neck	Not visible	Middle part of the neck	UE 2003/77
SN/16	Keay 25/35	Conjunto Industrial I (San Nicolas street, no. 3–5)	Fish salting plant Layers over the floor of the H-100 room pavement	2001	Late 5th c.	Rim-neck	Not visible	Upper part of the neck	UE 2003/67

Table 1 – Samples of Keay 25 / Africana 3 amphorae analysed for this study. Aix/40 to 79: French Mediterranean coastline wrecks and erratic finds (DRASSM). Aix/80 to 86: Arles, Rhône underwater excavations, and maritime erratic find (DRASSM/MDAA) [M. Bonifay (CNRS, CCJ) and Fl. Richez (DRASSM)]. SN/02-16: Samples from terrestrial contexts found at the Roman fish-salting plant at San Nicolas no. 3-5 street (Algeciras, province of Cádiz, south Spain). [D. Bernal-Casasola (UCA)].

The lipid extraction technique for ceramic samples was adapted from that used by Mottram *et al.* 1999. Each sample was twice solvent-extracted with 3 ml of chloroform/methanol (2:1 v/v) in an ultrasonic bath (for 45 minutes at 50°C). The samples were then removed from the bath and allowed to cool to room temperature before being filtered with a Millipore PVDF syringe filter (13 mm diameter, 0.20 µm pore diameter) in order to remove ceramic particles from the solvent extraction. The extraction solvent was then evaporated to dryness under a stream of nitrogen gas. Samples were stored at -20°C until analysis. Immediately prior to analysis, samples were derivatized with 50 µl of *N,O*-bis(trimethylsilyl)trifluoroacetamide (BSTFA) by heating at 70°C for 60 minutes. After derivatization, 10 µl of (tetratriacontane in hexane at a concentration of 0.5 mg/ml) was added.

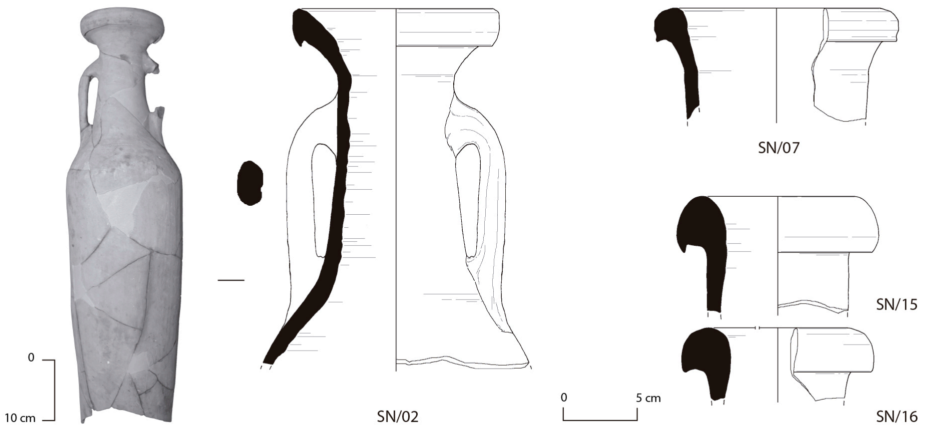

Figure 3 - Keay 25 amphora samples from Algeciras (Spain) excavations (D. Bernal).

Pitch samples were extracted using the lipid extraction technique above with several modifications. Samples were extracted once with 2 ml of chloroform/methanol (2:1 v/v) to approximately 2 mg of sample. Samples were then placed in an ultrasonic bath for 10 minutes at room temperature. From the extraction solvent, 20 µl was removed to an autosampler vial and briefly evaporated to dryness under a stream of nitrogen gas. Storage, derivatization and the addition of the tetratriacontane was conducted as per the lipid extraction technique above. Samples were diluted to 1 ml with dicholoromethane before GC/MS analysis.

The extraction technique used for the analysis of wine biomarkers was that developed by Pecci *et al.* (2013). Each powdered ceramic sample was extracted with 3 ml of an aqueous solution of potassium hydroxide (1 M) in an ultrasonic bath (for 90 minutes at 70°C). The samples were then removed from the bath and allowed to cool to room temperature before filtration (as above). The aqueous extraction solvent was then acidified to a pH of approximately 2 with hydrochloric acid. After acidification, the extraction solvent was twice liquid-liquid extracted with 3 ml of ethyl acetate. The ethyl acetate was removed to another vial and evaporated to dryness under a stream of nitrogen gas. Storage, derivatization and the addition of the tetratriacontane was conducted as per the lipid extraction technique above. Identification of tartaric and syringic acids was achieved by comparison of retention times and mass spectra against authentic standards as well as comparison against the NIST11 mass spectral database.

Analytical Conditions

Samples were analyzed by GC/MS using an Agilent 7820A gas chromatograph equipped with a Restek Rxi-5ms column (30 m length x 0.25 mm ID x 0.25 µm film thickness, 5% diphenyl/95% dimethylpolysiloxane stationary phase). The mass spectrometer was an Agilent 5975 quadrupole, operated in electron ionization mode (70 eV) and the scan range was *m/z* 40-650. The gas chromatograph conditions were as follows: inlet temperature 300ºC, flow rate 1.2 ml/min, transfer line temperature 280ºC. Helium was used as the carrier gas. The temperature program for the GC oven was a 50ºC hold for 2 minutes, 50-300ºC at 10ºC/min with a 10 minute isothermal hold at 300ºC. Injections were made by an Agilent 7693A autosampler and sample injection volume was 1 µl in splitless mode. For samples that appeared to be possibly too concentrated to inject in splitless mode, analysis was first conducted in split mode (50:1 or 25:1) in order to access the quantity of organic constituents before being run in splitless mode.

In addition to GC/MS analyses, one sample (see 'Pitch Samples' below) was analysed by scanning electron microscope with energy-dispersive X-ray spectroscope (SEM/EDX) using a JSM-5910 SEM equipped with an Oxford Inca 300 EDX. An accelerating voltage of 15 kV was used. Area scans of approximately 70 µm2 were conducted with a scanning period of 90 seconds.

RESULTS

Pitch Samples

Thirteen samples of visible residue from the interior of amphorae recovered from maritime contexts were analyzed and represented 9 Keay 25.1, 3 Keay 25.2 and 1 Keay 25.3 vessels (see 'pitch samples' in Table 1). Twelve of the samples were completely or nearly completely soluble in the organic solvent extraction. Chromatograms identified resin acids as observed in the ceramic samples, predominated by dehydroabietic acid (Figure 4). Other oxidation products included 7-oxodehydroabietic acid and 15-hydroxy-7-oxodehydroabietic acid. Heating markers (such as retene and anthracene compounds) were also observed in the 12 samples (Colombini *et al.* 2003, Pollard and Heron 1996). *Pinaceae* products such as abietic, pimaric and isopimaric acids were also observed in the samples, typically in low concentrations relative to the oxidation products and all three were not identified in all samples, most probably owing to differing extents of oxidation either during the production of the pitch or during deposition. Methyl dehydroabietate was also observed in all samples, in relatively significant amounts relative to dehydroabietic acid. This compound is indicative of method of production of the pitch: it is formed by heating resin-bearing wood, where the wood is burned to simultaneously extract resin from the source wood and convert it to pitch (Colombini *et al.*, 2003, Pollard & Heron, 1996).

One sample (Keay 25.1 Sample AIX/52), however, produced different results. Visually it was distinct from the other pitch samples in being a reddish-brown colour rather than a brown-black to black colour. Macroscopically, it was not appreciably soluble in either the chloroform/methanol or the aqueous extraction solvent used for wine biomarker analysis. Predominated by dehydroabietic acid, the oxidation by-product, 7-oxodehydroabietic acid, and methyl dehydroabietic acid were also identified. Unmodified resin acids, specifically abietic, pimaric and isopimaric acids, were also observed. Elemental analysis of the extracted sample by scanning electron microscope (SEM-EDX) indicated that the sample was principally composed of a silicate matrix. The two most apparent explanations for this material are either a deterioration of the vessel's surface due to poor firing or a concretion of clay sediment on the vessel's interior surface during deposition. The presence of biomarkers of pitch, albeit at low levels, suggests that, if the second possibility were to be true, a portion of the vessel's pitch lining was incorporated into the deposited sediment.

Keay 25.1 / Africana 3A ceramic samples

The lipid extractions of the Keay 25.1 ceramic samples identified significant amounts of *Pinaceae* products. The absorbed pitch residues bore a strong similarity to the visible pitch samples. The predominant resin acid was dehydroabietic acid and oxidation by-products of abietic acid, including 7-oxodehydroabietic and 15-hydroxy-7-oxodehydroabietic acids (Figure 5). Heating by-products such as retene were also apparent. Methyl dehydroabietate was present in all samples and unmodified resin acids, abietic, pimaric and isoprimaric acids, were also observed. The Keay 25.1/2 sample, AIX/47, failed during extraction. Similar to the pitch samples, all three resin acids were not present in all analysed samples, e.g. in the sample of AIX/64 (Figure 5) only abietic acid was identified. These results confirm that the amphorae lining was made of pitch and the presence of methyl dehydroabietate suggests, as stated above, that it was produced directly from the heating of wood (Colombini *et al.*, 2005).

The lipid extractions provided small amounts of fatty acids. Only the saturated fatty acids $C_{16:0}$ and $C_{18:0}$ were observed in the samples and were characterized by a greater or equal amount of $C_{16:0}$ compared to $C_{18:0}$ (Figures 5 and 6). No plant or animal sterols were observed in the samples. Therefore the presence of the acids is not indicative of specific contents of the amphorae, nor of the adding of specific fats to their organic coatings.

The Keay 25.1 ceramic samples were also analyzed for possible biomarkers of wine products using the extraction and analysis protocol of Pecci *et al.* 2013. Of the 10 samples, tartaric acid was positively identified in 4 samples (AIX/44, AIX45, AIX/74, AIX/80). In addition to tartaric acid, other compounds that are present in wine were also present in these samples: malic, malonic, fumaric, succinic acids (Table 2). These acids are present also in 5 samples in which tartaric acid could not be identified, AIX/57, AIX/64, AIX/68, AIX70, AIX/78 (Figure 7). In these cases, although it cannot be definitively established that wine was contained in the vessels, they merit further study. To further examine if the content is wine, different sampling strategies and possibly other methods could be tested in the future.

Syringic acid was identified in 4 samples (AIX/68, AIX/74, AIX/78, AIX/80).

Keay 25.2/Africana 3C ceramic samples

Two samples of Keay 25.2 amphorae were analyzed. Both samples (SN02 and SN07) were from the excavation at Cadiz and samples were obtained from the vessel necks. The lipid analyses of the samples revealed pitch residues identified by the presence of dehydroabietic acid and 7-oxodehydroabietic acid. No fatty acids apart from $C_{16:0}$ and $C_{18:0}$ were identified. The wine biomarker extraction of SN/02 did not identify tartaric acid, but identified malonic, fumaric, succinic and malic acids (Table 2). These compounds were absent in the extraction of SN/07.

Syringic acid and vanillic acid were identified in both samples. Syringic acid is considered to be another biomarker of grape products, derived from malvidin (malvidin-3-glucoside), the predominant anthocyanin (the pigment responsible for coloration) of red grapes (Singleton, 1996; Guasch-Jané *et al.*, 2004). In the samples where syringic

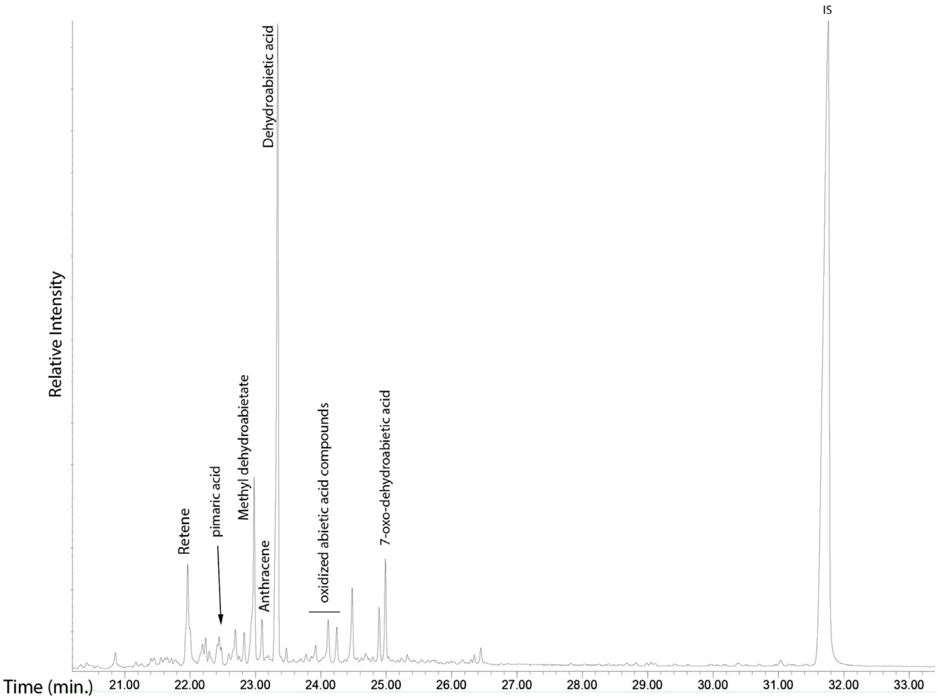

Figure 4 - Partial total ion chromatogram of a sample of visible pitch from Keay 25.1 sample AIX/51 (172LIP).

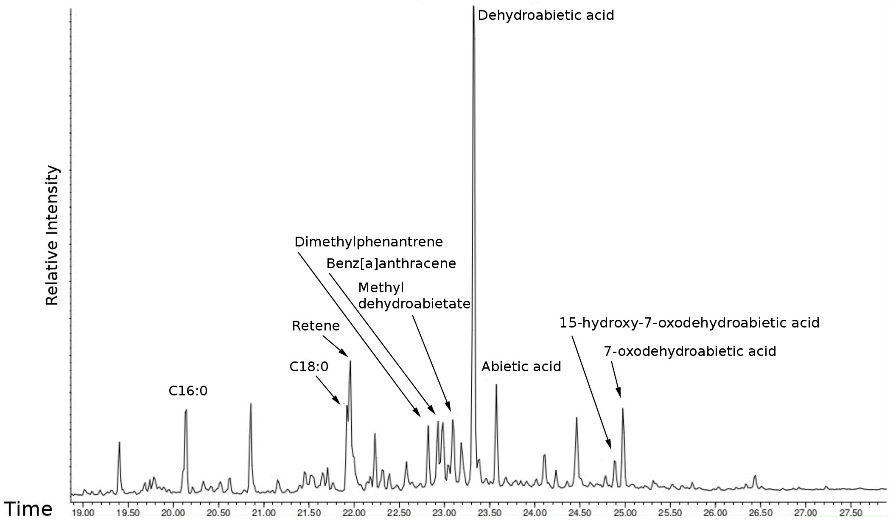

Figure 5 - Partial total ion chromatogram of the lipid extraction of Keay 25.1 sample AIX/64 (184LIP).

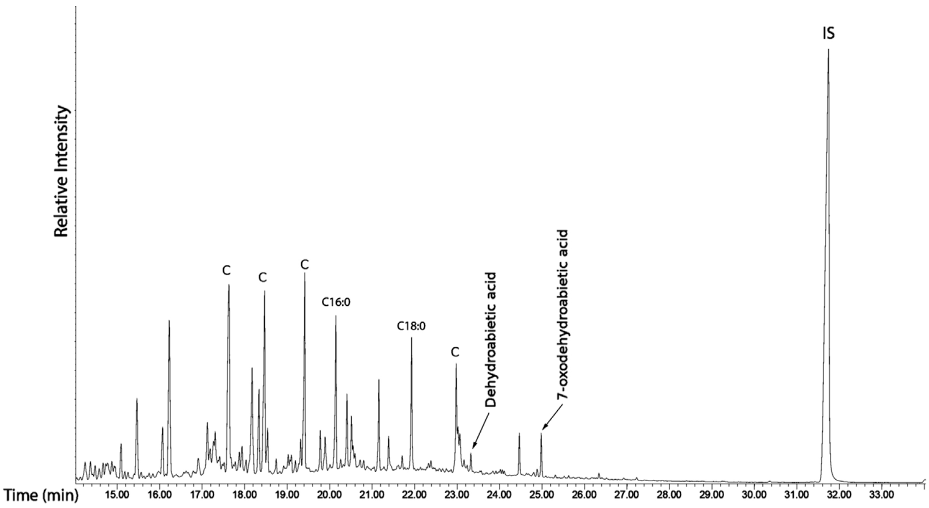

Figure 6 - Partial total ion chromatogram of the lipid extraction of Keay 25.1 sample AIX/66 (193LIP), 'C' represents contaminates.

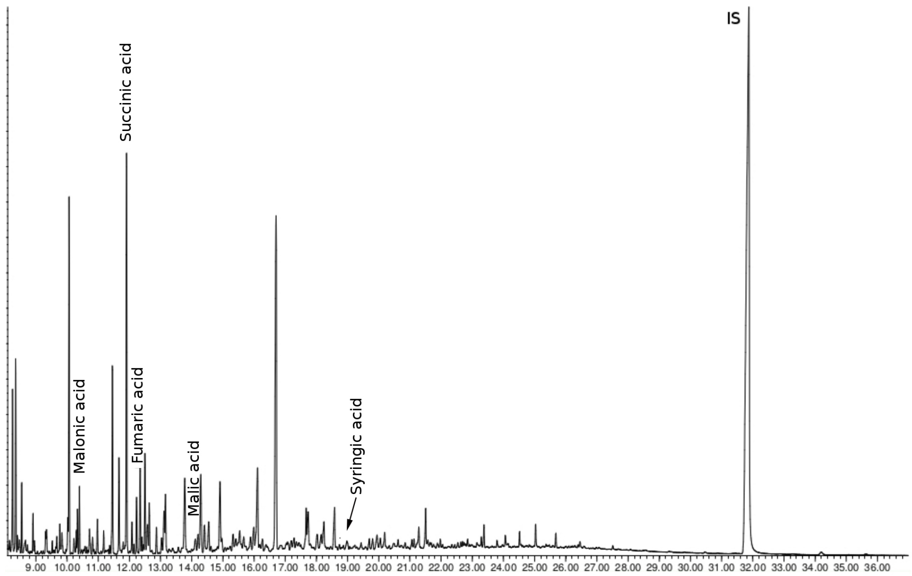

Figure 7 - Partial total ion chromatogram of the wine biomarker extraction of Keay 25.1 sample AIX/68 (117ETO).

Sample No.	malonic acid	fumaric acid	succinic acid	malic acid	vanillin	vanillic acid	tartaric acid	syringic acid	Wine	Animal fats	Pinaceae products
Keay 25.1											
AIX/44	X	X	X	X			X		X		X
AIX/45	X	X	X	X	X	X	X		X		X
AIX/57	X	X	X	X							X
AIX/64	X	X	X	X							X
AIX/68	X	X	X	X				X			X
AIX/70	X	X	X	X							X
AIX/74	X	X	X	X	X	X	X	X	X		X
AIX/78	X	X	X	X	X	X		X			X
AIX/80	X	X	X	X		X	X	X	X		X
AIX/82		X	X			X					X
Keay 25.2											
SN/02	X	X	X	X		X		X			X
SN/07						X		X			X
Keay 25.3											
AIX/54	X	X	X	X						X	X
AIX/59		X	X	X						X	X
AIX/66	X	X	X	X							X
AIX/67	X	X	X		X	X		X			X
Keay 25.3/35B											
SN/15	X	X	X					X			X
SN/16		X	X		X	X		X			X

Table 2 - Principal compounds observed in the wine biomarker extractions of Keay 25.1, Keay 25.2, Keay 25.3 and Keay 25.3/35B ceramic samples.

acid is present sometimes there are no tartaric acid nor other compounds present in wine (i.e. SN/07). Moreover, as Barnard *et al.* (2011) suggested, to be sure that syringic acid comes from wine a multi-step extraction method should be carried out. In the samples analysed, syringic acid is associated with vanillic acid and vanillin. This could suggest that in this case it is not related to the presence of wine but to something different.

It is of interest to note that in samples from the necks of vessels, including the two Keay 25.2 samples, SN/02, SN/07, as well as the two transitional Keay 25.3/35B samples, SN/15 and SN/16 (see 'Keay 25.3' below), there is a very apparent correlation with the detectable presence of syringic acid. In all 4 neck samples, syringic acid was identified. In particular, free syringic acid is found in some plant sources, including in small amounts in lignin. It is therefore possible that the syringic acid in the samples originates from another source such as cork stoppers that may have been used to seal the amphorae.

Keay 25.3 / Africana 3B ceramic samples

Samples from 4 Keay 25.3 amphorae were analyzed, AIX/54, AIX/59, AIX/66, AIX/67, all of which were obtained from maritime contexts. Two of the 4 lipid extractions did not indicate detectable fatty acids apart from common non-diagnostic fatty acids (i.e. $C_{16:0}$ and $C_{18:0}$) and no plant or animal sterols were detected. Like the other amphora types analyzed, the Keay 25.3 samples contained pitch residues indicated by the presence of dehydroabietic acid, 7-oxodehydroabietic acid, retene and methyl dehydroabietate.

Two samples, AIX/54 and AIX/59, demonstrated significantly different lipid profiles from the other samples analyzed. *Pinaceae* products, such as dehydroabietic acid, methyl dehydroabietate and 7-oxodeydroabietic acids, were also identified in both samples, indicating a pitch lining. Both samples contained significant quantities of $C_{18:1.}$ Long-chain saturated fatty acids $C_{20:0}$, $C_{21:0}$, $C_{22:0}$ and $C_{24:0}$ were identified in AIX/59 (Figure 8).

Cholesterol and cholesterol oxidation products were also detected in both samples. Cholesterol is a strong indicator of an animal origin for the content. The fatty acid $C_{22:1}$ was identified in Sample AIX/54. Definitive biomarkers of marine organisms, such as isoprenoid fatty acids, were not observed in the samples from either vessels. The presence of these fatty acids and cholesterol may be the result either of an animal content (possibly fish?) or possibly of the presence of animal fat that was added to the pitch used to line the vessels.

In the wine biomarker extractions of the Keay 25.3 samples, tartaric acid was not identified in any of the samples. Some compounds that are found in wine, other than tartaric acid, were observed in the samples: malic, succinic and fumaric acids were detected in all 4 samples (Table 2). Syringic acid was detected in one sample (AIX/67) as well as vanillin and vanillic acids. As with the Keay 25.1 samples, the quantity of syringic acid was very low. In this case, as for the Keay 25.1 samples, it is difficult to associate the presence of syringic acid with a wine content. While the presence of some acids found in wine were detected in the majority of the samples, the absence of tartaric acid in any of the samples prevents identifying the content as wine, at this time.

In addition to the Keay 25.3 samples, two samples of the transitional type between Keay 25.3 and 35B (SN/15 and SN/16) were analyzed. The lipid extractions indicated biomarkers indicative of a heated Pine product. The wine biomarker analyses identified malonic, fumaric and succinic acids in both samples, as well as syringic and vanillic acids.

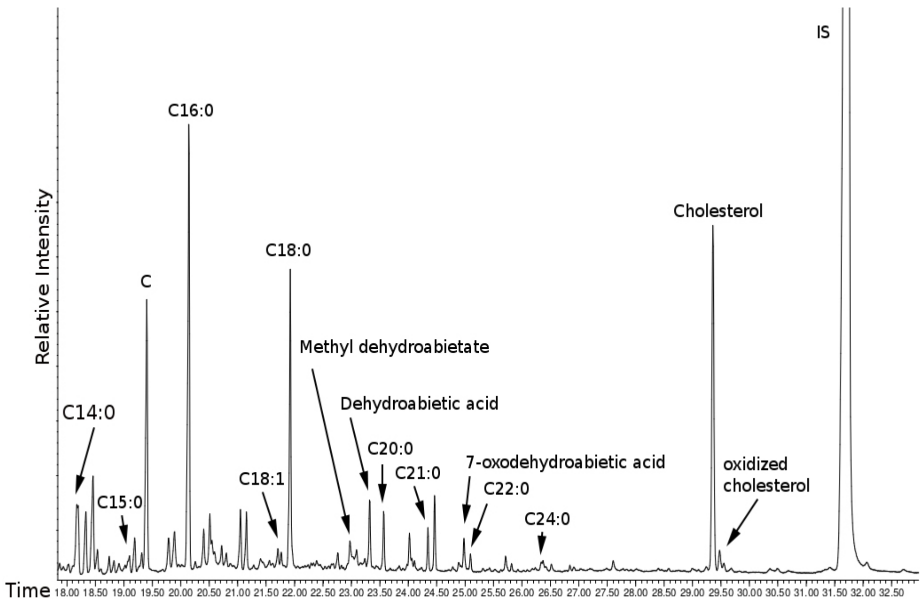

Figure 8 - Partial total ion chromatogram of the lipid extraction from Keay 25.3 sample AIX/59 (180LIP).

CONCLUSION

This first attempt to analyse a consistent series of Keay 25/Africana 3 amphorae, in order to solve the problem of the content of the most widely distributed African amphora type, provides exciting, if mixed, results.

First of all, no sample revealed definitive traces of vegetal oil. The same result has already been emphasised by a first, very limited, series of analyses (Garnier, 2007), but the absence of lipid markers is all the more important since the presence of pitch, as evidenced by chemical analysis, is not by itself a sufficient argument for excluding the assumption that olive oil was the content (Garnier *et al.*, 2011; Pecci & Cau, 2010; Romanus *et al.*, 2009). The absence of evidence for oil implies that the normal variants of Keay 25, i.e. sub-types 1, 2 and 3, did not carry olive oil. We cannot, however, exclude that some other variants of medium-sized African amphorae, for the moment attested in only tiny numbers, could have transported olive oil at the same time.

Second, the presence of discrete, but hardly questionable, markers of wine was confirmed in four samples of the variant Keay 25.1, the oldest variant of this type. These samples come from different find spots (the wrecks of Heliopolis I and Pointe de la Luque B, and the bed of the river Rhône), which gives more weight to this result. This suggests that at least part of the content of the Keay 25.1 was wine. If this were the case, it could explain the particularly wide distribution of this amphora type, from the very end of the 3rd century or the beginning of the 4th century onwards.

The problem of the content of the type Keay 25.3 is much more difficult to solve. The results are contradictory—no definitive evidence of wine (i.e. the detectable presence of tartaric acid) was observed, while some show traces of possible animal fat. Indeed, the typological similarity of this type with the type Keay 35B, which has been presumed to have been for the transport of fish products (Bonifay 2007, 20), as well as the probable origin from Nabeul of most of these amphorae, could provide good arguments for contents based on salted fish, or sauces derived from fish. Identification of the content of this type remains an area where future research needs to be targeted.

Finally, only two samples of Keay 25.2 and/or *spatheion* 1 were analysed. The results are not fully clear, with no definitive evidence of wine (i.e. tartaric acid) in either. The additional evidence of olive pits found in several examples from the Dramont E wreck (one of them analysed, with no specific biomarkers observed other than for pitch). Such confusion could reflect a possible interchangeability of content, these African amphora types becoming from the beginning of the 5th century onwards multipurpose types whose standardisation simplified the loading of cargoes in the holds of ships, as recently proposed (Bonifay forthcoming).

This survey partly confirms some recent hypotheses on the content of fourth-century African amphorae. It is probably too early to interpret this new evidence within the perspective of economic history, until we will be able to provide comprehensive and reliable distribution maps based on a critical review of the abundant but scattered published documentation. On the other hand, the question of a possible link between the appearance of this new generation of amphora at the transition of the third to the fourth century and the *canon uinarius* needs an extensive and detailed survey of the documentation from Rome, Ostia and *Portus*. What is certain is that such residue analyses provide hope for the future of amphora studies, and for a possible reappraisal of our understanding of the movement of goods in the Mediterranean during Roman times.

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