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Carole Mathe
Université d´Avignon et des Pays du Vaucluse
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Lebanese University
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Catherine Vieillescazes
Université d´Avignon et des Pays du Vaucluse
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THE STUDY OF NABATAEAN ORGANIC RESIDUES FROM MADÂ'IN SÂLIH, ANCIENT *HEGRA*, BY GAS CHROMATOGRAPHY – MASS SPECTROMETRY*

C. MATHE,[1] P. ARCHIER,[1] L. NEHME[2] and C. VIEILLESCAZES[1]

[1] *Laboratoire de Chimie Bio-Organique et des Systèmes Moléculaires Vectoriels, Faculté des Sciences, Université d'Avignon et des Pays de Vaucluse, 33 rue Louis Pasteur, F-84000 Avignon, France*
[2] *UMR 8167, Orient & Méditerranée*

Four Nabataean samples collected in some of the monumental tombs of Madâ'in Sâlih, ancient Hegra*, in Saudi Arabia, have been studied by gas chromatography coupled with mass spectrometry. These samples are textile fragments that are either covered with a black layer or bound together with some black amorphous substance. Fatty acids and triterpenoic compounds were detected. Eight triterpenic compounds were identified: ursa-9(11),12-dien-3-ol, ursa-9(11),12-dien-3-one, olean-9(11),12-dien-3β-ol, β-amyrone, β-amyrin, α-amyrone, α-amyrin and lupeol. The resinous chemical composition and these pentacyclic alcohols, in considerable proportion, indicate a resin of the Burseraceae family, possibly of the genus* Canarium.

KEYWORDS: MADÂ'IN SÂLIH, NABATAEAN SAMPLES, AMYRINS, TRITERPENOIDS, RESIN, GAS CHROMATOGRAPHY, MASS SPECTROMETRY

INTRODUCTION

Madâ'in Sâlih, ancient *Hegra*, in the Saudi Arabian Hijâz, belonged in Antiquity to the Nabataean kingdom, whose capital was Petra, in Jordan. *Hegra* is the southernmost site of this kingdom, which—in the first century BC—controlled most of the area between Damascus and the Hijâz from north to south and from the Negev to the Syro-Arabian desert from west to east. Ancient sources reveal that the Nabataeans were already installed in Petra at the end of the fourth century BC. They are described by Diodorus of Sicily as nomad pastoralists, who were involved in the long-distance caravan trade of spices, aromatics and frankincense, which they conveyed overland from South Arabia to the Mediterranean area. They took advantage of this profitable trade to a considerable extent and this, added to their progressive sedentarization, led them to develop urban centres such as Bosra in Syria, Petra in Jordan and *Hegra* in Arabia. A 'Nabataean culture', characterized by a religious pantheon, a specific coinage, a distinctive pottery and a particular script, as well as by identifiable carving techniques, hydraulic practices, architectural decoration habits and so on, blossomed in this region of the Middle East between the first century BC and the beginning of the second century AD. The Nabataean kingdom remained independent until AD 106, when it was annexed by Rome, under the reign of Trajan, in order to form the new Roman province of Arabia. The Nabataean presence in the region is therefore attested to for a period of 400 years at least, and probably much longer. Indeed, individuals who bear good Nabataean names are attested to in Petra until the sixth century AD, in documents written in Greek.

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The Madâ'in Sâlih archaeological project, which is financially supported mainly by the French Ministry for Foreign Affairs, undertook from 2002 to 2005, under the aegis of the Saudi Arabian Deputy Ministry for Antiquities and Museums, systematic archaeological and epigraphic surveys as well as a series of thematic studies, the aim of which was a better understanding of the site, of its urban organization, of the phases of its development and of the evolution of its population through the ages (Nehmé *et al*. 2006). This site is one of the three Nabataean sites, along with Petra and al-Bad', the latter also in Saudi Arabia, that contain rock-cut monuments, the most famous of which are hundreds of monumental tombs bearing a façade decorated with crowsteps, pediments, pilasters and various sculpted elements. Until the work done in recent years, Madâ'in Sâlih was known mainly through the work of A. Jaussen and R. Savignac, from the French Biblical and Archaeological School in Jerusalem, at the beginning of the 20th century (see Jaussen and Savignac 1909–14). Note that a joint Saudi–French archaeological expedition was due to start large-scale excavations on the site in January 2008.

During the 2004 season of the project, a pottery survey was undertaken at Madâ'in Sâlih. The ceramicists of the team collected in the tombs, among other objects such as pottery sherds, parts of wooden coffins, leather and rope fragments and a slab painted with Nabataean letters, a significant number of textile fragments. A preliminary description of the latter is published in the Saudi Arabian archaeological journal, *Atlal* (Augé *et al*. in press). The technical analysis of these fragments was carried out by P. Dal-Prà and what follows is a summary of her conclusions. Among the 40 textile fragments that have been selected and numbered, one fragment at least was dyed. The majority (75%) belong to the toile type and are more or less tightly woven, one of them being interlaced with leather, while the rest (25%) are plain toile derivatives. Five fragments of toile bear distinctive marks such as knot, pattern, seam and stitch holes and eight fragments are covered with or bound together through a blackish substance that intrigued the archaeologists. The plain toile derivatives are fragments of louisine with double warp threads, 20 mm wide, which can be interpreted as ribbons used for tying or binding. The identification of the textile fibre was done by Dominique De Reyer from the Laboratoire de Recherche des Monuments Historiques in Paris. He identified the textiles as being made of cotton, linen and of a hard vegetable fibre that probably belongs to a class of plants known as monocotyledons, such as palm trees and grasses, amongst others.

The fragments have been collected inside three monumental tombs of the site, IGN 20 and 28, which belong to the so-called Qasr al-Bint necropolis, and IGN 87, which belongs to the Jabal al-Khraymât necropolis. Only tomb IGN 87 is clearly dated, by the Nabataean inscription carved on its façade, to the years AD 71–2. IGN 20 is one of the largest tombs on the site, but despite the presence of a Nabataean inscription on its façade, it is not dated. Finally, tomb IGN 28 belongs to a particular type of tomb that does not bear a monumental façade. Its opening is carved several metres above the ground and it is certainly earlier than the neighbouring tombs, which are carved at ground level.

Very few Nabataean sites have yielded textile fragments, because in order to be preserved, this material required particularly dry climatic conditions. A few fragments have been discovered in Petra and Khirbat adh-Dharîh, but the most important site regarding this category of artefact is Khirbat Qazône, south of the Dead Sea in Jordan. A large number of textile fragments have been discovered there and have been published in several contributions (see Granger-Taylor 2007). Most of them are made of wool, but some of them are of cotton or linen. They belong to tunics, coats, scarfs and so on. However, it is probable that the textiles were used to wrap dead bodies. In this context, the presence of organic matter on some of the textiles from Madâ'in Sâlih may indicate that they were submitted, prior to their use as shrouds, to a special preparation aimed possibly at a better preservation of the body. In this view, four samples have been selected for analysis. They presented the following characteristics:

- IGN 20-1: a toile fragment covered with coarse blackish matter on one side and with a smooth coating on the other side.
- IGN 20-9: a toile fragment covering a layer of black matter.
- IGN 87-2 and 87-3: overlaid toile fragments bound together with black matter.

The Laboratoire de Chimie Bio-Organique et des Systèmes Moléculaires Vectoriels of the University of Avignon specializes in the study of natural substances used in cultural heritage contexts (Mathe *et al*. 2004a,b, 2007). Vegetal di- and triterpenic resins have been used since ancient times because they are available throughout the world and have particular properties that are useful for a wide range of applications. Indeed, they are employed in adhesives, incenses, painting varnishes, waterproofing and caulking agents, and so on. Therefore, it is not surprising that significant resin trade networks were established and that such resins are often found in organic remains on archaeological objects. However, their identification has always been difficult and often remains uncertain, the reason being that these substances are damaged in the course of time. Therefore, in order to identify them, it is necessary to determine the specific chemical markers of these vegetable species and/or their corresponding degradation compounds.

The aim of this study is to characterize the black amorphous substances covering the toile fragment. This analysis is archaeologically useful because it gives information on the techniques used by the Nabataeans to prepare dead bodies before they were buried in the tombs. Gas chromatography coupled with mass spectrometry (GC–MS) analyses were performed in order to detect possible organic compounds present in these samples and determine their chemical composition.

EXPERIMENTAL

Materials

Solvents and reagents, from Merck (Darmstadt, Germany), were all of analytical grade. For this chromatographic study, we have used the following commercial triterpenic standards: α-amyrin, β-amyrin and lupeol from Extrasynthese (Genay, France). From α- and β-amyrin, we have obtained α- and β-amyrone by a classical oxidation with PCC (pyridinium chlorochromate) in dichloromethane. Elemolic acids were isolated and characterized in our laboratory from frankincense. The methanolic extracts of a commercial ‘Eritrean-type’ frankincense resin (Les Encens du Monde-Asie Concept, Castelnau-le-Lez, France) were filtered and fractionated by liquid chromatography using silica gel (Merck). Fractions eluted respectively with EtOAc/cyclohexane 50/50 were further purified by HPLC (refractometric detection) on a C-18 reverse-phase column (Merck, Superspher 100 RP-18e, 250 × 4 mm). All structures were characterized on the basis of chemical and spectral evidence, including two-dimensional NMR experiments (COSY and NOESY ^{1}H-^{1}H, HMQC and HMBC) and mass spectrometric techniques (EI and HR–MS).

Sample preparation

A 5 mg amount of the sample analysed was trimethylsilylated with a solution consisting of pyridine (0.5 ml), hexamethyldisilazane (0.45 ml) and chlorotrimethylsilane (0.3 ml). The reaction was conducted at room temperature for 30 min and the solution was then dried with a stream of nitrogen while heating (<40°C). Thereafter, the residue was immediately dissolved in 0.6 ml of diethyl ether. Each sample was treated in triplicate.

Gas chromatography – mass spectrometry

GC–MS analysis was carried out in a Varian Saturn 3900 gas chromatograph, with a Varian 1177 injector, coupled with a Varian 2100 T ion trap mass spectrometer (Varian, Walnut Creek, CA, USA). The gas chromatograph was equipped with a 30 m × 0.25 mm I.D. fused-silica capillary column coated with a 0.25 μm film of poly (5% phenyl, 95% dimethylsiloxane): CP-Sil 8 CB Low Bleed/MS (Varian). The MS electron multiplier voltage was set at 1400 V and an ionization time of 25 000 μs was used, running in the electron impact (EI) mode, with transfer line, ion trap and manifold temperatures of 300°C, 200°C and 50°C, respectively. The mass spectrometer was set to scan 40–650 m/z with an ionizing voltage at 70 eV. Samples were injected (1 μl) with a splitless mode and the injector temperature was set to 250°C. A continuous flow rate of 1 ml min^{-1} of chromatographic grade helium was used. The column oven was initially heated to 50°C and this was held for 2 min after injection, followed by temperature ramping at 8°C min^{-1} up to 250°C, and 250–350°C at 3°C min^{-1}. No hold time was performed at the upper limit. The total run time was approximately 61 min. Identification of common fatty acids was performed using the NIST'98 mass spectral database.

RESULTS AND DISCUSSION

Analysis of trimethylsilylated pieces of the archaeological samples was performed using the described procedure. The four samples show very similar chromatograms regarding the various chemical families identified (Fig. 1). GC–MS analyses were thus used to determine the chemical composition of the substance present on the samples. The chromatograms obtained have two distinguishable areas that correspond to fatty acids on the one hand and to triterpenic compounds on the other. The constituents eluting between 14 and 30 min were attributed to fatty acids and their derivatives. All of them were clearly identified by a mass spectrometry library search (Table 1). Common fatty acids were identified in all of the samples, which contained the same chemical population in variable proportion. Samples IGN 20-1 and 20-9 contained only saturated fatty acids, whereas samples IGN 87-2 and 87-3 contained a large proportion of saturated fatty acids, but also one unsaturated derivative, oleic acid (C18:1). In all of the samples, palmitic acid (C16:0) is largely evident. The qualitative and quantitative composition of fresh oils or fats is significant with regard to their origin, animal or vegetable (Karleskind 1992). However, this distinction is not so easy when the fat substances are damaged, either naturally or under anthropic action. Indeed, saturated fatty acids are relatively stable through time, whereas unsaturated fat substances are much less so. Some analyses have used experimentally determined ratios of common fatty acids in order to determine whether certain types of containers from a particular region were associated with such broad resource categories as meat, fish, roots, greens or seeds (Malainey *et al*. 1999; Eerkens 2005). There are several reasons for the difficulties in interpreting these ratios: the ubiquity of most fatty acids, multiple sources of mixtures, and finally variations in the way fatty acids are damaged (e.g., unsaturated fatty acids, such as C18:1, are damaged much more quickly than saturated ones, such as C16:0). One of the main methods used nowadays for the identification of drying oils is based on the statistical evaluation of some selected ratios between fatty acids (e.g., azelaic/

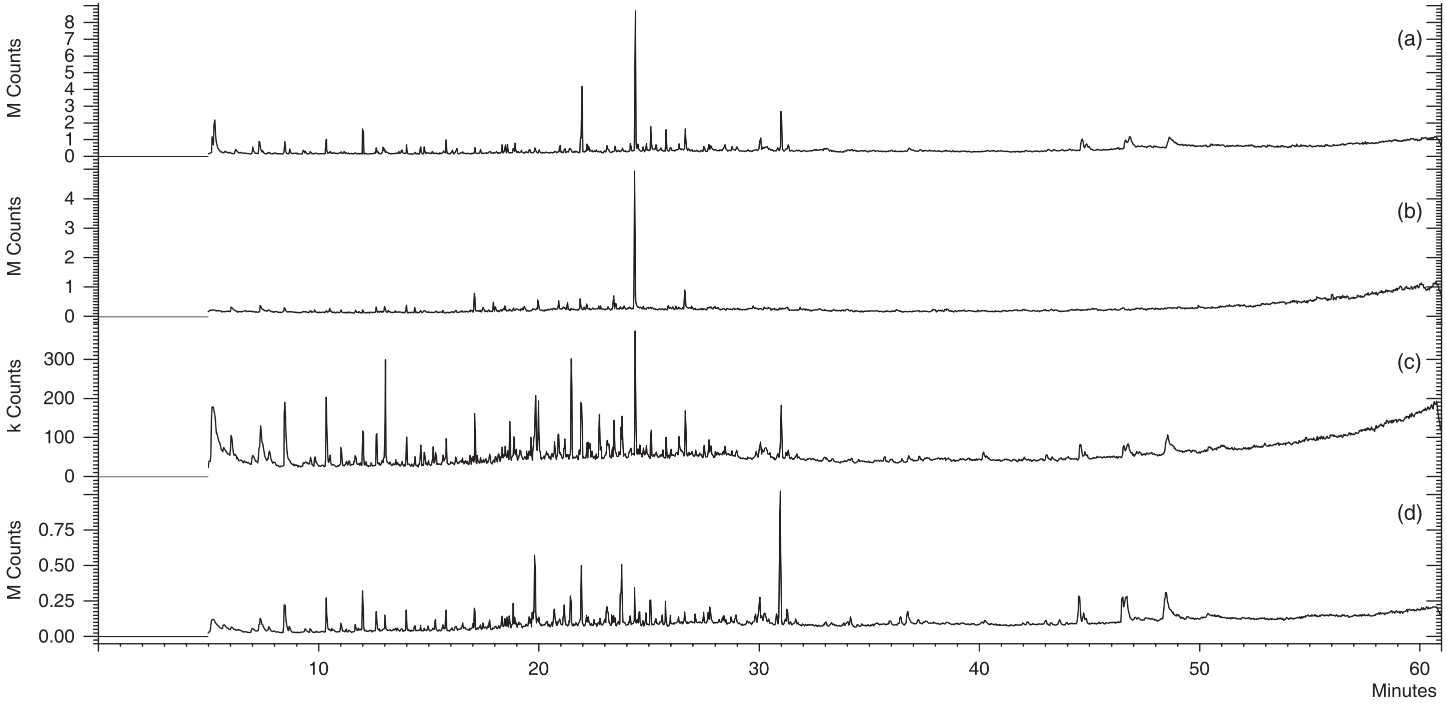


Figure 1 *The total ion current (TIC) chromatograms of the archaeological samples: (a) IGN 20-1; (b) IGN 20-9; (c) IGN 87-2; and (d) IGN 87-3.*

Table 1 *The chemical composition of fatty acids in relative percentages*

No.	t_R *(min)*	*Compounds*	*% IGN 20-1*	*% IGN 20-9*	*% IGN 87-2*	*% IGN 87-3*
1	14.64	Nonanoic acid	3.8	1.1	7.2	4.9
2	16.23	Capric acid	Traces	n.d.	Traces	Traces
3	19.20	Lauric acid	Traces	Traces	n.d.	n.d.
4	21.26	Azelaic acid	Traces	Traces	5.4	30.9
5	21.91	Myristic acid	6.8	7.5	Traces	Traces
7	24.38	Palmitic acid	74.4	80	57.9	40.5
8	26.36	Oleic acid	Traces	Traces	8.6	7.0
9	26.66	Stearic acid	15.0	11.4	20.9	16.7

n.d., Not detected.

palmitic acid and palmitic/stearic acid). Several analytical procedures for the analysis of drying oils in works of art have been presented in a few contributions (Mills 1966; Colombini *et al*. 1999; Tsakalof *et al*. 2006).

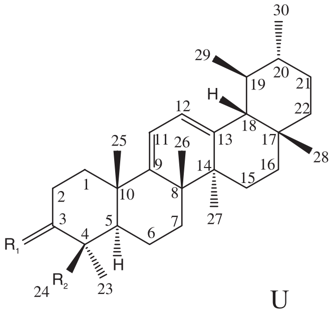
Azelaic acid is present in a considerable proportion in the sample collected in IGN 87. This compound is produced in small quantities from the oxidation of Δ9 fatty acids such as oleic (18:1), linoleic (18:2) and linolenic acids (18:3) (Passi *et al*. 1993), which are typically found in high proportion in seed oils (Buonasera 2007), in rancid oils and in ancient residues (Banks and Hilditch 1933; Regert *et al*. 1998). Moreover, the predominance of palmitic acid compared to stearic acid is an argument in favour of a vegetable oil.

The components characterized by retention times ranging from 40 to 45 min were identified as trimethylsilylated triterpenic derivatives (Tables 2 and 3). The characterization and/or the identification of the resinous parts of the samples are based on the triterpenic population corresponding to the most stable fraction. Therefore, due to its major analytical advantage for characterizing triterpenic resins, our study focused on the triterpenoid fraction. Likewise, because of their low volatility, the triterpenoids are present in larger amounts than the mono- and sesquiterpenoids, and they are thus more indicative of the presence of triterpenic resins in aged materials.

The GC–MS analysis carried out on the archaeological samples reveals that the triterpenic fraction corresponds to pentacyclic compounds with oleanane, ursane and lupane skeletons. All of the analysed samples have the same triterpenic composition, which means that the same resinous material has been used for sample preparation. In line with previous work (De la Cruz-Cañizares *et al*. 2005) and a mass spectrometry library search, one of the first compounds, which appeared at 42.04, 43.04 and 43.27 min, respectively, is tentatively assigned to ursa-9(11), 12-dien-3-ol (**I**), ursa-9(11),12-dien-3-one (**II**) and olean-9(11),12-dien-3β-ol (**III**) (Table 2 and Fig. 2). Other triterpenic compounds have been clearly identified by MS database and standard molecules: β-amyrone (**IV**), β-amyrin (**V**), α-amyrone (**VI**), α-amyrin (**VII**) and lupeol (**VIII**) (Table 3). They generally occur in the chemical composition of the main vegetal resins attested to in archaeology (Pollard and Heron 1996). The eluted triterpenic compounds noted (**IX–XIII**) are not clearly identified. It can also be noted that A-neooleana-3(5),12-diene is not present. The latter compound can be formed from β-amyrin as a result of thermal alteration, which means that the absence of this molecule could indicate the absence of this chemical oxidation process (De la Cruz-Cañizares *et al*. 2005).

Table 2 *Chemical structures of dienoic triterpenic compounds*

Structure types:



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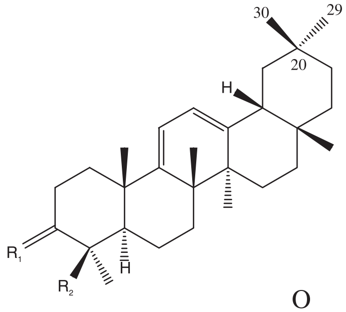

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No.	*Systematic name*	*Structure type*	*R_1*	*R_2*	*t_R (min)*	*Molecular weight*	*Mass spectrum*
I	Ursa-9(11),12-dien-3-ol, TMS derivative	U	-OTMS	CH_3	42.04	496	73(100)-147(34)-189(26)-207(22)-255(23)-295(11)-391(21)-496(73)
II	Ursa-9(11),12-dien-3-one	U	O	CH_3	43.04	422	55(19)-73(55)-133(24)-207(25)-255(38)-269(28)-281(19)-422(100)
III	Olean-9(11),12-dien-3-ol, TMS derivative	O	α-H, β-OTMS	CH_3	43.27	496	73(49)-147(18)-189(13)-207(10)-255(13)-391(11)-281(12)-496(100)

TMS, trimethylsilylated.

Generally, characterization and/or identification of a resin is based on the research for specific chemical markers of a family, a genus or a botanical species. Thus, abietic acid is specific of the Coniferae family and boswellic acids characterize the genus *Boswellia* (frankincense) (Mills and White 1994). In this present study, the triterpenic population of the resin does not contain chemical markers from a specific resin, but the presence of these compounds indicates a resin belonging to the Burseraceae family. Moreover, according to previous works, compounds **I** to **III** are not present in raw resinous material, but are the result of oxidation processes occurring on α- and β-amyrin during damage through time (Oros *et al.* 2002; De la Cruz-Cañizares 2005). In this context, β-amyrin drastically decreased from the raw resin, whereas β-amyrone and olean-9(11),12-dien-3β-ol appeared, amongst other compounds: β-amyrin could undergo an oxidation reaction during degradation process. However, it is important to note that α- and β-amyrin were characterized in fossil resins, which presupposes that these compounds present sufficient stability to remain unchanged over a long period of time and therefore can be used as markers.

According to the chemical triterpenic composition of the archaeological samples, it is possible that the black amorphous substance corresponds to elemi resin, a generic term applied to a large number of resins from the Burseraceae family, with a significant amount of amyrins. Several botanical genera belonging to the Burseraceae family exude elemi resins. This is the case of *Canarium* spp., *Bursera* spp., *Amyris* spp. and *Protium* spp. Nowadays, this term seems to be restricted to Manila elemi from *Canarium luzonicum* growing in the Philippines (Mills and White 1994). However, *Canarium* species are largely distributed from northern Australia through East and South-East Asia (including India) and Africa, while the other

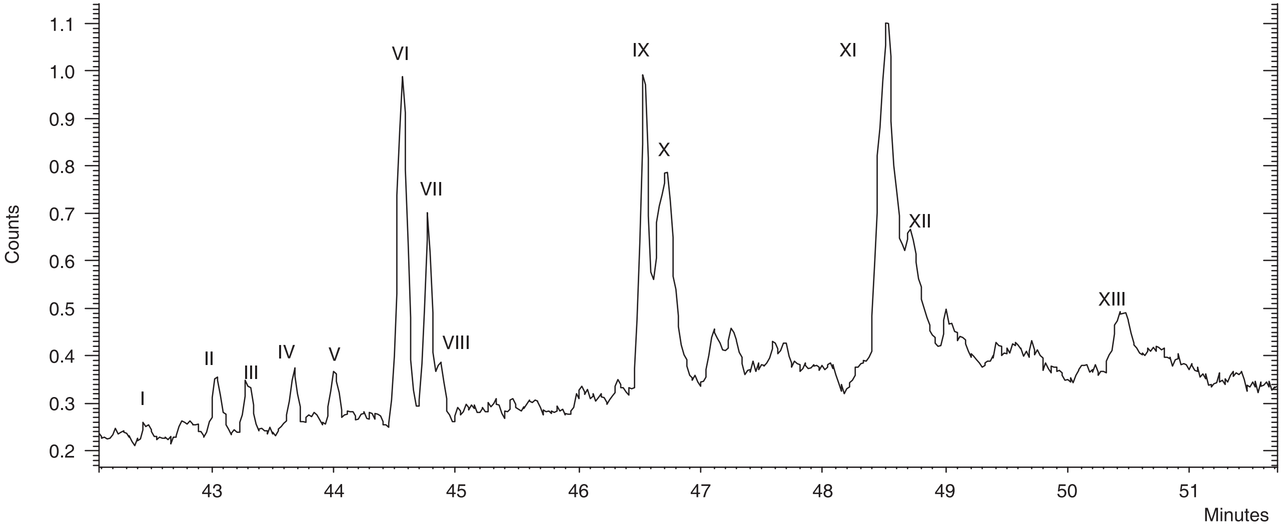
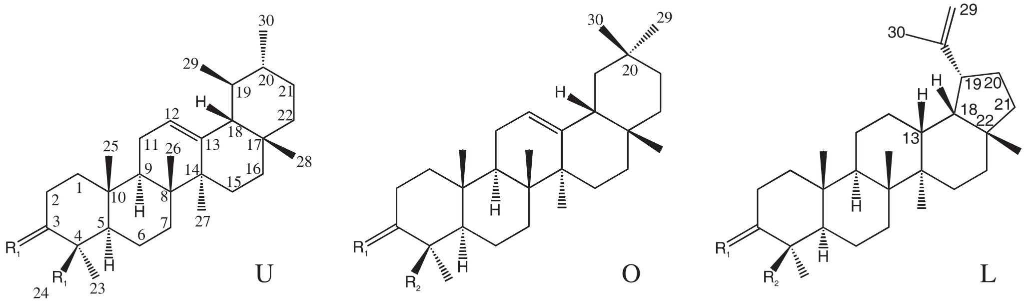


Figure 2 *The gas chromatogram of the triterpenic area of IGN 20-9.*

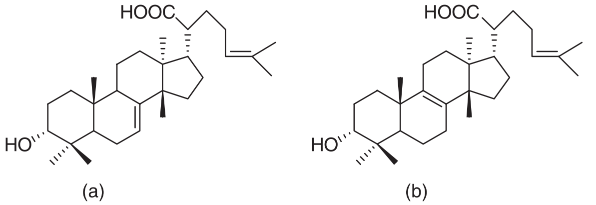
Table 3 *Chemical structures of triterpenic compounds*

Structure types:



No.	*Triterpenes*		*Structure type*	*R₁*	*R₂*	t_R *(min)*	*Molecular weight*	*Mass spectrum*
	Common name	*Systematic name*						
IV	β-amyrone	Olean-12-en-3-one	O	O	CH_3	43.67	424	55(20)-73(38)-107(22)-147(24)-189(40)-203(100)-218(62)-409(9)-424(9)
V	β-amyrin, TMS derivative	3β-hydroxy-olean-12-en-3-ol, β-OTMS ether	O	α-H, β-OTMS	CH_3	44.01	498	73(80)-107(27)-147(35)-189(60)-203(100)-218(70)-408(5)-483(5)-498(6)
VI	α-amyrone	Urs-12-en-3-one	U	O	CH_3	44.56	424	55(31)-73(24)-107(47)-147(46)-189(57)-203(63)-218(100)-409(25)-424(27)
VII	α-amyrin, TMS derivative	3β-hydroxy-urs-12-en-3-ol, β-OTMS ether	U	α-H, β-OTMS	CH_3	44.77	498	73(54)-107(23)-147(36)-189(62)-203(43)-218(100)-408(4)-483(2)-498(9)
VIII	Lupeol, TMS derivative	3β-lup-20(29)-en-3-ol, β-OTMS ether	L	α-H, β-OTMS	CH_3	44.87	498	73(9)-107(35)-147(28)-189(100)-203(59)-218(30)-408(12)-483(10)-498(16)

genera occur mainly in Central and South America. African elemi is exuded from *Boswellia frereana* (Strappaghetti *et al.* 1982), an endemic species from northern Somalia. The majority of elemi resins contain a large proportion of pentacyclic alcohols. The α- and β-amyrins, originally isolated and named after *Amarys* resins, have been identified as the main components of the triterpenoid fraction of elemi resins. The α- and β-elemolic acids, which correspond respectively to 3-α-hydroxytirucall-7,4-dien-1-oic and 3-β-hydroxytirucall-8,4-dien-1-oic acids (Fig. 3) were initially isolated from Manila elemi and are not systematically present in elemi resin (Cotterell *et al.* 1970; Tessier *et al.* 1982; Guang-Yi *et al.* 1988; Mora *et al.* 2001). These acids were also characterized in resins from the Burseraceae family, particularly in the genus *Boswellia* (Pardhy and Bhattacharyya 1978). Thus, they are not specific chemical biomarkers of the elemi resin. These elemolic acids, from the tirucallane group, were not found in any of the samples analysed.



(a) (b)

Figure 3 *The chemical structure of (a) α- and (b) β-elemolic acids.*

Copal resin has a chemical composition that is similar to those of the hopane skeleton compounds. According to mass spectrometry, characteristic fragments of hopanes are absent (Ogunkoya 1981). The archaeological samples analysed do not contain copal resin. Moreover, 3-epi-lupeol (3-α-lup-20(29)-en-3-ol), the major constituent of African elemi, is not detected, which means that we can exclude the presence of *Boswellia frereana* (Mathe *et al.* 2004b).

CONCLUSIONS

The study of archaeological organic samples is an analytical challenge because of the wide range of classes of molecules that are present, and because of the damage that has affected the products through the action of time. The result of four Nabataean samples shows the presence of a mixture of fatty acids and triterpenic compounds. The fat substance composition probably corresponds to a vegetable oil. The presence of α- and β-amyrins is evidence for a resin belonging to the Burseraceae family, and—excluding any other chemical markers—the occurrence of these pentacyclic alcohols in considerable proportion seems to indicate an elemi resin. It is difficult to identify the exact botanical origin of this resinous material, because there are a great number of corresponding species. However, African elemi is compatible with its chemical composition, while the *Bursera*, *Amyris* and *Protium* families of elemi only grow in America and can therefore be excluded. All in all, the results of the analysis seem to indicate that the resinous material contained in these archaeological samples belongs to the genus *Canarium*. It is important to note that, to our knowledge, elemi resins have not been reported in association with archaeological samples. Therefore, these results provide more information about the techniques used by the Nabataeans to prepare dead bodies during their funeral practices.

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