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'Choicest unguents': molecular evidence for the use of resinous plant exudates in late Roman mortuary rites in Britain

R.C. Brettell^{a,*}, E.M.J. Schotsmans^{a,b}, P. Walton Rogers^c, N. Reifarth^d, R.C. Redfern^e,
B. Stern^a, C.P. Heron^a

^a Archaeological Sciences, Life Sciences, University of Bradford, Richmond Road, Bradford, West Yorkshire, BD7 1DP, UK

^b PACEA UMR5199, Laboratoire d'Anthropologie des Populations Passées et Présentes, Université Bordeaux 1, Bât. B8, Avenue des Facultés, 33405, Talence, France

^c The Anglo-Saxon Laboratory, Bootham House, 61 Bootham, York, YO30 7BT, UK

^d Institut für Archäologie, Denkmalkunde und Kunstgeschichte, Universität Bamberg, Am Kranen 14, 96045, Bamberg, Germany

^e Centre for Human Bioarchaeology, Museum of London, 150 London Wall, London, EC2Y 5HN, UK

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ABSTRACT

Resinous substances were highly prized in the ancient world for use in ritual contexts. Details gleaned from classical literature indicate that they played a significant role in Roman mortuary rites, in treatment of the body and as offerings at the tomb. Outside of Egypt, however, where research has shown that a range of plant exudates were applied as part of the mummification process, resins have rarely been identified in the burial record. This is despite considerable speculation regarding their use across the Roman Empire.

Focusing on one region, we investigated organic residues from forty-nine late Roman inhumations from Britain. Using gas chromatography–mass spectrometry and the well-attested biomarker approach, terpenic compounds were characterized in fourteen of the burials analysed. These results provided direct chemical evidence for the presence of exudates from three different plant families: coniferous Pinaceae resins, Mediterranean *Pistacia* spp. resins (mastic/terebinth) and exotic *Boswellia* spp. gum-resins (frankincense/olibanum) from southern Arabia or beyond. The individuals accorded this rite had all been interred with a package of procedures more elaborate than the norm.

These findings illuminate the multiplicity of roles played by resinous substances in Roman mortuary practices in acting to disguise the odour of decomposition, aiding temporary soft-tissue preservation and signifying the social status of the deceased. Nevertheless, it was their ritual function in facilitating the transition to the next world that necessitated transportation to the most remote outpost of the late Roman Empire, Britain.

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1. Introduction

For centuries there has been speculation about the use of resinous substances in Roman mortuary contexts (Chioffi, 1998; Pearce, 2013). This has been fuelled by the brief insights provided by classical authors (Hope, 2009: 65–85) and unsubstantiated reports of scented substances in elaborate cremation and inhumation burials (e.g. Dunkin, 1844: 91–97; Gage, 1834, 1836; Waugh, 1962; Webster, 1947). As a result, a relationship between interment in

stone sarcophagi or lead-lined coffins, plaster body-coatings and the potential use of resins was proposed in the 1970s. The additional level of investment represented, which took the form of increased protection of the cadaver, appeared to be aimed at some degree of body preservation. Inferential links with the doctrine of resurrection and the presence of elements of this 'package' in late Roman burial contexts in north Africa and the Rhineland led to an association with the spread of Christianity (Ramm, 1971; Sparey Green, 1977). It was also suggested that this rite may have had gender correlates (Chioffi, 1998: 24–28) or been a signifier of social status (Philpott, 1991: 92; Toller, 1977: 2). The continuing debate over the nature of these burials has focussed on two key aspects, the botanical source and ritual significance of any resinous

* Correspondence. Tel.: +44 (0) 1274 234592.

E-mail address: r.c.brettell@student.bradford.ac.uk (R.C. Brettell).

materials present. Along the way, a mythology has developed through repetition of earlier suppositions despite the absence of any chemical confirmation for the use of resins. This state of affairs, in conjunction with recent continental finds, prompted us to undertake a molecular-based investigation of late Roman period burials from Britain.

2. The archaeological context

Natural resins are complex materials composed of a range of compounds of varying volatility (Howes, 1949: 87–89). The majority of these are terpenic moieties, polycyclic hydrocarbons with oxygen-containing functional groups (terpenoids) and their derivatives, which are classified according to the number of C₅ isoprene (CH₂=C(CH₃)CH=CH₂) units from which they are formed (Pollard and Heron, 2008: 241–242). Those of diagnostic value are primarily the higher molecular mass di- and triterpenoids (with twenty and thirty carbons, respectively) as these are limited in their botanical origins due to the different biosynthetic pathways employed by various plant families (Langenheim, 2003: 24–30). Fortunately, they also comprise the more degradation resistant water-insoluble fraction of 'true' resins and certain gum-resins and so can survive in the archaeological record (Colombini and Modugno, 2009).

Although the tissues of a vast array of species contain terpenic compounds, the number of trees and shrubs that exude aromatic resinous secretions in any abundance is more restricted (Howes, 1949: 86–87; Langenheim, 2003: 51–98). Reviews of those native to the Mediterranean region can be found in Howes (1950) with a discussion of their chemistry in Serpico (1996). With regards to the Roman period, these findings can be supplemented by information from classical texts concerning the properties and exploitation of these products (Hort, 1980: 223–247), ancient trade networks (Huntingford, 1980: 19–57; Rackham, 1968: 31–95), and the use of resinous substances as part of Roman mortuary rites (Groom, 1981: 1–15; Hope, 2007: 97–99, 111–119). The latter describe the anointing of the deceased with perfumed unguents (Fowler and Fowler, 2007: 511; Sullivan, 1977: 87–88), the heaping of aromatic substances on the pyre and within the tomb (AKJV, 2011; John 19: 39–42; Nagle, 2004: 87), the embalming of certain individuals (Grant, 1971: 384; Nagle, 2004: 157) and the burning of vast quantities of incense as part of funerary rituals (Rackham, 1968: 61–62). On a more practical level, these "*choicest unguents* [were also used] *to arrest the progress of decay*" (Fowler and Fowler, 2007: 511) and mask the odour of decomposition (Hope, 2009: 71–74). These texts imply that, while many plant exudates were employed in medicinal preparations or perfumery (Gunther, 1959: 5–92; Hort, 1980: 247–321), a more limited palette was deemed appropriate for the ritual sphere. Definitive evidence for this extensive use of resins has, however, rarely been recovered from the archaeological record (Pearce, 2013).

Organic residue analysis of embalming materials has previously been successful in establishing the role played by resinous substances in Ancient Egyptian mummification processes (Buckley and Evershed, 2001; Serpico and White, 2000; Tchaplá et al., 2004). Even in these complex mixtures, marker compounds have enabled the identification of a range of aromatic exudates. This research has shown that a modified form of this body treatment continued into the Roman period in Egypt with an increased use of imported conifer resins (Buckley and Evershed, 2001; Corcoran and Svoboda, 2010). Such findings prompted the analysis of visible residues from exceptional mortuary contexts elsewhere in the Roman Empire with biomarkers denoting the utilization of both diterpenoid coniferous products (Ascenzi et al., 1993; Devières et al., 2010; Papageorgopoulou et al., 2009; Reifarth, 2013), triterpenoid

Pistacia spp. (mastic/terebinth) resins (Bruni and Guglielmi, 2005; Devières, 2008: 115–131; Reifarth, 2013) and *Boswellia* spp. (frankincense/olibanum) gum-resins (Devières, 2008: 115–131). Most recently, brittle orange materials associated with the remains of an infant inhumed near Arrington, Cambridgeshire, UK have also been shown to be fragments of resin from the genus *Pistacia* (Brettell et al., 2013).

These finds display a number of commonalities which closely resemble the class of late Roman burial proposed by Ramm (1971). The deceased were generally interred in stone sarcophagi or lead-lined coffins and accompanied by high quality gravegoods (Ascenzi et al., 1993; Bruni and Guglielmi, 2005). Most appear to have been wrapped in some form of shroud and several were encased in plaster (Reifarth, 2009). Evidence for garments of damask silk, murex ('Tyrian') purple-dyed wool and spun gold threads has also been recovered (Devières, 2008: 181–182; Papageorgopoulou et al., 2009; Reifarth, 2013). Indeed, there is a striking correlation between contemporary descriptions of elite Roman funerary rites and these archaeological discoveries: "*Sabaeen frankincense, the Indian crop for burning, incense robbed from Palestine's temples ... and beads of myrrh ... [her body lies on a bier of] Chinese silk beneath the shade a purple canopy provides*" (Nagle, 2004: 157).

Thus, to test the hypothesis that the use of exotic resinous exudates formed part of mortuary practices introduced to Britain in the Roman period, samples were selected from both 'package' burials and normative inhumations. These were analysed using gas chromatography–mass spectrometry (GC–MS) and the well-attested biomarker approach (Evershed, 2008). The rationale behind this process is that suites of molecular markers have the potential to permit identification of source materials. With regards to plant exudates, it is the higher molecular mass terpenic components in the resin fraction that are both degradation resistant and diagnostic. These compounds may, therefore, survive over archaeologically relevant time periods and provide evidence for any resins utilized. Where our methodology differs is in the nature of the samples selected. Previously, research has only been conducted where visible residues or resin fragments were observed. Although these materials were sampled when present, the focus here was on the potential survival of biomarkers within the heterogeneous detritus, best described as 'grave deposits', found in the base of the more substantial containers or closely associated with the human remains.

Mortuary rites in late Roman Britain are particularly suited to this approach as they are sufficiently defined in both space and time to enable a systematic study to be undertaken. Moreover, the absence of visible residues as a result of the comminution of resinous deposits can be predicted due to a lack of surviving protective structures (e.g. mausolea, burial vaults or catacombs) and inclement environmental conditions. The recovery of biomarkers from these unprepossessing samples can, however, be attributed to archaeological sources with some confidence as only one native species, *Pinus sylvestris*, readily produces a terpenoid-containing resin and then not in any significant abundance (Howes, 1949: 109). As the natural distribution of this conifer is in northern, upland areas of Britain while issues of access and availability restricted sample acquisition to southern and/or lowland sites, the potential for contamination within the surrounding soil matrix was considered to be minimal. Molecular evidence for resinous exudates in these burials offers, therefore, a rare opportunity to discern the ritual actions that created this archaeological record. Traces of these highly valued commodities have important implications with respect to both practical and symbolic aspects of Roman mortuary rites and the relationship between Britain and the wider Empire in the 3rd–4th centuries AD.

3. Experimental

3.1. Sample selection and recording

92 residue samples were collected from 48 inhumation burials (Appendix Table S1). 32 of these represented individuals accorded more elaborate rites than the norm (stone sarcophagi, lead-lined coffins and/or plaster body-casings). In addition, the plait of hair recovered from the lead-lined gypsum burial, Crown Buildings, Dorchester was solvent-washed with the kind permission of Richard Breward, Dorchester County Museum (Table 1). The remainder of the burials assessed had been interred in a more normative manner (wooden coffin and/or shroud) sometimes with a 'bed' of plaster. Sample selection was constrained by the materials curated. This varied in relation to the date of discovery (1848–2007) and post-excavation practices with the human remains from earlier finds often reburied and much of the sediment from more recent finds discarded after sieving for artefacts. Thus, residues adhering to skeletal elements and plaster packing were sampled where visible and sub-samples of the grave deposits, comprising a mixture of inorganic and organic matter from the interior of the lead-liners or stone sarcophagi, collected when extant. Where possible, controls from areas not in direct contact with the human remains (e.g. external surface of the plaster, associated soil) were analysed to establish background values. The

materials sampled, with the exception of those from York, had been stored in sealed plastic or glass containers. Comparative, botanically and geographically certified, reference materials were obtained from Bristol Botanicals Ltd.

3.2. Sample preparation

Lipid residue analyses and interpretations were conducted using established protocols (Brettell et al., 2013; Stern et al., 2008). Briefly, ~0.5–2.0 g of the archaeological materials and ~0.05 g of the modern reference resins were solvent extracted in dichloromethane:methanol (DCM:MeOH, 2:1 v/v, 3 × 2 ml) aided by ultrasonication. The solvent-soluble fractions were combined and excess solvent evaporated under a stream of nitrogen. To produce trimethylsilyl derivatives a portion of each dry residue was treated with ~0.5 mL of *N,O*-bis(trimethylsilyl)trifluoroacetamide (BSTFA) with 1% trimethylchlorosilane (TMCS) (40 °C, 15 min; 25 °C, overnight). The derivatized samples were re-diluted in DCM for analysis by GC–MS. All solvents were high performance liquid chromatography (HPLC) grade.

3.3. GC–MS analysis

Analysis was carried out by combined gas chromatography–mass spectrometry (GC–MS) using an Agilent 7890A GC

Table 1

Details of late Roman period 'package' burials from Britain sampled. For further information and details of other burials assessed, see Table S1.

| Location | Container + fill | Sex | Age (yrs) ^b | Key findings |
|---|--|-----|------------------------|---------------------------------|
| Boscombe Down, Wiltshire, Grave 12785 | Stone sarcophagus | | Adult + child | Degraded bone |
| St. Martin's Close, Winchester, F57 (Morris, 1986) | Wood, lead-liner + gypsum | F | Middle adult | |
| Crown Buildings, Dorchester (Sparey Green et al., 1982) | Wood, lead-liner + gypsum | M | Young adult | Oil/fat |
| Poundbury, Dorchester, Dorset (Farwell and Molleson, 1993) | | | | |
| R2 mausoleum, Grave 8 ¹ | Ham Hill limestone + gypsum ^a | – | – | Pinaceae |
| R7 mausoleum, Grave 99 | Ham Hill limestone | M | Mature adult | |
| Site E, Grave 127 | Wood, lead-liner + lead substituted carbonate ^a | F | Adolescent | Pinaceae |
| R10 mausoleum, Grave 513 | Wood, lead-liner + plaster | M | Middle adult | |
| R10 mausoleum, Grave 517 | Ham Hill limestone + gypsum ^a | F | Middle adult | Pinaceae |
| R9 mausoleum, Grave 529 | Wood, lead-liner + plaster | F | Middle adult | Pinaceae |
| R9 mausoleum, Grave 530 ² | Wood, lead-liner + gypsum ^a | M | Middle adult | Pinaceae |
| Site E, Grave 817 | Wood, lead-liner + lead substituted carbonate ^a | F | Middle adult | |
| Site E, Grave 858 | Wood, lead-liner + lead substituted carbonate ^a | F | Mature adult | |
| Site E, Grave 862 | Wood, lead-liner + plaster | F | Middle adult | |
| Site E, Grave 867 | Wood, lead-liner + calcite (lime) ^a | ? | Early child | |
| Site E, Grave 868 | Wood, lead-liner + plaster | ? | Early child | |
| Site E, Grave 892 | Wood, lead-liner + some plaster | M | Middle adult | Pinaceae |
| Site E, Grave 1040 | Wood, lead-liner + lead substituted carbonate ^a | M | Middle adult | Pinaceae |
| Alington Avenue, Dorchester, Dorset (Davies et al., 2002) | | | | |
| Burial 4378, SF 1169 ³ | Wood, lead-liner | F | Mature adult | <i>Boswellia</i> spp. |
| Burial 3664, SF 1075 ⁴ | Wood, lid of sandstone + chalk rubble packing ^a | ? | Early child | <i>Boswellia</i> spp. |
| Burial grounds around London (Barber and Bowsher, 2000; Mackinder, 2000; MoL, 1999; Ridgeway et al., 2013; Swift, 2000; Thomas, 1999) | | | | |
| 280 Bishopsgate, E1, SK 15903 ⁵ | Limestone sarcophagus, lead inner coffin | F | Young adult | Pinaceae + <i>Pistacia</i> spp. |
| 49–59 Mansell Street, E1, Grave B355 | Wood, lead-liner + chalk | M | Young adult | |
| Northview Hospital, Purton, Wiltshire (Chandler, 1994; Nurse, 1992) | | | | |
| Grave 1, within walled structure ⁶ | Stone sarcophagus, lead inner coffin + ?plaster | ?F | Young adult | Pinaceae + <i>Pistacia</i> spp. |
| Grave 6 | Stone sarcophagus | F | Middle adult | ?Adipocere |
| Plaster burials around York, North Yorkshire (Ramm, 1971; RCHM, 1962) | | | | |
| YORYM: 2010.1201, unspecified | Inscribed stone sarcophagus, plaster | – | – | |
| YORYM: 2010.1219, Railway Excavations | Stone sarcophagus, plaster | – | – | <i>Pistacia</i> spp. |
| YORYM: 2007.6205i, Mill Mount | Stone sarcophagus, plaster | – | – | ? <i>Boswellia</i> spp. |
| YORYM: 2007.6206, Railway Excavations | Stone cist, cedarwood coffin, plaster | – | – | ? <i>Boswellia</i> spp. |
| YORYM: 2007.6207, ?Railway Excavations | Stone sarcophagus, plaster | F | Adult | |
| YORYM: 2007.6208, unspecified | ?Lead coffin or liner, plaster | – | – | |
| YORYM: 2007.6209, 89 The Mount | ?Stone sarcophagus, plaster | – | – | |
| YORYM: 2007.6210, unspecified | Lead coffin or liner, plaster | – | – | |
| YORYM: 2007.6211, Heslington Field | Stone sarcophagus, plaster | F | Adult | |
| YORYM: 2007.6212, Railway Excavations | Lead coffin or liner, plaster | – | Sub-adult | |

¹Fig. 2A, 1; ²Fig. 2A, inset; ³Fig. 2C, 1; ⁴Fig. 2C, 2; ⁵Fig. 2A, 2 and 2B, 1; ⁶Fig. 2B, 2.

^a Results from Raman spectroscopic analysis of available samples (ES).

^b For consistency, age determinations have been grouped into ranges, after Powers, 2012.

Table 2
Peak assignment with m/z values of the molecular ion (M^+), base peak (BP, most abundant peak in the mass spectrum) and key fragment ions of the trimethylsilylated terpenic compounds in the modern and archaeological samples shown in Fig. 2A–C. Peak = peak number in the chromatograms.

| Peak | M^+ | BP | Key fragment ions | Compound |
|--------------------------------|-------|-----|---|---|
| Fig. 2A: Pinaceae | | | | |
| 1 | 374 | 73 | 121, 133, 191, 207, 257, 299, 359 | Pimaric acid |
| 2 | 374 | 121 | 73, 91, 143, 241, 257, 359 | Sandaracopimaric acid |
| 3 | 374 | 241 | 73, 105, 143, 256, 257, 359 | Isopimaric acid |
| 4 | 370 | 237 | 73, 103, 143, 195, 209, 252, 355 | Didehydroabietic acid |
| 5 | 372 | 239 | 73, 129, 143, 171/3, 185, 240, 255 | Dehydroabietic acid |
| 6 | 374 | 256 | 73, 105, 185, 213, 241, 257 | Abietic acid |
| Fig. 2B: Pistacia spp. | | | | |
| 7 | 526 | 189 | 511, 409, 391, 320, 307, 219, 203, 133 | Moronic acid |
| 8 | 526 | 203 | 511, 408, 393, 320, 307, 219, 189, 133 | Oleanonic acid |
| 9 | 600 | 203 | 585, 482, 320, 279, 189, 129 | Oleanolic acid |
| 10 | 438 | 203 | 408, 320, 309, 232, 189, 175, 133 | Oleanonic aldehyde |
| 11 | ? | 203 | 408, 309, 279, 232, 190, 175, 131 | Olean-12-ene derivative |
| 12 | 600 | 203 | 585, 482, 320, 279, 189, 133 | Ursolic acid |
| 13 | 526 | 511 | 421, 393, 307, 257, 243, 213, 185, 169 | Isomasticadienonic acid |
| 14 | 526 | 511 | 421, 393, 311, 257, 213, 185, 169, 143 | Masticadienonic acid |
| Fig. 2C: Boswellia spp. | | | | |
| 15 | 394 | 218 | 379, 323, 257, 229, 203 > 189, 175, 161, 135, 119 | 24-norolean-3,12-diene |
| 16 | 394 | 218 | 379, 341, 281, 203 < 189, 175, 161, 133, 119, 107 | 24-norursa-3,12-diene |
| 17 | 498 | 218 | 393, 327, 279, 257, 203 > 189/190, 175, 121 | 3-epi- β -amyrin |
| 18 | 498 | 218 | 483, 408, 393, 229, 203 < 189/190, 175, 161, 121 | 3-epi- α -amyrin |
| 19 | 408 | 232 | 393, 353, 273, 255, 161, 135 | 24-norursa-3,12-dien-11-one |
| 20 | 424 | 218 | 409, 391, 367, 313, 257, 203, 189, 175, 135, 109 | β -amyrenone |
| 21 | 498 | 218 | 483, 468, 408, 393, 311, 241, 203, 189, 129, 69 | β -amyrin |
| 22 | 424 | 218 | 409, 393, 311, 257, 245, 203, 189, 175, 135, 121 | α -amyrenone |
| 23 | 498 | 218 | 483, 468, 408, 393, 279, 257, 203 = 189, 135, 119 | α -amyrin |
| 24 | 600 | 218 | 585, 510, 495, 382, 292, 203, 189, 161, 135, 107 | α -boswellic acid |
| 25 | 600 | 218 | 585, 510, 495, 382, 292, 203, 189, 161, 133 | β -boswellic acid |
| 26 | 570 | 218 | 555, 510, 495, 393, 352, 292, 218, 203 > 189 | 3- <i>O</i> -acetyl- α -boswellic acid |
| 27 | 570 | 73 | 555, 510, 495, 393, 352, 292, 203 <i>c.</i> =189, 133 | 3- <i>O</i> -acetyl- β -boswellic acid |

system, fitted with a 30 m \times 0.25 mm, 0.25 μ m HP-5MS 5% phenyl methyl siloxane phase fused silica column (Agilent), connected to a 5975C inert XL triple axis mass selective detector. The splitless injector and interface were maintained at 300 °C and 280 °C respectively and the carrier gas, helium, at constant flow. The temperature of the oven was programmed to rise from 50 °C (isothermal for 2 min) to 350 °C (isothermal for 10 min) at a gradient of 10 °C per min. The column was directly inserted into the ion source where electron ionisation (EI) spectra were obtained at 70 eV with full scan from m/z 50–800 amu.

3.4. Identification of lipid components

The GC–MS results obtained from the trimethylsilylated solvent extracts of the archaeological and modern samples are presented as total ion current (TIC) and extracted ion current (XIC) chromatograms. Each separated component is shown as a discrete peak with the area beneath representative of its relative abundance. Those compounds amenable to separation were identified through their characteristic fragmentation patterns and in comparison with the mass spectral literature and empirical data from modern reference samples. The key components discussed in the text have been labelled, with details given in Table 2. Only suites of terpenoid biomarkers were interpreted as positive evidence for resinous exudates despite the low probability of their natural occurrence in British soils.

4. Results

Analysis demonstrated that most samples were contaminated with phthalate plasticizers and related compounds, presumably derived from the plastic wrappings. In a number of cases ($n = 14$ burials) any organic compounds fell below the limits of detection.

The remainder of the normative inhumations, in common with the controls and 13 of the 'package' burials contained only ubiquitous lipid species (n -alkanes n -alkanols, carboxylic acids and steroidal compounds) indicative of contributions from degraded plant and animal tissues with some microbial input (Fig. 1; Appendix, Table S1). These are common components of soil organic matter with ingress into the stone and lead containers the result of poorly fitting joints or subsequent damage. Nonetheless, the combination

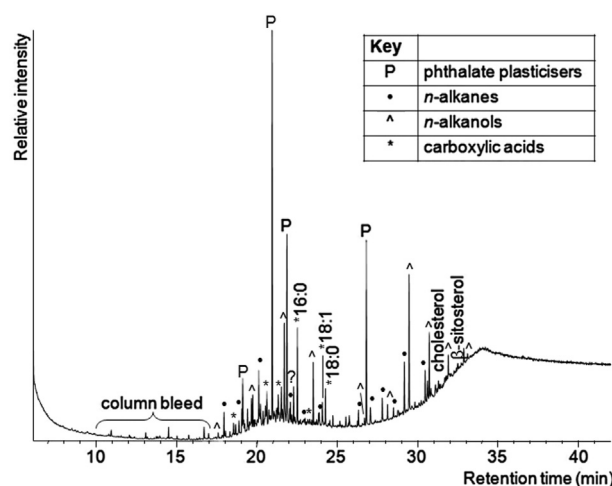


Fig. 1. Total ion current (TIC) chromatogram of the trimethylsilylated lipid extract of a sample from between the lead coffin and stone sarcophagus of the Spitalfields Lady, London. The compounds present comprise low abundance n -alkanes (*), n -alkanols (^), carboxylic acids (*C_{12:0–18:0}, C_{18:1}), cholesterol and β -sitosterol. These are components characteristic of soil organic matter. Modern phthalate plasticiser (P) contaminants were also observed.

of lipids (carboxylic acids, generally C_{14:0–18:0} and C_{18:1}, with cholesterol and its derivatives) in some of the more protected burials may represent end-products from the decomposition of the human remains. The significant abundance of diploptene, which relates to the bacterial degradation of bone (Evershed et al., 1995), in the Boscombe Down double burial is clearly intrinsic as hard-tissue preservation was particularly poor in this context. In addition, 14 of the burials incorporated terpenoids characteristic of plant exudates of archaeological interest. It is these findings together with the results from the analysis of the hair that are discussed below.

4.1. The human hair: Crown Buildings, Dorchester

Two solvent washes of the human hair from the plaster burial of the adult male interred in a lead-lined coffin, Crown Buildings site, Dorchester revealed a restricted number of carboxylic acids (C_{16:0}, C_{18:0}, and C_{18:1}). These ubiquitous components of animal fats and plant oils could derive from either natural or applied hair oils and are consistent with the greasy appearance of the plait (Sparey Green et al., 1982).

4.2. Pinaceae resins: Poundbury Camp, Dorchester

Residues from seven of the 19 burials analysed from the main late Roman cemetery at Poundbury Camp, Dorchester provided evidence of diterpenic compounds. These individuals had all been inhumed in stone sarcophagi or lead-lined coffins, five of which were located within mausolea. Mineral-replaced textiles were also present in conjunction with varying amounts of plaster (Farwell and Molleson, 1993). The resinous substances consisted of dark sticky patches located on the inner surfaces of these body coatings which were readily identifiable due to the retention of body contours and textile impressions (Fig. 2A inset). A control sample from the outer surface of the gypsum from Burial 8 was found to contain only traces of *n*-alkanes, ubiquitous hydrocarbons, and modern phthalate plasticizer contaminants.

The compounds of archaeological relevance were found to be diterpenes and diterpenoids with abieta(e)ne and pimara(e)ne skeletons (Fig. 2A, 1). These are biomarkers for coniferous resins from the sub-family, Pinaceae, which includes pines, firs, and larches (Langenheim, 2003: 35–37). Of widespread occurrence across Europe, all members of the Pinaceae produce resins with a similar molecular composition and many have been 'commercially' exploited since antiquity (Evershed, 1993; Hort, 1980: 223–241; Rackham, 1968: 413–427). Indeed, in the Roman period, it seems that conifers, *Pinus* spp. in particular, had a special significance in the mortuary sphere with pinecones carved on funerary monuments as symbols of immortality or mourning (Alcock, 1980; Mackinder, 2000: 14–16). This is supported by the growing body of chemical evidence from continental finds (Ascenzi et al., 1993; Reifarth, 2013) in conjunction with the substantial amounts of coniferous exudates transported to Roman Egypt (Buckley and Evershed, 2001; Maurer et al., 2002).

Survival of the primary resin acids in the residues from Burial 8 indicates that this exudate was relatively well-preserved. These compounds are often absent from archaeological materials due to natural oxidation processes with aged Pinaceae resins characterised by increased levels of dehydroabietic acid, 7-oxodehydroabietic acid and neutral derivatives (Mills and White, 1977). Such degraded abietane-skeleton moieties were also observed in the samples from Burial 8 and in those from Burials 892 and 1040. The remainder contained only stable end products, such as retene and methyl dehydroabietate. These findings are consistent with the presence of naturally aged Pinaceae exudates and,

although the more degraded examples could derive from the heating of Pinaceae resins or resinous woods (Egenberg et al., 2002), low abundance is considered a more significant factor in this instance. Confirmation of the nature of these residues was made through comparison with modern Pinaceae products (Fig. 2A, 4).

4.3. Pinaceae and Pistacia spp. resins: London, Purton and York

Of the 12 inhumations investigated from the burial grounds of Roman London only the exceptional inhumation known as the 'Spitalfields Lady' provided evidence for the presence of resinous substances. This female in her early twenties, who may have been a recent migrant to Britain (Montgomery et al., 2010), had been interred in a substantial limestone sarcophagus with decorated inner lead coffin (Thomas, 1999). Accompanied by high quality glass and jet artefacts, she had been clothed in silk damask and purple wool tabby with interwoven gold threads while bay leaves had been placed below her head (Thomas, 1999; Wild, 2012). The intact, undisturbed nature of this find and standard of excavation permitted numerous fully contextualized silt samples to be analysed (Fig. 2B inset). Those from between the sarcophagus and the lead coffin ($n = 12$) were viewed as controls since they were not directly associated with the body. They contained only molecular species commonly found in soils (Fig. 1). In contrast, those from within the lead coffin provided evidence of resin biomarkers. These again comprised diterpenic compounds with abieta(e)ne and pimara(e)ne skeletons characteristic of Pinaceae exudates (Fig. 2A, 2). Most abundant in the pelvic region but absent from the silt lateral to the lower arm bones, this resin was, again, relatively well-preserved as the primary resin acids and their initial degradation products were present while neutral derivatives indicative of extensive natural or thermal alteration (e.g. retene) were not observed.

Triterpenoids were also identified in all of the samples from inside the lead coffin, although those associated with the lower arms contained only traces of the more persistent neutral derivatives. In greatest abundance in the cranial and upper torso region, these compounds were found to have oleana(e)ne and tirucalla(e)ne skeletons based on their characteristic fragmentation patterns (Fig. 2B, 1). These moieties are biomarkers for resins from the genus *Pistacia*, otherwise known as mastic or terebinth (Assimopoulou and Papageorgiou, 2005a, 2005b). Survival of the resin acids and the absence of ocotillones (oxidised dammaranes with a base peak of m/z 143), which are often prevalent in heavily degraded *Pistacia* spp. exudates (Scalarone et al., 2003), again signified good preservation. The low abundance of 28-norolean-17-en-3-one and lack of peaks containing a significant ion at m/z 453, in contrast to changes observed during thermal degradation (Stern et al., 2003), indicated that they had not heated prior to deposition. Comparison with modern reference materials demonstrated a clear correspondence with the archaeological data (Fig. 2B, 3).

Samples from Grave 1, from the late Roman rural burial ground at Northview Hospital, Purton, Wiltshire likewise provided evidence of a mixture of Pinaceae and *Pistacia* spp. resins. This lead-lined stone sarcophagus located within a circular walled structure also contained the remains of a young adult female. Fine wool tabby-weave fabric with a coloured border, a high quality glass vessel and a pair of hobnailed shoes accompanied the deceased (Chandler, 1994). Moreover, there were indications that she had been embalmed (Nurse, 1992). Examination during sampling revealed a dark shiny coating on the post-cranial elements which bore a striking resemblance to residues on a comparable burial from Thessaloniki, Greece which had been treated with an aromatic unguent (Papageorgopoulou et al., 2009). Analysis confirmed these

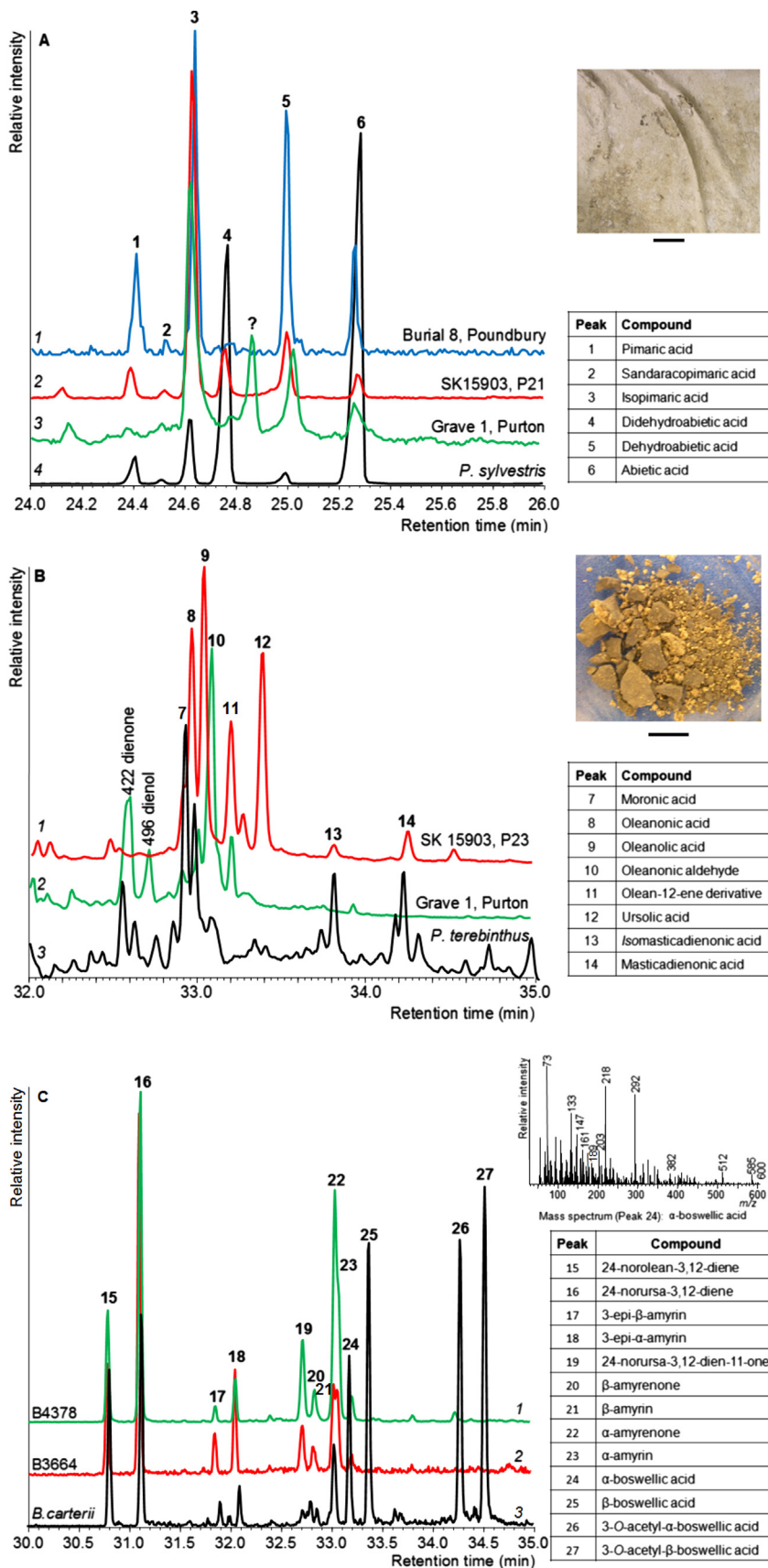


Fig. 2. Partial extracted ion current (XIC) chromatograms of trimethylsilylated lipid extracts of archaeological samples and comparative modern reference materials. Peak identifiers relate to Table 2. Also shown are images of characteristic samples (scale bar = 1 cm). A. XIC (m/z 241) of 1. residue from inner surface of plaster, Burial 8, Poundbury Camp, Dorchester. 2. debris from the pelvic region, base of lead coffin, SK15903, Spitalfields Lady, London. 3. debris associated with the cranium, Grave 1, Purton, Wiltshire. 4. modern *Pinus*

observations and established the nature of the resins utilized. The abieta(e)ne and pimara(e)ne skeleton diterpenic compounds, as before, indicated a naturally aged but relatively well-preserved Pinaceae exudate due to survival of an array of resin acids together with their initial degradation products (Fig. 2A, 3). Similarly, the triterpenes present were found to have oleana(e)ne, tirucalla(e)ne and dammara(e)ne skeletons and included moronic and oleanonic acid, key biomarkers of resins from the genus *Pistacia* (Fig. 2B, 2).

Pentacyclic triterpenes were also recovered from grave deposits associated with in-situ, plaster-encased, skeletal remains from a sarcophagus burial (YORYM:2010.1219, Railway Excavations) from York, North Yorkshire. Diagnostic compounds were identified as 28-norolean-12-en-3-one, formed by the decarboxylation of oleanonic acid, together with the more stable 28-norolean-17-en-3-one, the result of a subsequent hydrogen shift (Pastorova et al., 1998). The dominant moiety, as with the samples from Purton, was found to be oleanonic aldehyde. Although no definitive biomarkers were present, this combination closely reflects patterns observed in aged *Pistacia* spp. resins (Modugno et al., 2006). These degradation pathways result from natural environmental interactions (Colombini et al., 2000; Stern et al., 2003) and have been traced in modern exudates whose components are often highly variable (Assimopoulou and Papageorgiou, 2005a, 2005b).

The genus *Pistacia* (family Anacardiaceae) consists of a dozen species native to the Mediterranean and the Levant (Zohary, 1952). Molecular evidence for the use of this transparent, pale yellow resin comes from a range of Ancient Egyptian contexts, including varnishes on mortuary objects and embalming materials (Buckley and Evershed, 2001; Serpico and White, 2000, 2001). By the Roman period, the island of Chios seems to have been the main source of mastic, probably tapped from *Pistacia lentiscus*, although an even more desirable exudate was obtained from the 'terebinth' or 'turpentine tree'. In the 4th century AD, the latter was harvested from a variety, possibly *Pistacia terebinthus* L., which grew near Damascus, Syria (Gunther, 1959: 49; Hort, 1980: 223; Rackham, 1968: 267, 423). Thus, *Pistacia* resins were both highly prized and widely traded as verified by their identification in Roman period burials from Italy (Bruni and Guglielmi, 2005), Gaul (Devièse, 2008: 115–131), the Rhineland (Reifarth, 2013) and Britain (Brettell et al., 2013). The inclusion of this exotic exudate in the elaborate inhumations considered here closely matches the profile established by these previous finds.

4.4. *Boswellia* spp. gum-resins: Dorchester and York

Two third century AD inhumations from a rural burial ground at Alington Avenue, Dorchester were also investigated. The first comprised materials associated with the remains of an adult female who had been interred in a wooden coffin, seemingly packed with rubble derived from the chalk burial environment, and covered with a substantial stone slab (Burial 3664). The second consisted of residues from the lead-lined coffin (Burial 4378) of a child aged 4–6 years who had been furnished with a Black Burnished Ware jar, iron rod and curated coin (Davies et al., 2002). Traces of a white substance originally led to the suggestion that this was a gypsum burial but have been found to be calcium carbonate with adsorbed lead ions representative of chalk ingress. Adhering textile fragments, however, proved to be a wool plain-weave (tabby), probably undyed, with weft-faced sections dyed with shellfish (murex or

'Tyrian') purple. These probably derived from a tunic with two wide vertical purple stripes (*clavi*) on the shoulders (Walton Rogers, 2002) and are a strong indicator for the burial of a member of the elite (Wild 2012).

The majority of the residues from both inhumations contained a series of distinctive pentacyclic triterpenic moieties with oleana(e)ne and ursa(e)ne skeletons (Fig. 2C, 1 and 2). The significant ions at m/z 189, 203 and 218 are characteristic of olean-12-ene and urs-12-ene derivatives, molecular markers of the extensive resin-producing Burseraceae family (Modugno et al., 2006). In this instance, the botanical source can be more closely defined as the major fragment ion falls at m/z 218 with a significant ion at m/z 292 denoting an absence of functional groups on rings C, D and E and a carboxylic acid group at C-4 (Budzikiewicz et al., 1963). This structure is characteristic of the α - and β -boswellic acids and their degradation products which are unique to gum-resins from the genus *Boswellia*, better known as frankincense or olibanum (Evershed et al., 1997). Confirmation was made through comparison with modern botanically and geographically certified exudates (Fig. 2C, 3). Despite the close correspondence in the compounds identified, their relative abundances differed considerably as the archaeological samples contained only low levels of the boswellic acids, none of their *O*-acetyl oxidation products and an abundance of neutral derivatives. This reduction in acid moieties can occur as a result of changes wrought by pyrolysis (Mathe et al., 2007) although more research is required regarding the impact of natural taphonomic factors on this gum-resin before any such claim can be made here.

Residues adhering to plaster body-casings from two burials from York (YORYM:2007.6205i, Mill Mount; YORYM:2007.6206, Railway Excavations) were likewise found to contain triterpenic compounds, in contrast to a corresponding soil control. In addition to the widely distributed triterpenic alcohols, β - and α -amyrin, their oxidation products and epimers, the remainder of these moieties again consisted of olean-12-ene and urs-12-ene derivatives distinguished by a base peak at m/z 218. Unfortunately, no primary resin acids were present so the source could not be definitively identified but this sequence appears to reflect that observed in degraded *Boswellia* spp. gum-resins (Modugno et al., 2006).

The genus *Boswellia* comprises around 23 species of small deciduous trees with the main incense-producing varieties being *Boswellia carterii* (eastern Africa), *Boswellia sacra* (southern Arabia) and *Boswellia serrata* (north-western India) (Tucker, 1986). The role of their highly valued exudates in ancient mortuary rites has long been debated due to the ritual importance of this aromatic gum-resin (Groom, 1981: 1–14). Its main source, according to primary texts, was known as the 'Frankincense Kingdom' which appears to have been situated on the southern coast of Arabia (Hort, 1980: 233–241; Tucker, 1986). The aromatic bounty of this region earned it the epithet '*felix*' ('blessed') (Rackham, 1968: 37–47) and was widely traded throughout the eastern Empire and the Mediterranean (Huntingford, 1980: 35–38). Indeed, such was the demand that two harvests were needed each year to meet the requirements of increasingly lavish Roman funerary displays (Rackham, 1968: 41, 61). Nonetheless, despite extensive research, molecular confirmation of the use of frankincense has been scarce. Only a small number of studies have previously obtained positive results from a cosmetic unguent from Egypt (Mathe et al., 2004) and probable incense fragments from Nubia (Evershed et al., 1997) and Yemen

sylvestris resin. Inset: residue from inner surface of plaster, Burial 530, Poundbury. B. XIC (m/z 203) of 1. debris associated with the femora, Grave 1, Purton, Wiltshire. 2. debris from the upper torso region, base of the lead coffin, SK15903, Spitalfields Lady, London, shown in inset. 3. modern *Pistacia terebinthus* resin. C. XIC (m/z 218) of 1. debris from base of lead coffin, Burial 4378, Alington Avenue, Dorchester. 2. debris from cranial region, Burial 3664, Alington Avenue, Dorchester. 3. modern *Boswellia carterii* gum-resin. Inset: mass spectrometry of α -boswellic acid (Peak 24) from Burial 4378.

(Mathe et al., 2007; Regert et al., 2008). These regions all lie within the known geographical and commercial distribution of frankincense in antiquity. Thus, the identification of this exotic gum-resin in late Roman burials in France (Devièse, 2008: 115–131) and now Britain is remarkable.

5. Discussion

Our findings provide direct evidence for the use of resinous exudates as part of mortuary rites in late Roman Britain. This brings to an end the long-running debate regarding their presence in such contexts. The success of the project is, in part, due to its systematic approach which not only targeted visible residues but other samples with the potential to contain molecular markers related to the treatment of the body. As a result, we have revealed a previously unrecognized reservoir of chemical evidence in the debris from the base of these graves. This mixture of degraded materials retains traces of resinous substances long after the decomposition of macro-scale organic materials. In light of this, more strategic sample collection from future finds will be crucial in expanding our knowledge of this Roman mortuary practice and in enabling us to address increasingly intricate questions. For example, whether the apparent spatial variation in the relative abundance of the two resins applied to 'Spitalfields Lady' was due to taphonomic factors or a reflection of symbolic action.

The broader historical significance of this research is manifold touching on ritual, socio-cultural and economic aspects of life and death in the Roman world. So, what were the structuring principles behind this body treatment? The mortuary sphere has always provided an arena for socio-cultural display, an opportunity for those conducting the funerary proceedings to exhibit disparate aspects of an individual's identity. One such expression of difference in the late Roman period seems to have involved dressing the dead in richly-decorated garments and wrapping them in a shroud before interring them in a lead-lined coffin or stone sarcophagus with some individuals also encased in plaster (Sparey Green, 1977). This is best illustrated by the exceptionally well-preserved

inhumations from below the basilica of St. Maximin, Trier, Germany (Reifarth, 2013, Fig. 3).

What is now clear, is that resins formed a key element of this 'package' and could be employed in a variety of ways: as embalming agents (Ascenzi et al., 1993; Papageorgopoulou et al., 2009), sprinkled or pasted onto the textile wrappings (Brettell et al., 2013; Reifarth, 2009) or placed as offerings within the coffin (Bruni and Guglielmi, 2005). Applied to purify the body and facilitate the deceased's journey to the afterlife (Philpott, 1991: 118), all ages and both sexes seem to have warranted such consideration with a slight preponderance of females as noted by Chioffi (1998: 24–28). This desire for particular aromatic substances which resulted in their transport from one end of the Empire to the other, particularly at a time when long-distance trade is thought to have been in decline, attests to the ritual importance of these luxury goods. Their presence cannot, however, be correlated with specific religious beliefs as they were deemed to propitiate many pagan deities but also played a significant role in both Jewish and Christian rites (AKJV, 2011; Exodus 30:34, Leviticus 2:3, 6:15; Groom, 1981: 1–9).

Differentiation in terms of social status is more strongly supported as only the most elaborate inhumations from each region contained evidence of resinous substances. This may, of course, be an artefact of sample availability or taphonomy. Nonetheless, the incorporation of unheated resin fragments makes chemical sense in this context. Only in their natural state would the steady release of the volatile aromatic components have been able to mask the odour of decay and slow invasion of the body by decomposer organisms. Likewise, the anti-microbial properties of the resin fraction could then have acted to temporarily retard soft-tissue decomposition in conjunction with the use of plaster, whether gypsum or calcite-based, when present (Schotsmans et al., 2011). Such concerns would have been particularly pressing during the extended funerary rites accorded the social elite (Hope, 2009: 71–74). Moreover, unless these exotic materials were the personal property of migrants or returning provincials keen to display their acquired *romanitas*, our findings show that knowledge of this rite must have become embedded in the consciousness of the inhabitants of this remote province. Its selection, across Britain, from a spectrum of potential

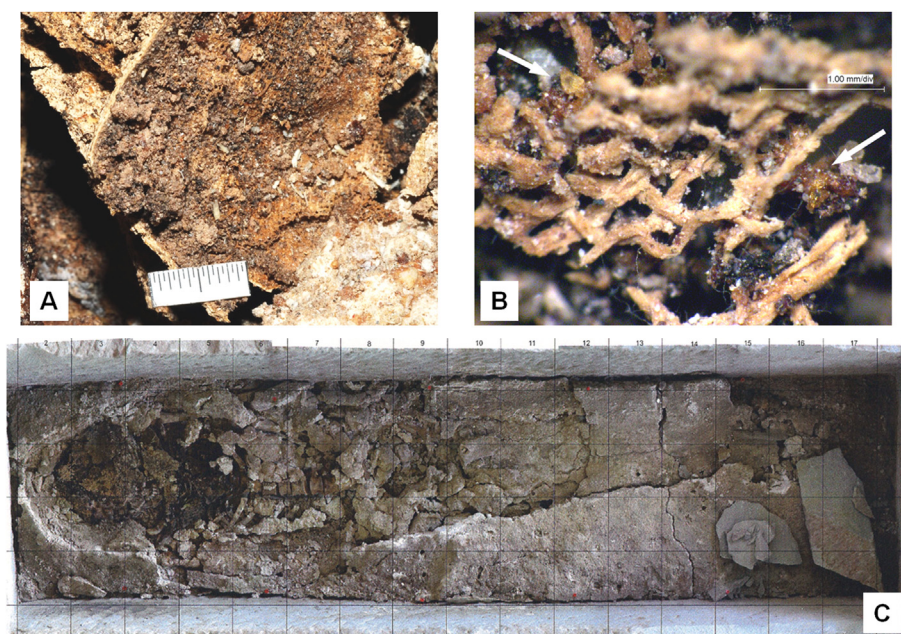


Fig. 3. Late Roman sarcophagus burial, Grave 107, St. Maximin, Trier, Germany. A. Associated textiles embedded with resin (macroscale). B. Embedded resin fragments (microscale, 100 mm/div). C. The skeletal remains encased in gypsum. Images courtesy of Nicole Reifarth.

mortuary practices demonstrates the fundamental influence of Roman mores even on this most conservative area of ritual action.

6. Conclusion

Molecular analysis of materials from late Roman period inhumations in Britain has demonstrated the potential for 'grave deposits' to retain invisible marker compounds and has revealed that only the most elaborate burials contained resinous substances. These were found to derive from three different botanical sources: European coniferous Pinaceae resins, Mediterranean *Pistacia* spp. resins and exotic *Boswellia* spp. gum-resins. This discovery resolves years of speculation concerning the importation of resins into Britain for use in mortuary contexts, although the manner in which they were transported has yet to be identified. It also demonstrates the profound impact of Roman eschatology throughout the Empire. The correlation with recent continental research, which has recovered evidence for the same three exudates in similar burials from across Europe, reinforces this view.

We conclude that these resinous materials performed multiple roles in the mortuary sphere. The most mundane was to disguise the odour of decomposition and aid temporary soft-tissue preservation prior to interment. During display of the body, they also acted as signifiers of social status in conjunction with the other components of these 'package' burials. In addition, the use of these specific substances implies a ritual dimension. The aroma of sanctity released, which was considered so pleasing to the gods, would have facilitated safe passage to the otherworld. Of equal importance, however, may have been their contribution through the agency of conspicuous consumption to a final exhibition of munificence. Enacted for the benefit of the survivors, this show would have served to promote memory and so helped ensure the continued existence of the deceased in the minds of the living.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at <http://dx.doi.org/10.1016/j.jas.2014.11.006>.

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