Ritual Black Drink consumption at Cahokia

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Edited* by Bruce Smith, National Museum of Natural History, Smithsonian Institution, Washington, DC, and approved June 29, 2012 (received for review May 18, 2012)

Chemical analyses of organic residues in fragments of pottery from the large site of Cahokia and surrounding smaller sites in Illinois reveal theobromine, caffeine, and ursolic acid, biomarkers for species of *Ilex* (holly) used to prepare the ritually important Black Drink. As recorded during the historic period, men consumed Black Drink in portions of the American Southeast for ritual purification. This first demonstrated discovery of biomarkers for *Ilex* occurs in beaker vessels dating between A.D. 1050 and 1250 from Cahokia, located far north of the known range of the holly species used to prepare Black Drink during historic times. The association of *Ilex* and beaker vessels indicates a sustained ritual consumption of a caffeine-laced drink made from the leaves of plants grown in the southern United States.

archaeology | organic residue analysis | caffeinated drinks | Mississippian ritual

Organic residue analysis of ceramics from Cahokia, the largest prehispanic site north of Mexico, reveals the presence of theobromine, caffeine, and ursolic acid, biomarkers for *Ilex*, or holly, species. We analyzed residue samples using liquid chromatography–mass spectrometry/mass spectrometry (LC-MS/MS), and show that occupants of Cahokia and surrounding small sites consumed *Ilex* drinks, documented historically as Black Drink, from beakers dating circa A.D. 1050–1250 (Fig. 1). Here we provide unique evidence of prehispanic use of *Ilex*, providing evidence for historical continuity in the use of caffeinated drinks in ritual activity in North America.

Cahokia was the earliest and greatest of the pre-Columbian native polities of the Mississippian era, dating to circa A.D. 1050–1600 (Fig. 2). This proto-urban center and its allied sites were spread across the fertile floodplains and uplands of the Mississippi River near modern St. Louis; a region marked by the conjunction of several major rivers and physiographic zones. By A.D. 800, numerous small farming villages dotted the landscape. In the mid-1000s, a religious, social, and political consolidation (Lohmann phase, A.D. 1050–1100) signified the rise of Cahokia as a discrete polity. Within a century, the central core, a Greater Cahokia administrative-political center, had grown in size to cover a 14.5-km^2 area. It encompassed more than 200 earthen mounds including the largest in North America, the multiterraced Monks Mound, ceremonial plazas, postcircle monuments, marker posts, borrow pits, dense habitation zones of elites and commoners, and a population of 15,000+ inhabitants. By the early to mid-1300s, Cahokia was abandoned (1–5).

Greater Cahokia includes three imposing ceremonial precincts. The largest precinct is at Cahokia, where an expansive 19–24 ha plaza is fronted on the north by Monks Mound rising more than 30 m in height. This precinct contained 120 other platform and burial mounds, residential areas, specialized workshops, borrow pits, and subsidiary plazas. Situated between Cahokia and the river, the East St. Louis ceremonial precinct with ∼50 mounds may have served as a special or high-status residential district for Greater Cahokia. On the opposite of the river the St. Louis ceremonial precinct, with its 26 mounds, provided access to the northern Ozarks. Within a day's walk of Monks Mound lay 14 subordinate single and multiple mound centers, and many hundreds of small rural farmsteads (4, 6, 7).

Cahokia's rapid growth was generated by considerable immigration drawn from regional populations of several adjacent states. This suggests that Cahokia was multiethnic and linguistically diverse. Early models of Cahokian growth postulated a wide trading network as pivotal to that process; however, more recent research has shown that large-scale acquisition and exchanges were focused on resources in the nearby Ozark highlands. Cahokia did, however, participate in the limited long-distance acquisition of items such as marine shell, sharks teeth, pipestone, mica, Hixton quartzite, exotic cherts, copper, and galena (4, 8–11). Consequently, Cahokia maintained a broad network of interactions with widely diverse groups ranging from the Gulf Coast and Southeast to the trans-Mississippi South (especially the Arkansas River valley Caddoans), the eastern plains, the upper Mississippi valley, and the Great Lakes.

At Contact, populations throughout much of the New World consumed beverages created from plant leaves, twigs, bark, or nibs and characterized by the presence of methylxanthines, including caffeine, theobromine, and sometimes theophylline. These drinks derived from two primary sources: *Theobroma cacao* and plants of the *Ilex* species, which are members of the holly family found on every continent except Antarctica. Because consumption of such drinks was widespread, an important question concerns when New World populations first created drinks from these plants, how they accessed the primary ingredients, and under what circumstances they drank them. The research reported here concerns consumption of such drinks at and near Cahokia.

The best-known New World drink with methylxanthines is chocolate derived from the *Theobroma cacao* tree. Native to the upper Amazon, the tree was cultivated in the tropics of Mesoamerica and exchanged widely, including into the American Southwest (12, 13).

Beginning in the 1500s, early explorers reported consumption of drinks containing caffeine in the area now comprising the southeastern United States (14). Over a large area from Florida to Texas, Arkansas to North Carolina, reports described Native Americans preparing and drinking beverages made from the toasted leaves of a variety of holly, probably *Ilex vomitoria* Ait., also called Yaupon (Fig. 2). Men often drank these beverages from cups made of marine shells. *I. vomitoria* grows to 8 m tall and is native to the Coastal Plain of the southeastern US from Virginia to Florida and west to Texas, as well as Bermuda, Chiapas and Veracruz (15). There is strong evidence that *I. vomitoria* was transplanted and cultivated by Native American populations to create this drink (14, 16, 17). A second holly, *Ilex cassine* L.,

Author contributions: P.L.C., T.E.E., W.J.H., and T.R.P. designed research; J.G. and T.W. performed research; P.L.C. and W.J.H. analyzed data; and P.L.C., T.E.E., J.G., W.J.H., T.R.P., and T.W. wrote the paper.

The authors declare no conflict of interest.

*This Direct Submission article had a prearranged editor.

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This article contains supporting information online at www.pnas.org/lookup/suppl/doi:10.1073/pnas.1208404109/-/DCSupplemental.

Fig. 1. Beakers from Greater Cahokia and the hinterlands (Linda Alexander, photographer). [Used with permission of the Illinois State Archaeological Survey].

known as Dahoon, might also have been used to prepare such drinks (15). *I. cassine* L. is native to the southeastern coastal area of North America from Virginia to southeastern Texas, Veracruz, and the Bahamas, Cuba, and Puerto Rico. This large shrub or tree grows up to 13 m in height, with evergreen leaves. Explorers called the beverage by various names, but today it is generally referred to as the Black Drink or cassina (18). Some scholars have suggested that both plants were used to create Black Drink, but most argue that only *I. vomitoria* was used in this way, an argument bolstered by the considerably higher amount of caffeine in *I. vomitoria* (15).

Populations in South American continue to make drinks from varieties of holly. Yerba maté made from *Ilex paraguariensis* St.-Hil., té o' maté made from *Ilex tarapotina* Loes., and guayusa made from *Ilex guayusa* Loes. are popular drinks with a deep history in South America (15). All of these holly drinks contain differing ratios of the methylxanthines caffeine and theobromine.

Because plant parts rarely preserve in archaeological contexts and because preparation of these drinks degrades or destroys the primary plant parts, direct evidence is scarce for the use of these plants to create prehispanic beverages. Fortunately, absorbed organic residue analysis allows identification of methylxanthines preserved in ceramics, permitting researchers to identify the presence of this class of beverage in the past. Absorbed organic residue analysis has been incorporated into archaeological interpretation for almost four decades. It relies on interpreting residues absorbed into porous, unglazed ceramics through use. Such residues can preserve for long periods of time because the interior pores are relatively protected from degradation. The residues are released upon grinding and extraction at elevated temperatures with ultrasonication. Researchers generally analyze multiple samples of ceramics to compare the results as a method of confirming initial findings. Absorbed organic residue analysis has identified cacao residues in a variety of contexts and vessel forms. Experimental work has demonstrated the persistence of caffeine from *I. vomitoria* in varied environmental contexts (19).

When caffeine and theobromine are encountered in absorbed organic residue analysis of archaeological materials, distinguishing which of the plants with methylxanthines left the residues may be difficult. First, residues may be differentially preserved, depending on various factors including the porosity of ceramic paste, original firing temperature of the pottery, use/cleaning of the vessel before deposition, preservation conditions at the site, and treatment/cleaning by archaeologists on recovery. Second, although each of the possible plants has a distinctive profile of methylxanthines compared with the others, the actual amounts also vary within each species. In *Ilex*, the amount of caffeine declines with the age of the leaves (18); shade (20) and latitude (21) may affect methylxanthine concentrations as well. Finally, preparation techniques, including the number of leaves used as well as the amount of water and other additives, alter the concentrations of methylxanthines in the drinks. For instance, tea leaves contain more caffeine than coffee beans, but brewed coffee has many times more caffeine than tea (15). The drinks with methylxanthines consumed in the New World were prepared in a variety of ways. Thus, drinks made with cacao ranged from thick to thin, hot to cold, with a variety of additives (22).

In contrast to a recent argument that caffeine alone is an adequate biomarker for distinguishing such drinks when combined with geography (19), we argue that caffeine alone shows only that a caffeinated drink was prepared in or consumed from the vessel, not the ingredients of that drink. For some parts of the New World, including South America, Mesoamerica, and the southeastern US, multiple plants were available for creating caffeinated drinks. In other areas, such as the American Southwest or the American Bottom (the flood plain of the Mississippi River in southern Illinois), no locally available plants were suitable for producing these drinks. The closest resources are not necessarily the resources used. So, we have searched for more secure means of distinguishing which plant left the residues found in pottery fragments.

Fortunately, research shows patterning in the ratios of methylxanthines in the *Ilex* species and cacao (15, 23, 24). Of the options known to have been present in North America, generally, *I. vomitoria* has a ratio of caffeine to theobromine of about 5:1 (15). In contrast, *I. cassine* has a ratio of caffeine to theobromine of 1:2, and low levels of both (15). *T. cacao* has a ratio of caffeine to theobromine of anywhere from 1:4 to as much as 1:7 (24). Specific compounds help to distinguish these as well. Although theobromine and caffeine are present in all of these plants, theophylline is not detected in *I. vomitoria* or *I. cassine*, but is present in low amounts in cacao. Our research indicates that ursolic acid is a biomarker for distinguishing *Ilex* from cacao.

Ursolic acid (3b-hydroxy-urs-12-en-28 oic acid) is a triterpenoid known to occur in a wide variety of plants in the form of a free acid or as an aglycone (25). It does not occur in cacao, although it does occur in many plants in addition to holly. We believe that use of a number of biomarkers provides an orthogonal approach to resolving the question of which plant left the organic residues; hence the inclusion of ursolic acid along with the methylxanthines.

In attempting to distinguish the source of organic residues in the Illinois material, we looked specifically at the ratios of caffeine to theobromine, the presence/absence of theophylline, and the presence/absence of ursolic acid. We note that theophylline occurs in low amounts in cacao and should not be taken as a necessary marker for chocolate drinks. If theophylline is present, chocolate was definitely present; if theophylline is absent, the ratios of theobromine to caffeine may help to determine the original substance.

One additional issue confounds the search for caffeinated drinks in the prehispanic New World: drinking vessels may or may not have been dedicated to use with only a single specific drink. It is entirely possible that populations with access to both chocolate drinks and *Ilex* drinks consumed both from the same vessel. Researchers need to be aware that vessels may have held multiple types of drinks, each potentially made from multiple ingredients.

Materials and Methods

For Cahokia, eight samples from beakers excavated at four separate sites were analyzed using LC-MS/MS at the Keck Center for Instrumental and Biochemical Comparative Archeology at Millsaps College. Initial sample preparation involved burring all exterior surfaces from fragments of ceramics ~1 cm^2 in size using a tungsten-carbide bit. Because the instrumentation is highly sensitive and the ceramics porous, samples may be contaminated from any contact

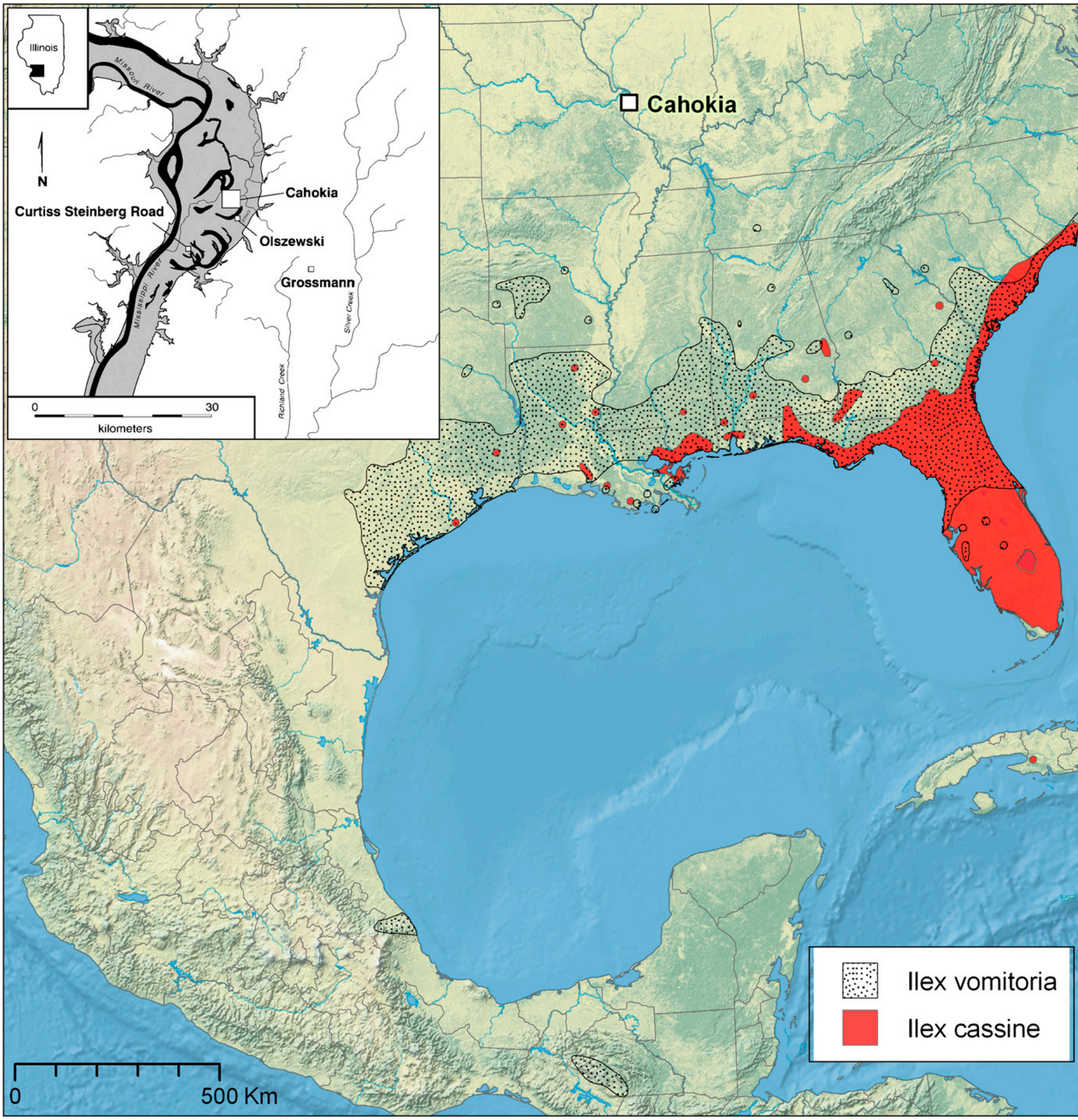

Fig. 2. Map showing distribution of *Ilex vomitoria* and *Ilex cassine* and Cahokia area sites used in study. Distribution data from http://esp.cr.usgs.gov/data/atlas/little/. (Ron L. Stauber, draftsman).

with caffeine, so it is critical that laboratory workers wear masks, gloves, and gowns and that no caffeinated drinks enter the laboratory. Samples were then ground into a powder for analysis.

For methylxanthine analysis, ~500 mg of ground sample was weighed out into a test tube and ~3 mL of hot deionized water was added to the test tubes. The samples were then heated in the heating block at 85 °C for 20 min. Samples were then cooled to RT and placed in a centrifuge for 10 min at a speed of at least 1,000 RPM. The supernatant was decanted into a 10-mL beaker and was reduced at 90 °C on the heating block until ~1.5 mL remained. Spiked solutions were created to confirm the trace peaks. Spiked solutions were made by taking a 495-μL aliquot of each 1.5-mL sample solution and spiking it with 5 μL of a standard solution of 1 ppm caffeine and theobromine. After mixing, this resulted in a spiked solution with an additional concentration of 10 ppb of caffeine and theobromine. All samples were transferred to auto sampler vials for LC-MS/MS analysis.

For the ursolic acid analysis, ~500 mg of ground sample was weighed into a test tube and ~3 mL of hot methanol added to the test tube. The samples were placed in an ultrasonic bath for 10 min, followed by a centrifuge for 10 min at a speed of at least 1,000 rpm in a Fisher Scientific Micro-V 7200 Gram Capacity Micro Centrifuge. The supernatant was decanted into a 10-mL beaker and reduced in an ultrasonicator until ~1.5 mL remained. Spiked solutions were made by taking a 495-μL aliquot of the 1.5-mL sample solution and spiking it with 5 μL of a 10-ppm ursolic acid standard solution. After mixing, this resulted in a spiked solution with an additional concentration of

Table 1. Mass fragments used to monitor caffeine, theobromine, theophylline, and ursolic acid in the ESI mode

Compound	Mode	Mass fragments
Caffeine	Positive	195.0 > 109.9
	Positive	195.0 > 137.9
Theobromine	Positive	181.0 > 138.0
	Positive	181.0 > 163.0
Theophylline	Positive	181.0 > 123.9
Ursolic acid	Positive	457.4 > 297.3
	Positive	457.4 > 411.3
	Positive	457.4 > 439.3
	Negative	455.0

100 ppb ursolic acid. All samples were transferred to auto sampler vials for LC-MS/MS analysis.

The samples were analyzed using a Varian 325 LC-MS/MS. For methylxanthine analysis, the drying gas temperature was set at 400 °C and the voltage on the detector was set at 1400 V. Separations were performed on a Grace 4.6 mm × 50 mm C18 reverse phase column with a mobile phase consisting of 85% (vol/vol) ammonium acetate buffer 0.1% (vol/vol) at pH of 4.2 and 15% (vol/vol) acetonitrile at a flow rate of 200 μL/min. For ursolic acid analysis, the drying gas temperature was set at 300 °C, the voltage on the detector was set at 1200 V. Separation was performed on a 4.6 mm × 50 mm FRULIC-C column with a methanol–water mobile phase [85% (vol/vol) methanol, 15% (vol/vol) water, 0.2% (vol/vol) ammonium hydroxide, and 0.3% (vol/vol) acetic acid] at a flow rate of 200 μL/min. Table 1 lists the mass fragments used to monitor caffeine, theobromine, theophylline, and ursolic acid in the ESI mode. In *SI Text* we provide the data for sample 2085, including chromatograms of standard, sample, and spiked sample for quantification respectively for caffeine and theobromine as well the total ion chromatogram of caffeine, theobromine, and theophylline for sample 2085 (Figs. S1–S3); two chromatograms of ursolic acid show the mass fragment (457.4–439.3) and the parent fragment (455.0) (Figs. S4 and S5).

All samples were routinely analyzed at least twice to ensure the accuracy of the results. Multiple blank samples were routinely analyzed between the archaeological samples to acquire a blank reference and to prevent contamination. There were no signals for any of the compounds detected in any of our blank solutions. Limits of detections (LODs) were calculated based on the International Union of Pure and Applied Chemistry (IUPAC) method: 3 * s (noise of the blank)/*m* (slope of the calibration curve). LODs are defined by both the noise and the signal intensity. We note that although all of the samples in this study produced positive results, in our larger study of 137 samples from other geographic areas, the number of samples analyzed with negative results (39 of 137) provides evidence that we are not simply monitoring background contamination.

Beakers are usually mug-shaped and -sized containers with relatively flat bottoms, near-vertical walls, lug handles, and depths and orifice diameters of less than 20 cm. Beakers are unknown in the greater Cahokia region before the 1000s (26, 27). These vessel forms might ultimately derive from similar vessels common among the ceremonial wares of various peoples in Arkansas, Mississippi, and Louisiana. After appearing in the early 11th century, beakers remain a ubiquitous, although infrequent, component of Cahokia's assemblages (28–30). They seldom comprise more than a small percentage of any assemblage, attesting to their use as special serving dishes.

The eight vessels in the present sample include beakers dating from A.D. 1050–1250 (Table 2). At least four beakers were shattered in ritual performances, including one beaker from the sub-Mound 51 pit (31), one from ritual refuse at a Cahokian "ritual-administrative" outpost (8), and two from platform mound contexts at Cahokia proper (32, 33). Two others were offerings buried with an adult at the Curtiss Steinberg Road site, a small rural cemetery 10 km south of Cahokia (34). The last two vessels are from likely domestic contexts from pits at both the Olszewski site, a large residential outlier on the southern border of Cahokia (35), and the Cahokia site's Dunham Tract area (30).

Results

The results in Table 2 demonstrate a combination of theobromine, caffeine, and ursolic acid, confirming the presence of *Ilex*. Although historic documents would suggest that the source was likely *I. vomitoria*, the detection of more theobromine than caffeine in many of the samples indicates that we cannot rule out the use of *I. cassine* instead, or even a combination of both species.

Discussion

Black Drink was recorded as used in historic period political, religious, and social contexts among many tribes of the US Southeast and trans-Mississippi South (14, 18, 36, 37). Its preparation involved the parching of holly leaves and small twigs that were subsequently placed in a large pot with water, boiled, and finally agitated into froth before consumption. For many groups, Black Drink played a central role in the ritual cleansing and purging of the body when combined with fasting and vomiting that was an essential precursor to conducting any important activities. Euroamerican observers documented the prodigious consumption of Black Drink, especially by men, often followed by bouts of ritual vomiting, before individual or community religious rituals, important political councils and negotiations, ballgames, or war parties.

There are five implications of this research that we outline below. First, the documentation of Black Drink at ~ A.D. 1050 is the earliest known precontact use. Second it demonstrates the

Table 2. Samples analyzed by HPLC-MS with contextual information and results

Beaker sample no.	Site	Context	Phase	Reference	Theobromine ppb*	Caffeine ppb*	Ursolic acid ppb*
2085	Cahokia	Submound 51, feasting pit	Lohmann A.D. 1050–1100	(31)	1.1	0.48	3
2086	Cahokia	Mound 33, general contexts	Late Stirling–early Moorehead A.D. 1150–1250	(32)	0.8	0	6
2087	Grossmann	Feature 308, ritual deposit in pit	Stirling phase A.D. 1100–1200	(8)	0	T	2
2088	Curtiss Steinberg Road site	Feature 8, adult burial	Late Stirling–early Moorehead A.D. 1150–1250	(34)	T	1.1	4
2089	Curtiss Steinberg Road site	Feature 8, adult burial	Late Stirling–early Moorehead A.D. 1150–1250	(34)	T	T	4
2090	Olszewski	Feature 11, refuse pit	Late Stirling phase A.D. 1150–1200	(35)	T	T	N
2091	Cahokia	Mound 11, general collection	Late Stirling phase A.D. 1150–1200	(33)	0	0	3
2092	Cahokia	Feature 34, Dunham Tract	Stirling phase A.D. 1100–1200	(30)	1	0.5	N

All samples are from collections of the Illinois State Archaeological Survey at the University of Illinois, Urbana–Champaign.
*T, trace (theobromine in trace level (0.15–0.3 ppb); caffeine in trace level (0.1–0.2 ppb); N, insufficient sample available to analyze; 0, nothing detected. No samples had theophylline. LODs for theobromine, 0.1 ppb; for caffeine, 0.06 ppb; and for ursolic acid, 0.7 ppb.

presence of Yaupon or Dahoon holly far north of their natural distributions, indicating their deliberate transportation. Third, it suggests that beakers may have been manufactured specifically to play a role in Black Drink ceremonies (38). Fourth, it reinforces other evidence for the existence of a fertility/life-renewal cult at Cahokia that included Black Drink ceremonies. Most importantly, it bolsters earlier suggestions that Cahokia played an important role in the subsequent religious developments in the Southeast.

Given its close association with important religious and political events in areas south of Cahokia, and given Cahokia's documented ties to those areas, especially with the Caddoan groups, Black Drink would logically seem to have been an integral part of Cahokian ritual activities (38). Cahokia, however, is well outside the historically recorded natural distribution of *I. vomitoria* or *I. cassine* (Fig. 2). Historical accounts place *I. vomitoria* in southwestern Arkansas and the southeastern corner of Oklahoma (south of the Arkansas River), in the southern one-half of Louisiana, and the southern one-third of Mississippi, distance ranging from ∼500–650 km overland from Cahokia. *I. cassine* would be found at an even greater distance from Cahokia.

Early accounts (14, 16, 36, 39) indicate that populations without access to natural stands of holly acquired dried Yaupon leaves through trade or transplanted Yaupon holly bushes near to their habitations in settings where it could survive the local climate. Unfortunately, archaeological signatures of such activities are difficult to identify. For example, University of Illinois archaeobotanical researchers have examined nearly 10,000 L of Mississippian period soil samples, searching for carbonized *Ilex* remains, to no avail. There is no ethnobotanical evidence for the presence of Yaupon or Dahoon holly in the American Bottom.

It appears then that when Cahokians imbibed Black Drink, it was an imported luxury. If Black Drink were as intimately interwoven into the spiritual and political life of Cahokians as it was among historic natives in the Southeast, they would have required large amounts of *Ilex*. Such an exchange network would have connected Cahokians to groups who had access to holly, such as their Caddoan neighbors in the Arkansas River valley or groups near the mouth of the Mississippi River.

Scholars have postulated that Black Drink ritualism may have been more widespread in precontact than in historic times based on its close association with marine shell cups and renewal and purification ceremonialism (39). Cahokians certainly possessed both shell cups and ceramic effigy shell cups, and some researchers have used this line of reasoning to argue for Black Drink at Cahokia (38). Others have suggested the centrally manufactured, highly iconic Ramey jars may have been used for the preparation and distribution of medicines, perhaps including Black Drink (40). The association of shell cups, Ramey jars, and possibly effigy shell cups with Black Drink consumption needs to be verified with residue analysis.

The conclusive evidence for the use of beakers as part of Black Drink ceremonialism raises the issue of whether this form is consistently evidence for Black Drink consumption. If so, then its appearance marks a significant elaboration in ritual Black Drink ceremonialism at Cahokia during the 11th to 13th centuries. In addition, the spread of this unique vessel form up the Illinois River valley and into southern Wisconsin with suspected American Bottom missionaries, colonists, or emigrants indicates another close but previously unrecognized religious tie to the Cahokia homeland's practices. It suggests that the beakers spread as part of a religious package including a suite of ritual accoutrements such as flint clay figures, Ramey Incised vessels, and accompanying icons and religious ceremonies. If their association with Black Drink is shown to be exclusive, then beakers add another distinctive artifact to those already associated with Cahokian religion during the Stirling phase (A.D. 1100–1200) (1, 41).

Fairbanks (36) has argued that Black Drink ceremonialism has time depth in the Eastern Woodlands, perhaps even dating to late Archaic times, but most probably present in Hopewellian times. Future research should consider this argument. If it is shown to be correct, it is possible that Cahokian beakers represent ritualized elaboration of earlier localized Black Drink consumption practices, practices that may have continued past the demise of the polity. Creation of a regional history of Black Drink consumption will require analysis of earlier and later ceramic wares. Our results represent unique proof that Black Drink ceremonialism has actual time depth—pushing its documented use from historic accounts in the 1500s back four centuries to the 11th century in the northern midcontinent. In the areas where *I. vomitoria*, and perhaps *I. cassine*, is native, its use may have very deep roots. Whether the historic meaning and ceremonialism have a similar time depth is unknown. We recognize that Cahokia was the primate center in the Eastern Woodlands, and that its ritualism, religion, and political and social milieu had a deep impact on surrounding groups. Although Cahokia residents are not likely to have invented Black Drink, they may well have shaped its role in native religious ceremonialism and served as a catalyst for its spread across the Eastern Woodlands.

Before this study, arguing for Black Drink purification ceremonialism in the American Bottom depended on proxy evidence. Such assertions were made based on the recognition of agricultural ceremonies involving fertility/life-renewal rituals at Cahokia (38). The demonstrated presence of *Ilex* in Cahokian beakers beginning around A.D. 1050 and continuing to around A.D. 1250, both in central Cahokia, and in outlying sites establishes that Black Drink consumption was intimately associated with the polity and that its use was pervasive across a number of political, social, and religious contexts.

ACKNOWLEDGMENTS. We acknowledge the Illinois State Archaeological Survey for their contributions to this project. Josh Henkin provided samples of *Ilex* species for our analysis. T.E.E. and T.R.P. selected the samples from the collections of the Illinois State Archaeological Survey at the University of Illinois, Urbana–Champaign. Ellen Sieg aided P.L.C. in preparing the archaeological samples. T.W. and J.G. analyzed the samples at the Millsaps College Keck Center for Instrumental and Biochemical Comparative Archaeology with help from undergraduates Erin Redman, Syed Ali, and Marlaina Berch. Linda Alexander photographed the beakers in Fig. 1 and Ron Stauber drafted Fig. 2. We thank Bruce Smith and two anonymous reviewers for helpful comments on an earlier draft of this paper. This research was conducted with funding from National Science Foundation Grant BCS-1012438 (to P.L.C. and W.J.H.) and the WM Keck Foundation (to Millsaps College).

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