Archaeometry **44**, 1 (2002) 95–105. Printed in Great Britain

THE PRESERVATION AND IDENTIFICATION OF PIÑON RESINS BY GC-MS IN POTTERY FROM THE WESTERN GREAT BASIN*

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Gas chromatography – mass spectrometry analysis of a sherd from the Nevada Test Site revealed a high quantity of terpenes, including sesquiterpenoids and diterpenoids, demonstrating that piñon resins were prepared in the pot in prehistoric times. The presence of these biomarkers allowed for a very specific identification of the products prepared in the pot, a level of detail not often achieved in lipid residue analysis. That the terpenes are relatively unoxidized demonstrates that they are quite stable over long periods of time. The study also shows that sherds on the surface of archaeological sites can preserve lipids and terpenes and are amenable to organic residue analysis.

KEYWORDS: NEVADA TEST SITE, GREAT BASIN, SHOSHONE, GAS CHROMATOGRAPHY – MASS SPECTROMETRY, POTTERY, DITERPENOIDS, PIÑON RESIN, *PINUS MONOPHYLLA*, ORGANIC RESIDUES

INTRODUCTION

Recent studies of ancient lipid residues in archaeological ceramics have met with mixed success. Often, the level of detail in the classification of preserved lipids is very general, with the analyst unable to pinpoint the exact food that was prepared or stored within a pot. This stems mainly from the fact that most plants and animals produce the same range of organic compounds, although in different relative quantities, forcing archaeologists to classify residues in general categories, such as ‘meat’ versus ‘plant’ or ‘mammal’ versus ‘fish’. Occasionally, however, the presence of specific biomarkers that are distinctive of certain species or genera allows for much finer identifications (Skibo and Deal 1995).

This report describes the results of a gas chromatography – mass spectrometry (GC-MS) study on a single sherd from Western Nevada, where such precision was achieved due to the presence of biomarkers. In this case, specific terpenes that are rare in nature suggest a very narrow range of sources for the preserved residues.

THE SAMPLE/SHERD

As part of a study investigating the origins of ceramics in the Western Great Basin (see Eerkens 2000), a sherd from 26-Ny-938 on the Nevada Test Site in Western Nevada was sampled for organic residues. JEC294 (Desert Research Institute catalogue number 1-37) is a body sherd (e.g., neither rim nor base) and was collected from the surface of 26-Ny-938. This site is a small rock shelter in the wall of a canyon below and south of Pahute Mesa, and lies within the piñon–juniper vegetation zone. Gambel oak (*Quercus gambelli*) grows nearby and an understorey

* Received 5 March 2001; accepted 26 April 2001.

of sage, ephedra, Indian rice grass and rabbit brush is present on site (Fowler *et al.* 1977). Following collection in the field, the sherd was not cleaned or treated in any way, but was catalogued and placed in a plastic bag for storage.

JEC294 is 5.8 mm thick. On the basis of curvature, the original pot is estimated to have had a diameter of between 230 and 250 mm near its mid-section. Because the sherd is not a rim, the diameter of the original mouth opening could not be estimated. Temper within the sherd is not particularly dense and is composed primarily of medium-sized quartz and other granitic inclusions. In outward appearance, the item looks much like any other brownware sherd from the region, although it does appear to be well-preserved (i.e., it is not sandblasted or weathered). However, during burring and crushing in preparation for extraction of organic residues (see below), a faint odour of pine was emitted.

The sherd was also examined by instrumental neutron activation analysis (INAA), to learn more about its production and provenance. Given similarities in the chemical composition of JEC294 compared with many other Nevada Test Site sherds, and the dissimilarity of these samples to other sherds from nearby regions, including Owens Valley and Death Valley, the INAA suggested that the sherd was made of a local clay (see Eerkens 2000).

Brownware pottery in the region is generally limited to the last 700 years of prehistory (Pippin 1986; Lockett and Pippin 1990; Rhode 1994). Unfortunately, there do not appear to be any significant changes in the shape and form of vessels in the Great Basin, and it is not possible to date the sherd to a more specific chronological period.

METHODS

Analyses were carried out on a 5890 Hewlett Packard Gas Chromatograph (GC) with splitless injection, coupled to a 5790 Hewlett Packard Mass Spectrometer (MS). A 30 m long fused silica wall-coated open-tube (WCOT) capillary column with an internal diameter of 0.25 mm was used (J and W DB5). Temperatures within the GC were raised from 100°C to 325°C at a rate of 12°C per minute, with the final 325°C temperature held for 8 minutes. Helium was used as the carrier gas.

The sherd was prepared by breaking off a 1 cm^2 piece and burring away the outer 1–2 mm of all exposed surfaces using an abrasive silicon carbide drill bit. This fragment was crushed to a powder in a small agate mortar and pestle, and 400 mg was transferred to a test tube. Then 200 ml of a 2 : 1 mixture of chloroform : methanol was added to the tube, gently agitated for 5 minutes and sonicated for 15–20 minutes. Following sonication, the test tube was placed in a centrifuge for 10 minutes to separate the solvent mixture from the inorganic clay particles. The solvent was then transferred to a second test tube and placed in a vacuum centrifuge. Finally, samples were derivatized to fatty acid methyl esters (FAMEs) by the addition of 100 μl of methanolic HCl and placement in a heating block set at 60°C for 1 hour. After heating, samples were dried within the vacuum centrifuge and stored in a freezer until ready for analysis, usually within two to three days. A solution containing known amounts of methyl benzoate (internal standard) and hexane (solvent) was prepared and added to each sample prior to injection in the GC-MS. Although other extraction techniques can produce higher yields (i.e., Stern *et al.* 2000), this technique can be successful in producing significant and interpretable levels of lipids.

Compounds were identified partially by their retention time within the GC, but mainly by their mass spectra. The National Institute of Standards and Technology (NIST) 98 Mass Spectral Library was used to match spectra obtained in archaeological sherds to reference

spectra. The NIST database contains over 100 000 standard reference spectra of high quality, including most FAMEs, terpenes and sterols of interest. The amount of each organic compound present in a sample was computed using the Automated Mass Spectral Deconvolution and Identification System program developed at NIST. The quantity of a particular compound was estimated by integrating and calculating the area under the GC total ion current peak associated with each compound.

RESULTS

Figure 1 presents a gas chromatogram for JEC294. The figure is divided into two sections to highlight various features of the chromatogram. The upper section represents the first half of the scan and the lower section the second half (note that the scale of the *y*-axis is not the same for the two sections). In some respects, the chromatogram looks similar to those for other sherds from the region. A range of organic compounds is present, including both saturated, mono-unsaturated and dicarboxylic fatty acids. However, in addition to these compounds, a range of terpenoids is also present, including sesquiterpenoids and diterpenoids. These compounds are represented by several smaller peaks between 7.4 and 9.4 minutes in Figure 1, for the former, and a dominating peak followed by several medium-sized peaks between 13.5 and 16 minutes for the latter. Sesquiterpenoids and diterpenoids were not encountered in 74 other archaeological sherds sampled from the Western Great Basin, including five others from the Nevada Test Site.

Sesquiterpenoids

At least four major and nine minor sesquiterpenoids were encountered during analysis. The mass spectra of many of these different compounds are similar, with base peaks occurring at $m/z = 161$ and molecular ions at $m/z = 204$. Figure 2 gives the mass spectra for the compound labeled S6 in Figure 1 (at 8.09 minutes) as well as the mass spectra of α-muurolene from the NIST mass spectral database. As can be seen from Figure 2, the match between compound S6 and α-muurolene is excellent. Zavarin and Snajberk (1980) list α-muurolene as one of the more common sesquiterpenoids in *Pinus monophylla* gum turpentine. However, α-muurolene is also present in a number of other plants (Langenheim 1981; papers in Pridham 1967), including juniper.

Unfortunately, I was unable to run authentic sesquiterpenoid samples from commercial sources through the GC to obtain retention times for comparison with JEC294. Moreover, many of the compounds have similar mass spectra and occur in low to trace amounts such that background noise complicates assignment of the compounds to discrete sesquiterpenoids on the basis of mass spectra alone. Tentative assignments of peaks on the basis of mass spectra were possible only for the more common and unique sesquiterpenoids. Table 1 lists the sesquiterpenoids encountered, as well as their abundances relative to the total sesquiterpenoid content (TSC) and total ion current (TIC).

Zavarin and Snajberk (1980) list over 30 different sesquiterpenoids in the higher-boiling gum turpentine of *P. monophylla* and *P. edulis*. The more common of these compounds include germacrene D, α-muurolene, γ-cadinene, γ-amorphene, α-copeane, cyclosativene and longifolene, although many more occur in trace quantities. As indicated in Table 1, many of these compounds are also present in the JEC294 sample.

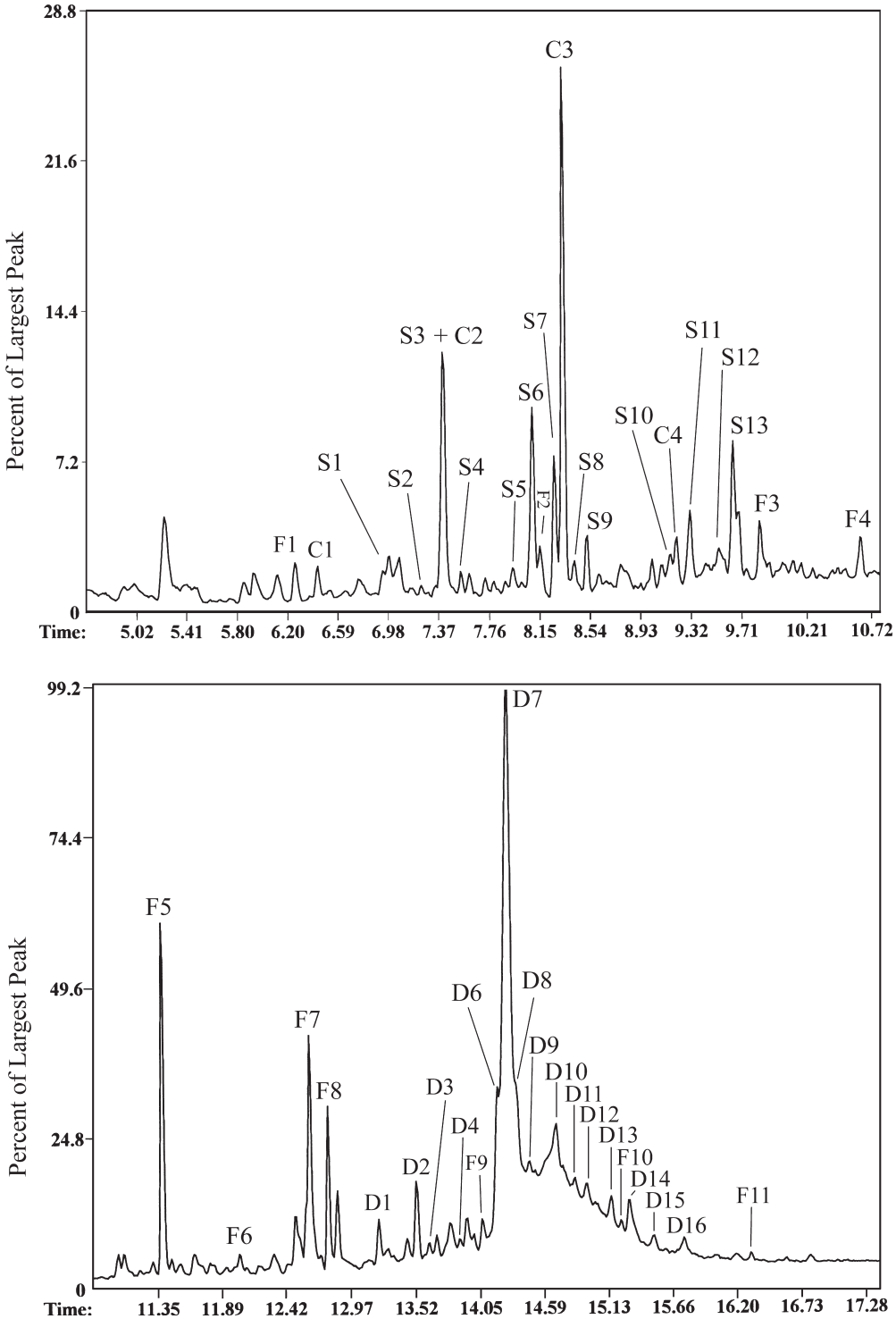

Figure 1 *The gas chromatogram of JEC294.*

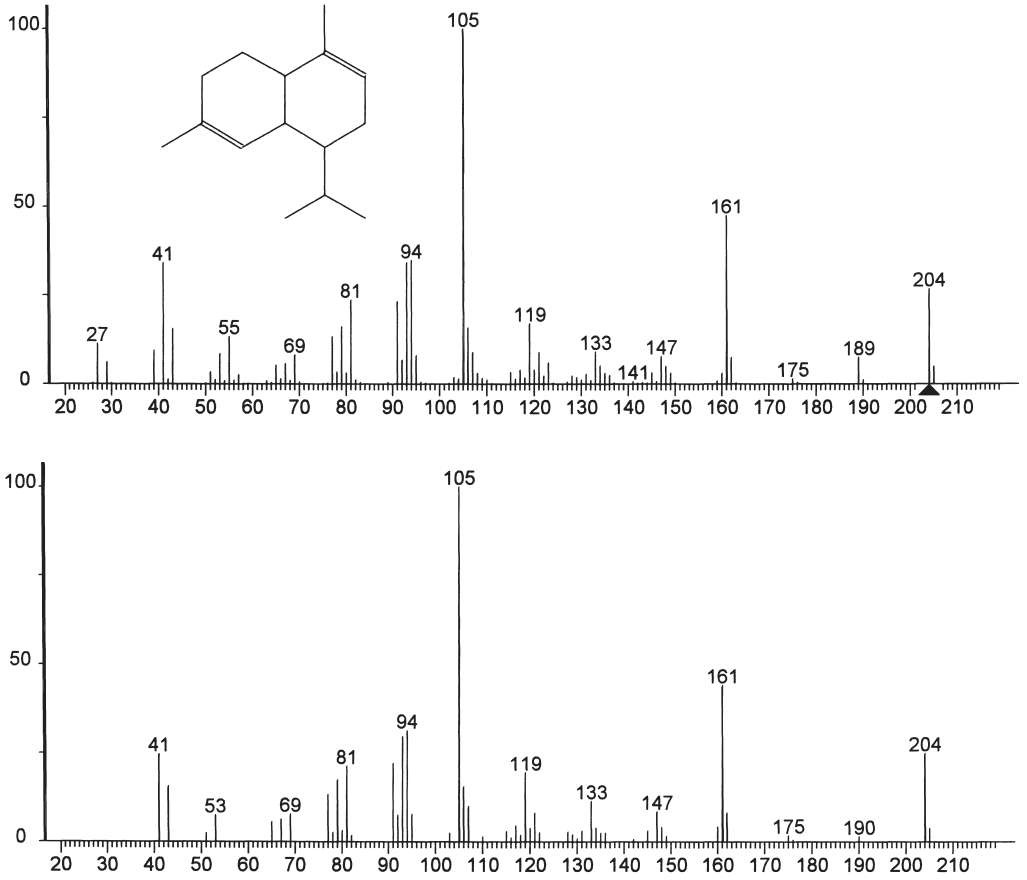

Figure 2 *The mass spectrum of α-muurolene from the NIST database (top) and the sesquiterpenoid observed at 8.09 minutes (scan 454) in Figure 1 (bottom).*

Table 1 *The distribution of sesquiterpenoids in JEC294*

Label in Figure 1	*Compound*	*% TSC*	*% TIC*	***Mass** and characteristic ions*
S1	Longifolene	1.7	0.019	**204**, 161, 189
S2	Unknown	0.6	0.007	**204**, 161, 175
S3	D-longifolene	31.3	0.360	**204**, 161, 133, 94
S4	Unknown	1.0	0.012	**204**, 147, 119
S5	*α*-guaine	1.2	0.014	**204**, 147, 105
S6	*α*-muurolene	21.1	0.243	**204**, 161, 105
S7	*δ*-cadinene	13.4	0.154	**204**, 161, 134
S8	Unknown	1.4	0.016	**204**, 200, 157, 119
S9	Cadala-1(10),3,8-triene	6.3	0.072	**204**, 157, 142
S10	Unknown	1.1	0.013	**204**, 161, 134, 105
S11	Unknown	7.3	0.084	**204**, 161, 121, 85
S12	Germacrene D	1.8	0.021	**204**, 161, 133, 119
S13	Cadalene	11.7	0.134	**198**, 183, 168

Note that the mass for unknown compounds is estimated on the basis of the appearance of the mass spectrum.

Table 2 *The distribution of diterpenoids in JEC294*

Label in Figure 1	*Compound*	*% TDC*	*% TIC*	***Mass** and characteristic ions*[1]
D1	Pimaral	3.0	0.129	**286**, 271, 257, 241, 105*
D2	Δ-8(9)-isopimaric acid, ME	6.6	0.283	**316**, 301, 257, 241*, 119
D3	Unknown	0.3	0.012	**314**, 299, 267*, 239
D4	Sandaracopimaric acid, ME	0.5	0.021	**316**, 301, 288, 121*
D5	Isopimaric acid, ME	0.3	0.012	**316**, 287, 257, 241*, 105
D6	Unknown	8.4	0.363	**300**, 284, 199, 187, 109*
D7	Δ-8(9)-isopimaric acid	65.6	0.820	**302**, 287*, 241, 185
D8	Dehydroabietic acid, ME	3.5	0.150	**314**, 299, 239*
D9	Unknown	0.5	0.020	**302**, 287, 249, 241*, 233
D10	Abietic acid, ME	2.7	0.116	**316**, 256*, 121
D11	Unknown	0.3	0.015	**300**, 241, 181*
D12	Unknown	0.5	0.020	**312**, 237*, 109
D13	Unknown	2.2	0.096	**315**, 299*, 283
D14	Oxy-Δ-8(9)-isopimaric acid, ME[2]	3.3	0.140	**328**, 315, 283, 149*
D15	Unknown	0.9	0.040	**328**, 286, 239, 148*
D16	7-oxo-dehydroabietic acid, ME	1.2	0.048	**328**, 253*, 213 187

[1] The mass for unknown compounds is estimated on the basis of the appearance of the mass spectrum; asterisks (*) indicate ion base peaks.

[2] An apparent compound; ME = methyl ester.

Diterpenoids

A range of pimaric and abietic diterpenoids was also encountered in the sample. The dominant member of this group (at 14.29 minutes in Fig. 1) was identified as Δ–8(9)-isopimaric acid and comprises over 65% of the diterpenoids. Other compounds include isopimaric acid, sandaracopimaric acid, abietic acid, dehydroabietic acid and 7-oxo-dehydroabietic acid. All of these compounds are mentioned by Zavarin and Snajberk (1980) and Fox *et al*. (1995) as being present in piñon resin. Table 2 lists the range and abundances of diterpenoid compounds observed in JEC294 relative to total diterpenoid content (TDC) and TIC.

The largest peaks seem to represent compounds in the unmethylated free acid state, although smaller peaks representing methyl esters are also evident. This indicates that the density of diterpenoids and fatty acids in this sherd was quite high. Thus, the amount of methanolic HCl (100 μl) added to this sample may not have been enough to completely derivatize the entire suite of compounds into methyl esters.

Fatty acids and other compounds

In addition to the terpenes found, a number of fatty acids were also observed. Primary among these include C18:1 (oleic acid), C16:0 (palmitic acid) and C18:0 (stearic acid). Table 3 lists different fatty acids as a percentage of the total fatty acid content. Other than a single mono-unsaturated fatty acid, all fatty acids represent saturated compounds. Polyunsaturated fats were not encountered, and are, indeed, rarely encountered in archaeological sherds due to their propensity for decomposition relative to saturated and mono-unsaturated compounds (Christie 1989; deMan 1992).

Table 3 *The distribution of fatty acids (as a percentage of the total fatty acid content)*

Label in Figure 1	*Shorthand*	*Common name*	*% TFAC*[1]
F1	C10:0	Capric	0.89
F2	C12:0	Lauric	0.22
F3	C14:0	Myristic	0.61
F4	C15:0	Pentadecylic	0.22
F5	C16:0	Palmitic	31.27
F6	C17:0	Margaric	0.22
F7	C18:1	Oleic	48.62
F8	C18:0	Stearic	12.83
F9	C20:0	Arachidic	1.33
F10	C22:0	Behenic	0.22
F11	C24:0	Lignoceric	0.22

[1] TFAC = total fatty acid content.

Table 4 *Dicarboxylic acids in JEC294, and the percentage of the total ion current*

Label in Figure 1	*Compound*	*% TIC*
C1	Heptanedioic acid, DE[1]	0.041
C2	Octanedioic acid, DE	0.171
C3	Nonanedioic acid, DE	0.559
C4	Decanedioic acid, DE	0.038

[1] DE = dimethyl ester.

In addition, several dicarboxylic acids were recovered as dimethyl esters and are listed in Table 4. Most prominent among these is azelaic acid (or nonanedioic acid). These compounds are common byproducts of oxidation of unsaturated fatty acids (Frankel 1980, 1987; Porter *et al*. 1981; Hudlicky 1990, 226). Indeed, azelaic acid, with nine carbon atoms, probably represents decomposition of an unsaturated fat with a double bond at the ninth carbon atom position (e.g., C18:1Δ9 or C18:3Δ9). Regert *et al*. (1998) suggest that dicarboxylic compounds are more likely to survive in archaeological sherds in arid climates. For example, they find a range of these compounds, especially azelaic acid, in a sherd from a site in Egyptian Nubia. Similarly, azelaic acid was one of the most common compounds in lipids extracted from a 4000-year-old Nubian burial (Gülacar *et al*. 1989) and was also common in sherds from a site in Syria (Shimoyama *et al*. 1995). The presence of these compounds in the current sample and others from the arid Western Great Basin (see Eerkens 2000) supports the conclusions of Regert *et al*. (1998) that dry environments tend to promote preservation of dicarboxylic acids.

Identification

On the basis of the conspicuous presence of sesquiterpenoids and diterpenoids, it is clear that the pot is associated with resinous materials of pine. The predominance of Δ-8(9)-isopimaric acid among the diterpenoids makes it clear that the pot contains the resins of piñon, either

P. monophylla or *P. edulis*. This compound is comparatively rare in nature and is found only in small quantities in most other species of pine. Zavarin and Snajberk (1980) and Fox *et al*. (1995) both find that this isomer is the major diterpenoid component in piñon resin. Its presence here in high quantities, comprising over 60% of the diterpenoids, demonstrates that JEC294 contains the resins of one of these species of piñon. From the GC-MS data alone, it is not possible at this point to discriminate between the two species. However, on the basis of the local environment in which JEC294 was collected, the former seems most likely.

The discovery of piñon resins in this sherd is not surprising, given the location of 26-Ny-938 within the piñon–juniper zone and the ready access to piñon that the site would have afforded. At the same time, the pot also contains a range of fatty acids, indicating exposure to other sources of oil. Elsewhere (Eerkens 2000, 113), I have interpreted the distribution of fatty acids in JEC294 as consistent with the preparation of a seed or berry product. This suggests that the pot probably performed in several capacities, perhaps first serving as a cooking vessel to prepare seeds or berries, and later to render pitch and tar from piñon resins.

DISCUSSION

As mentioned, JEC294 appears much like any other brownware sherd from the region. There were no visible residues adhering to the interior or exterior surfaces of the sherd and it was clear during preparation for GC-MS analysis that pine resins had been absorbed *within* the interior walls of the pot. Indeed, the outer and inner surfaces were burred away to a depth of 1 mm, demonstrating that the resins recovered during GC-MS analysis are within the core fabric of the sherd. Moreover, the sherd was prepared twice for GC-MS analysis and once for INAA analysis, and the scent of pine was strong during all three events, suggesting that the pitches are not localized within one part of the sherd, but are ubiquitous throughout. Stern *et al*. (2000) suggest that external contaminants from the surrounding soil are unlikely to penetrate beyond 2 mm into the interior of a sherd. Thus, such contaminants would have been largely removed during sample preparation. It also seems unlikely that the resins could have been introduced by sap falling on the sherd while it lay on the surface of the site, since the resins occur throughout the entire sherd, and nothing was visible on the surface to suggest that this was the case. Together, these items suggest that the pine resins are not the result of postdepositional contamination, but were introduced prehistorically through use by Shoshone people.

It is also clear that the terpenes are not the product of boiling piñon nuts within the pot. Figure 3 shows a gas chromatogram for a modern pot in which piñon nut mush was brought to the boil and cooked for approximately 2 hours. Compounds in common with JEC294 are given the same labels as in Figure 1 and Tables 1–4 (e.g., C1–C4, F1–F11), while compounds unique to this sample are labelled within the figure. Although a range of fatty acids and other organic compounds are present, the figure and associated mass spectra demonstrate that piñon nuts contain no terpenes (which is not surprising, since they constitute a favoured and pleasant-tasting food resource). Thus, the processing of this food resource could not have contributed to the GC pattern observed in JEC294.

For three reasons, it seems unlikely that piñon resin would have been added to the pot itself in an attempt to waterproof or repair it. First, it is clear that the resin is not on the surface of the pot, as you might expect for a sealant to make the pot waterproof. Second, the fact that pots were used regularly to boil foods suggests that they were already waterproof without any added sealant. Finally, no glue appears along the edges of the sherd, as might be expected if piñon resin were used to mend a broken pot.

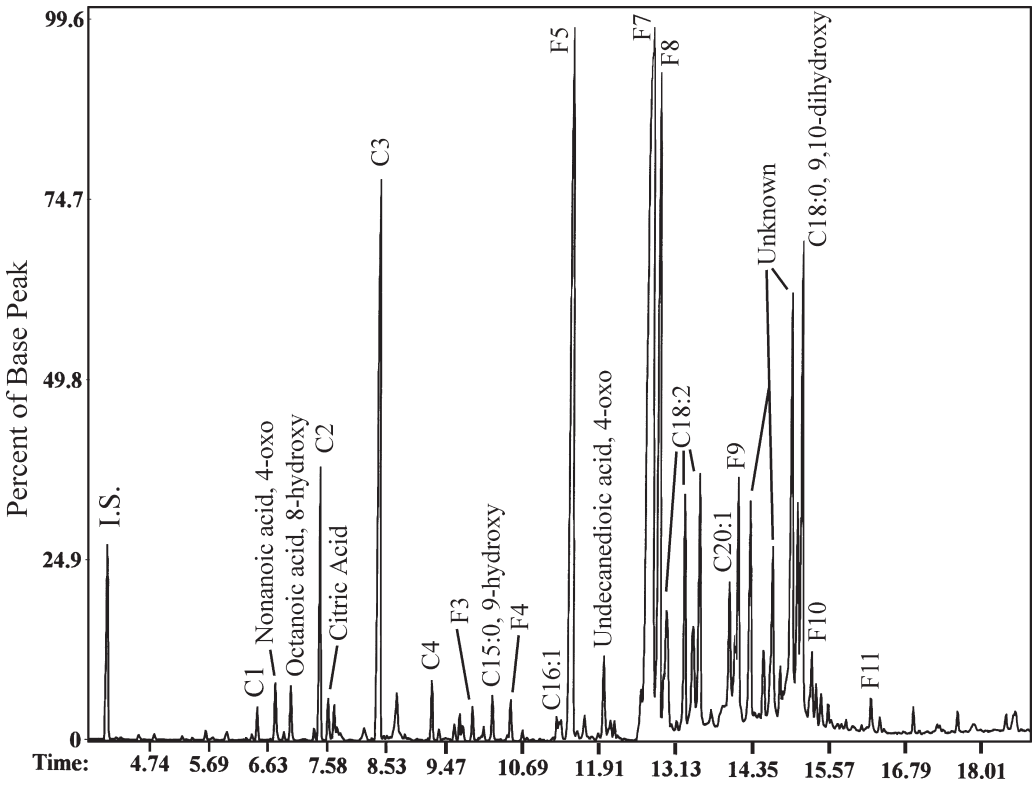

Figure 3 *The gas chromatogram of a test cooking pot containing piñon nuts.*

Instead, it appears that the pot itself was used to render pitch or tar through the boiling of piñon resin. The ethnographic record in the Great Basin gives a number of uses for piñon tars and pitches (see, e.g., Stewart 1942, 271; Kelly and Fowler 1986; Zigmond 1986). For example, pitch was commonly used as a sealant on baskets to make them watertight and to reinforce and seal the soles of shoes. Pitch was also used as a glue; for example, to haft projectile points to foreshafts, to cement bone points to harpoon foreshafts and to glue other items together. Boiled piñon resin was also used to dress wounds and sores, and it was used as a cure for rheumatism, tuberculosis and indigestion (Balls 1962, 28; Bennett and Zingg 1976, 60).

Unfortunately, the method of rendering pitch is not given in any of these ethnographic accounts. However, the current findings suggest that the boiling of piñon pitch in pots was at least one method that Shoshone people used to accomplish this task.

CONCLUSIONS

The finding of diterpenoids in a sherd from the Western Great Basin adds to our knowledge of the range of uses for prehistoric Great Basin pots. A study of organic residues in 75 sherds (including this sherd) from the Western Great Basin suggested that pots in this area were primarily used to cook plants, especially seeds (Eerkens 2000). JEC294 was the only sample containing terpenoid acids, indicating that the boiling of pine resins in ceramic pots was uncommon, but occasionally undertaken. On the basis of JEC294, it is clear that the range of

uses for prehistoric ceramic vessels in the Great Basin extended beyond merely cooking food, and included the processing of other materials as well.

The fact that these compounds were found in a sherd collected from the surface of an archaeological site suggests several points of relevance to organic residue studies. First, it is clear that coniferous terpenes, particularly pimaric and abietic acids, survive quite well for centuries or more in archaeological pottery samples. There does not appear to be significant deterioration of these compounds relative to that extracted from raw piñon resin (cf., Fox *et al*. 1995). For example, dehydroabietic and 7-oxodehydroabietic acids, common byproducts of diterpenoid oxidation (see Pollard and Heron 1996, 247), form only a minor fraction of the compounds recovered in JEC294. Indeed, others report that diterpenoids, especially pimaranes, are quite stable over long periods of time (Pollard and Heron 1996; van den Berg *et al*. 1996).

Second, the fact that this sherd was recovered from the surface of an archaeological site, rather than in an excavated context, demonstrates that surface sherds can still yield important information concerning pot use and function. Certainly, the arid nature of the region, with rainfall averaging less than 15 cm per year, does not promote degradation of organic residues through hydrolysis. Furthermore, these sherds are not surrounded by soil, and are less apt to be contaminated by fatty acids and other organic compounds found within the soil matrix. In this respect, surface sherds that have not been subjected to sandblasting and other degradative processes may provide the archaeologist with excellent opportunities to extract intact and unaltered organic residues.

Finally, the discriminatory power of lipids residue analysis can occasionally be quite good, allowing genus- or even species-level identification of the foods or other products cooked, processed or prepared in ceramic vessels. Unfortunately, the level of identification achieved in JEC294 was not realized in 74 other sherds analysed by GC-MS from the region (Eerkens 2000). The identification of biomarkers that are unique to different species and genera, then, will be crucial in this regard given the analytical methods currently available to archaeologists.

ACKNOWLEDGEMENTS

This research was funded by a dissertation improvement grant from the National Science Foundation (no. 9902836). I kindly thank Dr Robert Yohe, Dr Hector Neff and Dr Elizabeth Dixon for reading and reviewing earlier versions of this paper. I also thank Dr Colleen Beck of Desert Research Institute for access to the Nevada Test Site ceramic collections, and Dr Stuart Smith and Dr James Pavolovich for access to and assistance with the GC-MS and other analytical equipment.

REFERENCES

Balls, E. K. 1962, *Early uses of Californian plants*, University of California Press, Berkeley.

Bennett, W. C., and Zingg, R. M., 1976, *The Tarahuamara: an Indian tribe of New Mexico*, University of Chicago Press, Chicago.

Christie, W. W., 1989, *Gas chromatography and lipids*, Oily Press, Ayr, Scotland.

deMan, J. M., 1992, Chemical and physical properties of fatty acids, in *Fatty acids in foods and their health implications* (ed. C. K. Chow), 17–45, Marcel Dekker, New York.

Eerkens, J. W., 2000, *The origins of ceramics among late prehistoric hunter–gatherers in California and the western Great Basin*, Unpublished Ph.D. dissertation, Department of Anthropology, University of California, Santa Barbara.

Fowler, D. D., Bath, J., and Budy, E., 1977, *Report on the cultural resources inventory on the Nevada Test Site July through November 1977*, Report submitted by Desert Research Institute to US Department of Energy NV-00 under contract EY-76-C-08-1253.

Fox, A., Heron, C., and Sutton, M. Q., 1995, Characterization of natural products on Native American archaeological and ethnographic materials from the Great Basin region, USA: a preliminary study, *Archaeometry*, **37**, 363–75.

Frankel, E. N., 1980, Lipid oxidation, *Progress in Lipids Research*, **19**, 1–22.

Frankel, E. N., 1987, Secondary products of lipid oxidation, *Chemistry and Physics of Lipids*, **44**, 73–85.

Gülacar, F. O., Buchs, A., and Susini, A., 1989, Capillary gas chromatography – mass spectrometry and identification of substituted carboxylic acids in lipids extracted from a 4000-year old Nubian burial, *Journal of Chromatography*, **479**, 61–72.

Hudlicky, M., 1990, *Oxidations in organic chemistry*, American Chemical Society, Washington, DC.

Kelly, I. T., and Fowler, C. S., 1986, Southern Paiute, in *Handbook of North American Indians*, vol. 11: *Great Basin* (ed. W. L. d'Azevedo), 368–97, Smithsonian Institution, Washington, DC.

Langenheim, J. H., 1981, Terpenoids in the Leguminosae, in *Advances in legume systematics* (eds. R. M. Polhill and P. H. Raven), 627–55, Royal Botanic Gardens, Kew.

Lockett, C., and Pippin, L., 1990, Re-examining brownware ceramics in the central and southern Great Basin, in *Hunter–gatherer pottery from the far West* (ed. J. M. Mack), 67–82, Nevada State Museum Anthropological Papers no. 23, Carson City, NV.

Pippin, L. C., 1986, Intermountain brown wares: an assessment, in *Pottery of the Great Basin and adjacent areas* (ed. S. Griset), 9–21, Anthropological Papers, no. 111, University of Utah, Salt Lake City.

Pollard, M. A., and Heron, C., 1996, *Archaeological chemistry*, Royal Society of Chemistry, Cambridge.

Porter, N. A., Lehman, L. S., Weber, B. A., and Smith, K. J., 1981, Unified mechanism for polyunsaturated fatty acid autoxidation, *Journal of the American Chemical Society*, **103**, 6447–55.

Pridham, J. B. (ed.), 1967, *Terpenoids in plants: proceedings of the Phytochemical Group Symposium, Aberystwyth, April 1966*, Academic Press, New York.

Regert, M., Bland, H. A., Dudd, S. N., van Bergen, P. F., and Evershed, R. P., 1998, Free and bound fatty acid oxidation products in archaeological ceramic vessels, *Proceedings of the Royal Society of London B*, **265**, 2027–32.

Rhode, D., 1994, Direct dating of brown ware ceramics using thermoluminescence and its relation to the Numic spread, in *Across the West: human population movement and the expansion of the Numa* (eds. D. B. Madsen and D. Rhode), 124–30, University of Utah Press, Salt Lake City.

Shimoyama, A., Kisu, N., Harada, K., Wakita, S., Tsuneki, A., and Iwasaki, T., 1995, Fatty acid analysis of archaeological pottery vessels excavated in Tell Matsuma, Syria, *Bulletin of the Chemical Society of Japan*, **68**, 1565–8.

Skibo, J. M., and Deal, M., 1995, Pottery function and organic residue: an appraisal, in *Conference on Archaeology in South-East Asia* (eds. C. Yeung and W. B. Li), 321–30, University Museum and Art Gallery, Hong Kong.

Stern, B., Heron, C., Serpico, M., and Bourriau, J., 2000, A comparison of methods for establishing fatty acid concentration gradients across potsherds: a case study using late Bronze Age Canaanite amphorae, *Archaeometry*, **42**, 399–414.

Stewart, O. C., 1942, Culture element distributions: XVII. Ute – Southern Paiute. *University of California Anthropological Records*, **6**(4), 231–356.

van den Berg, K. J., Pastorova, I., Spetter, L., and Boon, J. J., 1996, State of oxidation of diterpenoid Pinacae resins in varnish, wax lining material, 18th century resin oil paint, and a recent copper resinate glaze, in *ICOM Committee for Conservation 11th Triennial Meeting, Edinburgh, Scotland 1–6 September 1996: Preprints*, vol. 2 (ed. J. Bridgland), 930–7, James and James, London.

Zavarin, E., and Snajberk, K., 1980, Oleoresins of pinions, *Journal of Agricultural and Food Chemistry*, **28**, 829–34.

Zigmond, M., 1986, Kawaiisu, in *Handbook of North American Indians*, vol. 11: *Great Basin* (ed. W. L. d'Azevedo), 398–411, Smithsonian Institution, Washington, DC.