Absorbed Residue Evidence for Prehistoric *Datura* Use in the American Southeast and Western Mexico

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Scholars have been studying absorbed residues for many years looking for evidence of ancient food-related cultural practices (Barnard and Eerkens 2007; Evershed et al. 1990; Evershed et al. 1992). This is especially the case in the archaeological search for evidence of subsistence changes associated with the origins of agriculture, seasonal variation, or environmental shifts (for example, Copley et al. 2003;

ABSTRACT

Absorbed residue studies have been used in subsistence research for decades. Only more recently have the chemical methods employed been used to explore the consumption of ritual concoctions such as those including cacao, yaupon holly, and alcohol. In this article we use mass spectrometry to identify *Datura* residues in prehistoric contexts from western Mexico and the American Southeast. *Datura* is a genus of flowering plants that contain hallucinogenic alkaloids. Their use in both regions is known historically and still continues today. This study sampled 55 pottery vessels and 18 shell vessels using both a traditional burr method and a water-based sonicator sampling method. *Datura* residues were found in 13 pottery vessels and 14 shell vessels using both sampling approaches. These results demonstrate that it is possible to identify *Datura* residue in pottery and shell vessels and that the use of *Datura* extends back into prehistory in both regions. The form and decoration of pottery vessels with *Datura* residues show correlations with specific motifs and themes. Historically, shell vessels were used in the Southeast for the consumption of another ritual beverage, called the Black Drink. The presence of *Datura* shows that those vessels were used for other kinds of beverages as well.

Los estudios de residuos absorbidos han sido utilizado en la investigación de la subsistencia durante décadas. Sólo más recientemente se han utilizado los métodos químicos empleados en estos estudios para explorar el consumo de brebajes rituales tales como los que incluyen cacao, acebo de Yaupon, y alcohol. En este trabajo se utiliza la espectrometría de masas para identificar los residuos de *Datura* en contextos prehistóricos del oeste de México y el sureste de Estados Unidos. *Datura* es un género de plantas florecientes que contiene alcaloides alucinógenos. Su uso en ambas regiones es documentado históricamente y continúa en el presente. Para este estudio se analizaron muestras de 55 vasijas de cerámica y 18 vasijas de concha utilizando tanto el método tradicional de rebaba como el método de muestreo en baño de sonicación con agua. Residuos de *Datura* fueron encontrados en 13 vasijas de cerámica y 14 vasijas de concha utilizando los dos sistemas de muestreo. Estos resultados demuestran que es posible identificar los residuos de *Datura* en vasijas de cerámica y concha y que el uso de *Datura* se remonta a la prehistoria en ambas regiones. La forma y decoración de las vasijas de cerámica con restos de *Datura* muestran correlaciones con motivos y temas específicos. Históricamente, se utilizaron vasijas de concha en el Sureste para el consumo de otra bebida ritual, llamada la Bebida Negra. La presencia de *Datura* muestra que las vasijas se utilizaron también para otras bebidas.

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Dudd et al. 1999; Reber and Evershed 2004). More recently, absorbed residues have become a tool in identifying the use of nonfood substances made from plants such as cacao (*Theobroma cacao*; Hall et al. 1990; Hurst et al. 2002; Powis et al. 2002; Zarrillo et al. 2018) and yaupon holly (*Ilex vomitoria*; Crown et al. 2012; King et al. 2017), as well as fermented beverages such as wine (McGovern et al. 2013) and pulque (Correa-Ascencio et al. 2014). These studies give us a means for finding the remains of ritual beverages, exploring associated material culture, and understanding their contexts of use. This, in turn, makes possible the exploration of issues relating to medicine traditions, ritual practices, elite culture, and how these were adopted, changed, and moved across cultural boundaries.

In this essay we apply mass spectrometry to the identification of residues of *Datura* in late prehistoric shell and pottery vessels from western Mexico and the southeastern United States. *Datura* is a genus of flowering plants that contain hallucinogenic alkaloids that are still in use by native peoples in the region today. The use of *Datura* before the coming of Europeans has been confirmed by direct archaeological evidence and indirectly through iconography and early historical texts. However, identifying its use through absorbed residue studies has not been accomplished until now. Using liquid chromatography mass spectrometry, we have identified a key *Datura* alkaloid (atropine) in samples collected from 14 shell cups and 7 pottery vessels found in Mississippian and Caddoan Mississippian (AD 1000–1600) contexts in Arkansas and Oklahoma, as well as in an additional 6 pottery vessels recovered from precolumbian contexts in West Mexico. Our results go beyond simply demonstrating that *Datura* residues can be identified. They also reveal correlations between the presence of *Datura* and particular vessel forms and modes of decoration.

DATURA USE IN NORTH AND CENTRAL AMERICA

Humans have been using hallucinogens derived from plant and fungi sources for millennia (Ratsch 2005:37–41). In North America, one of the most potent hallucinogens was, and still is, produced from plants in the genus *Datura*. Members of the Solanaceae family, *Datura* are a series of flowering plants whose leaves, seeds, and flowers contain tropane alkaloids such as atropine, scopolamine, and hyoscamine (Lester et al. 1991). These alkaloids produce hallucinogenic effects but also are toxic when consumed in high enough doses.

Datura plants are found around the world, from North and South America to Asia, Europe, and Africa. However, its natural range is difficult to determine because it has become so widespread (Lester et al. 1991). *Datura* is cultivated commercially in Central America, North Africa, Ethiopia, India, and England for pharmaceutical purposes (Gerlach 1948) and is a common ornamental plant found throughout North America.

Its use in North America before the coming of Europeans has been confirmed by various kinds of archaeological evidence. *Datura* seeds have been found in Archaic period contexts in the Lower Pecos of Texas (Boyd and Dering 1996), in Ancestral Pueblo (AD 1250–1300) contexts in Colorado (Litzinger 1981), and in Mississippian (AD 1100–1200) contexts in Illinois (Emerson and Jackson 1984). Further, it has been argued that imagery and *Datura* fruit effigy pottery vessels confirm its presence from Mexico (Kan et al. 1989) into the Southwest and even into the Southeast (Lankford 2012, 2014). The sixteenth-century Aztec-language Florentine Codex includes *Datura* as an important medicine plant among the Aztecs (Ratsch 2005:41), and *Datura* continues to be part of Native American medicine ways and ritual practices to this day (Ratsch 2005:480–482).

Descriptions of modern-day practice indicate that *Datura* is used for a variety of purposes, depending on the dose and method of consumption (see Ratsch 2005:477–485). In Mexico and the American Southwest, *Datura* is included in topical medicines designed to treat physical injuries and ailments. Low doses, often delivered by smoking the leaves, are considered an aphrodisiac and also used in ritual divination. Tea produced from the leaves and seeds delivers the highest doses of alkaloids and creates the powerful hallucinatory experiences associated with shamanism and spiritual journeys (Ratsch 2005:477).

THE SAMPLE AND METHODS

In an attempt to find evidence of *Datura* use through absorbed residues, we sampled 18 shell and 55 pottery vessels held in the collections of the Gilcrease Museum at the Thomas Gilcrease Institute of American History and Art in Tulsa, Oklahoma. The tables below include information on each vessel included in this study. Because all were acquired by Thomas Gilcrease from dealers in the early twentieth century, we do not know the full history of each piece, and this creates limitations that are apparent in the tables as well as others that we will discuss below.

Thomas Gilcrease was an enrolled member of the Creek Nation who had a lifelong passion for western American art, especially as it related to the history of the west and his own Native American culture. As a tribal member, Gilcrease wanted his collections to exemplify the artistry, sophistication, and genius of Native Americans, past and present, and his collecting was guided by that goal. Thanks to wealth generated by his oil businesses, Gilcrease became a very successful collector of art, historical documents, and Native American artifacts. In 1955, after decades of collecting and an attempt to establish a museum in San Antonio, Texas, Gilcrease transferred his collection to the city of Tulsa, Oklahoma, and the museum that bears his name was established (Gilcrease Museum 2018). The Gilcrease archaeology collections are products of their time, when standards guiding the recovery and acquisition of artifacts were different. Although we do not have specific information for each vessel included in this study, we presume that many were recovered using methods that today would be considered destructive. Further, we suspect that many were

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TABLE 1. Mississippian and Caddoan Mississippian Ceramic Vessels Sampled.

Vessel Accession Number	Sample Number	Method	Culture	Period/Date	Type	Form	Location	*Datura*
5425-250	9	Sonicator	Caddoan	AD 1400–1500	Hardman Engraved	Turtle-frog effigy	Middle Ouachita River	X
5425-296	21	Sonicator	Mississippian	Late Mississippian/ Protohistoric	Mississippi Plain	Bottle	Eastern Arkansas	
5425-482	18	Sonicator	Caddoan	AD 1200–1300	Haley/Glassel Engraved	Bottle	Lafayette County, Arkansas	
5425-576	13	Sonicator	Mississippian	Protohistoric	Mississippi Plain	Bottle with medallions	Chickasawba (3MS5)	
	108	Burr						
5425-595	23	Sonicator	Mississippian	Protohistoric	Mississippi Plain	Pedestaled bottle	Chickasawba (3MS5)	
	110	Burr						
5425-669	116	Sonicator	Caddoan	AD 1400–1500	Hatchell Engraved	Bottle	Lafayette County, Arkansas	
5425-809	17	Sonicator	Mississippian	Protohistoric	Walls Engraved	Bottle	Friend (3MS6)	
	107	Burr						
5425-824	15	Sonicator	Mississippian	Late Mississippian/ Protohistoric	Walls Engraved	Bottle	Friend (3MS6)	X
5425-858	22	Sonicator	Mississippian	Protohistoric	Walls Engraved	Bottle	Friend (3MS6)	
	105	Burr						
5425-885	1	Sonicator	Caddoan	AD 1500–1700	Hodges/Natchitoches Engraved	Compound bowl	Lafayette County, Arkansas	X
5425-1105	24	Sonicator	Mississippian	Late Mississippian/ Protohistoric	Mississippi Plain	Cat monster effigy bowl	Castile Landing (3SF12)	
5425-1213	20	Sonicator	Mississippian	Late Mississippian/ Protohistoric	Mississippi Plain	Bottle	Bradley (3CT7)	
	104	Burr						
5425-1220	12	Sonicator	Mississippian	Late Mississippian/ Protohistoric	Mississippi Plain	Frog effigy bowl	Notgrass (3MS15)	X
5425-1270	11	Sonicator	Caddoan	AD 1400	East/Friendship Engraved	Compound bottle	Montgomery County, Arkansas	
5425-1294	10	Sonicator	Mississippian	Protohistoric	Untyped	Red and white head pot	Notgrass (3MS15)	
	111	Burr						

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TABLE 1. Continued

Vessel Accession Number	Sample Number	Method	Culture	Period/Date	Type	Form	Location	*Datura*
5425-1303	6	Sonicator	Mississippian	Protohistoric	Carson Red-on-buff	Head pot	Rose Mound (3CS27)	X
	109	Burr						
5425-1327	5	Sonicator	Mississippian	Protohistoric	Untyped	Female effigy bottle	Mississippi County, Arkansas	
5425-1334	2	Sonicator	Mississippian	Middle Mississippian	Untyped	Female effigy bottle	Eastern Arkansas	
5425-1358	112	Sonicator	Mississippian	Protohistoric	Rhodes Incised	Bottle	Rose Mound (3CS27)	
5425-1426	117	Sonicator	Mississippian	Late Mississippian/ Protohistoric	Bell Plain	Bottle	Eastern Arkansas	
5425-1510	4	Sonicator	Mississippian	Late Mississippian/ Protohistoric	Untyped	Female effigy bottle	Eastern Arkansas	
5425-1514	3	Sonicator	Mississippian	Middle Mississippian	Untyped	Female effigy bottle	Eastern Arkansas	X
5425-1582	114	Sonicator	Caddoan		Untyped	Bottle	Southwest Arkansas	
5425-1590	16	Sonicator	Caddoan	AD 1400	Hatchell Engraved	Bottle	Lafayette County, Arkansas	
5425-1938	7	Sonicator	Caddoan	AD 1600–1700	Hodges/Natchitoches Engraved	Composite bottle	Red River Great Bend	
5425-1949	27	Sonicator	Mississippian	AD 1500–1600	Walls Engraved	Bottle	Bradley County, Arkansas	X
5425-2030	115	Sonicator	Caddoan	AD 1500–1600	Hatchell Engraved	Bottle	Lafayette County, Arkansas	
5425-2541	113	Sonicator	Caddoan	AD 1400–1500	Untyped bottle	Bottle	Tillar Farms (3DR30)	
5425-3619	8	Sonicator	Mississippian		Old Town Red	Bottle	Perr County, Arkansas	
5425-4894	14	Sonicator	Mississippian	Late Mississippian/ Protohistoric	Mississippi Plain	Bottle with medallions	Banks (3CT13)	
5425-5161	19	Sonicator	Mississippian	Late Mississippian/ Protohistoric	Mississippi Plain	Bottle	Banks (3CT13)	
	106	Burr						

TABLE 2. Mississippian and Caddoan Mississippian Shell Vessels Sampled.

Shell Cup Accession Number	Sample Number	Method	Sample Location	Date (AD)	Style	Form	Location	*Datura*
9025-544	63	Burr	Cut columella	1200–1450	Craig B	Decorated top	Spiro (34LF40)	X
9025-546	65	Burr	Cut columella	1350–1450	Craig C	Decorated top	Spiro (34LF40)	X
9025-553	68	Burr	Cut columella	1200–1450	Craig B	Decorated top	Spiro (34LF40)	
9025-554	78	Burr	Interior back	1200–1450	Craig B	Decorated fragment	Spiro (34LF40)	X
9025-587	75	Burr	Lip left center	1100–1200	Braden A	Decorated fragment	Spiro (34LF40)	X
9025-598	73	Burr	Cut columella			Whole undecorated	Spiro (34LF40)	X
9025-598	79	Burr	Lip left center			Whole undecorated	Spiro (34LF40)	X
9025-599E	67	Burr	Cut columella			Whole undecorated	Spiro (34LF40)	
	84	Burr	Lip left center			Whole undecorated	Spiro (34LF40)	
9025-599H	25	Sonicator				Whole undecorated	Spiro (34LF40)	X
	60	Burr	Cut columella			Whole undecorated	Spiro (34LF40)	X
9025-599H	74	Burr	Lip left center			Whole undecorated	Spiro (34LF40)	
9025-599I	71	Burr	Cut columella			Whole undecorated	Spiro (34LF40)	
	83	Burr	Lip left center			Whole undecorated	Spiro (34LF40)	X
9025-1472	64	Burr	Cut columella	1350–1450	Craig C	Decorated top	Spiro (34LF40)	X
9025-1473	66	Burr	Cut columella	1200–1300	Braden B	Whole decorated	Spiro (34LF40)	X
	80	Burr	Lip left center	1200–1300	Braden B	Whole decorated	Spiro (34LF40)	X
9025-1554	62	Burr	Cut columella	1200–1400	Craig A	Decorated top	Spiro (34LF40)	X
9025-1665	69	Burr	Cut columella	1350–1450	Craig C	Decorated top	Spiro (34LF40)	X
9025-1670	61	Burr	Cut columella	1350–1450	Craig C	Decorated top	Spiro (34LF40)	X
9025-1718	77	Burr	Interior	1200–1450	Craig B	Decorated fragment	Spiro (34LF40)	
9025-1720	70	Burr	Cut columella			Whole undecorated		X
	81	Burr	Lip left center			Whole decorated	Spiro (34LF40)	X
9025-1727a	76	Burr	Lip left center			Undecorated outer whorl	Spiro (34LF40)	
9025-1749	72	Burr	Cut columella			Whole undecorated		X
	82	Burr	Lip left center			Whole undecorated	Spiro (34LF40)	X

acquired by Gilcrease in ways that do not meet today's ethical standards (Society for American Archaeology 2018).

Thirty-one of the ceramic vessels sampled (Table 1) derive from various Mississippian and Caddoan Mississippian sites in Arkansas. Contextual information associated with each vessel is quite limited. However, thanks to the assistance of David Dye of Memphis University and Ann Early and George Sabo of the Arkansas Archaeological Survey, we were able to assign each vessel to a type, time period, and, in some cases, a known archaeological site. All vessels date to the period between AD 1400 and 1700 and were chosen because of their unique forms (most often bottles and effigies) and recognizable imagery (engraved or incised in two dimensions or as three-dimensional forms). Our operating assumption was that bottles and symbolically loaded vessels are likely to have contained special liquids in the past. In most cases, we know little more about their context than the state and county where they were found.

The shell cups and fragments sampled (Table 2) all reportedly come from the famous Mississippian period Spiro site in LeFlore County, Oklahoma (see Brown 1996). Spiro is well known for its Craig Mound, built between AD 1250 and 1450, and was first brought to the attention of professional archaeology in the 1930s by treasure hunters who leased the mound from the Pocola Mining Company (La Vere 2007). Subsequent systematic excavations and decades of work to reconstruct the nature of the features it contained have revealed a unique set of mortuary practices that included incredible quantities of engraved and plain shell cups along with an unequaled cache of various sacred objects made of pottery, shell, stone, and copper (Brown 2010). We presume that the shell samples at the Gilcrease are part of that Craig Mound assemblage, but we do not know for certain. Alex Barker of the University of Missouri's Museum of Art and Archaeology assisted with stylistic associations for each of the decorated cups included in Table 2.

It has long been assumed that shell cups, made from the outer whorl of whelk shells (genus *Busycon*), were used as dippers for serving and consuming ritual beverages. This assumption is based on the many historic-period descriptions of Native Americans using shell cups in ritual settings, particularly for the consumption of a Native American tea made from yaupon holly called the Black Drink (see Hudson 2004).

Twenty-four of the ceramic vessels we sampled were collected from various sites in western Mexico dating between the Early Formative (1400–1000 BC) and Early Postclassic (AD 900–1250) periods. While very little contextual information is associated with the vessels (Table 3), Chris Beekman of the University of Colorado

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TABLE 3. Western Mexico Ceramic Vessels Sampled.

Accession Number	Sample Number	Method	Period/Phase/Date	Form	Location	*Datura*
54.20362	44	Sonicator	Comala phase, AD 1–500	Dog head effigy	Colima	
54.20459	28	Sonicator	Terminal Early Formative, 1400–1000 BC	Bottle		X
54.20461	30	Sonicator	Early or Middle Formative, 2000–900 BC or 900–300 BC	Tall-neck jar	Comala	
5444-1713	101	Burr	Comala phase, AD 1–500	Squash effigy	Comala	
5444-2092	99	Burr	Comala phase, AD 1–500	Vegetable effigy	Comala	
5444-2094	100	Burr	Comala phase, AD 1–500	Vegetable effigy	Colima	
5444-7346	42	Sonicator	Middle Formative, 900–300 BC	Two-tiered gourd effigy jar	Coastal Jalisco	
5444-7366	33	Sonicator	Late Formative, 300 BC–AD 200	Bowl		X
5444-7411	38	Sonicator	Late Formative–Early Classic, 300 BC–AD 500/600	Trophy head effigy	Colima	
5444-7551	35	Sonicator	Late Formative–Early Classic, 300 BC–AD 500/600	Zoomorphic	Jalisco	
5444-8180	102	Burr	Comala phase, AD 1–500	Squash effigy	Colima	
5445-1706	103	Burr	Postclassic, AD 900–1500	Spouted vessel	Aztatlan	
5445-3551	39	Sonicator	Comala phase, AD 1–500	Squash effigy globular jar	Colima	
5445-3571	32	Sonicator	Comala phase, AD 1–500	Squash effigy globular jar	Coastal Colima	
5445-3585	34	Sonicator	Postclassic, AD 900–1500	*Molcajete*		X
5445-3597	29	Sonicator	Late Formative–Early Classic, 300 BC–AD 500/600	Shallow bowl	Nayarit	X
5445-3800	31	Sonicator	Postclassic, AD 900–1500	*Molcajete*		X
5445-3947	43	Sonicator	Late Formative, 300 BC–AD 200	Anthropomorphic effigy jar		
5445-3986	41	Sonicator	Late Formative–Early Classic, 300 BC–AD 500/600	Small tripod human effigy	Possible Jalisco	
5445-4002	37	Sonicator	El Grillo phase, AD 500–900	Anthropomorphic face effigy	Central Jalisco	
5445-4004	40	Sonicator	Comala phase, AD 1–500	Small bottle	Colima	
5445-4039	36	Sonicator	Late Formative–Early Classic, 300 BC–AD 500/600	Foot effigy		
5445-4256	45	Sonicator	Comala phase, AD 1–500	Frog effigy	Colima	
5445-7384	26	Sonicator	El Grillo phase, AD 500–900	Shallow bowl		X

assisted in assigning each vessel to a type, cultural affiliation, and time period. The vessels were chosen for sampling because of their unusual shapes—often specialized functional types or effigy vessels—and because we assume that these unique vessels may have held special liquids.

Our samples were collected using one of two methods (Tables 1–3). The first, known as the burr method (Powis et al. 2002), removes a portion of the interior surface and vessel through abrasion. For this sampling method, a clean piece of sandpaper was used to collect each sample, and gloves were worn and changed between samples. Each sample was collected on a fresh piece of multipurpose paper and transferred to a clean vial for transport. Although sample sizes varied, our target size was at least 1 ml.

The second method employed a Branson 2510 Ultasonic Benchtop Cleaner. Traditionally used to clean objects, a sonicator has a water tank that is vibrated using sound waves. In our sampling, we placed 100 ml of distilled water in each vessel and then placed the vessels in a shallow plastic container that floated on the surface of the water in the tank. The water and the vessel floating on it were then vibrated for 30 minutes, after which time the samples were transferred to clean vials for transport.

Each sample collected was analyzed using mass spectrometry, looking for peaks indicative of atropine and scopolamine. These are two of the primary alkaloids found in *Datura* giving the plant its hallucinogenic properties. In North and Central America, no other plants are known to contain these particular alkaloids, making it possible to use them as exclusive biomarkers for the presence of *Datura* (Duke 2015).

All of the objects from which our samples were taken are stored at the Gilcrease on open shelves. As a result, there is the

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TABLE 4. Contamination Samples.

Location	Sample Number	Sample Type
Curation shelving: D3F right	46	Water
Curation shelving: D3F right	85	Cotton ball
Curation shelving: D3G center	47	Water
Curation shelving: D3G center	86	Cotton ball
Curation shelving: D3 top center	48	Water
Curation shelving: D3 top center	87	Cotton ball
Curation shelving: C3D right	49	Water
Curation shelving: C3D right	88	Cotton ball
Curation shelving: H9G left	50	Water
Curation shelving: H9G left	89	Cotton ball
Curation shelving: H5G right	51	Water
Curation shelving: H5G right	90	Cotton ball
Curation shelving: I1 top left	52	Water
Curation shelving: I1 top left	91	Cotton ball
Worktable foam	53	Water
Worktable foam	92	Cotton ball
Curator's desk	54	Water
Curator's desk	93	Cotton ball
Conservation desk	55	Water
Conservation desk	94	Cotton ball
Conservation room worktable	56	Water
Conservation room worktable	95	Cotton ball
Conservation room coffee maker area	57	Water
Conservation room coffee maker area	96	Cotton ball
Flat drawers near freezer and cabinets	58	Water
Flat drawers near freezer and cabinets	97	Cotton ball
AV room	59	Water
AV room	98	Cotton ball

possibility that they have been, and continue to be, exposed to modern sources of contamination through air circulation systems and human activities. For example, artifacts stored in this way are exposed to caffeine-laden dust, which confounds the search for cacao residue (Washburn et al. 2012). As a way of assessing the potential for modern contamination in the Gilcrease samples, we collected and analyzed dust from shelving units and work areas at the museum (Table 4). Contamination samples were collected by swabbing areas with cotton balls that had been cleaned with alcohol and dampened with distilled water and by wetting areas with distilled water, which was then collected using clean pipettes.

LABORATORY METHODS

Atropine, scopolamine, and formic acid were purchased from Sigma-Aldrich Chemical Co. (St. Louis, Missouri). All solvents were mass spectrometry grade, and all other chemicals used were of the highest grade available. An ACQUITY UPLC C18 1.7 μm (1 × 150 mm) column was purchased from the Waters Corporation, Milford, Massachusetts.

A total of 117 samples were extracted using the following procedure. For the burr samples, 90 to 200 mg of burr from each sample were added to a 0.25 ml water:methanol mixture (1:1). Samples were vortexed and incubated at 80°C for 30 minutes. After incubation, samples were sonicated (one minute), vortexed (one minute), and centrifuged (three minutes). The resulting precipitate from each sample was removed, and the supernatant was filtered with 5 kD membrane filters. Water-based samples obtained by sonicator were freeze-dried completely. A 0.25 ml water:methanol mixture (1:1) was added, and the samples were sonicated (one minute), vortexed (one minute), and centrifuged (three minutes). The supernatant was filtered with 5 kD membrane filters. The filtrates from both extraction methods were transferred to vials for ultra performance liquid chromatography (UPLC)/tandem mass spectrometry (MS-MS) analysis.

Linearity, accuracy, precision, limit of detection, and limit of quantitation parameters were determined for atropine and scopolamine. To calculate limits of detection, various concentrations—0.05, 0.10, 0.25, 0.50, 1.0, 2.5, 5.0, 10, 25, 50, 100, 250, 500, 1,000, and 5,000 pg/ml—of the analytes were injected to UPLC/MS-MS. The injected amount that resulted in a peak with a height at least three times as high as the baseline noise level was used as the limit of detection. Intraday precision and accuracy were determined by calculating the percent coefficient of variation and relative error of the measurement of five replicates of each of the validation standard concentrations, analyzed on the same day.

A Waters Xevo TQ triple quadrupole mass spectrometer was used to record MS and MS-MS spectra using electrospray ionization in positive ion mode, with capillary voltage of 3.0 kV, an extractor cone voltage of 3 V, and a detector voltage of 500 V. Cone gas flow was set at 50 L/hour, and desolvation gas flow was maintained at 600 L/hour. Source temperature and desolvation temperature were set to 150 and 350°C, respectively. The collision energy was varied from 6 to 13 to optimize four different daughter ions. The acquisition range was 20–350 D. Pure standards (Figure 1; atropine and scopolamine) were introduced to the source at a flow rate of 10 μl/minute by using a methanol:water (1:1) and 0.1% formic acid mixture as the carrier solution to develop a multiple reaction monitoring method for UPLC/MS-MS operation.

UPLC/MS-MS analyses of all the samples were carried out with a Waters ACQUITY UPLC System connected with a Xevo TQ triple quadrupole mass spectrometer. Analytical separations on the UPLC system were conducted using an ACQUITY UPLC C18 1.7 μm column (1 × 150 mm) at a flow rate of 0.15 ml/minute. The gradient started with 100% A (0.1% formic acid in H_2O) and 0% B (0.1% formic acid in CH_3CN) changed to 50% A over three minutes, followed by a four-minute linear gradient to 10% A, resulting in a total separation time of seven minutes. The elutions from the UPLC column were introduced to the mass spectrometer, and the resulting data were analyzed and processed using MassLynx 4.2 software (Figure 1). A pure standard mixture was used to optimize the UPLC conditions prior to analysis. Blanks

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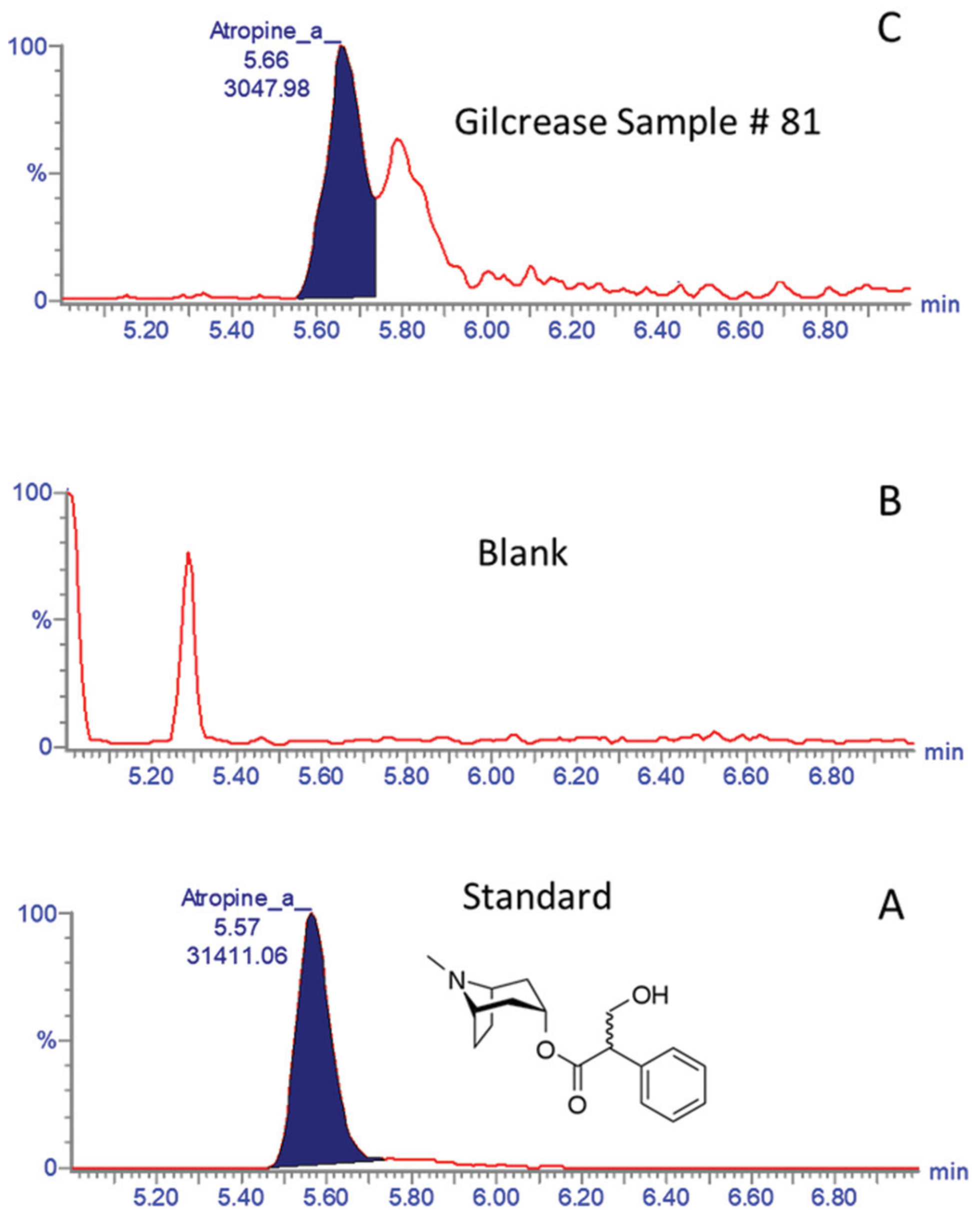

FIGURE 1. Ultra performance liquid chromatography/tandem mass spectrometry chromatograms: (A) standard atropine; (B) blank; (C) representative Gilcrease sample confirming the presence of atropine.

were run in duplicate at the beginning, middle, and end of each run after the standards.

The developed method was validated by determining limit of detection, limit of quantification, linear dynamic range, precision, and accuracy (Table 5). The analytical parameter determinations of the atropine and scopolamine were done using standard mixtures. The results indicated that the limit of detection was 0.5 and 1 pg and the limit of quantitation was 1.5 and 3 pg for atropine and scopolamine, respectively, suggesting that the developed method is highly sensitive. The linearity of the method was determined from the calibration curves constructed for atropine and scopolamine. The data for accuracy, precision, and recovery are presented in Table 5. These results provide support that this method is dependable and reproducible for the analysis of atropine and scopolamine.

RESULTS

As Table 1 shows, seven of the 31 Mississippian ceramic vessels returned results positive for atropine, while none contained scopolamine. The presence of atropine was further confirmed by co-chromatography (Supplemental Figure). All of these positives were identified in samples collected using the sonicator. This might suggest that the burr method somehow systematically selected against recovering atropine. However, the eight vessels sampled using both the sonicator and burr method produced

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TABLE 5. Validation of Detection Method.

Variable	Atropine		Scopolamine	
Limit of detection	0.5 pg		1 pg	
Limit of quantitation	1.5 pg		3 pg	
Linearity	0–5,000 pg		0–5,000 pg	
Standard concentration (pg)	% CV Precision	% RE Accuracy	% CV Precision	% RE Accuracy
0.05	23.1	3.0851		
0.10	41.1	5.2151	51.4	11.9111
0.25	12.9	0.6866	43.7	10.1706
0.50	15.6	0.5274	34.2	4.1028
1.0	8.0	0.1772	21.2	1.3881
2.5	12.1	0.1349	12.1	0.4348
5.0	5.2	0.0499	6.0	0.1797
10	5.8	0.0386	8.9	0.2092
25	2.5	0.0113	6.4	0.0988
50	1.5	0.0051	6.9	0.077
100	3.1	0.0069	7.1	0.0554
250	1.8	0.0025	3.1	0.0143
500	5.9	0.0052	3.8	0.0121
1,000	3.0	0.0013	4.0	0.0067
2,500	1.6	0.0004	1.3	0.0008
5,000	0.7	0.0001	1.2	0.0005

Note: % CV = percent coefficient of variation; % RE = percent relative error.

consistent results, in this case all negative. These findings, along with the results from the shell samples, suggest that both methods are equally effective in recovering atropine residue. Among the positive Mississippian vessels are bottles and bowls, including special effigy forms, potentially representing both the storage and consumption of *Datura* beverages.

Fourteen of the shell cups and fragments we sampled contained traces of atropine (Table 2), while none contained scopolamine, a pattern also seen with the ceramic vessels. Only one shell cup was sampled using the sonicator (Cat. #54250-1949). Because the vessel had to float in the sonicator for this method to work, curators at the Gilcrease agreed to allow this sampling method with only one shell. The sonicator sample found traces of atropine in the cup, but the two subsequent burr samples from the same cup did not. Seven other cups or fragments were sampled in two locations using the burr method. Those locations included the area along the outer whorl of the cup and the cut surface at the top of the shell where the inner column had been removed. Our results show that both areas have the potential to contain atropine traces.

Of the 24 vessels deriving from western Mexico, 6 had traces of atropine, but scopolamine was absent in all cases (Table 3). These vessels range from the Early Formative (1400–1000 BC) to the Early Postclassic (AD 900–1250), showing that *Datura* use persisted over a long period in the region. Three of the vessels are simple bowls that we can assume were used to consume *Datura* beverages. Two of the vessels are tripodal *mocajetes* with a grinding surface built into their form, suggesting that they were used in the preparation of *Datura* beverages. The last sampled vessel is a small bottle that may have been used to hold prepared *Datura* beverages.

As noted above, we also collected and analyzed samples of dust recovered from 14 different locations in the curation and work areas of the Gilcrease Museum (Table 4). In all cases, no traces of atropine or scopolamine were found in the dust samples. The absence of these alkaloid residues on the shelves suggests that the atropine we found in the shell and pottery vessels was likely not introduced through contamination within the museum. While it is still possible that the atropine residues detected are the result of other kinds of contamination, we see that as unlikely. Atropine is not found in high levels in groundwater like other alkaloids such as caffeine (Fram and Belitz 2011), so postdepositional contamination through water percolation or postrecovery contamination through washing seems unlikely. Given that we do not know the full history of the Gilcrease Museum objects sampled, it is possible that they were contaminated with modern sources of atropine through intentional or unintentional human actions. This is unlikely because, outside of recreational use, the most common uses of atropine are in the medical field, where it is most often administered intravenously or through injection (American Society of Health-System Pharmacists 2016).

DISCUSSION

All of the Mississippian vessels that tested positive for atropine either are decorated with iconographic motifs or are effigy vessels in forms related to the Mississippian cosmological concept of the Beneath World—one of three realms of the Mississippian cosmos (Lankford 2007a, 2011; Reilly 2004). The others are the sky realm, or Above World, and the surface realm, This World. Each of these three realms was associated with its own supernaturals, unique powers, and ritual themes. The Beneath World

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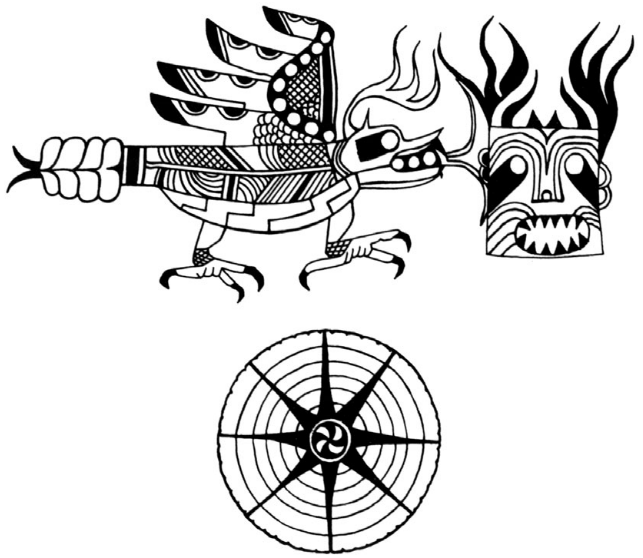

FIGURE 2. Drawing of imagery on Walls Engraved bottle, GM 5425–824, Gilcrease Museum, Tulsa, Oklahoma (Perino et al. 1960:149; reproduced with permission of *Central States Archaeological Journal*).

was a place of chaos and the realm of the dead, as well as the source of water and the growth of the natural world. Each day when the setting sun descended into the Beneath World, the day sky and the Beneath World switched places. Beings such as the Lord of Death and an earth mother figure associated with the moon inhabited this underwater and night sky realm. Given these associations, Beneath World powers would have been involved in rituals associated with death, but they were also important as sources of rain and the productivity of the earth and human populations.

One vessel that tested positive for *Datura* is a medium-sized bottle incised with images representing the Underwater Panther and the Great Serpent (Figure 2; Perino et al. 1960). According to Lankford's (2007b) research with Native American sacred narratives, both of these images represent avatars of the Lord of Death in the Beneath World. Keeping with this thematic association, two additional *Datura*-positive vessels are effigies of frogs or a turtle, both of which are associated with the watery Beneath World, and another is engraved with a swirl design also arguably associated with that same realm (Lankford 2011).

Another of the *Datura*-positive Mississippian vessels is an effigy of a female figure, understood to be a cognate of historic-period earth mother figures such as Old-Woman-Who-Never-Dies (Figure 3). Many groups in the Eastern Woodlands of North America consider this woman to be the moon in the night sky, so she is often associated with the Beneath World realm and its powers (Duncan and Diaz-Granados 2004). Dye (2015) has argued recently that female effigy vessels found in the Lower Mississippi Valley may have helped create a connection between women and female guardian spirits. These bottles were effigies of those spirits as well as living manifestations of them. In her study of guardian spirits, Ruth Benedict (1923) noted their widespread occurrence in North America and the use of *Datura* as a source of dreams through which women found and communicated with such spirits. Our discovery of atropine residue in a female effigy bottle from Arkansas lends support to this argument.

FIGURE 3. Female effigy bottle, GM 5425-1514, Gilcrease Museum, Tulsa, Oklahoma.

The remaining *Datura*-positive vessel is a human head effigy (Figure 4). Similar heads appear as early as the twelfth century in Mississippian art, and they have been interpreted as belonging to an Above World creator figure who was decapitated battling monsters in the Beneath World (Brown 2007; Walker 2004). This decapitated creator figure was brought back to life by his sons after they brought his head back from the Beneath World. While there are multiple potential associations of this human head effigy, it may also be related to powers and rituals of the Beneath World. If that is the case, then it may have been part of rituals focused on the return of spirits after death.

Ten of the shell cups and fragments with atropine residue were decorated, and their stylistic assignments are listed in Table 1. Only one of these is a whole vessel, and it was decorated in the Braden B or Late Braden style (Phillips and Brown 1978). It exhibits the amphisbaena theme, which is a creature with snake-like characteristics associated with the Beneath World (Figure 5).

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FIGURE 4. Carson Red-on-buff human head effigy bottle, GM 5425-1303, Gilcrease Museum, Tulsa, Oklahoma.

FIGURE 5. Braden B shell cup, GM 9025-1473, Gilcrease Museum, Tulsa, Oklahoma.

The other eight atropine-positive samples are vessel fragments and all exhibit imagery executed in the Craig A, B, or C style (Phillips and Brown 1984). The theme of the imagery on each is difficult to understand given their fragmentary nature. It may be important to note that, regardless of attached imagery, shell is associated with water and therefore the Beneath World.

There is also some interesting patterning in the decoration and form of the *Datura*-positive vessels from West Mexico. Two of these bowls are painted with designs that appear to mimic *Datura* seedpods. One of the vessels is a shallow bowl (Cat. #5444-7366) dating to the Late Formative period (300 BC–AD 200) with imagery resembling a maturing *Datura* seedpod (Figure 6). Another vessel is a shallow bowl (Cat. #5445-3597) dating to the Late Formative/Early Classic period (300 BC–AD 500/600), and it is decorated with motifs that resemble an opening *Datura* seedpod (Figure 7). Ceramics in the region are well known for taking recognizable effigy forms of plants, fruits, and even animals. It is often assumed that the vessel form is related to its use (Schondube 1998), and this pattern appears to be the case for these two bowls with *Datura* residue in them.

It is worth noting that two of the *Datura*-positive vessels take the *mocajete* form (Figure 8) and date to the Early Postclassic

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FIGURE 6. Late Formative bowl, GM 5444–7366, Gilcrease Museum, Tulsa, Oklahoma.

FIGURE 7. Late Formative–Early Classic bowl, GM 5445-3597, Gilcrease Museum, Tulsa, Oklahoma.

period (AD 900–1250). While this may be an artifact of our limited ceramic sample, it appears that earlier uses of *Datura* in West Mexico were associated with bowls decorated with *Datura* motifs. Later *Datura* preparation and consumption are associated with another kind of specialized vessel, this time one with a prepared grinding surface built in.

SIGNIFICANCE

Until now, the only direct means of identifying *Datura* use in prehistoric contexts was through the recovery of preserved portions of *Datura* plants. Charred seeds have been found in context (Emerson and Jackson 1984:333), and there is the possibility that *Datura* phytoliths may be recoverable as well. The use of *Datura* has also been inferred more indirectly through the interpretation of imagery as well as the presence of what some have argued are *Datura* fruit effigy vessels. With our demonstration that *Datura* is preserved as absorbed residues in both pottery and shell vessels, we have introduced another means of directly identifying the plants' use in prehistoric contexts. Absorbed residues have the benefit of being preserved more commonly than actual plant remains and being recoverable from a ubiquitous type of material culture (pottery), whose form and style allow it to be dated

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(a)

(b)

FIGURE 8. *Molcajete* vessels, Gilcrease Museum, Tulsa, Oklahoma: (a) GM 5445-3800 (b) GM 5445-3585.

and sourced fairly readily. Also, often vessels are directly associated with imagery that further allows for some understanding of the connection between *Datura* use and ritual themes.

In this study, we have demonstrated the efficacy of the sonicator sampling method for recovering absorbed residues from pottery and shell, especially residues containing atropine. While other water-based sampling methods have been used to recover caffeinated beverage residues from pottery (Washburn et al. 2012), the sonicator method has not been used to sample pottery for absorbed residues until now. However, Zarrillo and colleagues (2018) did use a sonicator to collect starch grains from ceramic vessels and also to sample cacao absorbed residues from stone bowls. The sonicator method is less destructive than burr methods, which actually remove a portion of the vessel. It also has the benefit of using vibration to release absorbed residues from vessel matrices, while other wash sampling methods rely only on solubility to collect samples.

Admittedly, the provenience information available for the objects we sampled does not meet modern standards, and that places limits on what we are able to infer from our results. Despite this limitation, we can make some useful inferences from the data generated. We can, with reasonable confidence, conclude that *Datura* was used in Mississippian and Caddoan contexts in Arkansas and from the Middle Mississippian (AD 1350–1450) to the Protohistoric (AD 1500–1700) period in Oklahoma in both ceramic and shell vessels. We already had an idea that it might have been used in this area because of mentions in ethnographic reports and archaeological data, but now we have confirmed it. We can also conclude that *Datura* consumption involved ceramic bottles and bowls, as well as shell vessels. Based on historical information, it has long been assumed that shell cups were used in ceremonies that included the Black Drink—a tea made from yaupon holly leaves. Now we must be open to the idea that these vessels were used in the consumption of more than one kind of ritual beverage.

Because we chose Mississippian and Caddoan Mississippian vessels that carried imagery (through their form or decoration), we made it possible to explore the correlation between what we understand about the thematic associations of particular imagery and *Datura* consumption. In these areas there is a clear correlation between the presence of *Datura* and Beneath World beings and locatives on Mississippian and Caddo pottery vessels. That association holds for the shell cups decorated in the Braden stylistic tradition. The Craig imagery is different in that it depicts ritual events that likely involved both people and supernaturals. These are important observations because they give us clues to the rituals that included *Datura* use in these cultural settings.

We can also be reasonably confident that *Datura* was prepared and consumed in West Mexico during the Late Formative to the Early Postclassic period. The presence of atropine in the *mocajete* vessel form suggests that grinding was part of the preparation of *Datura*. Because modern *mocajetes* are used to grind spices, including chili, it is often assumed that prehistoric versions were used for the same purpose. While this has yet to be demonstrated, it is apparent that the vessel form was used to grind other plants included in specialized beverages, particularly *Datura*. Those beverages were served in, and probably consumed from, simple bowls decorated to resemble a *Datura* seedpod.

Probably the most important contribution this study makes is in the potential it opens for future research. We have shown that atropine preserves in ancient shell and pottery vessels, it can be recovered using both sonicator and burr methods, and it can be measured using liquid chromatography mass spectrometry. The inferences we can draw from our current sample are limited because of a lack of contextual information. However, future targeted studies using objects with more detailed contextual information can begin to explore a whole host of interesting questions.

Several researchers (Lankford 2012; Litzinger 1981), along with this study, have suggested that there may be particular vessel types and decorative forms associated with *Datura* use. These ideas can now be evaluated. We have introduced some inferences about the thematic associations of *Datura* use in the Mississippian period. Future studies that correlate *Datura* presence and iconographic interpretations will surely give us a fuller understanding of how and why *Datura* beverages were prepared and consumed in different cultural contexts. We know a good deal about recent uses of *Datura* (Ratsch 2005), but we know much less about its older contexts of use and where it fit into ritual practices and ancient social systems. With targeted sampling of more vessels from various places and contexts, it will be possible to generate a clearer understanding of the use of *Datura* in time and space.

Finally, special beverages like those made from *Datura* are associated with ritual practices. Like so many other aspects of human behavior, these practices often have a place of origin and history of spread, alteration, and adaptation to new settings. The study of ritual beverages allows for the exploration of ritual complexes, their histories, and how they are part of culture-making on local and regional scales.

Acknowledgments

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Data Availability Statement

The mass spectrometry data generated using samples collected from pottery vessels, shell vessels, and surfaces at the Gilcrease Museum are archived at the Gaikwad Steroidomics Laboratory (http://gaikwadsteroidomics.com/) and are available upon request. The unanalyzed portions of the physical samples collected from archaeological specimens and surfaces at the Gilcrease are also stored at the Gaikwad Steroidomics Laboratory.

Supplementary material

To view supplementary material for this article, please visit https://doi.org/10.1017/aap.2018.30
Supplemental Figure. UPLC-MS/MS chromatograms of sample extract (bottom) and sample extract spiked with standard atropine (top).

REFERENCES CITED

American Society of Health-System Pharmacists
2016 Atropine. Electronic document, http://www.drugs.com/monograph/atropine.html, accessed March 1, 2016.

Barnard, Hans, and Jelmer W. Eerkens (editors)
2007 *Theory and Practice of Archaeological Residue Analysis*. BAR International Series 1650. Archaeopress, Oxford.

Benedict, Ruth F.
1923 *The Concept of the Guardian Spirit in North America*. Memoir No. 29. American Anthropological Association, Washington, DC.

Boyd, Carolyn E., and J. Philip Dering
1996 Medicinal and Hallucinogenic Plants Identified in the Sediments and Pictographs of the Lower Pecos, Texas Archaic. *Antiquity* 70(268):256–275.
Brown, James A.
1996 *The Spiro Ceremonial Center: The Archaeology of Arkansas Valley Caddoan Culture in Eastern Oklahoma*, 2 vols. Memoir No. 29. Museum of Anthropology, University of Michigan, Ann Arbor.
2007 On the Identity of the Birdman within Mississippian Period Art and Iconography. In *Ancient Objects and Sacred Realms: Interpretations of Mississippian Iconography*, edited by F. Kent Reilly and James F. Garber, pp. 56–106. University of Texas Press, Austin.
2010 Cosmological Layouts of Secondary Burials as Political Instruments. In *Mississippian Mortuary Practices: Beyond Hierarchy and the Representationist Perspective*, edited by Lynne P. Sullivan and Robert C. Mainfort Jr., pp. 30–52. University of Florida Press, Gainesville.
Copley, M. S., R. Berstan, Stephanie N. Dudd, G. Docherty, A. J. Mukherjee, V. Straker, S. Payne, and Robert P. Evershed
2003 Direct Chemical Evidence for Widespread Dairying in Prehistoric Britain. *Proceedings of the National Academy of Sciences* 100:1524–1529.
Correa-Ascencio, Marisol, Ian G. Robertson, Oralia Cabrera-Cortésc, Rube Cabrera-Castrod, and Robert P. Evershed
2014 Pulque Production from Fermented Agave Sap as a Dietary Supplement in Prehispanic Mesoamerica. *Proceedings of the National Academy of Sciences* 111(39):14223–14228.
Crown, Patricia L., Thomas E. Emerson, Jiyan Gru, W. Jeffrey Hurst, Timothy R. Pauketat, and Timothy Ward
2012 Ritual Black Drink Consumption at Cahokia. *Proceedings of the National Academy of Sciences* 109(35):13944–13949.
Dudd, Stephanie N., Robert P. Evershed, and Alex M. Gibson
1999 Evidence for Varying Patterns of Exploitation of Animal Products in Different Prehistoric Pottery Traditions Based on Lipids Preserved in Surface and Absorbed Residues. *Journal of Archaeological Science* 26:1473–1482.
Duke, James A.
2015 Dr. Dukes Phytochemical and Ethnobotanical Databases. Electronic document, http://www.ars-grin.gov/duke/, accessed March 1, 2016.
Duncan, James R., and Carol Diaz-Granados
2004 Empowering the SECC: The "Old Woman" and Oral Tradition. In *The Rock Art of Eastern North America: Capturing Images and Insight*, edited by Carol Diaz-Granados and James R. Duncan, pp. 190–215. University of Alabama Press, Tuscaloosa.
Dye, David H.
2015 Earth Mother Cult Ceramic Statuary in the Lower Mississippi Valley. Paper presented at the 72nd Annual Southeastern Archaeological Conference, Nashville, Tennessee.
Emerson, Thomas E., and Douglas K. Jackson
1984 *The BBB Motor Site*. University of Illinois Press, Urbana.
Evershed, Robert P., Carl Heron, Stephanie Charters, L. John Goad
1992 The Survival of Food Residues: New Methods of Analysis, Interpretation, and Application. *Proceedings of the British Academy* 77:187–208.
Evershed, Robert P., Carl Heron, and L. John Goad
1990 Analysis of Organic Residues of Archaeological Origin by High Temperature Gas Chromatography and Gas Chromatography–Mass Spectrometry. *Analyst* 115:1339–1342.
Fram, Miranda, S., and K. Belitz
2011 Occurrence and Concentrations of Pharmaceutical Compounds in Groundwater Used for Public Drinking-Water Supply in California. *Science of the Total Environment* 409:3409–3417.
Gerlach, George H.
1948 *Datura innoxia*, a Potential Commercial Source of Scopolamine. *Economic Botany* 2:436–454.
Gilcrease Museum
2018 History. Electronic document, https://gilcrease.org/about/history/, accessed July 16, 2018.
Hall, Grant D., Stanley M. Tarka, William J. Hurst, David Stuart, Richard E. W. Adams
1990 Cacao Residues in Ancient Maya Vessels from Rio Azul, Guatemala. *American Antiquity* 55:138–143.
Hudson, Charles M. (editor)
2004 *Black Drink: A Native American Tea*. University of Georgia Press, Athens.
Hurst, William J., Stanley M. Tarka, Terry G. Powis, Fred Valdez, and Thomas R. Hester
2002 Cacao Usage by the Earliest Maya Civilization. *Nature* 418:289–290.
Kan, Michael, Clement Meighan, and H. B. Nicholson
1989 *Sculpture of Ancient West Mexico: Nayarit, Jalisco, Colima*. Los Angeles County Museum of Art, Los Angeles.
King, Adam, Terry G. Powis, Kong F. Cheong, Nilesh W. Gaikwad
2017 Cautionary Tales on the Identification of Caffeinated Beverages in North America. *Journal of Archaeological Science* 85:30–40.
La Vere, David
2007 *Looting Spiro Mounds: An American King Tut's Tomb*. University of Oklahoma Press, Norman.
Lankford, George E.
2007a Some Cosmological Motifs in the Southeastern Ceremonial Complex. In *Ancient Objects and Sacred Realms: Interpretations of Mississippian Iconography*, edited by F. Kent Reilly and James F. Garber, pp. 8–38. University of Texas Press, Austin.
2007b The Great Serpent in Eastern North America. In *Ancient Objects and Sacred Realms: Interpretations of Mississippian Iconography*, edited by F. Kent Reilly and James F. Garber, pp. 107–135. University of Texas Press, Austin.
2011 The Swirl-Cross and the Center. In *Visualizing the Sacred: Cosmic Visions, Regionalism, and the Art of the Mississippian World*, edited by George E. Lankford, F. Kent Reilly, and James Garber, pp. 251–278. University of Texas Press, Austin.
2012 Weeded Out the Noded. *Arkansas Archaeologist* 50:50–68.
2014 Following the Noded Trail. *Arkansas Archaeologist* 53:51–68.
Lester, R. N., M. Nee, and N. Estrada
1991 Solanaceae III—Taxonomy, Chemistry, Evolution. In *Proceedings of Third International Conference on Solanaceae*, edited by J. G. Hawkes, pp. 197–210. Royal Botanic Gardens, Kew, UK.
Litzinger, William
1981 Ceramic Evidence for Prehistoric *Datura* Use in North America. *Journal of Ethnopharmacology* 4:57–74.
McGovern, Patrick E., Benjamin P. Luley, Nuria Rovira, Armen Mirzoian, Michael P. Callahan, Karen E. Smith, Gretchen R. Hall, Theodore Davidson, and Joshua M. Henkin
2013 The Beginning of Viniculture in France. *Proceedings of the National Academy of Sciences* 110(25):10147–10152.
Perino, Gregory, Charles C. Willoughby, and Harry L. Lemley
1960 The Piasa Design in Arkansas. *Central States Archaeological Journal* 7(4):146–150.
Phillips, Philip, and James A. Brown
1978 *Pre-Columbian Shell Engravings from the Craig Mound at Spiro, Oklahoma*, Part 1. Peabody Museum Press, Cambridge, Massachusetts.
1984 *Pre-Columbian Shell Engravings from the Craig Mound at Spiro, Oklahoma*, Part 2. Peabody Museum Press, Cambridge, Massachusetts.
Powis, Terry G., Fred Valdez Jr., Thomas Hester, and William J. Hurst
2002 Spouted Vessels and Cacao Use among the Preclassic Maya. *Latin American Antiquity* 13:85–106.
Ratsch, Christian
2005 *The Encyclopedia of Psychoactive Plants: Ethnopharmacology and Its Applications*. Park Street Press, Rochester, Vermont.
Reber, Elenora A., and Robert P. Evershed
2004 How Did Mississippians Prepare Maize? The Application of Compound-Specific Carbon Isotope Analysis to Absorbed Pottery Residues from Several Mississippi Valley Sites. *Archaeometry* 46:19–33.
Reilly, F. Kent
2004 People of Earth, People of Sky: Visualizing the Sacred in Native American Art of the Mississippian Period. In *Hero, Hawk, and Open*

https://doi.org/10.1017/aap.2018.30 Published online by Cambridge University Press

Hand: American Indian Art of the Ancient Midwest and South, edited by Richard Townsend and Robert V. Sharp, pp. 125–138. Yale University Press, New Haven, Connecticut.

Schondube, Otto
1998 Natural Resources and Human Settlements in Ancient West Mexico. In *Ancient West Mexico: Art and Archaeology of the Unknown Past*, edited by Richard F. Townsend, pp. 204–215. Thames and Hudson, New York.

Society for American Archaeology
2018 Principles of Archaeological Ethics. Electronic document, http://www.saa.org/AbouttheSociety/PrinciplesofArchaeologicalEthics/tabid/203/Default.aspx, accessed July 16, 2018.

Walker, Chester P.
2004 The Art of the Central Mississippi Valley. In *Hero, Hawk, and Open Hand: American Indian Art of the Ancient Midwest and South*, edited by Richard Townsend and Robert V. Sharp, pp. 219–259. Yale University Press, New Haven, Connecticut.

Washburn, Dorothy K., William N. Washburn, and Petia A. Shipkova
2012 Cacao Consumption during the 8th Century at Alkali Ridge, Southeastern Utah. *Journal of Archaeological Science* 40(4):2007–2013.

Zarrillo, S., Nilesh W. Gaikwad, C. Lanaud, Terry G. Powis, C. Viot, I. Lesur, O. Fouet, X. Argout, E. Guichoux, F. Salin, R. L. Solorzano, O. Bouchez, H. Vignes, P. Severts, J. Hurtado, A. Yepez, Louis E. Grivetti, T. M. Blake, and Fred Valdez
2018 Mid-Holocene *Theobroma cacao* Use Highlights Upper Amazon as Early Center of Domestication. *Nature Ecology and Evolution*, in press.

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