ARCHAEOLOGICAL SITE REVEGETATION, ORGANOCHLORIDE BASED PESTICIDES, PCB’S AND THEIR RELATIONSHIPS TO RESOURCE PRESERVATION AND PROTECTION

Prepared by

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With

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for

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ARCHAEOLOGICAL SITE REVEGETATION, ORGANOCHLORIDE BASED PESTICIDES, PCB’S AND THEIR RELATIONSHIPS TO RESOURCE PRESERVATION AND PROTECTION

INTRODUCTION

BACKGROUND

Archaeological sites are being lost or significantly degraded due to natural and cultural impacts that have greatest effect upon exposed or unconsolidated surfaces. Erosion has long been recognized as the mechanism which is most damaging, but in addition to the physical losses due to erosion mechanics there are other changes than can be detrimental to the remnant deposits. Soil properties, including morphological, physical, chemical, mineralogical, and biological characteristics, are thrown into disequilibrium resulting from mineral weathering, clay genesis, leaching, eluviation/illuviation, salt accumulation, pH fluctuation, and gain or loss of organic carbon.

While wind and water erosion, bioturbation, construction, and agriculture are typically cited as the sources of the problem, archaeological excavations themselves can lead to site erosion and negative effects on soil properties. Poorly chosen locations for excavations relative to natural or cultural threats, lining excavations with inappropriate materials, and backfilling without protecting the reconditioned ground surface can all contribute to rapid, immediate, and significant losses. There are also negative effects that take longer to occur as the rates of soil formation slow.

Revegetation is one of the most important, cost-efficient
stabilization methods to counter these unwanted changes (particularly to halt the immediate catastrophic losses caused by erosion), but there is little data available to evaluate the long-term results on sites, artifacts, and other culturally deposited materials. Evaluating the long-term effects is important because environmental change at archaeological sites cannot be stopped, but an acceptable, knowable level of change can be achieved. Management decisions to plan and implement revegetation projects must consider a range of feasible alternatives, choose solutions based upon identified benefits, and undertake appropriate maintenance activities over a foreseeable time span.

A principal element of this project was to consider archaeological sites as micro-environments within larger ecological contexts. By that we intended to gain new knowledge about how the particular qualities of archaeological sites and materials are affected by ecological changes, especially land-use practices as one of the most significant types of environmental impacts (Collins et al., 1995; Foss et al., 1995:9-12). We elected to concentrate on the effects of organochloride compounds because they represent one of the most significant, discreet, and highly observable chemical changes to soils (Flury 1996).

Though soils include physical, morphological, mineralogical, and biological as well as chemical properties, organochloride analysis were chosen to provide the widest range of useful data for evaluating the condition of the archaeological deposits and materials and for considering the elements appropriate to design
Preservation Technology and Training. This project was undertaken as a cooperative effort among the University of Mississippi, Battelle Pacific Northwest Laboratories, and the National Park Service Archaeology and Ethnography Program. My colleagues in this effort are Richard Waldbauer, NPS Archaeology and Ethnography Program, and Paul Nickens, Battelle Corp. Additional funding to support laboratory analysis was provided by the Archaeology and Ethnography and Ranger Activities Program of the National Park Service. Significant in-kind support was provided by the University of Mississippi Environmental Toxicology Laboratory; the Tennessee Valley Authority; and each of the managers, curators, and property owners of the project field locations.

Briefly, our interest in agricultural chemicals centered around the question of the level of impact that these agents might have on the in situ conservation process. Our initial interest included pesticides and herbicides, but both fiscal and laboratory constraints limited us to a study of pesticides alone. We proposed an analysis of artifacts and soil samples from three archaeological sites that would be central to the study. The pesticides that are the focus of this analysis are organochloride based compounds, some of which are in use today and some that have been withdrawn from sale and use. Compounds of mercury and arsenic were used as pesticides during the late nineteenth and early twentieth centuries and these were included in the analysis of some samples.

The three primary sites in the study were consciously selected, each for a different reason. The Albany Mound Group (11-
WT-1) in Illinois was selected because it has been a managed and planned conservation effort that is directed toward the return of the vegetation within the Park's confines to long grass prairie. The Moccasin Bend site (40-HA-63) to the south of Chattanooga was selected because it is immediately downstream from the industrial district of the city, and was suspected of containing a number of pollutants. In addition, a portion of the site was in active agriculture some years ago. The third site that we selected is located to the south of Natchitoches, Louisiana and was an active cotton plantation until recently. Farming of the two prehistoric sites has not been ongoing for at least a decade, while portions of the plantation fields are still in active agriculture.

Our field methodology included the collection of a one inch soil core that extended downward for a depth of two feet. Where possible, artifacts from the surface as well as from an undisturbed subsurface context were collected. We received full cooperation from the Putnam Museum in Davenport, Iowa and the Illinois State Museum in Springfield, Illinois and were given permission to test materials excavated in 1907 and 1988 at Albany Mounds. The McClung Museum at the University of Tennessee at Knoxville, Tennessee provided us with provenienced sherds from the 1964 excavations at Moccasin Bend.

Field and laboratory treatment of the primary samples was designed to reduce the oxidation of the various pesticides between their recovery and the beginning of the analytical procedure. Soil samples were retrieved in metal tubes, capped and sealed and
kept chilled until they were delivered to the lab where they were frozen. Sample storage in an anaerobic environment is important in pesticide studies since the breakdown of organochloride based compounds is more rapid in an oxygen rich environment. Museum samples were given no special treatment beyond storage in locking plastic bags, since prior storage had been aerobic in nature. Only slight heating of the tubes has been necessary for the removal of the core and the analytical sample. After removal from the steel tubes, the cores are wrapped in plastic and aluminum foil and refrozen for any subsequent analyses that may be appropriate.

The Environmental Toxicology Laboratory at the University of Mississippi has developed and established a laboratory procedure that can identify 13 organochloride based pesticides and the five metabolites produced by the break down of DOT as a part of the same analytical procedure. In addition, this process will identify 21 polychlorinated biphenyls (PCB’ S) as a part of the same run.

The analysis is completed with a gas chromatograph, in our case, a Hewlett-Packard 5890 Series II with a data station. Instrument run time is slightly less than 170 minutes (2 hours and 50 minutes). Compounds and PCB’ s contained in the analytical library of the data station are identified at specific times during the analytical run. As a result, the GC will identify a PBB at a specific point in the run, while in reality the identified material could be a compound or one of its metabolites that are not included in the library. Pesticides included in the standard CC analysis employed in this study are those that are found to have been
commonly used in the southern U. S following World War II.

Sherds and other artifacts that were used as sampling material were reduced to powder using a 3/8° electric drill turned at slow speed. The resulting powder is collected and processed for analysis in the GC. Soil samples are processed without reduction of particle size. Laboratory procedures have been described by Allgood, et al. (1994).

ALBANY MOUND GROUP (11-WT-1)

A total of six samples was recovered from the Albany Mound Site and submitted for analysis. Three of these were soil samples that were recovered from the field, one was a sherd from the 1988 salvage excavations at Mound 38, and the other two consisted of a soil sample and a sherd that was recovered during the 1908 excavations completed by Nickerson. Two of the soil samples were recovered by coring while the third was a grab sample. The locations of the various sampling sites are shown on Figure 1.

Core sample Number 1 was recovered from an extinct meander levee of the Mississippi River and is well within the floodplain. This is a midden deposit that lies between Mounds 69 and 72. Core sample Location Number 2 lies on the northern side of a hollow that is slightly northeast of Mound 7 at a distance of approximately 450’. The third contemporary soil sample was the grab sample that was taken from the vicinity of Mound 44. Three additional samples were assessed from the Albany Mound Site, and were taken from collections housed at the Putnam Museum in Davenport. Two of these
Figure 1. Sample Collection Locations, Albany Mound Group (11-WT-1) after Kimball (1907)
three samples were recovered by Nickerson (Herold 1971) during his 1908 excavations of Mound 20. One of these samples was a small sherd and the other was a soil sample from near what apparently was a hearth. The final sample analyzed from the Albany site was a sherd that was recovered in 1988 during the professional excavations of a looters hole in Mound 38.

MOCCASIN BEND (40-HA-63)

The archaeological deposits at the Moccasin Bend site were of particular interest to this study because of the two-fold potential that both the midden deposit and concomitant artifacts would contain both pesticide and other chemical contaminants. The industrial section of the City of Chattanooga (Figure 2) lies immediately across the Tennessee River from the site and prior to the alteration of the River channel in about 1964, the lower portion of the site that was closer to the river channel was subject to only periodic flooding. The portion of the site excavated by Graham in 1964 was farmed until the late 1950’s.

Contemporary soil samples and a single sherd were collected from the cutbank above the portion of the channel that had been moved to the north. Three core samples from two locations were submitted for assessment as was a single sherd. Four artifact samples from Graham’s excavations were also analyzed. These were vertically stratified samples that had been recovered from adjacent trenches. This portion of the site was removed by channel realignment and comparable soil samples could not be collected for
Figure 2. Sample Locations at the Moccasin Bend Site (40-HA-63)
this study

OAKLAND PLANTATION

Oakland Plantation is located on the Cane (Red) River valley floodplain south of Natchitoches, Louisiana in the west-central portion of the state. The site was selected because of its history of almost continuous cotton agriculture since 1790. Like the Dedic Site, Oakland Plantation is a National Register property and is a part of the Cane River National Historical Site and National Heritage Area.

Following the analysis of the samples from the three primary sites, it became evident that we could expand the geographical range of sampling locations and stay within the budgetary structure for the project. Additional analytical sites were selected in the Tennessee River Valley, in the Mississippi and Connecticut Valleys, and at Casa Grande National Monument. With the exception of the Tennessee River location, all are or have been in agriculture, and use of pesticides or their water borne introduction appeared likely.

RED FOX MOUND (1-LI-is)

This site consists of a small mound and an associated midden deposit that has been partially excavated (Krause: Personal Communication). A portion of the mound, probably 40-50%, has been eroded away by wave activity. The cultural complex is situated on what appears to be the first terrace above the Tennessee River on
the northern side of Wheeler Lake. The midden is buried beneath a culturally sterile level of overbank flood deposited sand and silt, and is effectively sealed from the surface by at least a foot. A single grab sample was recovered from a freshly excavated and cleaned profile.

JAKETOWN SITE (22-HU-505)

This site is located in the lower portion of the Yazoo Basin which is north of Vicksburg, Mississippi and tributary to the Mississippi River. This is a well chronicled site that includes both mounds and associated midden deposits that begin with the Poverty Point Period and extend through the Mississippian (Figure 3) [Phillips, Ford and Griffin 1951:274]. A portion of the site is now owned by the State of Mississippi and has been out of agriculture since 1984. Prior to that time, the application of agricultural chemicals would have been common and aerial drift of pesticides applied to adjacent fields remains a possibility to the present time.

Two sampling loci were selected at this site. One location is situated between the two mounds and the other was on the western side of the western-most of the two mounds. The sample point between the mounds was approximately midway between the two earthworks. Two soil samples were take from this location, with the first being taken from the surface of the ground to a depth of two feet, and the second was taken from the base of the 0.5 ’ deep plowzone to a depth of two feet. Artifactual material collected
Figure 3. Sample Locations at the Jaketown Site (22-HU-505)
with these soil samples was fired clay, and was collected from below the plowzone.

The second sample location was approximately as far away from the present base of the western mound as the first location had been between the mounds. Artifacts collected for analysis were fired clay that was collected from beneath the plowzone. The companion soil sample was taken from the surface of the site to a depth of two feet.

HOLLYWOOD SITE (22-TU-500)

This site is also in state ownership, and consists of at least four Mississippian Period mounds, a midden deposit and what has been interpreted as a plaza area enclosed by the mounds. The site was taken out of active agriculture at the time of state acquisition in 1992. Farming still continues in the immediate vicinity and pesticide introduction is still possible as a result of aerial overspray from the nearby fields. The site was tested archaeologically during the summer of 1993. The test unit locations are visible as depressions and these served as the guide for the collection of soil and artifact samples.

Three sample locations were selected (Figure 4). The first was near the toe of a small mound remnant and four feet to the west of the 1993 test trench. Analytical artifacts collected from this loci were fired clay (daub) and were collected from below the plowzone. The soil sample was initiated at the base of the plowzone—approximately 0.5′ below the level of the ground surface.
Figure 4. Sample Locations at the Hollywood Site (22-TU-500)
The second sample was taken one foot to the south of a test unit that had been placed in the alignment of the suspected ramp which provided access to the top of the large mound. Analytical artifact samples included fired daub and a single sherd, all of which were retrieved from beneath the plowzone. The top of the soil core was at the base of the plowzone, again approximately 0.5’ below the present ground surface.

The third sample was recovered from a point 100’ to the south of the second sample. This location was selected because it is the lowest point in the plaza area, and was thought to be a likely point of concentration of surface water runoff. Artifacts collected for analysis were fired clay that was collected from below the plowzone. The companion soil sample was taken from the present ground surface.

CASA GRANDE NATIONAL MONUMENT

Casa Grande National Monument was selected as one of the additional sites to be assayed for residual pesticides for a number of reasons. The setting of the stands in stark contrast to the balance of the locations selected for this study. The Monument is situated in a semi-arid environment while all of the other sites that were tested are in major stream valleys with sufficient rainfall for non-irrigation farming to be successful. Casa Grande, as the remains of a Hohokam settlement that was occupied between circa A.D. 1150 and 1400, represents successful prehistoric irrigation agriculture. The system of canals and ditches that were
a part of Hohokam farming practices made productive agriculture possible.

While the National Monument was set aside in 1892, modern agricultural practices have encroached to the extent that the Monument is essentially a protected rectangular island surrounded by disturbed terrain. Until recently, cotton fields were planted up to the Monument boundaries, separated only by intervening canals and highways. Modern developments have encroached towards the Monument boundaries, including a housing development to the south and a commercial venture to the east. Active cotton fields still surround much of the Monument. Highways and their rights-of-ways border on the east and north, and a canal and its parallel access road lie immediately adjacent along the southern and southwestern perimeter. The adjoining agricultural tracts were of particular interest since these would be the source of any organochloride based pesticides that might be present in the samples. Analytical soil samples were collected from four locations around the Monument. One sample loci was near the northwest corner of the Great House and the cores were accompanied by a caliche sample that had spalled from the original construction fabric of the structure.

DEDIC SITE (19-FR-157b)

The Dedic Site was selected because of the need to develop general comparative analytical data associated with agricultural change and extensive alluvial deposition that are significantly different from the principal project locations in the
Mississippi and Tennessee River Valleys. The archaeological deposits at the Dedic Site, which is a National Register Property, include deep, intact Paleo Indian features, and the area may have been in agricultural use during the past 300 years. The site is situated within the floodplain of the Connecticut River and is on a virtually flat terminal Pleistocene lake bed surface. Lake Hitchcock was formed when the Connecticut River was blocked by an ice dam in the vicinity of Rock Hill, Connecticut (Ulrich 1978:5). Soil in the lake bed is very sandy and sand dunes and dune remnants are still visible in the area.

The Dedic site location was initially identified as a consequence of a cultural resources assessment prior to the development of the Deerfield Industrial Park. At that time, the site loci was in tobacco fields and tobacco drying barns were located on the southern side of the industrial park development area. Two of the barns were subsequently moved to the south of the dune, with Barn 8 being seated over the distribution of artifacts from the site. Barn 5 lies furthest to the east and is separated from Barn 7 by a distance of about 100’.

ANALYTICAL RESULTS

ALBANY MOUNDS

Soils Analysis

The first of the sites that was visited was the Albany Mound area and these samples composed the initial assessment of materials recovered for this study. A protocol analysis on sherds that had
been taken from collections curated at the University of Mississippi was followed since the identification of organochlorides had been successful. Analysis of the recovered cores followed a protocol that had been previously established by the Toxicology Laboratory.

The first sample, Location 1 (Figure 1) that was assayed was the soil sample that had been recovered from the midden deposit on the abandoned Mississippi River levee on the western side of the site. Samples were assayed from near the top and bottom of the core and both were void of any pesticide compounds or PCB’s. These results were not expected since all traces of Mounds 69 and 70 are gone and the levee appears to have been part of an agricultural plot.

The second core sample, Location 2, assayed was the one taken from the west of Mound 7. This core location is situated in an area that was in corn prior to state acquisition, and the likelihood of pesticides or their metabolites being present appeared greater. This sample contained a low level of para para DDT (p,p-DDT) both at the top and the bottom of the core (2.1 and 3.3 ppb respectively). The top portion of the core contained the PCB’s c-28 and c-195 (2.2 and 1.3 ppb respectively) while the basal portion of the core only contained c-28 (4.8 ppb). The presence of PCB’s was unexpected in this location, partially because of our ignorance of the atmospheric behavior of these materials. The assayed values are low level, however, and we initially attributed their presence to airborne transport from the Great Lakes region.
The analysis of the grab sample, Location 3, indicated the presence of p,p-DDT and the PCB c-28 (4.8 ppb), with the same

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Figure 5. Analytical Values From Soil Sample and Artifacts From Albany Mounds

question regarding the point of origin of the c-28. This sample location is near the present alignment of a bituminous surface highway which could be a PCB source. It is well above the level of the Mississippi River and should have never been the subject of overbank flooding which could have introduced non-point source PCB’s.

All three samples were analyzed for arsenic and mercury. None of the reported values were sufficiently high to suggest that either of these elements had been used at Albany Mounds in a pesticide formulation. The results of the arsenic and mercury
analyses for all of the Albany Mound samples are shown in Figure 6 (indicated in ppb). In general terms, the naturally occurring arsenic content in soil ranges between 1,000-40,000 ppb, well above the levels present at Albany. Mercury levels generally range between 20-625 ppb, placing all of the Albany levels within the background distribution.

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<th>Sample Type</th>
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<th>Hg (ng/g)</th>
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<td>Sherd</td>
<td>AR15236</td>
<td>(a)</td>
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<td>Md</td>
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Figure 6. Arsenic and Mercury Values From Albany Soil Samples and Artifacts

Museum Sample Analyses

This analysis covers both artifacts (15236a) taken from the Putnam Museum and the Illinois State Museum (Sq 2-02), as well as the soil sample from Nickerson’s excavations (#112). Figure 5 indicates the analytical values of the soil and various museum samples from Albany Mounds.

The sherd (sq 2-02) recovered from the 1988 excavations was devoid of organochlorides, but contained identified PCB’s which included c-28 (6.8 ppb), c-99 (2.6 ppb), c-101 (5.7 ppb) and c-195 (17.3 ppb). Since this sherd was recovered from its primary matrix during the 1988 excavations, and was stored in a plastic bag following its archaeological analysis, the presence and point of origin of the PCB’s seemed to require additional inspection. The
analytical program used in the GC will not identify any materials other than organochlorides, their metabolites, and PCB’s. Two viable alternatives to explain their presence are apparent: (1) these were PCB’s that were aerially deposited and absorbed prior to excavation; or (2) a form of organochloride pesticide had been used on crops, and was not incorporated into the analytical database of the GC. The former explanation appeared less likely in the face of the c-195 count of 17.3 parts per billion, but still remained a viable possibility.

MOCCASIN BEND (40-HA-63)

Soil Artifact Analysis

Core samples from two of the three test locations indicated on Figure 2, were submitted for analysis. Two cores were recovered from Location 1. The first sample was taken at the top of the cutbank, some 12’ above the level of the River. The second core taken at this location was adjacent to a surface collected sherd that lay at about the middle of the cutbank on what appeared to be a naturally eroded surface. The upper sample is labeled as Sample 1, the mid-cutbank sample is labeled as S-2-Loc 1, and the associated surface sherd is identified as MB 3-2 in Figure 7 which follows. The top of the upper core yielded p,p-DDT (0.8 ppb) and the PCB c-28 (2.1 ppb), while the lower portion of this core yielded p,p-DDT (2.1 ppb) and the PCB’S c-28 (3.6 ppb), c-126 (2.9 ppb), c-195 (16.7 ppb), and c-206 (1.0 ppb). These analytical
differences and the relatively low values suggest that the analyzed compounds have migrated downward into the soil at the top of the cutbank. The mid-cutbank sample contained only the PCB c-28 (1.0 ppb) at the top of the core and was clean of organochlorides and PCB’s at the bottom of the core. The sherd that was recovered adjacent to the mid-cutbank sample was also devoid of analytes.

Core sample Location 3 (Figure 2) was situated near the top of the cutbank and above the riprap armor of this portion of the river bank. Only PCB’s were present as analytes (Figure 7) with the top portion of the core indicating the presence of c-28 (2.1 ppb), c-
170 (2.1 ppb) c-180 (1.1 ppb) , and c-195 (2.1 ppb) . In contrast, the base of the core produced a single PCB, c-28 (1.8 ppb) . The absence of multiple organochlorides in these samples is somewhat surprising since we had anticipated their presence as a consequence of aerial transport from the nearby industrial area of the city.

**Museum Sample Analysis**

Analytical artifacts were acquired from two trenches that had been excavated by Graham in 1964, and were from stratified locations. Three were selected from adjacent trenches that allowed us to assay materials from depths that measured 0-. 5’ below the surface, 2.0-2.5’ below the surface, 4.3-4.8’ below the surface, and 7.3-7.8’ below the surface. As noted earlier, these materials were recovered from the lower portion and flooding of this portion of the occurred only rarely.

As can be seen from Figure 7, the extent of organochloride contamination of the samples appears to have been an ongoing

<table>
<thead>
<tr>
<th>Batch No</th>
<th>Sample Type</th>
<th>Location</th>
<th>Chemical ID</th>
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**Figure 8.** Analytical Values of Arsenic and Mercury at the Moccasin Bend Site for all Samples

process to the point in time of their excavation. With the
exception of the p,p'-DDT metabolite, quantitative values of all of
the pesticides is higher in the sherd from the 0.0-0.5' level than
for the sherds from the three lower levels. The similar or slightly
higher values found in the sherds from the two lower levels as
opposed to the sherd from the 2.0-2.5' level suggests that the
interior portion of this class of artifacts may serve to slow the
metabolization of the chemical compounds. A similar inference can
be drawn for the various PCB's present in these materials.

Figure 8 includes the arsenic and mercury level analyses from
all of the samples at Moccasin Bend. As was the case with the
Albany Mounds site samples, all of the values at Moccasin Bend are
within the range of background levels.

OAKLAND PLANTATION

Soil and Artifact Analysis From Oakland Plantation

Four loci were selected for the recovery of soil samples and
accompanying artifacts from near the still active agricultural
areas at Oakland Plantation (Figure 9). Sample Area 1 lies to the
west of the plantation house and the slave quarters area and was
inside of the edge of a currently active field. The margin of the
field was the site of a turn road, and numerous historic period
artifacts were present on the surface. The core sample collected
from this location contained no pesticides at the top of the core,
while the bottom of the core contained o,p'-DDT (13.3 ppb), p,p'
DDE (82.5 ppb), o,p'-DDE (1.4 ppb) Mirex (6.3 ppb) and the PCB c
The presence of Mirex is not surprising since fire ants have been in the area for years. The artifact analyzed with this core sample was historic period sherd which contained only the DDT metabolite p,p'-DDE (2.6 ppb). Sample Location 2 was to the north of Location 1 near the remains of a building that had been used as a cotton gin. At the time that the sample was recovered, this area was incorporated in a plowed field. Only the metabolite p,p'-DDE was identified in the analysis of the top of the core, and was at a very low level (6.1 ppb). The artifact associated with this soil sample was a brick fragment that also contained a low concentration of p,p'-DDE (3.4 ppb). Sample Location 4 was situated along the fence line that separated the barnyard space near the house from the field. This location is geomorphically described as backswamp, indicating that it lies below and at the back side of a natural levee built by the river. This soil is referred to locally as "gumbo" because of its high content of fines which causes it to be very sticky when wet. The bottom of the core recovered from this location was devoid of pesticides. The upper portion of the core sample yielded only p,p'-DDE (2.2 ppb).
artifact recovered for companion analysis was a brick fragment whose pesticide content matched the analysis of the top of the core --- p,p’-DDE at a concentration of 2.2 ppb.

RED FOX (1-LI-15)

Soil Analysis

As noted, this site was selected for sampling because of its proximity to the Tennessee River and overbank flooding that would have occurred, and because of the buried nature of the midden deposit. A single grab sample was collected for analysis. No organochlorides or PCB’s were present. This sample was not examined for the presence of arsenic or mercury.

JAKETOWN SITE (22-HU-505)

Soil Artifact Analysis

Two locations were selected for sampling at the Jaketown Site, with care being taken to insure that the two area represented the major aspects of the topography of the site. Two core samples were collected from Location 1, with the first being taken from the surface of the ground while the second was recovered from the base of the plowzone. Prior to taking the soil samples, a small test unit (15 cm x 15 cm) was excavated to establish vertical stratigraphic control and to allow the recovery of artifacts to be tested for residual pesticides. The plowzone depth was established in this manner, and its removal guided the recovery of the second sample. The primary reason that the below plowzone sample was
recovered was that it allowed us to extend the depth of the core approximately six inches, which proved to be an instructive decision. Figure 10 provides the analytical values of the core artifact samples that were recovered from both Locations.

As noted earlier, this site came into state ownership in 1984 and pesticides have been applied directly to the site since that time. Only two pesticides were identified, DDT and its metabolites, and Mirex. Prior to its withdrawal from approval application, the latter was used for a number of years as a control for fire ants. While the Mirex levels in the soil samples are relatively low, they are indicative of the environmental persistence of this material. The metabolites of DDT (o,p'-DDT, p,p'-DDE, and o,p'-DDE) occur at the top of the first core taken at Location 1. The sample taken from the base of the plowzone contained p,p'-DDT, o,p'-DDT, and p,p'-DDE. Only p,p'-DDE is present at the bottom of core Number 1. The bottom of the second core taken at this location was six inches below the base of the first core and was devoid of any pesticides or any of their metabolites. At this juncture, it seems logical to assume that these compounds had not percolated to that depth.

Mirex was present at the top of both of the samples taken at this location. None was present at the base of either core sample. The residues were present at low levels, 3.5 ppb and 1.7 ppb respectively. These low levels may indicate that the use of Mirex was limited at these locations, and may have been introduced as aerial drift. The primary use of this compound was for fire ant control.
control and the unfarmed margins of the earthworks would have been prime locations for the ant hills. Treatment of any hills would likely have been close enough that contamination at the two core locations could have occurred.

Sample Location 2, on the western side of the western-most mound produced a slightly different suite of pesticides. Para-para DDT and its metabolite o,p′-DDE were found, as was A-chlordane, Dieldrin, Endosulfan, and Mirex. The greater variety of pesticides present at Location 2 may be a consequence of this location's proximity to currently active fields.

The artifacts used in the Jaketown analysis were pieces of daub, that while fired, are not to the level of hardness of ceramics. These were subsurface artifacts that were from below the base of the plowzone and would have been in more of an anaerobic depositional environment. The artifact from sample 1, Location 1

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contained only Mirex, while the artifact from Sample 2, Location 1 was devoid of pesticides. The daub from sample Location 2 contained p,p’-DDT and Mirex at levels that are higher than those found in the accompanying soil sample.

HOLLYWOOD SITE (22-TU-500)

Analysis of Soil Samples From Hollywood and Artifacts From Jaketown and Hollywood Sites

Archaeological testing of the Hollywood site included a soils analysis that was completed by the Soil Conservation Service. Their analysis suggested that the surface of the area within the site had been intentionally filled by the developers of the mound complex. Plowing and other historic activities have removed the ramp leading to the top of the large mound, and the area has been farmed for a number of years. The acquisition by the state has left the site out of agriculture for only a few years.

As may be seen from Figure 11, there are some differences between the distribution of pesticide residues from other sample analyses that have been presented previously. The first area sampled was near the end of an archaeological test trench and there was surface evidence that a historic structure had been on or near the crest of the adjacent mound. The plowzone was clearly evident at this location and both prehistoric and historic cultural material were present. These did not extend below the depth of the plowzone. The pesticide analysis of the top of the core from this location was void of any evidence of organochlorides and PCB’s. The base of the core yielded both DDT and three PCB’s. The lack of
any pesticide retention in the upper portion of the sample may be the consequence of increased aerobic levels in the soil.

Sample Location 2, which is reported to be in the alignment of the ramp, yielded DDT and two of its metabolites at the upper level, while the lower portion of the core was reported to be clean. This suggests that the downward translocation of the pesticides was reduced for some reason. There were no indications in the soil profile of an impermeable strata that would have prevented downward percolation. The pesticide levels in the upper portion of the core are not particularly high, however, and another explanation may be appropriate. The ground surface to the south of Sample Location 2 is noticeably lower and surface runoff may have moved deposited pesticides away from this Location before they could be absorbed in appreciable quantities. A test core was recovered from the base of the plowzone, but was not included in this analysis.

Figure 11 also includes the analysis of the artifacts that...
were recovered from the two sampling locations at Hollywood. There are several differences in the analytical results when the core and artifact samples are compared. The metabolites of DDT in the daub samples represent different points in the breakdown process from both soil sample locations, and the number of PCB-like materials is not as great. Mirex is not present in either daub sample even though it is present in the top of sample Location 2.

CASA GRANDE NATIONAL MONUMENT

Analysis Soil Samples Artifacts From Casa Grande

Five soil samples, one sherd and a piece of adobe from the Great House were collected for analytical purposes (Figure 12). Sample 1 was collected at the southwest corner of the Monument adjacent to the boundary fence. To the west of this location is a modern canal and an access road. Immediately to the southwest and west of the test loci are active cotton fields. This sample, like the other three that were submitted for analysis contained a surprisingly small suite of pesticide residues. The top of the core yielded only para, para – DDE (19.0 ppb), while analytes at the bottom of the core were para, para – DDE (89.9 ppb) and o,p’ – DDE (6.8 ppb). Sample Location 2 was near the northeastern corner on the Monument property, with cotton fields to the north and east. The upper portion of the core yielded p,p’ – DDE (113.7 ppb), while the basal sample was devoid of any organochloride residues. Sample Location 4 was recovered from near the northwest corner of the Great House. As was the case At Locations 1 and 2, pesticide
residues were unexpectedly low, with only the upper portion containing 15.0 ppb of the metabolite p,p’-DDE. This was the sampling loci that was near the point of the source of the piece of Great House wall plaster that was submitted for analysis. This material contained o,p’- DDT (51.4 ppb) and p,p’- DDE (71.8 ppb), as well as the PCB’s c-28 (91.9 ppb) and c-180 (8.7 ppb). The archaeologist A. Bandolier completed excavations in the midden to the south of the Monument headquarters building, with sherds from that work being left in a disposal pile. These artifacts have lain on the surface during the entire period of modern cotton cultivation and appeared to be an excellent source for comparative material. A single sherd was collected and submitted for analysis. It was void of both PCB’s and organochlorides. The final sample submitted for analysis was a soil sample collected from the Grewe Site to the north of the Monument. This area is within the right-of-way of a highway overpass that is scheduled for immediate construction. Until recently, the area was in active cotton fields. Analysis of the sample yielded only p,p’-DDE (52.5 ppb) in the upper portion of the core.

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Figure 12. Analytical Values From Soil Samples and Artifacts Recovered From Casa Grande National Monument

33
Analysis of Soil Samples From The Dedic Site

At the time that the present samples were collected, the tobacco crop had been harvested, the field had been plowed, and the barns located on the site was filled with hanging tobacco. A portion of one of the sand dunes is still present to the north of the Barns 7 and 8 and is separated from these structures by a field road. Soil on the site is very sandy with the upper portion of the deposit being composed of reworked eolian materials which overlie the deeper lacustrine sands that were deposited in the lake. The dune is stable, and is covered with a growth of grasses and weeds. Five soil samples were collected from the general vicinity of the two barns. The first was taken to the north of the northeastern corner of Barn 7. This test was placed at the toe of the dune at the edge of the field road, and its purpose was to provide visible stratigraphic control for succeeding samples. This sample was not included in those subsequently submitted for pesticide analysis. The four remaining coring Locations were sampled for inclusion in the analytical process. Location 2 was outside of the actively plowed portion of the field in a grassed area that was immediately to the north of the field road, near the northeastern corner of the field, and north of the northeastern corner of Barn 8. Location 3 was placed 170’ due south of Location 2 in a shallow swale. Location 4 was on a slight rise on the same north-south axis and was 170’ to the south of Location 3. Location 5 was placed inside of Barn 8, 16’ from the western end of the
structure. The floor inside of the structure was dirt, and was the same soil that was in the surrounding field.

The Dedic Site was purchased as a part of Sanderson Farms, Inc. in 1984, and the history of pesticide use for the last 12 years was familiar to the property owner. He indicated to us that the primary pesticides that had been used were carbyl (Sevin), asephate (Orthene), and endosulfan (Thiodan). The first of these compounds is included in the suite of pesticides that can be routinely identified in the current analytical procedure, while the other two are not. None of the three compounds were present in the analyzed samples.

The analysis of the samples (Figure 13) from the four Locations indicated there were no residual pesticides in the lower portions of any of the cores. Because of the sandy nature of the field, irrigation is a necessary part of crop production and it is possible that any organochloride residues have been leached or washed to the base of the lake bed. The recovered cores only reached a depth of 2.2' below the surface, and if the suggestion is accurate, heavier concentrations of pesticides may well be present as much as 20' below the surface. The owner of the farm had

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Figure 13. Analytical Values From Soil Samples Recovered From the Dedic Site at Sample Locations 2 - 5
indicated to us that the sand deposit reaches a depth of approximately 20' in the area of the Dedic Site.

Core Location 2 yielded o,p’-DDT (57.2 ppb), p,p’-DDE (128.6 ppb), Heptachlor Ep (6.2 ppb) and Mirex (5.5 ppb). Location 3 yielded a slightly higher concentration of o,p’-DDT (63.5 ppb), but a lower concentration of p,p’-DDE (98.0 ppb). Mirex and Heptachlor Ep were absent at Location 3. Intuitively we suspected that the levels of pesticides present at Location 4 might be higher since it was placed in the lowest point of a swale. Again Mirex was absent, but Heptachlor Ep was present (11.1 ppb). The analytical level of o,p’-DDT was only slightly higher (62.8 ppb) while the level of p,p’-DDE was higher than at Location 3 at 121.4 ppb, but lower than the level at Location 2. Location 5, which was inside of the barn produced Mirex (5.2 ppb) and o,p’-DDT (61.8 ppb) and p,p’-DDE (66.1 ppb). Some archaeological excavations have been completed within the barn and we were careful to try to select an area to be sampled that had not been disturbed in this manner.

CONCLUSIONS And RECOMMENDATIONS

It is our impression that this study represents one of the first attempts to evaluate the relationships that exist between artifacts their cultural material bearing matrix and introduced man-made chemicals. The original intent of this study was specifically directed toward the quantification of pesticides and herbicides, and relate the levels of these materials to the potential for in situ site conservation through revegetation. Cost
considerations reduced the analysis to pesticides and PCB’s. The numeric results of the various analyses presented above clearly indicate that the residual levels of pesticides are low enough to no longer be toxic, either to humans or to the organisms which aid in soil development. This is a critical element since bioturbation and the reduction of organic materials are major contributors to the development of the A-O and A-1 soil horizons which are necessary for vegetation growth. Similarly, organochloride based pesticide residues are not present at levels that would be toxic to field archaeologists or laboratory personnel. The demonstrated presence of these materials, particularly those that have not been actively used for a decade or more, may require that these materials be taken into consideration as the analysis of archaeologically recovered materials becomes more sophisticated.

As noted, the gas chromatograph detection of chemical compounds is based on a time analysis and the identification of specific compounds is dependent on the library within the data station that accompanies the machine. The samples that were recovered from the Albany Mound complex and the excavated materials from that site contained a broad spectrum of organochlorides and compounds that were initially identified as PCB’s. A simple explanation for the presence of PCB’s in modern soil samples could easily attribute their presence to aerial transport of these materials from the manufacturing areas of Chicago and Detroit. In the context of this analysis, modern artifacts and soil samples and those collected in 1907 from the Mound excavations contained
essentially the same suite of PCB’s, thus bringing this simplistic explanation into question. Simply stated, organochloride based pesticides and PCB’s are exclusively man-made compounds that were not in use in 1907.

A positive identification of these compounds as PCB’s was necessary. Ohmicron Environmental Diagnostics, a developer and manufacturer of chemical test kits, recently made available a PCB identification procedure marketed as PCB RaPID Assay. GC samples assayed for the initial analysis were subsequently tested for specific PCB identification. Use of this test procedure clearly demonstrated that the materials originally identified as PCB’s were not PCB’s. The correct interpretation therefore is that the compounds that were time-identified as PCB’s are organochloride based compounds that are not included in the library of our GC. In all likelihood, these compounds are metabolites of complex organochloride formulations that have resulted from the process of compound decay. As the decay process for some compounds proceeds, identification of the component parts becomes increasingly complex. For example, the pesticide Atrazine, which is not included in our library, decays into 11 component compounds which may have the same GC time signature as one of the PCB’s.

The precise source of the unidentified organochloride (or metabolites) in the materials from Albany Mounds remains to be determined. It appears safe to assume that the materials held by the Putnam Museum were exposed to these compounds after they were placed in storage, especially since organochloride pesticides were
not introduced to the vermin control market until well after
curation was initiated. Since there are no useful records
regarding Putnam's pesticide control program, it seems reasonable
to assume that a broad spectrum of chemicals was used over the
years. Introduction of these materials would likely have been the
consequence of random pesticide applications with the compounds
penetrating the paper sacks and cloth bags that were used as
storage containers.

Sherds from the Moccasin Bend collection presented a similar
set of compounds that were identified as PCB’s by the GC, but were
not identified as such by the PCB test kit. Several sources for
these contaminants can be proposed: (1) they could have been
introduced to the Moccasin Bend Site and artifacts from the
industrial district of Chattanooga; (2) they are metabolites of
agricultural pesticides that were applied to local crops grown on
the site; or (3) they were introduced into the artifacts after
they were placed in the Museum at the University of Tennessee.

Artifact curation at the University of Tennessee has been
ongoing since 1961, and successive sprayings could have introduced
contaminants, even in the relatively closed bags and boxes, and
metabolization of the compounds detected in the sherds occurred
over nearly two decades. The 1964 Moccasin Bend collections were
accessioned into the McClung Museum and stored in paper bags in
museum quality boxes with lids. The Museum has been included in
the University’s pest control program since 1961, with treatment
for pests completed semi-annually. The unidentified metabolites
could have been introduced into the collections as a result of the regular treatment program. Another possibility is that pesticides were introduced into the building and then into the collections through the air exchange system, with the pesticides actually being applied around the exterior of the building. The relatively deep vertical provenience of these sherd and the low compound concentrations suggest that these are likely metabolites of an organochloride based pesticide rather than the pesticide proper. Translocation of the pesticide to the depths of the lowest sample likely occurred over a long period of time, with the compound breaking down progressively through time.

Unfortunately, the area excavated by Graham is now inundated and could not be sampled as a part of this study. Soil sample taken at locations well removed both horizontally and vertically from Graham’s excavations, Locations 1 and 3, contained 4 unidentified metabolites each. There is not a 1:1 correspondence between the two soil samples or between either of the soil samples and the analyzed sherds.

Mirex was found in the materials from the Jaketown Site, the Hollywood Site, and the Dedic Site. This compound, as noted earlier, was widely used in the southern United States in an attempt to control the spread of fire ants. It appears to have never been used to control pests on growing crops. Its presence in the Dedic Site soil samples is a somewhat anomalous occurrence for the Connecticut River Valley, which is well removed from the distribution of fire ants.
In addition to furthering our basic knowledge of contemporary effects on archaeological materials, data collected as a part of the present study can be used to developed that will serve to further the protection of archaeological sites that are being looted. Protection can come through a carefully programmed chemical fingerprinting of any damaged site, particularly those that have a long history of continued looting. Site specific chemical fingerprints can serve as evidence to support prosecutions of site looting. Forensic investigations are intended to reconstruct past criminal activities that are based on real or physical evidence which is defined as being circumstantial or indirect (Hyland 1996:3). Swanson (1992) indicated that circumstantial evidence is used to infer the existence and particulars of an unknown fact from known facts. The unknown facts are the criminal activities, and the known facts are physical evidence of the activity. From the prosecution perspective, the fingerprint can serve as forensic evidence that will be site specific.

The use of qualitative-quantitative studies of sites and artifacts can be used to develop very strong circumstantial evidence that could be used to relate either artifacts or soil to their point of deposition. At this juncture, two scenarios can be proposed for the use of quantitative chemistry as a means of developing site fingerprints. Analytically, both approaches would be essentially the same as those used for this study.

The first scenario would require that a site be gridded on
predetermined dimensions, and both soil and artifact samples chemically defined. Clearly, the smaller the grid pattern, the more discerning the analytical definition. In this approach, identified chemicals would be those that are deposited as a result of anthropogenic activities such as farming as well as those that might be present as a result of natural movement.

Some of the organic compounds appear to move through the earth’s atmosphere from relatively warm climates and are condensed and then deposited at colder, higher latitudes. The point of deposition can be vegetation, soil, or in bodies of water. This global distillation process appears to be most pronounced for organochlorine compounds, frequently used as pesticides, and are of intermediate volatility (Simonich and Hites 1995:1851). The direct consequence is that some organochlorides or their metabolites may be present in archaeological sites well removed from the point of use of the chemical compound. Simonich and Hites (1995) have quantified the presence of these compounds in tree bark from such unlikely locations as the southern portion of the Alaskan peninsula and north central Russia. The value of the identification of any of the organochlorides in archaeological sites as forensic evidence is that these are exclusively man-made compounds that have no natural occurrence. The determination of a carefully defined site fingerprint could then be used to place physical evidence near its point of origin.

The second scenario would involve the intentional development of a site specific fingerprint through the introduction of a marker
compound or compounds. Ideally, the marker should be inert, nontoxic, harmless to the environment, and have a relatively long half-life. The medium used to introduce the marker into the cultural deposit should have essentially the same characteristics, and neither the marker nor the carrier should have an adverse effect on artifacts or the culture bearing matrix. Similarly, neither should have the capacity to alter the background chemistry of the cultural deposit. The marker compound would be introduced at known and nontoxic levels, and its presence subsequently quantified at various stratigraphic levels within the area that was treated. Periodic analyses would be required to maintain an accurate forensic base. As an example of how this scenario might work, the PCB coniger c-28 might be selected. It meets the criteria established above, and like other PCB’s is not water soluble, and would not be carried off by rainfall. PCB’s are manmade compounds that have a relatively short history of broad use, and their presence can be easily detected. The introduction of c-28 into an archaeological deposit might be accomplished through the use of a 10% methanol - 90% water mixture, with the methanol serving as the means to carry the coniger into solution. Both the water and the methanol would evaporate, and as may be seen from the analytical data presented earlier, c-28 is readily held in the soil as well as in porous artifacts such as prehistoric ceramics.

As a final step in the development of the forensic base, it appears appropriate that any site/s so treated be clearly marked. Such signs would not be intended as a fright tactic to warn looters.
away although this purpose might equally be served. It would serve as a simple warning to anyone intending to desecrate a resource that a more sophisticated technique is now available as a policing agent.

There is little doubt that the application of a fingerprinting program would vary through various physiographic and climatic zones. Testing and site specific determinations would be necessary, although the basic approach would be the same regardless of the location. Since archaeological sites tend to be small vis-a-vis their climatic definition, additional parameter could be established that would aid in the development of forensic evidence. Most would fall within the size range of microclimate (Stuller 1995:103) which would further serve to limit the forensic definition.
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