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Journal of California and Great Basin Anthropology

Title

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Permalink

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Journal

Journal of California and Great Basin Anthropology, 20(1)

ISSN

0191-3557

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Publication Date

1998-07-01

Peer reviewed

The Chemistry, Archaeology, and Ethnography of a Native American Insect Resin

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The resin exuded by the North American lac scale insect Tachardiella larrae Comstock has been used in the past by peoples of the Great Basin region as a sealant, an adhesive, a modelling material, and for medicinal purposes. Such use has been reported ethnographically, but identification of the resin surviving on archaeological materials has been problematic until recently. This study seeks to examine the chemistry and use of the resin by examination of its working properties and through the application of a range of analytical techniques to authentic, experimental, and archaeological samples.

ETHNOGRAPHIC studies (Coville 1892:361; Essig 1931:19-21, 1934:182) and archaeological evidence (Sutton et al. 1987:240-242; Pinto 1989; Fox 1993) have shown that many natural products were used in antiquity in the Great Basin region, such as lac, pine, and juniper resins. Lac resin, secreted by the lac scale insect (*Tachardiella larrae*), was used for a variety of purposes; as a sealant, an adhesive, and for medicinal purposes (Essig 1931:19-21; Euler and Jones 1956; Sutton 1990:262). This article reports the analysis and experimental preparation of insect resin from the southwestern Great Basin region.

The extensive use of plant and insect resins throughout the world can be attributed to three factors: the wide availability of the materials, their durability and resistance to degradation (a characteristic crucial to their function in host organisms), and most importantly, to the array of uses for which they can be employed. Notable attributes include adhesiveness, insolubility in water, plasticity, vitreosity, colorability, and resistance to spoilage (Gianno 1990:1). Unequivocal identifications of archaeological resin samples are not common, but it is likely that considerable numbers of residues have simply been overlooked. If the material can be characterized

by chemical means, then more artifacts displaying the use of lac may come to light. Specific identification of lac resin would increase the potential for studies of distribution, technology, and patterns of exchange/mobility.

Recent work has examined natural resins from the region and demonstrated the potential for their differentiation on the basis of chemical characteristics (Fox et al. 1995). However, since North American lac has never been commercially exploited, its chemical composition has remained largely unknown; the work of Fox et al. (1995) identified some major components, but not the minor constituents or the nature of the insoluble component. The aim of the work reported here is to further elucidate the composition of the resin, to create a fingerprint that will facilitate confident identification of the resin in archaeological materials, and to examine the effect of preparation practices on both the chemistry and properties of the resin.

INSECTS AND INSECT PRODUCTS AS A RESOURCE

The material culture of the Great Basin has been characterized by an efficient exploitation of the available resources. The extensive use of all

types of insects by the native peoples of this region has been well documented ethnographically, in particular by Essig (Essig 1931:12-39, 1934; also see Bodenheimer 1951; Sutton 1988). Insects were exploited as an important food source; termites, leather jackets, maggots and the larvae of bees, wasps, ants, and wood boring beetles were all eaten, and have been shown to be of substantial nutritional value (Bodenheimer 1951; Brothwell and Brothwell 1969:68-69; Ebeling 1986:400; Sutton 1988, 1995:259). Some insects were harvested in large numbers, providing a considerable food supply; for example, the caterpillars of the Pandora moth (*Coloradia pandora* Blake), and grasshoppers when they swarmed (Essig 1931:23-24; Bodenheimer 1951:292; Madsen and Kirkham 1988:593; Sutton 1988). Insect consumption is extensively documented in trapper journals and pioneer diaries of the middle to late nineteenth century, and a similar variety of preserved insect remains has been observed at archaeological sites around the Basin (Madsen and Kirkham 1988:593, 596-597).

Insect products such as cocoons were also utilized (Essig 1931:20-21; Sutton 1995:261), but the products of the scale insects (Coccoidea) present the greatest diversity of use potential, and where available, have been used throughout the world for resins, waxes, dyes, and honeydew.

Insect Resins: A Brief Overview

The exploitation of the various products of the scale insect is not a phenomenon unique to Native American culture. Several familiar red dyes can be obtained from different species of scale insect. The red dye kermes from *Kermococcus vermilia* Planch, native to the Middle East and Mediterranean, and cochineal from the Mexican insect *Dactylopius coccus* have a long history of use (Linsenmaier and Chadwick 1972:118; Borrer et al. 1976:320; Goffer 1980:186-187; Ross 1986).

The waxes and resins produced by the Coc-

cidae have also been well utilized. Because of its commercial value, probably the most well known of these is "shellac," a derivative of the resin produced by a number of Asian scale insects, notably *Laccifer lacca* Linnaeus, which feeds on *Butea monosperma* Lamk. This substance has been used as a varnish on a wide range of materials, particularly furniture and paintings, and has found a broader range of uses in the twentieth century; for example, as an insulating material (Mills and White 1994:115-116). Candles have been produced from the pure white wax of *Ericerus pe-la*, the Chinese wax scale insect, and in Africa a root infesting genus, *Margarodes*, produces waxy cases with an attractive metallic or golden gloss. They are mined as "earth pearls" and worn as jewelry (Linsenmaier and Chadwick 1972:118; Borrer et al. 1976:315-318). In some areas, scale insect exudates have been utilized for their nutritional value. The honeydew excreted by the tamarisk manna scale (*Trabutina mannipara*), which feeds on desert tamarisk trees of the Mediterranean and Near East, is a particularly rich carbohydrate source and has long been used as a foodstuff (Linsenmaier and Chadwick 1972:118; Brown 1975:227; Borrer et al. 1976:319-20).

Lac Resin: Ethnography and Archaeology

There is more than one resin-producing scale insect in the Great Basin region. The oak wax scale (*Cerococcus quercus* Comstock), which infests oak trees (*Quercus* spp.) in the semiarid mountain regions of California, and the irregular wax scale (*Ceroplastes irregularis* Cockerell), which inhabits various species of saltbush (*Atriplex* spp.), were also exploited (Essig 1931:183; also see Sutton 1990:262). The oak wax scale produces globular masses of wax of bright yellow color and was commonly used as a form of chewing gum (Borrer et al. 1976:319). The genus *Tachardiella* is represented by three species, each specific to a particular plant. *Tachardiella fulgens* Cockerell inhabits the *sámota*

bush (*Coursetia glandulosa*) (Felger 1977:159; Felger and Moser 1985), and *Tachardiella glomerella* Cockerell has been observed to infest sagebrush (Chamberlin 1923:180). *Tachardiella larrae*, associated with the creosote bush (*Larrea tridentata*), is by far the most abundant, though all were utilized.

Essig (1931:19-21, 1934:182) reported the use of insect resins as an adhesive for fastening the sinew backing of bows, as a sealant in the waterproofing of baskets, and as a chewing gum. Use of these resins has also been described for the hermetic sealing of pots (Euler and Jones 1956; Felger 1977:158-159). Coville (1892:361) noted its use in the fastening of stone arrowheads in their shafts and observed the lid of a sugar bowl that had been mended with the adhesive. Further uses from the ethnographic literature include the gluing of feathers to arrows, and hafting of knives and awls (Sutton 1990:265). The resin was also exploited for other purposes; for example, infusions of melted lac resin in water were drunk by Seri women as a contraceptive (Felger and Moser 1985:386).

There is some inconsistency in nomenclature in the literature. For example, the resins are often referred to as "greasewood gum" and "creosote gum." The creosote bush itself does not yield any useful resin (Sutton 1990:267), although phenolic-type resins derived from the leaves have in recent years been examined for their commercial potential as antioxidants and fungicides (Campos-López and Román-Alemany 1980). However, while the creosote bush foliage has medicinal applications, there is no ethnographic indication that any resins or other substances have ever been isolated from the plant itself; the various names can therefore be assumed to describe lac resin.

The few archaeological samples of lac that are documented support the ethnographic reports of use. However, these identifications tend to be based on attributes such as color, and while consistent with the properties of the resin ob-

served in this study, their specific chemical identity remains unknown. For example, Pinto (1989) identified lac as the mastic used in the hafting of blade artifacts from excavations at Mitchell Caverns. Pottery vessels from Southcott Cave, dating from A.D. 1000 to 1900, have shown evidence of hermetic sealing with dense red pitch in the covering of a storage vessel and lid. Analysis by gas chromatography (GC) suggested a lac origin (Sutton et al. 1987:240-242). Particularly interesting is the reported recovery of an intact sealed vessel dating from A.D. 1300 with preserved contents of mescal, again with a resin sealant identified as lac (Euler and Jones 1956).

Lac resin has also been reported on artifacts from a Pueblo III burial, where the resin was used as a mounting material for the formation of turquoise mosaic objects, as well as being modelled to produce bird-shaped ornaments. There was also evidence of its use as a hafting material on stone points, and as an adhesive in the manufacture of ceremonial objects (McGregor 1943:288-289, 292-294). More recently, Fox (1995) identified lac on several archaeological samples, again by gas chromatographic fingerprinting. Residue from the inside (presumably as a sealant) of an intact and painted prehistoric Cahuilla ceramic olla was positively identified as lac. Adhesives from the base of an obsidian point and from native repair of an olla recovered from a cave in Imperial County, California, were also assigned a lac scale origin (Fox 1995:82).

TACHARDIELLA LARRAE, THE LAC SCALE INSECT

The creosote bush (*Larrea tridentata*), host to *Tachardiella larrae*, is the most conspicuous of desert plants in the southwest region (Leake et al. 1993:85). It is widespread in the Mojave, Sonoran, and Chihuahuan deserts and the south (Sutton 1990:262; Grayson 1993:22). The multiple stemmed plant has very hard wood and produces bright yellow flowers and resinous foliage

in response to any rainfall, as well as a strong odor from which it derives its name (Felger and Moser 1985:384; Ebeling 1986:126). Products of the bush itself were utilized, notably a yellow pigment that was used for painting and dyeing. Medicinal applications of the foliage included smoldering poultices for the relief of swelling and pain, and a tea brewed for the treatment of breathlessness. Infusions were also prepared from the roots to cure dizziness (Felger and Moser 1985:386; Leake et al. 1993:85).

The distribution of the lac scale insect follows that of the host plant (Colton 1943; Sutton 1990:262), though infestation of the bushes has been reported to be densest in southern Mojave County, Arizona, and the northern part of Yuma County (Colton 1943:23). Colton (1943) conducted the first detailed study of the life history of the insect. *T. larrae* broods but once a year, with small red larvae produced in late February or early March. The young leave the female lac cell immediately after birth. They do not move far from the parent cell and are unwilling to infest new branches. Once attached to the branch with their mouthparts they immediately begin to secrete lac (Colton 1943:25).

Lac is a product of gland cells distributed in the integument of the insect, and is secreted as a protective covering to prevent desiccation and predation (Imms et al. 1957:170; Jackson and Blomquist 1976). Sufficient lac is produced to cover the insect entirely, forming a lac cell, within which the male will metamorphose to emerge as a tiny red fly. The female remains within the lac cell for the duration of her life, and the male copulates with her there while she is still immature; he dies shortly after mating has occurred.

The embryos developing within the female may number 50 to 100, and as they mature the internal organs will reduce and eventually disappear so that when the larvae are born and leave the lac cell, the female body collapses and dies (Colton 1943:27-29). Since the larvae tend

to infest only the parent branch, thick layers of the red lac cells may be laid down, encasing the whole branch. Colton (1943:25) observed the wide variation in degree of infestation of bushes, which on his inspection ranged from 0 to 54%.

TECHNOLOGY, PROPERTIES, AND PROCUREMENT

The earliest investigations of lac were undertaken in the 1940s, when interest was aroused as to the potential of the substance to supplement the use of shellac in the manufacture of varnishes (Colton 1943; Westgate 1945:190-191). Economic studies had been undertaken (Ferris 1919), which indicated that in one day one person could collect 75 pounds of branches well covered with wild lac. This would then yield a pound of lac resin. Further studies of this nature were abandoned once it was ascertained that lac was not economically viable for commercial purposes. More recently, Sutton (1990:263) reported being able to collect several ounces of resin from the wild in a matter of minutes; raw lac is then removed manually from the branches. Indeed, ethnographic accounts have reported the lac being picked from the branches and put into a receptacle, with a hot coal held nearby so that the pieces melted together into one globule, more suitable for carrying. Seri men apparently kept such pieces in their arrow quivers ready for use (Felger and Moser 1985:384-385).

Raw lac, as scraped from the branch, is hard and brittle, and contains a lot of woody material, bark from the twig, live and dead scale insects, and possibly other infesters of the host plant (Westgate 1945:197). Westgate (1945) reported a variety of laboratory determinations, including solubility, volatility, and drying properties, but no detailed accounts were given. Similar tests by Sutton (1990:263-264) are reported in more detail. These mostly involve heating, during which the resin softens at around 121° C, becoming darker in color as well as pliable and sticky, but does not melt below about 221° C.

Sutton (1990:264) also reported rapid cooling of the material, while regaining its brittleness and hardness. Coville (1892:361) noted that in preparing the resin, Native Americans would grind the resin with pulverized rock. Since it seems evident that the resin would have been heated before use (Sutton et al. 1987:240), it seems likely that to aid in the heating process, powdered rock was added to grind the resin more finely. In addition, the rock should have conserved heat and hence cooling would have been retarded, thereby allowing the resin to be more easily manipulated. Sutton (1990:264) conducted some simple preparation experiments to test these ideas, but found no alteration to the substance by such additions. The fact that small nodules of resin were carried for "on-the-spot" use suggests that melting of the resin for use cannot have been an elaborate procedure.

THE CHEMISTRY OF LAC RESIN

All insects secrete some kind of waxy coating, primarily as a protection against desiccation. In the scale insect, this capacity is taken to an extreme, since sessile existence for a large part of its life cycle necessitates a response to the problem of predation; and in the desert environment experienced by *Tachardiella*, the additional problem of water loss is exacerbated. Compositionally, insect waxes share a general range of molecular constituents; hydrocarbons, wax esters, aliphatic alcohols, and free fatty acids. However, their specific chemistry is highly variable and the products of the scale insects are usually characterized by long chain ester and hydroxyfatty acid components. Branched hydrocarbons that are usual for insect waxes are much less common in the scale exudates, and longer chain primary alcohols (C_{22} - C_{24}) are particularly common (Jackson and Blomquist 1976: 211-213).

The chemistry of the resin produced by *Laccifer lacca* Linnaeus, the Asian lac scale, is particularly well established due to its commercial

importance in the production of shellac (for detailed accounts of the chemistry of the material, see Singh et al. 1969, 1974a, 1974b; Wadia et al. 1969; Khurana et al. 1970; Upadhye et al. 1970a, 1970b; Yates and Field 1970; Yates et al. 1970). Shellac is known to be made up of low molecular weight polymers (oligomers) of esterified polyhydroxy carboxylic acids. The acid constituents are of two types; aliphatic compounds related to common fatty acids and alicyclic sesquiterpenes. The major aliphatic constituents are aleuritic acid, (9,12,15-trihydroxypalmitic acid) and butolic acid (6-hydroxytetradecanoic acid). The alicyclic acids are derivatives of the sesquiterpene cendrene. These constituents are assembled into polymers and dimers to form a continuous range of oligomers with a range of molecular weights (Mills and White 1994:117).

The soluble dye components of Asian lac have also been examined. Water soluble dye components responsible for the red and brown colors of the resin have been identified (Wright 1949; Vollman 1957). Elsewhere, however, the red dye has been reported as a product of the crushing and washing of the dead insects (Goffer 1980:187), strongly suggesting that the dye constituents derive from the remains of dead insects trapped within the lac rather than from the resin secretion.

Compared with the wealth of information concerning shellac, the chemistry of the resin produced by *T. larrae* is poorly understood. The earliest studies were carried out by Colton (1943), who established the percentage composition of the components of the resin by fractionation. He also noted the compositional difference between lac and Asian lac, the latter having a higher proportion of resin and less wax (Colton 1943). It has been observed that *L. lacca* flourishes best on plants containing gums or resins or which are rich in certain types of latex. Furthermore, the food plant influences the color and quantity of the lac produced (Imms et al. 1957:

170). If this is the case, then the compositional differences between the resins produced by the different insect species could be as much a result of the chemical nature of the food plant as of the difference between the insects themselves.

Ethnographic accounts have reported that the lac produced by the different species of *Tachardiella* have identical properties and uses (Felger and Moser 1985:326), despite their preference for different plant hosts. However, the chemistry of these resins is completely unknown, and so the relationship between composition and technological properties remains to be understood. The soluble dye components of the resin have been reported briefly and shown to be highly similar to that of Asian lac, though with a much more abundant yellow dye portion (Wouters and Verhecken 1989:194). This reflects the yellow dye content of the host plant, and emphasizes the way in which the plant and resin chemistry may be linked.

Although gas chromatography has been used in basic characterization of lac (Sutton et al. 1987), there have been no comprehensive attempts to characterize the resin until recent years (see Fox et al. 1995; Stacey et al. 1995). These preliminary studies used gas chromatography (GC) and combined gas chromatography/mass spectrometry (GC/MS) to examine both raw samples of the resin and a variety of archaeological samples, in order to investigate chemical composition and identification of the material. The principal constituents of the resin were identified in these studies; tetradecanoic (myristic) acid, hexadecanoic (palmitic) acid, 7-hydroxyhexadecanoic acid, and 7-hydroxytetradecanoic acid, the latter comparing well with the 6-hydroxytetradecanoic acid reported in Asian lac (Mills and White 1994:117; Fox et al. 1995:371). The minor constituents remain unidentified, and a portion of the samples is suggested to comprise a biopolymer similar to that identified in Asian lac, although this remains to be confirmed.

SAMPLES AND SAMPLE PREPARATION

Table 1 summarizes the samples and analyses undertaken. The authentic samples derived from a twig of creosote bush, fully encrusted with resin up to 4 mm. thick in places. Samples of between 20 and 100 mg. were removed with a fresh scalpel blade and transferred to sample vials for extraction. Three samples were taken from the raw lac resin, so that the degree of chemical homogeneity of the resin could be observed.

A sample of melted lac, originating from the heating experiments conducted by Sutton (1990:264), was also available for analysis. Since the conditions and precise heating temperatures for the preparation of this material was not documented in detail, the analysis of these samples served more as a support for samples deriving from the more detailed heating experiments undertaken as a part of this investigation. These experiments are discussed below.

An archaeological sample of possible lac origin was also examined. This sample originates from Newberry Cave in the Mojave River Valley (Davis and Smith 1981). This site is dated between 1,780 and 1,020 B.C., and has been interpreted as having had a magico-religious hunting function. One of the artifacts recovered from the site is a quartz crystal with resinous material adhering to one end, which possibly served to haft the stone to form a wand (Davis and Smith 1981:36). The small quantity of this hafting material that was available was sampled as described above, though sample sizes were smaller (approximately 3 mg.).

EXPERIMENTAL METHODS

The crumbled resin samples were extracted with 0.3-0.8 ml. chloroform and methanol (v/v 2:1) and warmed gently on a heating plate. Some samples were subjected to extended immersion in the solvent at room temperature for several days. Between 50 and 100 μ l of the su-

Table 1
SAMPLES AND ANALYSIS

Lab Reference No.	Description	Analysis
618	Authentic Native American lac resin (raw), coating a twig of creosote bush	GC, GC/MS, Py/GC, saponification
19	melted raw lac resin, from the experimental heating study carried out by Sutton (1990:264).	GC
18200	Melted sample from heating experiments, heated to 200° C	GC
18400	Melted sample from heating experiments, heated to 400° C	GC
17	Resin adhering to a quartz crystal from Newberry Cave in the Mojave Desert	GC, GC/MS, Py/GC, saponification

pernatant were then transferred, by Pasteur pipette, into a fresh vial and reduced to dryness under a stream of nitrogen.

Saponification procedures were applied to both the authentic and archaeological samples, principally to examine how difficult the polymerized component of the resin was to break up, but also to examine the extent of preservation of this portion in the ancient material. Samples as described above were heated (c. 70° C) for about two hours with 1 ml. of 0.5 M potassium hydroxide in methanol. It was observed that more material dissolved under this treatment, but a small quantity of insoluble matter persisted. On cooling, samples were acidified with a drop of 5 M hydrochloric acid and around 0.5 ml. hexane added.

Brisk agitation followed to redissolve as much soluble material as possible to the hexane layer, which was removed with a Pasteur pipette to a fresh vial. This process was repeated twice to ensure maximum extraction of soluble components. The hexane was evaporated by heating under a stream of nitrogen to produce a dry residue which was prepared for GC analysis following the procedure outlined below. A control sample of pure hexane was also dried and derivatised.

Samples were derivatised by the addition of 50 ml. of *N,O*-bis (trimethylsilyl) trifluoroac-

tamide (BSTFA) + 1% trimethylchlorosilane (TMCS), with 50 μ l of pyridine to facilitate the full dissolution of the residue. The sample was heated to 60° C on a heating plate, before being thoroughly redried under a stream of nitrogen and redissolved in 50 ml. of cyclohexane, prior to injection into the GC.

Gas chromatography was carried out using a Hewlett Packard 5890 series II gas chromatograph, equipped with flame ionization detector. Samples were introduced by on-column injection into a 60 cm. x 0.32 mm. i.d. retention gap of deactivated polyamide clad fused silica connected to the analytical column via a glass capillary connector. The analytical column was a polyamide clad 12 m. x 0.22 mm. i.d. fused silica capillary coated with BPX5 stationary phase (modified 5% phenyl 95% methyl siloxane, 0.25 μ m film thickness; SGE, UK). The carrier gas was helium at a constant flow of 1 ml. min⁻¹. The temperature of the oven was programmed from 50 to 370° C at a rate of 10° C mins⁻¹ following a two-minute isothermal hold at 50° C after injection, with the final temperature held for eight minutes. The data were stored and processed using Hewlett Packard 3365 Chemstation software.

Combined GC/MS was carried out using a VG 12-253 quadrupole instrument operating at 70 eV. The instrument contained a splitless

injector at 250° C, and a column of polyamide clad fused silica capillary (25 m. x 0.32 mm. i.d.) coated with OV-1 stationary phase (film thickness 0.1 μm). The carrier gas was helium, and temperature programming of the oven was from 50 to 350° C at a rate of 10° C min^{-1} . The mass spectra were recorded over a mass range of 50 to 650 μ .

The application of pyrolysis gas chromatography (Py/GC) to some of the samples offered the opportunity to examine the polymerized component of the resin. Such detailed analysis of the lac resin had never been undertaken before and these analyses constitute a preliminary study to examine the potential of this approach.

The pyrolysis was carried out at 700° C using an SGE PIU-II pyrolysis unit fitted to a Hewlett Packard 5890 Series II gas chromatograph with a flame ionization detector. Samples were introduced to the pyrolysis unit by solid sampling kit punch probe and entered into the chromatograph column via a split injector (150:1) at 375° C. The analytical column was a 25 m. x 0.22 mm. i.d. fused silica capillary coated with BPX5 stationary phase (0.25 μm film thickness). The carrier gas was helium and the oven temperature was programmed from 40 to 360° C at a rate of 10° C min^{-1} following a 0.5-minute isothermal hold at 40° C after injection, with the final temperature held for five minutes.

The Heating Experiments

The experimental heating, undertaken to examine the behavior of the resin during processing, sought to identify how the resin might have been utilized in the ways that have been described. Sutton's (1990) experiments were repeated in order to examine the melting process at a wider range of temperatures under more controlled conditions, and to provide melted samples with known melting temperatures for GC analysis.

Two 5-g. samples of the raw resin were finely ground with a ceramic pestle and mortar,

one with an equal weight of sand and one without. The samples were placed in glass crucibles in a preheated furnace at 150° C, and left to melt for 20 minutes. They were then removed, and the color, consistency, and any other notable observations recorded. They were then returned to the oven at the higher temperature of 200° C and allowed to melt for a further 20 minutes. This procedure was repeated increasing the temperature by stages to 250° C, 300° C, and 400° C, allowing 20 minutes at each stage. After heating to 200° C and 400° C, 2 to 5 mg. samples were taken from the pure melted resin for analysis.

RESULTS

Heating Experiments

Observed changes in the resin as a function of temperature are shown in Table 2. The heating experiments raised a number of questions concerning the use and preparation of the resin. It was found that the resin could not be melted to produce anything resembling a liquid. At its most advanced stage of melting, the resin formed a tacky plastic mass; further heating resulted in charring. The addition of sand did not appear to improve fluidity or benefit the heating process in any way, and the sand content of the mixed sample served more to hinder than to enhance utility of the resin. The ethnographic observation of crushing with rock (Coville 1892: 361) bears witness to the hardness of the material and the difficulty in grinding it rather than to any thermal benefit.

GC and Py/GC

Figure 1 shows a chromatogram of samples of authentic lac. Comparison of the various samples that were analyzed indicated a good degree of chemical homogeneity in the material. The correlation of the principal peaks is good, although the much smaller peaks interspersed between these, especially before 15 minutes, are

Table 2
 PHYSICAL AND CHEMICAL CHANGES OF LAC RESIN WITH INCREASING TEMPERATURE^a

Temperature (°C)	Physical Changes: Ground Lac Resin	Physical Changes: Ground Lac Resin with Sand	Chemical Changes
150	Darkening in color, dark red. Sticky, fragments of resin sticking together and sticking to the dish. Soft, but not "gluey." Strong smell similar to burning rubber.	Becoming sticky, adhering to sand grains and to container. Darkening in color to red/brown.	Not analyzed
200	Very dark red. Further softening, but not fluid; soft enough to manipulate, plastic and tacky. Cools to brittle hardness almost immediately.	Dark red/brown. Sand and resin sticking together to form a soft, granular, gluey mass. Cools rapidly to brittle hardness.	Reduction in Peak 1. Peaks 3 and 5 unaltered. Peaks eluting after 20 mins. Also well-defined.
250	As above but more sticky, no increase in fluidity, firm and plastic. Rapid cooling to brittleness, dark red color retained on cooling.	Increased stickiness, but adhering more firmly to sand as a result. Swift cooling to form brittle, sand-encrusted granules.	Absence of Peak 1. Increased unresolved material.
300	Starting to burn, still soft. Almost "springy" in its firmness.	Increased softness, still granular, sticky and plastic.	Not analyzed
400	Burned, blackened, and smoking, still slightly sticky.	Burned, blackened, and firmly adhered to sand. Appearance and behavior similar to caramelized sugar.	No peaks, high molecular weight material eluting after 20 mins. Poorly resolved.

^a For peak identities, see Table 3.

more variable. The majority of the authentic samples exhibited a broad unresolved peak eluting after 25 minutes. However, in the example given in Figure 1, a number of distinct peaks was seen in this region. It appears from this evidence that the first half of the chromatogram is the most diagnostic, and forms a more reliable GC reference for comparison with aged material.

Samples deriving from the heating experiments were analyzed by GC and these results are summarized in Table 3 in relation to the physical changes that were observed with increasing temperature. Chemical changes that occur below 250° C are no more significant than differences associated with the general compositional variability of the resin; above this temperature, however, there is substantial alteration and decomposition.

In the chromatogram of saponified lac resin

(Fig. 1), it can be seen that while the three main components are still present, their relative abundance is severely reduced and the chromatogram has been transformed into a spread of several smaller and more well-defined peaks. The peaks present at longer retention times correlate well with minor peaks seen in the authentic samples.

Comparison of the data from authentic samples with that of the archaeological sample from Newberry Cave indicates substantial differences in both presence and abundance of the smaller peaks. However, the three main peaks, though slightly altered in relative abundance, show up well, and the similarity of the chromatogram to those of the authentic samples clearly indicates a lac scale origin. This is supported by analysis of the saponified extract of the Newberry Cave sample, which shows an excellent match for the principal peaks seen in the authentic lac subjected to the same procedure. These samples

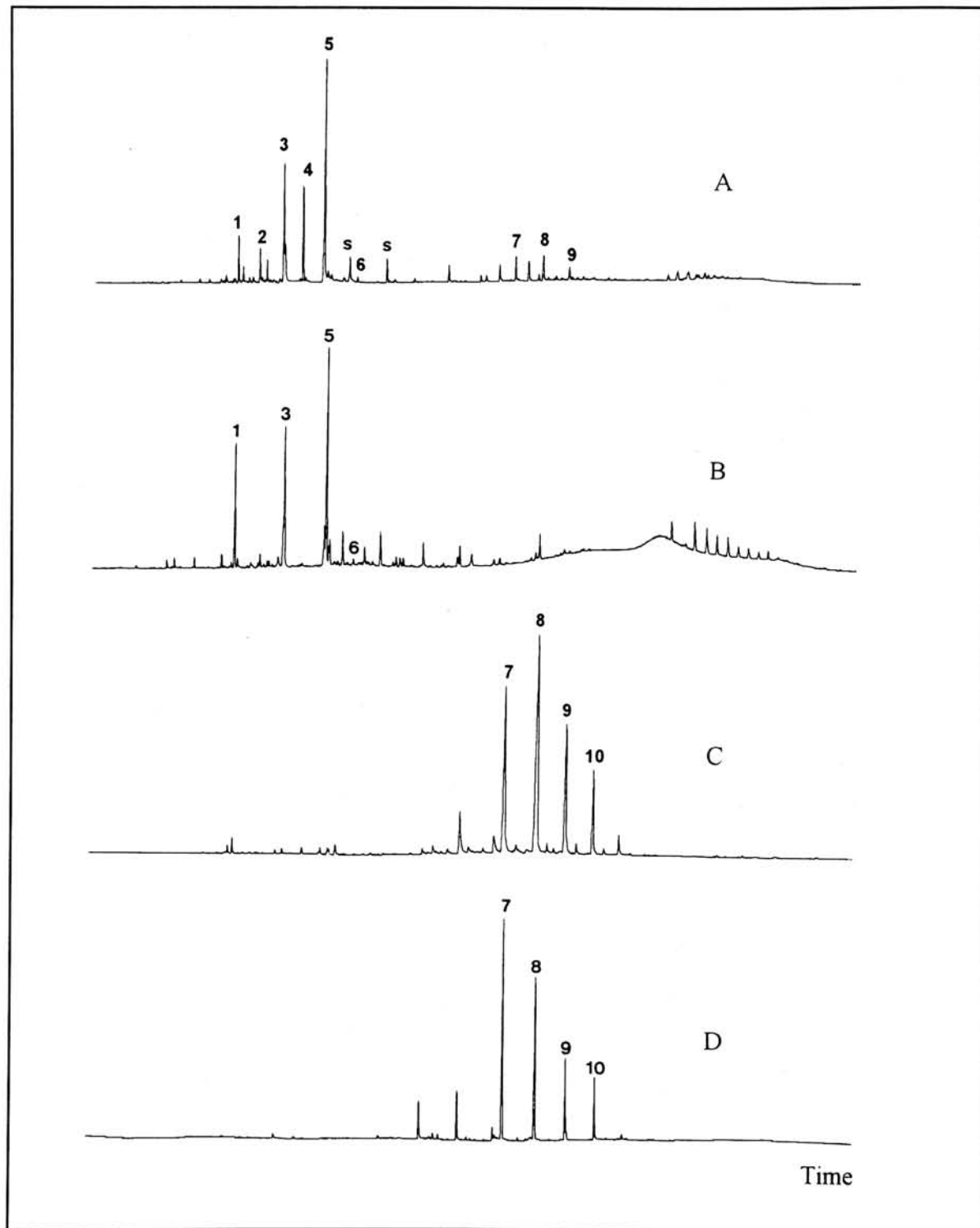
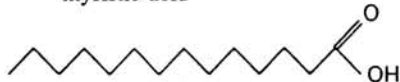
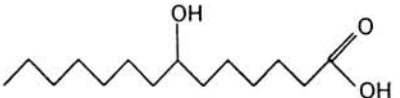
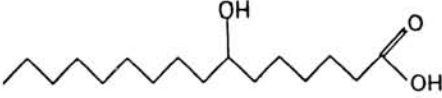
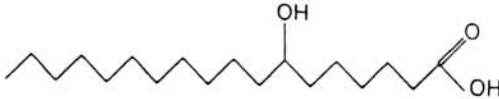






Fig. 1. Gas chromatograms: (a) authentic lac resin; (b) archaeological sample from Newberry Cave; (c) saponified lac resin; (d) saponified resin from Newberry Cave. For peak identities, see Table 3. Peaks marked "s" are possible sugars. Retention times for all four chromatograms correlate.

Table 3
PEAK IDENTIFICATIONS AND STRUCTURES BASED ON THE GC/MS OF TMS DERIVATIVES^a

Peak No.	Diagnostic TMS Fragment Ions ^b (<i>m/z</i>)	Structure and Identity
1	117, 129, 132, 145, 285	myristic acid 
2	201, 231, 315	not determined
3	201, 289, 373	7-hydroxytetradecanoic acid 
4	229, 231, 343	not determined
5	229, 289, 401	7-hydroxyhexadecanoic acid 
6	257, 289, 429	7-hydroxyoctadecanoic acid 
7	75, 83, 103, 125, 467	octacosan-1-ol 
8	75, 83, 103, 125, 495	triacontan-1-ol 
9	75, 83, 103, 125, 523	dotriacontan-1-ol 
10	73, 83, 103, 125, 551	tetratriacontan-1-ol 

^a *Italicized* ions were used for structure elucidations, ***bold italicized*** ions are [M-15]⁺.

^b All samples were analyzed as trimethylsilyl (TMS) derivatives. Details of the fragmentation patterns for TMS derivatives and method of structural elucidation of these ions are given in Fox et al. (1995).

were also examined by Py/GC. The extreme fragmentation patterns produced a radically altered chromatogram with an abundance of well-defined peaks. Again, the similarity of the Newberry Cave sample and the authentic lac can be emphasized; the correlation between the two samples appears to improve upon the results of the standard GC analysis.

GC/MS

The GC/MS analyses produced a far wider set of data than has previously been accomplished in the examination of this material (Fox 1993; Fox et al. 1995). In particular, three further compounds which comprise part of the diagnostic region identified on gas chromatograms were tentatively identified. Tentatively, two unsaturated hydroxyfatty acids were identified, although further analysis is required to determine the location of the double bond in the carbon chain. A higher molecular weight hydroxyfatty acid, 7-hydroxyoctadecanoic acid, was also identified. Table 3 gives details of the GC/MS data and structural elucidations. All of the peaks that have been identified as a result of this and previous studies are indicated in Figure 1 (with the exception of palmitic acid, which does not feature in the authentic samples reported here).

The GC/MS data obtained from the Newberry Cave archaeological material were comparable to the authentic sample in its abundance and complexity. The data allow confident matching of many of the major peaks to samples of authentic lac, although their relative abundances are considerably altered. However, a number of other peaks that are as yet unassigned are also evident in this material. Similarly, a wide range of minor components not seen in the authentic lac are also present, although the quality of the spectra for these is not of a standard to allow confirmation at this stage.

The GC/MS data of the saponified samples facilitated the identification of the series of peaks seen to elute after 25 minutes in the GC analy-

ses. These are long chain primary alcohols. Structural information from GC/MS from these molecules is limited but the m/z 103 peak is diagnostic and $[M-15]^+$ ions were observed in all cases (see Table 3). The four alcohols identified had chain lengths of C_{28} , C_{30} , C_{32} , and C_{34} .

A number of peaks was observed (marked with an "s" on Figure 1) with fragment ions of m/z 129, 217 and 204. These are tentatively assigned to sugars. In addition, a number of n -alkanes were observed.

DISCUSSION

Reports of the use of lac resin are not entirely consistent with the way in which it melts. The resin has been observed to regain its hardness very rapidly upon cooling, thus acting as a strong bonding agent. One can envisage its use as a sort of adhesive "putty" in the hafting of tools, fastening of feathers to arrow shafts, and mending of pottery. In this form, it could also be used to seal baskets by molding onto the surface to produce an uneven, but watertight layer. This is consistent with the observation by Fox (1995:83) that the lac was more commonly used as a mastic or adhesive, whereas higher plant resins were usually exploited more for sealing purposes. It is plausible that this relates to differing technological properties of the materials.

The archaeological example of lac on a ceramic vessel from Southcott Cave, where "some of the material had 'run down' the inside of the jar indicating that the material had been applied in a liquid state, probably while hot" (Sutton et al. 1987:240), is less understandable in light of the experimental results. The vessel was part of a collection of ceramics with putative lac residues adhering to them, one of which was analyzed by GC. The example just described shares the rich red color of the other lac residues, and so the presence of lac in this residue seems likely. How the material was processed to form a liquid can only be explained conjecturally. Ethnographic sources note the use of lac resin as a

chewing gum and it is possible that such behavior represents part of the processing of the resin and that salivary enzymes break down the material into a more easily melted form. Alternatively, the residue may be a product of the mixing of lac with other resins, to produce more favorable thermal properties.

Combined GC/MS has been demonstrated to be a useful technique for the identification of lac resin (Fox 1993; Fox et al. 1995), and this is borne out by the results of this study. The variability in chemical composition of the material is clear, although the improvement in results for samples analyzed after a much longer period of immersion in solvent indicates that the standard extraction methods are perhaps inadequate for maximum extraction of soluble components. The variance in gas chromatograms produced by the lac has implications for the identification of lac where it is mixed with other resins. To date, only one such sample has been analyzed, an archaeological resin sample from a chuckwalla barb recovered from Breakfast Canyon, Death Valley, which was identified by GC/MS as a lac and pine mixture (Fox 1995:82; see also Yohe and Valdez 1996).

Fox (1993) has demonstrated that plant resins from the Death Valley region have substantially different chromatograms, but the variability of lac, combined with the unknown concentrations of different resins in mixed samples, may produce results which are problematic to interpret. Analyses involving saponification techniques present a possibility of overcoming this problem. A series of long chain alcohols was identified in both the authentic and archaeological samples. The saponified fingerprint is particularly useful for the purpose of identification because this region of the chromatogram is distinctive for lac compared with other resins that have been analyzed (see Fox et al. 1995). In addition, the alcohols could be diagnostic for lac insect resin. Analyses of other insect waxes show less abundance of such alcohols and differ-

ent chain lengths (Faurot-Bouchet and Michel 1964; Tulloch 1973). It is anticipated, therefore, that the fingerprint that has been defined is a suitable indicator for lac deriving from species of *Tachardiella*. However, this cannot be confirmed until other insect waxes from the area have been examined.

Though only very small samples have so far been analyzed, encouraging results have been obtained for the application of pyrolysis to this material. The preliminary analyses show excellent correlation between authentic and aged samples, and in combination with MS would allow a full analysis of chemical composition and thus facilitate detailed understanding of the way in which the material degrades with time. It would also offer an opportunity to determine the nature of the polymerized component. Such structural information may shed light on the response of the material to heat, the differences in properties of the material as compared with Asian lac, and especially comparisons with other resins from the Great Basin region, since it is the properties of the material that dictate the way in which it can be manipulated. If the pyrolysis fingerprint could be refined, it may provide a more reliable fingerprinting method than GC because it involves all of the material, including the insoluble polymer.

The GC/MS data have provided an opportunity to examine the relationship between the chemistry of the soluble portion of aged and authentic samples. The material has been shown to reach its maximum melted state at around 200° C. No major chemical alterations are induced by this, so it seems improbable that preparation by heating will influence the degradation process or products. Thus, the samples have been directly compared. Many of the compounds characteristic of authentic lac are retained in the Newberry Cave resin. The exceptions are the unsaturated compounds, which are prone to oxidation. The degradation products of the identified unsaturated molecules have not

been located in the Newberry Cave data. This is partly due to the complexity of many of the mass spectra produced. It is probable that these spectra would be improved by the use of a "soft" ionization technique, such as chemical ionization, which generates the mass spectra by means of ionic reactions rather than electron bombardment, resulting in much reduced fragmentation and more molecular weight information.

Sufficient numbers of constituents have now been identified to allow speculation as to the origin of these lipids, which may offer an explanation for the chemical heterogeneity of the resin. The fact that the plant food of the lac scale insect influences the chemistry of the resin produced (Imms 1960:170; Mills and White 1994:116) is particularly relevant. The lac cells build up on the plant over a period of years to produce the thick encasing of the branch. Annual fluctuation in growing conditions, (i.e., "wet" versus "dry" years) will influence the chemical content of the plant sap upon which the insects feed, and likewise will influence the resins they produce. Such fluctuation will naturally be very small, but the analysis of tiny samples from the raw source will enhance the apparent heterogeneity. In theory, such variability should be reduced within a single archaeological sample because a large quantity of resin will have been more thoroughly "mixed" during preparation processes. It seems sensible to recommend that where GC alone is being used as an identification technique that several samples of authentic resin should be employed for the purposes of comparison, to account for this variability. That a single sample of archaeological material is sufficient is encouraging, since often only minute quantities of such material are available for analysis.

An understanding of the relationship between the plant and insect resins would be very useful given that the other species of *Tachardiella* feed on different plants. The properties and

uses of the exudates of these other species are reported as being basically the same. Whether they also share the same chemistry is not known. No other insect resins from the region have been chemically examined, and hence the characterizations documented cannot reliably be used to distinguish between insect resins of different origin. The extent to which the fingerprint that has been produced is distinctive for *Tachardiella* remains to be demonstrated.

The relationship between plant food source and secreted resin is certainly reflected in the compounds that have so far been identified in the authentic lac. The unsaturated acids reflect the unsaturated nature of plant lipids, and the hydroxyacids may be derivatives of similar compounds. It has been observed in the investigation of European buried peat bog bodies that unsaturated fatty acids can be hydrated to hydroxyfatty acids by the action of microbes (Evershed 1990:145, 1992). While microbes will be present in the resin, associated with the breakdown of the female insect, the rapid hardening of the resin, combined with dry desert conditions, means that microbial alteration of the lac after secretion is unlikely. It is more likely that a similar process is being initiated by endogenous enzymes; this is consistent with the reported presence of hydroxyfatty acids as components of insect waxes (Jackson and Blomquist 1976:213).

The newly identified 7-hydroxyoctadecanoic acid is particularly relevant in the examination of such changes. It probably derives from 7-octadecenoic (petroselinic) acid, a common naturally occurring monoene acid (Christie 1989:14-16; Gunstone 1992:3). Petroselinic acid is closely related to 9-octadecenoic (oleic) acid, a common constituent of plant lipids, the two acids being distinguished by the location of the double bond in the carbon chain. It is likely that petroselinic acid is also a constituent of the plant lipids of *L. tridentata*, or that it represents an intermediate stage in the conversion of oleic acid to 7-hydroxyoctadecanoic acid by the insect. Further

work is necessary to explore these ideas.

The origins of the molecular species that have been identified are interesting when examined in light of the changes in the lipid profile after saponification of the samples. The alcohol series becomes very prominent, and this is accompanied by the virtual disappearance of the fatty acids and hydroxy acids in most cases. The alcohol series appears to derive from the wax fraction of the resin, a very similar series has been observed in the wax fraction of Asian lac (Faurot-Bouchet and Michel 1964:418; Tulloch 1973:369). It is possible that the alcohols derive from the breakup of wax ester constituents, but this seems unlikely given the reduced abundance of fatty acids seen in this fraction. Alternatively, the alcohols may be preferentially liberated from the polymerized material. Saponification did increase the degree of extraction but some insoluble material persisted. The disappearance of the other identified components with saponification is nevertheless confusing. A reasonable explanation would be that the alcohols are present in such abundance that the acid peaks are dwarfed. However, this is highly inconsistent with the composition of Asian lac. Exploration of this issue would be possible given higher quality GC/MS data. Regardless of their origin, the observation of these alcohols in the saponified portion is a significant finding because they are well preserved in the ancient material and so offer an excellent possibility for fingerprinting. Nevertheless, the consistency of their appearance in ancient material will need to be tested.

It is possible that the chemistry of the resin is influenced by the presence of insect material, as inclusions may be readily observed in the raw resin. It is known that the principal body fat of aphids is myristic acid, as well as other short chain acids, and that the body fat content of insects is related to the fatty nature of their plant food. Aphids, like scale insects, are phloem feeders, and so it is quite feasible that the myristic acid present in the lac derives from the

remains of the female insect trapped within the cell, rather than from lac secretions. This may also be true of the palmitic acid content of the resin, but at this stage such suggestions can only be tentative. No such conclusions can be drawn without a thorough understanding of the insect physiology. If some of the fingerprint molecules derive from insect remains in the resin, there may be implications for the identification of archaeological samples.

Ethnographic evidence concerning preparation of the resin is limited. If the ground resin was thoroughly washed before melting, then much of the insect material might be removed, altering the chemical profile of the material. None of the archaeological samples analyzed here or by Fox (1995) shows evidence of such alteration, either because this type of processing did not take place, or because the insect content of the raw resin does not influence the chemistry of the material. In either case, the impurity of the resin secretion is worth noting and purification during preparation may be worthy of further investigation.

The presence of *n*-alkanes and sugar molecules in the resin is interesting. It has not been possible at this stage to identify these components, but the presence of *n*-alkanes is consistent with the chemistry of insect waxes generally (Jackson and Blomquist 1976). The abundance and range of sugar components is not surprising given the reported use of the resin as a chewing gum. It is also consistent with reports of a carbohydrate "laccose" in Asian lac (Richards 1978:221).

ARCHAEOLOGICAL IMPLICATIONS AND POTENTIAL

It is clear that this study has raised a number of issues that require elucidation by further investigation. Nevertheless, progress has been made; most of the principal components of the soluble part of the resin have been assigned, so a good reference base now exists for future ex-

amination of archaeological samples. Heating experiments have shed light on the thermal properties of the material and have suggested how it may have been manipulated in antiquity. In addition, application of new techniques to natural products archaeology has been explored with great success, with saponification techniques and Py/GC offering reliable fingerprinting data. Full characterization of the soluble material would probably be possible if chemical ionization techniques were employed. Further examination of authentic and ancient samples in this way would almost certainly lead to the identification of further constituents and would be particularly useful for gaining insight into the ways in which the constituent molecules degrade with time.

Application of Py/MS may also be appropriate now that the potential of this technique has been demonstrated. Investigations of this nature would facilitate development of a full chemical characterization of the lac. The studies so far have suggested that a pyrolysis fingerprint more closely matches aged samples. This needs to be tested further, including comparison with more archaeological samples, from both lac and non-lac sources.

There is enormous potential for the use of these analytical techniques, combined with an experimental approach to understanding the technological processes involved in procuring and preparing natural organic substances. Experiments involving heating and mixing of this and other resins from the area would be very valuable in understanding the way in which such materials were exploited.

While the chemistry of other insect waxes suggests that the fingerprints produced are suitable for the identification of lac resin from *Tachardiella*, further investigation of insect products from the region would be useful. A number of other noninsect derived resins, such as brittlebrush and arrowweed, has also reportedly been used, though to a lesser extent, and these have not been analyzed at all (Euler and Jones

1956:90-91; Felger 1977:159).

A number of other issues has been raised by this study that, while somewhat out of the range of archaeological interest and application, are nonetheless worthy of further study for the purpose of enhancing the understanding of lac resin chemistry. These issues include investigation of how the resin chemistry is related to the phloem chemistry of the creosote bush, and the ways in which the chemical profile of the lac is influenced by dead insect material inclusions. Now that a reliable reference base is available, there is the potential for analysis of archaeological samples of suspected lac resin in order to establish the extent and nature of use of such material in antiquity, as implied in the ethnographic record.

ACKNOWLEDGEMENTS

The authors are indebted to the ESPRC Mass Spectrometry Service Center at the University of Swansea for the GC/MS analyses, and Scientific Glass Engineering (SGE), UK for undertaking the pyrolysis analysis. Thanks are also due to Carol Rector of the San Bernardino County Museum for providing the sample from Newberry Cave.

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