# SOIL CHEMICAL ANALYSIS OF ACTIVITY AREAS IN THE ARCHAEOLOGICAL SITE OF PIEDRAS NEGRAS, GUATEMALA

by

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#### ABSTRACT

## SOIL CHEMICAL ANALYSIS OF ACTIVITY AREAS IN THE ARCHAEOLOGICAL SITE OF PIEDRAS NEGRAS, GUATEMALA

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## Department of Agronomy and Horticulture Master of Science

This thesis contains three manuscripts aimed at the interpretation of activity areas and ancient human behavior through soil chemical analyses. The association of chemical signatures with ancient human activities allowed us to correlate artifacts recovered from excavation with soil chemical patterns. By doing this, we were able to determine the effectiveness of interpretations based solely on chemical signatures. We found certain chemical patterns that reflect the ancient activities performed in the Classic Maya period. We compared phosphate concentration with ceramic data obtained from 37 test pits excavated in areas of varying phosphate concentration in three residential sites. This allowed us to determine the effectiveness of chemical analysis as a predictor of ancient refuse disposal patterns. We also used principal component analysis to compare trace metal patterns from a strip excavated floor to determine the possible interpretive capability of chemical analysis. This study also provided an opportunity to compare total

and extractable methods of phosphate and trace metal analysis, and to determine which method most accurately predicted ancient activity areas. Finally, soil chemical analysis revealed that chemical patterns between known activity areas (from a modern Kekchi Maya household at Las Pozas, Guatemala) are related to current documented activities. These same patterns also compared with chemical patterns from a rapidly abandoned site (Cerén, El Salvador) and a slowly abandoned site (Piedras Negras, Guatemala). Cluster analysis was used to compare the chemical patterns of the three sites. We found a positive correlation (r = 0.72) between phosphate concentration and ceramic density. This correlation indicates that there is significant potential of using chemical signatures to define and orient excavations of residential areas. At the strip excavated site, we were able to correctly identify areas of food preparation as well as craft production. We also determined that extractable DTPA metal analysis and Mehlich P determination methods were preferable to total analyses due the detail of resolution provided by these methods in suggesting areas of ancient activity. We found a strong relationship between modern activities and chemical signatures, particularly phosphate associated with food preparation, consumption, and disposal. Some chemical signatures found in the ethnographic case were not applicable to the ancient sites, due to contamination from modern activities as well as loss of the chemical signature over a short period of time. The vigorous application of these methods in archaeological excavations can strongly enhance the interpretive capabilities of researchers and help guide excavation strategies.

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The Piedras Negras Project takes place within a regional concession graciously granted by the Instituto de Antropología e Historia (IDAEH). The Consejo Nacional de Áreas Protegidas (CONAP) and officials of the Parque Nacional del Lacandón permitted work within the park. The Piedras Negras Project is co-directed by Dr. Stephen Houston (BYU) and Lic. Héctor Escobedo (Univ. del Valle), with Dr. David Webster (Penn State) as additional senior staff. Funds for the research of this project were provided in part by grant #9974302 from the National Science Foundation.

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# THE USE OF IN-FIELD PHOSPHATE TESTING FOR THE RAPID IDENTIFICATION OF MIDDENS AT PIEDRAS NEGRAS, GUATEMALA

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ABSTRACT

The detection of ancient Maya sites has been promoted in recent studies through the use of soil phosphate analysis to help delimit areas of occupation based on decomposed organic matter. Organic matter is elevated around areas of ancient human habitation due to refuse associated with human activity. Theoretically, the highest concentration of organic matter-- detectable by phosphate analysis-- is directly associated with areas of refuse disposal. Thus, the objective of this study is to investigate the potential of phosphate analysis as a detection tool for residential middens in Piedras Negras, Guatemala. This paper presents the results of a sensitive, in-field phosphate analysis method applied as a midden prospection tool in residential areas. Phosphate concentration is correlated with artifact data obtained from a total of 37 test pits excavated in areas of varying phosphate concentration in three residential sites. A positive correlation between phosphate concentration and ceramic density indicates the potential of this method in defining and orienting excavations of residential areas.

#### **INTRODUCTION**

Though archaeologists working in the Maya area have always focused a great deal of their attention on the elaborate masonry architecture of the region, the study of more modest houses and households has also played a significant role in the field for much of this century (e.g., Wauchope, 1934; 1938; 1940). The archaeology and

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ethnoarchaeology of households in recent decades predominantly focuses on the house or household cluster as a set of activity areas (e.g., Bawden, 1982; Bermann, 1994; Deetz, 1982; Drennan, 1988; Manzanilla, 1987, 1996; Santley and Hirth, 1993; Robin, 1999; Smith, 1987*a*; Tringham, 1991; Wilk and Ashmore, 1988; Wilk and Rathje, 1982). Analysis of such spatial groupings is critical for understanding a wide variety of social phenomena including gender relations, loci and modes of production, social hierarchy and heterarchy, ethnicity and demography (M. Blake, 1988; S. Blake, 1988; Deal, 1988; Hammel, 1980; Hammel and Laslett, 1974; Haviland, 1985; Hayden and Cannon, 1983; 1984; Hendon, 1996; Laslett, 1969; Laslett and Wall, 1972; McAnany, 1995; Nakane, 1970; Netting, 1965, 1993; Netting, et al., 1984; Robin, 1999; Tourtellot, 1988; Webster and Gonlin, 1988; Wilk and Ashmore, 1988; Wilk, 1983, 1985, 1989, 1991; Yanagisako, 1979). This broad scope of work, however, depends largely on physical analyses of the data recovered in excavation, whether on the macroscopic or microscopic level. In order to increase the efficiency of archaeological excavations in locating areas abundant in physical artifacts, the target of this work is the detection of rich deposits of cultural material, or middens containing information on household activities. Our working hypothesis is that due to the increased organic matter that was placed in residential middens as refuse, the analysis of soil phosphorus concentrations will be useful in detecting these areas as targets for residential excavations. Using the Maya site of Piedras Negras, Guatemala as a case-study, the objective of this research was to use soil chemical patterns to locate residential middens prior to excavation.

Significance

The study of residual chemical signatures can shed important new light on many of the daily activities of the ancient Maya, including cultivation and storage of crops and preparation of food, among others. Traditional archaeological methods that attempt to locate middens based on the surface remains of artifacts and architecture remain problematic (Flannery, 1976; Killion, et al., 1989). As Manzanilla and Barba (1990) remark: "Although drawn from examinations of household structure, conclusions about surface distributions of artifacts and domestic mounds do not often rest on firm functional, contextual, social, or chronological evidence". Yet, logistical problems of time and money often prohibit the excavation needed in order to confirm the results of surface testing. Historically, archaeologists have tended to relegate soil chemistry to a passive role in the investigation of ancient cultures. Chemical analysis of soils is generally used to confirm, deny, or expand upon the results achieved through other techniques (though see Cowgill, 1961; Cowgill and Hutchinson, 1963; Popenoe, 1957). Bethell and Máté (1989) point out that archaeologists often use chemical data to support conclusions already drawn from excavation and rarely use them to develop more detailed interpretations or to refine research strategies. By *beginning* with chemical analysis as a primary step in the development of an excavation strategy, a more effective program of research can be enacted by the archaeologist (Dunning, 1993; Manzanilla and Barba, 1990). Thus, the use of soil phosphate analysis as a technique in orienting excavation strategy prior to excavation can lead to more detailed results that serve in making more fine grained cultural interpretations.

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Methodological history of phosphate analysis in archaeology

Several studies of archaeological sites demonstrate a relationship between phosphate concentrations found in the soil and areas of specific household activities (Barba, et al., 1987; Cavanagh, et al., 1988; Craddock, et al., 1986; Dunning, 1993; Killion et al., 1989; Konrad, et al., 1983; Manzanilla and Barba, 1990; Middleton and Price, 1996; Ortiz and Barba, 1993; Proudfoot, 1976; Robin, 1999; Sánchez, et al., 1996; Terry, et al., 2000; Wells, et al., 2000). Ethnographic studies, likewise, illustrate the relationship of phosphate concentration with domestic activities (Barba and Ortiz, 1992). Activities such as preparation, storage, and disposal of food and the fertilization of soil for agriculture leave distinct chemical impressions that can remain relatively unmoved in the soil for centuries (Barba and Ortiz, 1992). Soil samples collected prior to excavation can be analyzed and chemical patterns determined, mapped, and compared with these studies to give insight into spatial land use.

The application of phosphate analysis in archaeology was first developed in Europe in the 1930's, where pioneering efforts by Arrhenius (1931) and Lorch (1940) were followed by work of numerous scholars, including Dauncey (1952), Sieveking et al. (1973), and Provan (1973). Their results indicated that areas of ancient occupation contained elevated concentrations of phosphate. Through this discovery came the development of a new tool in the archaeological repertoire. The general acceptance and application of chemical methods in archaeology was relatively slow, particularly in America, until the influential work of Eidt (1973). Since that time, increasing numbers of researchers have focused on the relationships between the chemical signatures left in the soil and ancient, as well as modern, activities (e.g., Ball and Kelsay, 1992; Barba and Ortiz, 1992; Coultas, et al., 1993; Dunning, 1993; Jacob, 1995; Manzanilla and Barba, 1990; Wells, et al., 2000).

The most common application of soil phosphate analysis in archaeology is as a means for pre-excavation prospection to locate or delimit archaeological sites (e.g., Ball and Kelsay, 1992; Dauncey, 1952; Eidt, 1984; Hammond, 1983; Provan, 1973; Sieveking, et al., 1973). Soil chemical data provides important clues as to the activity of a given area when it has been difficult to judge from artifact data alone (e.g., Ball and Kelsay, 1992; Cavanagh, et al., 1988; Coultas, et al., 1993; Dunning, 1993; Lippi, 1988). In contrast to these studies locating general sites, this study concentrates on a more detailed approach in locating specific activity areas within a site.

The association of phosphate with human activities lies in the organic remains of food waste. When the plants are harvested and transported, the phosphate is relocated with them in the form of cellular membranes and other molecular structures. As the plants in the form of food waste or fecal materials decompose, the mineralized phosphate is readily sorbed or fixed on the surface of the soil particles where they remain for centuries (Barba and Ortiz, 1992). Agricultural practices of removing crops from outfields and disposing of food waste and night soil in gardens and fields close to the home leave an imprint of elevated soil phosphate (Eidt, 1984; McManamon, 1984; Woods, 1977). The phosphates accumulated over long periods of time at midden sites are still present in the soil and easily extractable.

The chemical analysis of phosphate has proven successful in applications to soils formed under a wide variety of geological conditions and subject to a range of erosional and depositional processes (see White, 1978). Soil phosphate testing in the Maya region is particularly promising given the highly calcareous nature of the soils derived from the karst topography of the region, as calcium serves to fix phosphate compounds in the soil for long periods. Theoretically, high relative phosphate concentrations in close association with architectural features should indicate high probability of residential middens, which are important for developing a site or a regional ceramic chronology (Gifford, 1976; Smith, 1987*b*). The results of this study will help determine the efficacy of this in-field soil phosphate test as a prospection tool for residential middens using ceramic density and artifact assemblage as indicators.

In 1997, Terry, et al. (2000) introduced to archaeology an economical field procedure for quantitatively determining phosphate concentrations that is more quantitative than Eidt's ring test method (e.g., Eidt, 1973, 1977, 1984) and more fieldcompatible than other quantitative methods (Hassan, 1981). This method permits archaeologists to achieve phosphate results in the field at low cost. The procedure consists of extracting the soluble and readily labile, or extractable, soil P and determining the concentrations quantitatively with the aid of a colorimeter. This method was found superior to in-field ring techniques because of the higher resolution and detail provided (Terry, et al., 2000). The ring test procedure is limited to an ordinal scale based on the judgement of the viewer and the concentration range of results is limited. The Mehlich II (Mehlich, 1978) extraction procedure provides objective, quantitative results highlighting subtle details that are missed with a ring test. Bethell and Máté (1989:17) point out the requirement of quantitative soil chemical methods for the further refinement of feature analysis. The analysis of multiple samples collected over an entire area can present patterns of subtle changes in elemental concentrations indicating areas of ancient soil modification due to repeated activities. This method allows for the determination of more subtle gradations in phosphate concentrations over a wide range of very low to high levels that may prove culturally significant.

#### Study Site

Piedras Negras is located on the eastern bank of the Usumacinta River in the northwestern Petén rainforest of Guatemala. Piedras Negras was initially settled around 400 BC and rose to become a significant dynastic center after about AD 450. By about AD 808, however, the site ceased to be a seat of royal authority with the capture of its last known ruler by forces from the site of Yaxchilan (Stuart, 1998). A gradually diminishing occupation of the site persisted through much of the 9<sup>th</sup> century AD. By the end of the 9<sup>th</sup> century the site had been almost entirely abandoned, though it later became a site of ritual importance to the Lacandon Maya (Satterthwaite, 1943; see also Houston, et al., 1999).

In 1997, Brigham Young University and the Universidad del Valle began a planned five-year study of this Classic Maya center. In addition to intensive excavations in and around the monumental architecture of the site, archaeological investigations have included expansive excavations in more modest, non-elite structures. Within the sitecore, these buildings tend to be organized into spatially distinct clusters of patio groups compacted between ceremonial plazas, monumental buildings, and the riverbank. These clusters each exhibit a distinctive history reflecting the complexity of the daily activities that took place there during the course of occupation. The project has focused its excavation efforts on numerous residential and ceremonial areas within the city. To date, analyses of the data recovered from these locations have yielded important insights regarding household size and composition through the excavation of human burials, production activities revealed by artifact distribution, and residential growth documented by the construction stages of domestic buildings. (Aguierre, 1998; Arredondo, 1998, Escobedo and Houston, 1997, 1998; Fitzsimmons, 1999; Golden, et al., 1999; Houston, et al., 1998, Houston, et al., 1999; Monteroso, 1998; Urquizu, 1997; Wells, 1998).

The location and topography of Piedras Negras, Guatemala, proved advantageous in this study. Because of the remote location of Piedras Negras, there has been little cultural activity since the time of the ancient Maya that would contribute to contamination of the chemical analysis results. The karst topography generated limited soil development and deposition since the time of abandonment. The soil types around the area of Piedras Negras consist mostly of Argiudolls (Fernandez, 2001). The soil surface has remained relatively unchanged since occupation in this area making, sample collection relatively easy. However, in areas of high soil development or deposition subsequent to abandonment, such as flood plain areas, this prospection procedure may not be as effective.

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#### METHODS

Data Collection and Analysis

Surface soil samples (0-10 cm below leaf litter layer) were collected from the residential house-mound groups in quadrants "O", "N", "C", and "J" within Piedras Negras (Figure 1). Grid size varied depending on the desired detail of each particular area and the size of the individual site, but did not exceed 5 m. Grids were established using a compass and measuring tape. Over four hundred soil samples collected from these groups were analyzed for extractable phosphate indicative of ancient activities. A geostatistical analysis was performed on the patterns resulting from the chemical analysis and a map was interpolated with the aid of Surfer software (Golden software, Golden, CO)

The size of the grid from which soil samples were taken depends on the detail sought. We have found that grid spacing of up to 5-m shows the significant areas of elevated phosphate, however the resolution is not as fine as a tighter grid provides (i.e. a 2-m grid). We suggest that the grid size should correspond to the density of structures, the total area of the site tested, and the location with respect to hypothesized activity.

The "C" group was most likely a non-royal elite residential cluster of housemounds located in the northern edge of Piedras Negras (Vassaux, et al., 2000). A total of 62 soil samples were collected on a 5-m grid for the purpose of testing the midden prospecting method. The grid spacing in this group was large because of the size of the group and the low density spacing of the mounds.

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The "J" group, or the Northwest Court (structures associated with J-24, -25 and -26) of the Acropolis, appears to have been a residential group, perhaps representing a preparation area for food consumed in the royal court, though the identity of its occupants remains unclear (Golden, 2000). A total of 92 samples were taken from the platforms and patios of this area using a 2-m grid.

The "O and N" residential group has been hypothesized to have had significant ties to the royal palace of Piedras Negras (Houston, et al., 1999). Due to the extensive size of this group, soil samples were collected using a 5-m grid. Over 250 soil samples were collected and analyzed from this area.

#### Chemical Analysis

The method of extractable phosphate analysis used both in the field laboratory and BYU laboratory is based on the Mehlich II extraction solution (Mehlich, 1978) and Hach reagents (Hach, Co., Loveland, CO). The complete phosphate analysis procedure is described in Terry, et al. (2000).

Two grams of sieved (<2 mm), air-dried floor or soil sample is extracted with twenty ml of the Mehlich II solution for 5 minutes. The samples are then filtered. One ml of the extract is dispensed to a vial, diluted to 10 ml, and the contents of a "PhosVer 3" powder pillow are added to the vial. The sample is shaken by hand for one minute and allowed to stand an additional 4 minutes for color development. The concentration of phosphate in the samples is determined on a battery operated Hach DR 700 colorimeter at a wavelength of 880 nm. The readings from the colorimeter are compared with a standard curve of known P concentration with corresponding transmittance levels.

#### Test Pit Analysis

Subsequent to the analysis of soil phosphate content, test pits were placed in areas of varying phosphate concentration throughout the "C" and Northwest Court groups. Phosphate concentration was plotted against ceramic density recovered from each test pit to determine effectiveness of extractable phosphate as a residential midden prospection tool. The software package SPSS company (Chicago, Illinois) was used for statistical analysis.

The comparison of ceramic density with phosphate concentration was carried out over all three test groups, "C", "J" and "O & N". The ceramic density of each of 37 test pits was calculated for correlation analysis with phosphate concentrations. The stratigraphy of the test pits was not considered, rather, due to the arbitrary excavation of test pits, the final ceramic density was combined to give one value of average ceramic density per test pit. In order to compare the relative concentration of both ceramics and phosphates from each residential group, data from each group was standardized. The data from each residential group was separately analyzed to give the mean and standard deviation corresponding to each variable of the dataset. The standardized value is the number of standard deviations from the mean. By standardizing both the phosphate concentration and ceramic density values, we were able to statistically compare the data collected from the three sites. The standardized variables of both phosphate

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concentration and ceramic density were analyzed using simple linear regression to determine correlation.

It is obvious that ceramic density alone is not an indication of a residential midden, however, there is a generally accepted relationship between the two where the artifact assemblage indicates domestic use (Golden, 2000; Killion, et al., 1989; Robin, 1999). In most cases in this study, the ceramic assemblage includes a full range of ceramics such as serving vessels, water jars, basins that indicate household use. Expended metates were also present, as well floral and faunal remains and lithic debitage, finished tools and cores. There are several advantages to using the ceramic density for statistical correlation; first, ceramics tend to persist in the soil for long periods, whereas bones and other residential refuse may decompose; and second, ceramic density (kg/m<sup>3</sup>) is quantifiable on a continuous scale that is able to indicate small variations that can be correlated with phosphate concentration.

Following the phosphate analysis of the soil samples collected from the each residential group, the spatial pattern of the concentration was analyzed. Areas of high phosphate levels, indicative of long-term disposal of kitchen waste, were easily discernable. Such areas were then selected for placement of test pits. As a control, test pits were also placed in areas of low and moderate phosphate concentrations. This allowed us to obtain a background or reference with which we could compare the highest concentrations.

#### RESULTS

Sector C residential group

The highest concentrations of soil phosphate in this group were localized around the structures in the southeast portion of the group. The lowest concentrations were found southwest portion (Figure 2). The phosphate concentration throughout the group ranged from about 9 to 241 mg/kg P.

Test pits 2, and 4 were placed in areas of high soil phosphate concentration (241, and 120 mg/kg P). Each one of these test pits contained a high density of ceramic material (Table I). These two pits also contained bone fragments and carbonized bone, obsidian blades as well as evidence of waddle and daub. The ceramic density of the test pits in these two areas was 22.9 kg/m<sup>3</sup> and 9.9 kg/m<sup>3</sup>, respectively.

Two test pits (6 and 9) were also placed in areas containing relatively moderate levels of phosphates (41, and 116 mg/kg P). Test pit 6 revealed a very high density of ceramics (20.7 kg/m<sup>3</sup>). Interestingly, there were few bones or other materials that would be diagnostic of a domestic kitchen midden. This test pit also contained more obsidian cores than obsidian blades. Due to the location of this area with respect to the structures, we hypothesize that this area was used for storage of materials that had possibilities for other applications (Sheets, 1992; Smyth, 1990). The other test pit of intermediate phosphate concentration, test pit 9, yielded a ceramic density of 6.6 kg/m<sup>3</sup>. The two test pits (7 and 8) placed in the area of lowest phosphate concentration (<25 mg/kg P) yielded ceramic of less than 5.9 kg/m<sup>3</sup>.

Sector J (Northwest Court) residential group

The highest concentrations of soil phosphate in the northwest court group of sector J were located on the edge of a steep downslope, possibly indicating disposal of refuse over the edge (top of Figure 3). The lower concentrations were generally found on the south side of the group, the likely pathway leading to the neighborhood (bottom of Figure 3). The range of phosphate concentration here differs dramatically from group "C". Although the phosphate concentration range of the "C" group was very broad (9 mg/kg to 241 mg/kg), the range of this group seemed very narrow (from about 7 to 33 mg/kg P). The narrow range of phosphate concentration of the northwest court group, although unexpected, gave us an excellent opportunity to demonstrate the sensitivity of the phosphate method being used. The difference in phosphate concentration of the individual areas.

A total of eight test pits were excavated in this patio group near the Acropolis, corresponding to variations in phosphate. The phosphate concentration of this site ranged between 7 and 33 mg/kg P (Table II). Two test pits were placed on the northeast side of structure J-24 where relative phosphate concentration (33 mg/kg P, and 22 mg/kg P) indicated the possibility of midden locations (pits PN-46A and PN-46C, respectively). Animal bones, high concentration of ceramics, charcoal, obsidian and other artifacts were unearthed in both test pits indicating the presence of a kitchen midden (Golden, et al., 1999). This was confirmed by horizontal excavations of this patio group in the 2000 field season.

Six test pits were placed in areas moving from low to higher concentrations of phosphates (pits PN-46B and PN-46D ranging from 12 to 21 mg/kg P). One of the test pits, PN 46B-1 provided an exceptionally high density of ceramics (20 kg/m<sup>3</sup>) where phosphate concentration was relatively moderate (12 mg/kg P). Interestingly, except for excavations conducted as PN-46C, all other midden materials uncovered represented a relatively thin deposit of, on average, less than 50 cm overlying construction fill.

The highest phosphate concentrations do not necessarily appear in the areas of highest artifact density, but in areas where the midden is not covered by overlying collapse. Since soil samples were collected from the humus layer below the leaf litter, stucco and other material from post-occupational collapse would mask or dilute phosphate signatures. But it is important to note that they do not entirely eclipse these signatures.

#### Sector O & N residential group

The "O and N" residential group was by far the largest group analyzed for this study. The soil phosphate concentration of samples from this group ranged from 9 mg/kg P to 240 mg/kg P. The highest concentrations were found near the southeast corner of structure N-4 (NW group of structures, Figure 4). The lowest concentrations of phosphate were detected in the northeast corner of the study area, the platform O-23 on the University of Pennsylvania map (NE corner of Figure 4). This area was most likely swept clean of organic debris as it served as an entrance into the residential group. Test pits in the "O & N" residential group were placed in areas of varying soil phosphate concentrations. Of the 23 test pits excavated, eight (Table III) were located in areas of high relative phosphate (>100 mg/kg P), five were placed in areas of intermediate concentration (60-100 mg/kg P), and ten were placed in areas of low relative phosphate concentration (<60 mg/kg P). The test pits were well scattered throughout the group providing a good representation of activity over the entire area. The ceramic density in this group was much lower than that of the other two groups analyzed, most likely indicating the different functions of each group. However, the areas of highest phosphate concentration (>100 mg/kg P) yielded the highest ceramic density (average 4.7 kg/m<sup>3</sup>, n=8). The test pits in areas of moderate and low phosphate concentration (<100 mg/kg) contained relatively low ceramic density (average 1.1 kg/m<sup>3</sup>, n=15). The high amount of animal bone found in the three pits of highest phosphate concentration indicates the disposal of kitchen waste in these areas (PN 24A-9, PN 10F-2, PN 10F-1).

Every area with a high phosphate peak that was excavated in this study contained high relative ceramic density. There were several areas where the phosphate concentration was low and ceramic density was high. This pattern would indicate several different types of disposal. Kitchen middens, where organic waste was discarded show high phosphate and high ceramic density. The other type of residential midden encountered had high ceramic concentration but negligible evidence of organic debris.

#### Statistical Analysis

Following the analysis of the chemical data and the calculation of ceramic density

from each test pit, the variables were standardized by site. The different ranges of phosphate and ceramic density due to factors such as length of habitation, group function, and archaeological excavation techniques made unstandardized comparison impractical. By comparing standardized values, we were able to minimize the differences between the residential groups and focus on the relationship of soil phosphate to ceramic density.

Statistical analysis of the standardized ceramic data and phosphorus test results indicated a positive correlation between phosphate concentration and ceramic density from test pits of the three groups (Figure 5). There is a strong association between phosphate concentration and ceramic density. The r value is 0.72 with significance of p< 0.05. Extractable soil phosphate accounted for 51 % of the variation in the ceramic density. The positive correlation between the two shows that there is phosphate can be used as an effective prospecting tool to locate kitchen middens prior to excavation.

#### CONCLUSIONS

Each site investigated in this research was sampled because there was little superficial information indicating the history of these residential groups. In each case, logistical considerations, most pressingly the limited duration of the field season, meant that these areas could not be excavated extensively. With the in-field phosphate test, it was possible to locate important features such as kitchen middens prior to excavation. Such features can provide an important data set for developing an initial understanding both of the construction chronology and occupational use-life of a structure or group of structures, and thus help to structure excavations in subsequent seasons. Total in-field analysis time, from sample collection to computer generated concentration maps was as little as two days (mostly for soil sample drying). We were able to analyze up to 80 samples per day. The positive correlation between phosphate concentration and ceramic density demonstrates the value of this method in kitchen midden prospection. Because of the ease and rapidity of obtaining positive results, the implementation of phosphate prospecting in the residential areas of archaeological sites provides a valuable tool in the development of an excavation plan.

Given soil developmental conditions that include low to moderate soil development and low deposition, in-field soil phosphate testing is an effective prospecting tool for kitchen middens. Some of the test pits yielded much higher ceramic concentration than explained by the phosphate test. These test pits, however, tended to be void of other artifacts that would indicate a refuse midden such as bone fragments, carbonized materials, or waddle and daub. We suggest that these pits may not represent kitchen middens as such, but rather some other type of midden in which organic remains were not heavily deposited. In every other case, the areas of highest phosphate concentration yielded high ceramic density as well as bone fragments, charcoal, shells and other artifacts indicative of a kitchen-refuse midden. Artifacts from these pits are presently being analyzed. There is a statistically significant correlation between areas of high phosphate levels and the ceramic remains that are indicative of domestic refuse. This research also indicates that different kinds of middens exhibit distinct chemical signatures that may correlate with their contents, and thus with ancient behaviors. Future research, therefore, will focus on those deposits that yield high artifact density

with moderate phosphate concentrations. In so doing, it is anticipated that phosphate analysis of archaeo-anthrosols will provide a pro-active technique for interpreting the archaeological record in conjunction with more traditional mechanical methods of analysis.

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The PNP is co-directed by Dr. Stephen Houston (BYU) and Lic. Héctor Escobedo (Univ. del Valle), with Dr. David Webster (Penn State) as additional senior staff. Alejandro Guillot supervised excavations in the C group. Monica Pellecer and Fabiola Quiroa helped with the excavations in the Northwest group (Op 46). The O and N group excavations were supervised by Isabel Aguierre, Ernesto Arredondo, James Fitzsimmons, and Monica Urquizu. Alejandro Guillot and Fabiola Quiroa performed ceramic analysis and lab work with help from Rene Muñoz.

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Pit Designation	Soil P Concentration	Ceramic Density
	mg/kg	kg/m <sup>3</sup>
1	241	22.9
2	120	9.9
6	116	20.7
9	41	6.6
7	21	5.9
8	9	3.4

Table I. Soil phosphate concentration and ceramic density of test pits located in Sector C (Op 41C).

located in Sector J (Op. 46).				
Pit Designation	Soil P Concentration	Ceramic Density		
	mg/kg	kg/m <sup>3</sup>		
PN 46A	33	14.3		
PN 46C	22	8.81		
PN 46D-3	21	6.8		
PN 46D-2	20	6.3		
PN 46D-1	19	11.1		
PN 46B-2	19	9.9		
PN 46B-3	15	5.1		
PN 46B-1	12	20.4		

Table II. Soil phosphate concentration and ceramic density of test pits located in Sector J (Op. 46).

Pit Designation	Soil P Concentration	Ceramic Density
	mg/kg	kg/m <sup>3</sup>
PN 24A-9	140	6.1
PN 10F-2	122	3.2
PN 10F-1	120	3.1
PN 24A-12	116	6.2
PN 24A-2	111	3.3
PN 10K-1	108	3
PN 24A-11	102	8.7
PN 10C-3	102	4
PN 24A-14	93	1.1
PN 10G-1	90	0.3
PN 10H-2	80	1.3
PN 24A-4	80	1.5
PN 10H-1	70	1.1
PN 24A-13	56	1.5
PN 24A-5	51	1.6
PN 24A-10	40	1.9
PN 10J-1	33	0.7
PN 10I-2	28	0.8
PN 24A-3	28	0.7
PN 24A-1	23	1.5
PN 10E-1	20	1
PN 10B-1	15	0.9
PN 24A-6	9	0.7

Table III. Soil phosphate concentration and ceramic density of test pits located in the Sector O/N (Ops. 10 and 24).

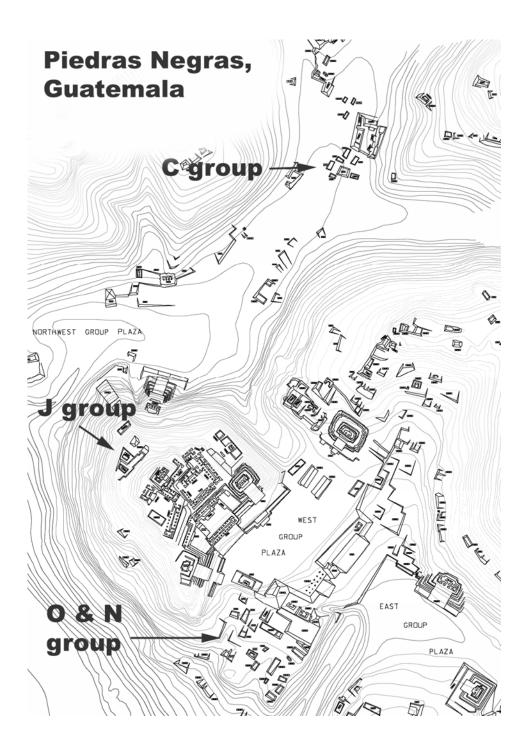


Figure 1. Residential groups "C", "J", and "O/N" involved in this study. University of Pennsylvania map of Piedras Negras, Guatemala.

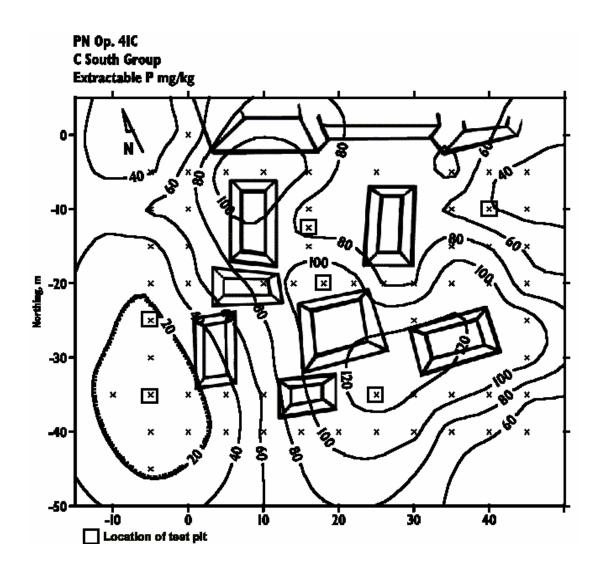


Figure 2. Map of "C" group showing location of test pits and isolines of P concentration.

Parnell, figure 3

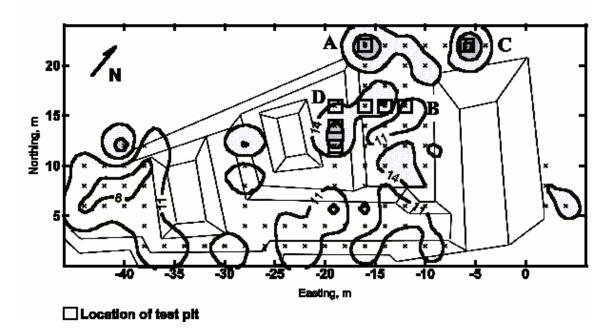


Figure 3. Map of 'J' group showing location of test pits and isolines of P concentration.

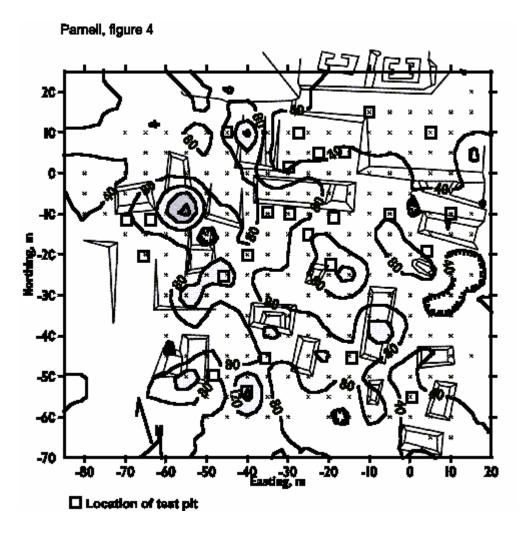


Figure 4. Map of 'O/N' group showing location of test pits and isolines of P concentration.

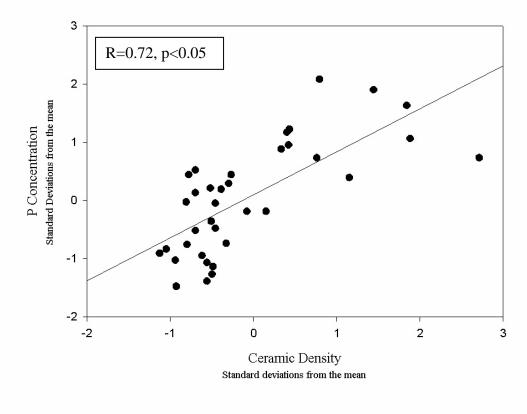


Figure 5. Linear regression comparing the standardized values of ceramic density and P concentration.

# SOIL CHEMICAL ANALYSIS APPLIED AS AN INTERPRETIVE TOOL FOR ANCIENT HUMAN ACTIVITIES IN PIEDRAS NEGRAS, GUATEMALA

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## ABSTRACT

The applicability of soil chemical analysis to the interpretation of ancient human activity

areas in the Maya region was studied for potential implications in anthropogenically modified soils. We studied chemical signatures associated with a strip excavated site in Piedras Negras, Guatemala. The focus of this study is on soil phosphate and trace metals as indicators of ancient activity. We compared total phosphate with Mehlich extractable phosphate to see which method was superior. We also compared total (nitric-perchloric) digestion with the DTPA extraction procedure of heavy metal determination to resolve which method most accurately indicates ancient activity areas. We used principal component analysis to compare the metals and give probable locations of ancient activities. Statistical analysis of chemical patterns indicated possible areas of food preparation and craft production as well as sweeping patterns. We found that elevated phosphate, barium and manganese levels indicate areas of organic refuse disposal. Mercury and lead concentrations indicate areas of craft production. These observations were supported by the artifact data. We were also able to determine areas associated with similar activities using chemical analysis. The comparison of the total and extractable element analysis demonstrated that both methods yield important information regarding activity areas. However, because of the resolution, convenience, economy, and safety, of the extractable DTPA and Mehlich procedures, we suggest these methods.

Keywords: Activity Areas, Household Archaeology, Heavy Metals, Maya, Phosphate INTRODUCTION

The interpretation of human behavior from the material remains of artifacts deposited on the landscape is the lifeblood of archaeology. Recent advances in the

chemical analysis of residues on archaeological floors and soils supply another course of research for behavioral interpretation of household groups (see Barba and Ortiz, 1992, Manzanilla and Barba, 1990, Middleton and Price, 1996). Application of such analysis to remains of households, the fundamental social unit, is considered vital for understanding various aspects of human societies and behavior (Hammel, 1980; Hammel and Laslett, 1974; Laslett, 1969; Laslett and Wall, 1972; Nakane, 1970; Netting, 1965, 1993; Netting et al., 1984; Wilk, 1983, 1989, 1991; Yanagisako, 1979). Traditionally, the distribution of artifacts characteristic of distinct activities provide a basis for interpretation of household groups and their surrounding areas of activity. However, there are several disadvantages to grounding interpretations exclusively on artifact distribution (Flannery 1976; Killion, et al 1989). Most Maya settlements were abandoned gradually, allowing inhabitants time to transport a portion of their possessions to the next residence (Cameron and Tomka 1993; Schiffer 1987; Stevenson 1982). Manzanilla and Barba (1990) comment that: "Conclusions about surface distributions of artifacts and domestic mounds do not often rest on firm functional, contextual, social, or chronological evidence". The eventual location of an artifact in an area may have little to with its original area of use because of discard or cleaning prior to abandonment. Such abandonment activities do not affect chemical residues of floors and soils. Where historical records on domestic life are not available or possibly misleading, chemical data provide clues to the organization of households, the correlation of architectural remains to social groups, the articulation of various types of activities, and the perception and organization of domestic spaces. We use a housemound group within the site of Piedras

Negras, Guatemala as a case study to investigate the potential of soil chemical analysis to the interpretation of domestic behavior. The objectives of this paper were: 1) to compare the soil chemical signatures with the artifact distribution within a large horizontally excavated household group, and 2) to compare different methods of soil phosphate and trace element analysis.

The underlying premise of soil chemical analyses is that activities performed in the same place over a long period of time leave behind distinct chemical signatures, or residues that become trapped in the soil. These chemical residues remain relatively unchanged for centuries although time and weathering destroy other avenues of research. Unlike traditional artifacts, the spatial pattern of chemicals fixed in the floor or soil remains unaffected by abandonment processes and closely reflects the use of space in the past. Soil chemical data provide important clues to the activity within a given area when interpretation has been difficult to determine from artifact data alone (e.g., Ball and Kelsay, 1992; Cavanagh et al., 1988; Coultas et al., 1993; Dunning, 1993; Lippi, 1988). As a research tool, soil chemical analysis provides an additional opportunity to understanding past activities, and is a valuable interpretive tool, which will be demonstrated in this paper.

The potential validity of soil chemical analysis as an interpretive tool in archaeology lies in the ability to predict archaeologically significant artifacts based on chemical signatures. There are two ways to assess its validity: by looking from the artifacts to the soil analysis, and vice versa. By studying both approaches, we ensure that significant deposits of artifacts and significant chemically enriched areas will not be missed. Ideally, the chemical analysis will correlate highly with artifact frequencies within the same area, and will also indicate some areas that do not correlate with significant artifacts, denoting patterns of behavior that leave no physical artifacts behind. Conversely, it is possible that there are areas of high artifact concentration where chemical signatures are low, denoting the discard of artifacts with low residual chemical concentration. The other extreme would be significant artifacts that were undetected via chemical signatures, representing a failure on the part of chemical detection. Significant artifacts for archaeologists would include high concentrations of individual types of artifacts, such as ceramics and lithics, as well as burials (with any accompanying burial goods) and rare or unusual specimens possibly from storage of artifacts (Smyth, 1987).

Past studies involving soil chemical analyses have demonstrated significant interpretive potential in the study of prehistoric land-use and activity patterns (e.g., Cavanagh et al. 1988; Coultas et al. 1993; Dunning 1993; Ball and Kelsay 1992; Lippi 1998; Manzanilla and Barba 1990; Wells et al. 2000). Ethnographic studies aimed at the association of specific activities with chemical signatures demonstrate the interpretive value of chemical analysis (Barba and Ortiz 1992; Hayden and Cannon 1983; Manzanilla1996; Smyth 1990). Some of the strongest correlations of chemical residues with activities come from ethnographic studies that look at groups or individuals that have remained isolated from modern-day technological advances. The assumption involved in many of these cases is that the activities and social structure of these groups will have remained somewhat unchanged from the time of their ancestors. Chemical data from floor and soil samples collected and analyzed from modern houses is related to the ethnographic information on space use and activity patterns. These modern-day activities include gardening, waste handling, the storage and preparation of food, and sweeping that pushes organic material to the patio peripheries (Barba and Ortiz 1992; Fernandez, et al., 2000). Investigations carried out by Manzanilla and Barba (1990) establish some general chemical patterns of various ancient activities such as rest or sleeping areas, areas of cooking, meal preparation and consumption, and areas of heavy foot traffic. Once the correlation between human activities and chemical concentrations is refined through ethnographic studies, these techniques can be applied to the study of ancient settlements (Middleton and Price 1996).

Although chemical analysis is generally accepted, there are still several issues that concern archaeologists, geographers, and soil scientists pertaining to some aspects of the application of this analysis to archaeological settings. The validity of the chemical analysis of soils as a tool for archaeological interpretation in many sites depends on several issues, such as the depth at which the sample is collected, the method of analysis used, and the strength of behavioral interpretation. Deposition and soil formation can often bury the chemically enhanced occupational level. Also, there are many distinct forms of chemicals in the soil. The methods used to detect anthropogenic modifications of soil chemical levels are generally adaptations of agronomic procedures for determining available nutrients for plants. We hope this research will help established more precise methods of measuring anthropogenic chemicals.

### **Phosphorus Analysis**

The association of phosphate and areas of human activity lies in the phosphorus cycle. Soil phosphorus (P) exists in a complex equilibrium of different forms, including fixed inorganic P absorbed to aluminum, calcium, and iron compounds, soluble and labile (extractable) inorganic P, and organic P. Plants obtain their required phosphate from the soluble and labile inorganic P fraction of the soil phosphate. When the plants are harvested and transported, phosphate is carried with them in the form of cell membranes and other molecular structures. As the plants, in the form of organic garbage (fecal residues or food waste), decompose, the phosphate becomes readily sorbed or fixed on the surface of the soil particles where they remain for centuries (Barba and Ortiz, 1992). This process depletes the outfield areas where plants utilized soil phosphate while enriching the soil phosphate concentration of the areas of compost, consumption, and deposition in areas of occupation. This principle indicates that areas of accumulation of organic waste, such as food disposal, preparation, and storage areas would present elevated concentrations of phosphate while areas of intensive agriculture which did not accumulate phosphate of decomposing plants or remains would have lower concentrations when compared with background levels (Eidt 1984; McManamon 1984; Woods 1977). These differences in the chemical concentration can be analyzed, mapped, and interpreted according to their spatial pattern. (Reviews and methods of soil phosphate analysis in archaeology can be found in Bethell and Máté 1978, Eidt 1984, Parnell, et al., n.d., Terry, et al., 2000, and Wells, et al., 2000.)

The application of soil phosphate analyses in archaeology can be divided into four general areas of study. First, soil phosphate levels are determined in pre-excavation

prospection to locate or delimit archaeological sites before excavation, which has been the most common use of phosphate analysis in archaeology (e.g., Ball and Kelsay, 1992; Dauncey, 1952; Eidt, 1984; 1973; Hammond, 1983). Second, researchers use phosphate analysis as a prospection tool to locate kitchen middens and aid in the orientation of excavations (Parnell, et al. n.d.). Third, phosphate analysis is used as a tool in landscape archaeology to examine past agricultural practice (Coultas et al., 1993; Dunning, 1993; Dunning et al., 1997, 1998). The fourth area is the delineation of features and activity areas through phosphate analysis during and after excavation (Manzanilla and Barba, 1990).

The chemical analysis of phosphate has proven successful in applications to soils formed under a wide variety of geological conditions and subject to a range of erosional and depositional processes. Soil phosphate testing in the Maya region is particularly promising given the highly calcareous nature of the soils derived from the karst topography of the region, as calcium serves to fix phosphate compounds in the soil for long periods.

### Metal Analysis

Methods for analysis of other activity-diagnostic chemicals are providing other clues to ancient space use patterns. Particular interest in metal analysis has grown within the last decade, focusing on copper (Cu), iron (Fe), mercury (Hg), manganese (Mn), lead (Pb), and zinc (Zn). Trace metals demonstrate interpretable results in soils associated with ancient settlements (Blintiff, et al., 1990; Entwistle, et al., 1998; Lewis, et al., 1993; Lambert, et al., 1984). Metals are readily adsorbed or precipitated on the mineral surfaces of calcareous soils and stuccos commonly found at Maya archaeological sites. Metallic ions remain stable in alkaline soils for long periods in the form of adsorbed and complexed ions on clay surfaces, and as insoluble oxides, sulfides, or carbonates (Lindsay, 1979; see also Wells, et al., 2000).

The use of trace metal analysis in Mesoamerican archaeology has been quite limited, yet this analysis represents a significant tool for archaeologists. Activities of the ancient Maya involved the use of a variety of metallic substances. High iron (Fe) concentration in soils has been found in areas associated with ancient agave processing or animal butchering as well as kitchen areas (Manzanilla 1996). Hematite (iron oxide,  $Fe_2O_3$ ), and iron ochre (hydrated ferric oxide,  $Fe_2O_3 \bullet H_2O$ ) were used in pigments. Cinnabar (mercuric sulfide, HgS) is a bright red mineral often used by the Maya as decorative paint or dye for ceremonial purposes, such as burials or caches. Additional minerals used as pigments included pyrolusite (manganese dioxide, MnO<sub>2</sub>) for blacks, malachite (copper carbonate,  $CuCO_3 \bullet Cu(OH)_2$ ) for greens, and azurite (copper carbonate, 2CuCO<sub>3</sub>•Cu(OH)<sub>2</sub>) for blues (see Goffer, 1980:167-173 for a review of pigment chemistry; see also Vásquez and Velázquez, 1996a, 1996b for examples). Thus, trace metal analysis of soils in and around residential and ceremonial architecture will prove useful in identifying the chemical signatures of pigment processing and other activities. Of particular interest to the research in this study, heavy metals analysis of soils close to residential architecture has proven useful for Wells et al. (2000) in identifying possible signatures of paint residues from polychromatic facades of ancient

Maya dwellings that may have served to publicly mark local identities such as status, rank, or lineage affiliation.

Researchers have used a variety of extractions and total elemental analysis procedures to understand archaeological site formation. Bintliff et al. (1990) analyzed trace metal accumulation in soils near ancient Greek settlements and concluded that trace metals accumulate at very significant excess levels on and around ancient sites, and that these patterns can be useful for archaeological prospecting and site survey, as well as for reconstructions of land-use histories. They caution however, that many closely-spaced soil samples need to be taken in order to evaluate the usefulness of trace metal assays for past land-use intensity. Lewis et al. (1993) and Scudder et al. (1996) used a dilute acid extraction of trace elements from soils of Native American sites in the Eastern U.S. and from ancient Roman sites in Italy and Tunisia. Middleton and Price (1996) also used a dilute acid extraction procedure in analysis of archaeological floors in Mexico. Entwistle et al. (1998) performed total elemental analysis of anthrosols from Scottish historical sites by digesting soil samples in nitric and perchloric acid. Lambert et al. (1984) used a chelate extraction procedure to determine trace metal concentrations in soils of the New River flood plain of Belize. All of these procedures yielded significant interpretations about the past via trace metal analysis. There are currently many procedures involving the analysis of metals in archaeological soils that potentially look at different fractions of the total metal makeup of soils.

Total analysis procedures, such as nitric-perchloric digestion measure the chemicals naturally found in the mineral forms within the soil particles. However, Middleton and Price (1996) indicate that the high concentrations of metals from a digested sample overwhelm the comparably minute concentration of anthropogenic metals. The total analysis measures the total chemical concentration, including chemicals that are naturally fixed within the lattice of clays, whereas anthropogenic chemicals would most likely be found adsorbed to the surfaces of soil particles. They argue that the extractable fraction of soil chemicals would theoretically give us better interpretive capabilities. Chelate extraction procedures are effective for calcareous soils found in the study area because they don't react with the calcium carbonate in the parent material. Methods using an acid extractant are problematic because dilute acids are neutralized by the calcium carbonate and strong acids dissolve the calcium carbonate freeing metals that are naturally occluded in the soil particles (Lindsay and Norvell 1978). The favorable solubility constants of DTPA over other chelating agents for the elements of interest in this study (Lindsay and Norvell 1978) as well as the behavior of the extractant in the calcareous soils of the Petén suggested this method as most favorable. In areas of high calcium carbonate, a chelate extraction procedure would seem most effective in extracting anthropogenic metals. This study will compare the chelate extractable metals and the total metals to artifact distribution and activity areas.

### DATA AND METHODOLOGY

Soil chemical tests are indicative of chemicals trapped within the soil matrix that may not pertain to actual artifacts unless the artifact is contributing to the chemical signature of the soil. The main possibilities or causes of chemical signatures are (1) artifacts, (2) architectural features, or (3) behavior. To study this theory, the hot spots of chemical concentration found by any given soil test are considered in order to interpret the relationship to a particular type of artifact (or a complex of artifacts in association). For each case, areas of both chemical enrichment and chemical depletion were examined to study the relationship between each kind of test and particular artifacts, architectural features, or behaviors.

The study area for chemical signatures in an archaeological setting was located in Piedras Negras, Guatemala. Piedras Negras is a large Maya site located on the Usumacinta River in Northern Guatemala. The site is in the northwestern concession of the Petén biological preserve. The area around Piedras Negras lies in a humid tropical climate with annual rainfall up to 200 cm and falls in the Afwg classification of the Köeppen system (Lee and Hayden 1988). Soils of the area are generally well drained and fertile and consist principally of Argiudolls (Fernandez, et al., 2001) that formed from the underlying limestone bedrock.

Maler in 1901 and Morley in 1937-1938 originally visited the site of Piedras Negras. Piedras Negras is known for its large temples and abundance of stela. Further, Piedras Negras is the birthplace of Maya epigraphy. Tatiana Proskouriakoff, while working with the monuments, discovered that the dates mentioned in the monuments referred to the life-spans of rulers interred within the site (1960). Through the large-scale excavations of the University of Pennsylvania during the 1930's a great deal of information is known about the large elite buildings within the site, but little about the more modest ones (Escobedo and Houston, 1997). Household excavations were limited in the past, but are an important part of ongoing excavations at the site. Recent excavations by David Webster and Amy Kovak (1998, 1999), Luis Romero (1999), and Christian Wells (1999) have contributed enormously to our understanding of ancient Maya life.

Excavations at Piedras Negras, under the direction of Christian Wells, completely uncovered several household mounds that consisted of residential buildings belonging to a possible elite/sub-elite family, based upon the high quality ceramics found within the site. Ceramic associations indicate a Late Classic date (650-800 AD) of occupation. The main structure, U-16, was excavated using a 2m grid. The building was 15 m long by 10 m wide, and had three principal rooms in its final phase. The research strategy was to completely strip' the mound, layer by layer, in an attempt to gather all the artifacts associated with local residential households within the site core. In accordance with this design, some of the material was screened through 1/4-inch mesh to discover the minute artifacts that would have been associated with daily living. Although it was impossible to excavate down to bedrock in every square, most of this mound was excavated.

The stratigraphy of this building in general consisted of three layers (Fig. 1, stratigraphy). The top layer corresponded to a humus level, comprised of organic jungle debris mixed with roots, loose stones, and terminal deposits. The next level, layer two, pertained to the floor of the building when it was in use. This layer provides a wealth of artifacts of all kinds, with ceramics, obsidian, chert, and figurines in particular. The third layer and subsequent layers comprised the ancient fill of the building, replete with ceramic sherds and intrusive burials.

Building U-16 in Late Classic times was a large three-room structure with an associated platform in front. The residents had access to wares bearing hieroglyphic inscriptions and fine imports. They used and worked obsidian and chert tools within the confines of the building. The south room and north room may have been used for food storage or perhaps small-scale craft production, while the main rooms were used for domestic purposes, including food preparation, entertaining guests, and sleeping. Burials were also discovered through excavation. The remains of seven people were found interred within the building. These people died over the life cycle of this house, perhaps within a span of two centuries. Their remains, buried under floors and benches, indicate the intricate ties, which bind an ancient Maya family together in life and death through associations of artifacts and grave preparation.

The artifacts discovered in the course of the excavations hint at the range of activities that would have been carried out within the household group. Several metates and manos were found, including a granite metate that may have been used for grinding ore into powder. A small piece of jade/jadite was discovered, which is very rare for Piedras Negras and supports the idea of an elite context. Pumice, magnetite, and pyrite show the range of imported stone, while the presence of spinning whorls suggests domestic cloth production.

Artifacts associated with U-16 are all domestic in nature and strongly suggest a local, small-scale household economy with elite ties. These artifacts are unusual within the confines of Piedras Negras for their quantity and variability. The spatial distribution of these artifacts over the site reveals patterns of waste disposal and suggests loci of

domestic action.

We are uncertain whether the chemical signatures are responsive to individual stratigraphic layers. Because the deposition at Piedras Negras is quite shallow (0-10cm), we were unable to test this hypothesis. Therefore, we lumped the artifacts from all stratigraphic layers together in this analysis and compared them to loci with a chemical concentration peak. This provided an opportunity to see the relationship between artifacts (and type of artifacts) and particular chemical signatures. Ideally, such a relationship should exist, as in the case of pottery thrown away with residual organic material inside, or knives lost or discarded with remnants of organic material. Chemical signatures could also suggest behavior that may not have any artifacts associated with it, such as patterns of sweeping, places where food is served or prepared, evidence of wall ornamentation, and other activities. Alternatively, chemical signatures could indicate the use of artifacts with respect to their location, such as whether a metate was used as a grinding tool or as part of the architecture.

### Sample Collection and Analysis

An important part of the excavation strategy was the collection of soil samples for chemical analysis. We collected a soil sample from the floor level (i.e., the old living surface) of each unit yielding a total of 91 samples. Additional samples were taken from areas of suspected activity (near benches and inside rooms). The soil samples consisted of approximately 0.25 L of soil that was air-dried, crushed using a mortar and pestle, and

sieved to 2 mm using polyester sieves and subsequently analyzed for their chemical signatures.

### *Extractable phosphate procedure*

The primary method of extractable phosphate analysis we used is based on the Mehlich II extraction solution and Hach reagents (Terry et al 2000). Two grams of airdried, sieved (<2 mm) floor or soil sample was placed in one of six 50 ml jars attached to a board that facilitated shaking and processing of multiple samples. Each soil sample was extracted with twenty ml of the Mehlich II solution for five minutes. The samples were then filtered and the filtrate collected in clean 50 ml jars. One ml of the extract was dispensed to a vial, diluted to 10 ml, and the contents of a PhosVer 3 powder pillow were added to the vial. The sample was shaken by hand for exactly one minute and allowed to stand an additional four minutes for color development. The concentration of phosphate in the samples was determined on a Hach DR 700 spectrophotometer at a wavelength of 880 nm. A control sample was analyzed with each run. The results were compared with a standard curve of known P concentration and percent transmittance. A more detailed description of the procedure can be found in Terry et al. (2000).

## Extractable trace metal analysis

We analyzed the samples for extractable trace metal concentrations using the DTPA (diethylenetriaminepentaacetic acid) extraction procedure developed by Lindsay

and Norvell (1978). Ten grams of air dried, sieved (<2mm) soil was mixed with 20 ml of 0.005 *M* DTPA solution buffered at pH 7.3 to extract the metals from the soil. The samples were shaken for two hours, after which the extracting solution was separated from the soil by centrifugation and filtration. For the present analysis, the concentrations of barium (Ba), cadmium (Cd), copper (Cu), iron (Fe), mercury (Hg), manganese (Mn), lead (Pb), and zinc (Zn) were determined simultaneously on a Thermo Jarrell Ash ICP atomic emission spectrometer. We made a standard solution for calibration of the ICP of 1 mg/kg of Ba, Cd, Cu, Hg, Pb, and Zn and 10 mg/kg of Fe and Mn diluted in DTPA extracting solution. DTPA solution was used as a blank and a control sample was analyzed with each run. Following analysis, we washed all glassware with a phosphate free detergent and rinsed with HCl (6  $\underline{M}$ ) and distilled water.

### Total trace metal analysis

We also analyzed the soil samples for total trace metals for comparison using the double acid digestion procedure (see Entwistle, et al. 1997, 1998). Soil samples were ground by mortar and pestle to pass a 60-mesh polyester sieve. Five ml of concentrated nitric acid was used to digest a 0.5 g portion of the soil sample. Because of the high calcium carbonate of the samples, nitric acid was added in two ml and then three ml aliquots to prevent an overflow of the sample from the effervescence. The sample was allowed to sit overnight in a ventilating hood and then heated in a digestion block at approximately 200°C to digest the organic material for approximately 15 minutes. Three ml of concentrated perchloric acid was added to the cooled solution. The mixture was

again heated in the digestion block at approximately  $200^{\circ}$ C for one hour. The mixture was diluted, agitated and allowed to settle prior to ICP analysis. We made a standard solution for calibration of the ICP of 1 mg/kg of Ba, Cd, Cu, Hg, Pb, and Zn and 10 mg/kg of Fe and Mn diluted in distilled water. Distilled water was used as a blank. Total phosphorus was measured colorimetrically from this extract (see Kuo, 1996). Two NIST certified control samples (numbers 2710 and 2711) were analyzed with each run to test the reproducibility of the analysis. Following analysis, we washed all glassware with a phosphate free detergent and rinsed with HCl (6 <u>M</u>) and distilled water.

The repeated analysis of the standard sample by total and extraction methods demonstrate the high reproducibility of results. Extractable DTPA copper, iron, manganese, mercury, lead and zinc ranged from 8-12 percent relative standard deviation (n = 10). Barium and cadmium, however, were extreme in variation with relative standard deviations greater than 25 percent. The relative standard deviation of barium, cadmium, iron, manganese, lead and zinc ranged from 4-10 percent in the total digestion procedure (n = 8). The relative standard deviation of copper and mercury using the total digestion method exceeded 20 percent.

### Statistical Analysis

By isolating the individual chemical signatures from the patterns of related chemicals, we may enhance the interpretive capability of chemical analysis. We used a correlation matrix to analyze the similarities between chemical concentration patterns using SPSS software (Chicago, Illinois). We standardized chemical signatures that were

significantly correlated and reduced the data with factor analysis (principal components analysis) to generate a three-dimensional map of chemical trends with surfer software (Golden, Colorado). This allowed us to simultaneously compare the chemical pattern of multiple elements. Because of the natural variations in the chemical makeup of the soil, anthropogenic chemical signatures may be hard to detect. Most naturally occurring variance in chemical patterns is theoretically reduced by principal component analysis. The new variable produced through this analysis accounts for the natural variation and co-linearity of the different variables. We saved the factored scores as a new standardized variable and plotted a map for comparison with the individual variables comprised in each component to determine anomalies in the natural variation that could be interpreted as anthropogenic. By comparing the individual standardized chemical signatures with the patterns of the related chemicals corresponding to its principal group, we were able to analyze areas of specific enrichment possibly resulting from ancient human activity. Using least squares regression, we were able to locate areas where the individual elemental concentrations were higher than expected or predicted by the pattern model produced through principal components (Fig. 2). We saved the standardized residuals from this regression analysis and plotted them to determine outliers. We compared areas of specific elemental enrichment to the artifact type and distribution, and architecture for the interpretation of archaeological activities.

## RESULTS

### *Phosphate*

65

We found high concentration peaks of extractable phosphate (Fig. 3) in the north room, central room, south room, and south patio area with highest concentrations in the north patio area. Low extractable P concentrations seem to follow the edges of the structure. Total P analysis shows a somewhat similar pattern of high phosphate in the south patio and north patio area (Fig. 4). As with extractable phosphate, we generally found low total phosphate concentrations toward the peripheries of the structure. Although both methods show similar patterns, extractable P exhibits much greater detail in this case. From the elevated concentrations of extractable P surrounding the north room of the structure, we can assume this was a possible area of food preparation. The high concentrations of extractable P associated with the benches may indicate food consumption. The pattern of elevated P levels moving from the rooms toward the patio, as indicated by both P procedures, suggests a habitual sweeping pattern from the kitchen area, culminating in two spots where refuse was dropped off the edge of the living space as kitchen middens. Low phosphate around the peripheries could indicate the presence of ancient roof drip lines that would have washed P enriched organic debris away (Fernandez, et al., 2000).

#### DTPA Trace Metals

#### Group 1

We found that cadmium (Cd), copper (Cu), manganese (Mn), and barium (Ba) data from the DTPA analysis were strongly correlated (see table 1). We used principal components analysis to reduce the data to one variable describing the similar pattern of

the four individual elements. The new variable accounted for 69.9% of the variance of the four elements. We found the highest concentrations of these elements in the north room of the structure and the north and south patio areas (Fig. 6). The area of lowest concentration of these elements was located north of the north room. The high signatures seem to be related to areas of high extractable P. The highest values were found associated with deposits where organic materials would have been continuously swept. Hence, these metals are most likely associated with the organic resources used by this household as food or as part of their craft production. We use craft production generically to suggest activities related to any kind of "craft" that would leave a chemical residue, i.e., painting ceramics, body paint, or applying pigment to ritual objects. The excavated artifacts do not suggest any special activity in this location, so we propose that the chemical analysis shows an area where paint or pigment was possibly spilled on the floor and swept out of the way. The highest concentration indicates the eventual build-up of materials associated with craft related activity. The moderate concentrations in the north room and the south patio may be a result of sweepings or less accumulation of organic debris.

The chemical patterns of DTPA copper and manganese were isolated from the chemical pattern produced by their group to indicate areas of abnormally enriched concentrations. The principal bench of the central room and in the south patio showed areas where the standardized copper residual significantly exceeded the principal component variable (Fig. 7). The central bench of the structure would have been a locus of activity of the structure. This most likely would have been the reception area for

guests and dignitaries. The south patio area was very high in artifacts and appears to be another locus of activity. This area may have been associated with workshop activities such as pigment grinding or the use of mineral substances. The area of greatest positive standardized residual of DTPA manganese, compared with related elements in group 1, was found in the north room and in the northeast corner of the structure (Fig. 8). The elevated signature of manganese seems to correspond with sweeping patterns from the north room to the edge of the north patio.

### Group 2

Iron (Fe), mercury (Hg), lead (Pb), and zinc (Zn) data obtained from the DTPA extraction procedure were strongly correlated (Table 1). Again, we used principal components analysis to reduce the data to one variable describing the similar pattern of the four individual elements. The new variable accounted for 65.2% of the variance of the four elements (Fig. 9). The area of highest concentration of these elements was most pronounced around the south patio area. Other areas of elevated chemical concentration were the south room, the central room, the north room and the edge of the north patio. The lowest chemical concentration was located to the north of the structure. The pattern of high elemental concentration appears to be a sweeping pattern perhaps dealing with craft production in the southern platform of the structure.

The chemical pattern of DTPA iron was isolated from the chemical pattern produced by its group to show areas of unusually high iron. The isopleth lines indicate

standardized residual values where the iron pattern was significantly dissimilar to the other elements of group 2. The areas where the standardized iron residual significantly exceeded the principal component pattern of the other related elements of group 2 were south of the south patio (Fig. 10). This area contains a major cultural deposit including artifacts diagnostic of possible workshop activities (such as worked bone and obsidian). There is also a region of moderate iron concentration in the north room possibly resulting from food preparation activities. The highest standardized residual value of DTPA lead, where it differed most from other related elements in its group, was in the south room and the south patio (Fig. 11). Since lead is not associated with food, rather with pigment or craft activities, this chemical pattern confirms the location of the craft production area. The large concentration area may represent a sweeping pattern related to craft production. Similar to the DTPA copper pattern, the pattern of DTPA mercury was significantly different from those elements related to it on the principal bench of the central room and in the south patio (Fig. 12). As with other metal elements, mercury is indicative of pigment or craft activity, more specifically ritual activity (Vasquez and Velasquez1996a).

## Total Trace Metals

### Group 1

The concentration data on manganese (Mn), barium (Ba), iron (Fe), and zinc (Zn) collected from the total digestion were all significantly correlated (Table 2). We used principal components to reduce the data from the four elements into one variable. The

new variable accounted for 83.2% of the variation of the four elements. We found that the highest elemental concentration, reported as a standardized value, was associated with the area north of the north patio (Fig. 14). Moderate levels were associated with the south patio area. This pattern seems similar to the high total P found in this same area indicating a relationship between these elements and organic refuse.

The chemical pattern of total iron was isolated from the chemical pattern produced by its group to indicate anthropogenically-enriched areas of iron. The isopleth lines indicate standardized values where the iron pattern was significantly dissimilar to the other elements of group 1. The areas where the standardized iron residual exceeded the principal component variable was north of the north room and the north patio (Fig. 15).

The isolated pattern of total manganese was different from those elements related to it in the south room and the south patio as well as the north bench in the central room (Fig. 16). This pattern, similar to the standardized DTPA lead residual, indicates an area that may have been used in craft production. As with DTPA copper and mercury residual patterns, the highest concentration of total zinc, compared with related elements, was found in the central room, focusing on the principal bench, and in the south patio (Fig. 17). These chemical patterns support the findings from the DTPA extraction that intense activity was associated with the central bench and workshop activities. This is an important finding because it indicates that both methods show the same relationship between the south patio and the principal bench of the central room. It also suggests that the activities of these two areas were somehow directly related.

## Group 2

Cadmium (Cd), copper (Cu), lead (Pb), and mercury (Hg) analyzed using the total digestion procedure also show patterns that are significantly correlated (Table 2). The data from these elements were reduced using principle components to produce a variable that would describe the similar patterns of the four elements. The new variable accounted for 83.1% of the variance of the four elements. The area of highest concentration of these elements was most pronounced south of the south patio (Fig. 18), where there was an accumulation of ceramics and other artifacts. These elements seem to correspond with a midden related to craft production.

The isolated pattern of standardized residuals of total copper compared with the other elements of group 2 indicated use of copper rich items. The greatest difference in these patterns was north of the north patio, in the south room, and peaked in the south patio (Fig. 19). As with the DTPA copper and total manganese patterns, this chemical signature may be associated with sweeping patterns related to craft production. The areas of highest standardized residual of total mercury, compared with related elements, was found near the principal bench of the central room and seems to follow a path out to the central patio area (Fig. 20). This pattern appears to be some type of activity involving heavy metals in the main room of the structure. As this room was swept out, there was an accumulation of the chemicals following the path of sweeping leaving the room and finally the deposition of refuse outside the patio area.

The above groups demonstrate that each separate chemical signature relates to different features of the archaeological record. Through a combination of multiple elements associated with activities, the probability increases of discovering the significant features of a site. Phosphorus has proven very reliable in the field to discern deposits of organic waste, which yield the most variety of artifacts. Heavy metal analysis also successfully detects areas of organic waste and also tends to reveal craft production, burials, and help in locating areas that were possibly painted or areas where pigments were prepared and utilized.

### Artifact distribution

In order to evaluate the relationship between cultural deposits containing valuable artifact information and chemical analyses, the areas of high counts of archaeologically recovered artifacts were compared with the gamut of chemical analyses. This comparison provided us with an opportunity to see how known loci of artifacts (from excavated units) compare with predicted loci of importance from soil chemical analysis. In this first comparison there was no attempt to understand which individual chemical analysis would 'predict' a certain kind of artifact; all are lumped together indiscriminately (Table 3).

We found deposits rich in ceramic pieces (greater than 1000 pieces) in all three rooms, in the south patio, south of the south patio, and north of the north room. With exception of the area north of the north room, these same areas were rich in obsidian blades. Chert deposits were found in the south patio, the south room, the central room, and the north room. We found deposits rich in animal bones south of the south patio, in the south patio, in the south room, in the central patio and in the central room. With exception of the central room, these same areas contained deposits rich in figurine fragments. Although manos and metates have often been used by the ancient Maya in construction or fill, the location of these were also recorded to look at any possible clues involving their use. Manos were found south of the south patio, in the south room and on the east edge of the north patio. Metates were found in the south room, in the central patio, in the central room, and the north patio. These areas of accumulation of cultural materials indicate areas of intense use or eventual disposal. Burials were found in the south room, in the central room, and in the central patio.

Based on artifact distribution results (Table 3), which take all chemical analyses performed into account, excavations oriented according to elevated chemical signatures would find all important artifacts and burials. The artifacts recovered from these areas include a large sample of both utilitarian and rare goods, as well as burials and architecture.

#### DISCUSSION

Soil chemical analyses provide archaeologists with a tool that can be used in two ways: prospection, and interpretation. Through sampling prior to excavation, chemical analysis gives archaeologists a general idea of the activity areas in the group to help orient their work. Post excavation interpretation applies these analyses to determine activity areas where artifacts are scarce or misleading, or where subtle changes in artifact distribution or content may be enhanced. Many of the details that were originally present when the artifact was deposited have been altered through weathering or other processes. Through the use of chemical analyses in the interpretation of ancient activities, archaeologists can acquire an impression of the loci of activities and the different functions of those areas.

The chemical analysis of soils in this study was very effective in locating activity areas when compared with traditional methods based on architecture and recovered artifacts. There seems to be a very strong relationship between the areas of high chemical concentration and major cultural deposits. In fact, there was only one deposit, north of the north room, that did not correspond to elevated chemical concentrations. All of the middens found during excavation were successfully predicted by phosphate and trace element analysis.

Through the use of phosphate analysis, we were able to distinguish the north room as the food preparation area of this group. The high concentration of phosphate would indicate that this area was involved in possible food preparation. The lack of heavy metals indicative of craft production or ritual activities supports this finding. The high quantity of ceramic pieces, obsidian blades and chert in the north room also substantiates this claim (see table 3). Likewise, the south room and patio were revealed to be areas of craft production. Although moderate in phosphate concentration, these areas had high concentrations of metals such as lead and mercury, which would not be associated with food production because of their toxicity. The artifacts from the deposits in these areas, such as high number of ceramic pieces, figurine fragments, animal bones, chert and obsidian blades in the south room and patio sustain this find. We were also able to distinguish similar activities between the principal bench of the central room and the south patio area because of their chemical signatures. This pattern appeared in both total digestion and DTPA extraction procedures, indicating that this relationship is not merely coincidental. This may indicate activities related to the use of pigments or dyes used for reception of guests or other important events. The elevated concentration of metals that follows the back wall of the structure may be an indication that this structure was decoratively painted.

Furthermore, from the chemical analysis methods, we have evidence of important features that were not indicated by the distribution of artifacts such as the outlines of the roofed area, sweeping patterns, as well as loci of major activities associated with food preparation and craft production. Although these areas may have been presumed by researchers, chemical analysis gives evidence to support assumptions where artifacts are absent.

Another advantage to using chemical analysis is the detection of subtle differences in the artifact distribution from the chemical signatures present. The analysis of chemical signatures helps focus on minute differences in deposits of cultural material (a kitchen midden vs. a craft production midden) to help refine research strategies. For example, from the high concentration of P in the north room, we would assume this area was used for food production and can then look at artifacts as evidence of this activity. Likewise, from the lack of P and the high concentration of metals indicative of craft production, we have a better interpretation of the use of cultural material.

#### CONCLUSIONS

The data gathered throughout the course of this study indicates that extractable phosphate is more revealing than total phosphate. Although chemical data produced by the two procedures demonstrated similar patterns, extractable phosphate data presented more detail that was applicable to interpretation such as sweeping patterns and smaller cultural deposits. The extractable P method has several advantages over the total P procedure. The extractable P method can be readily adapted to a field-lab situation. The equipment for the extractable P method is lightweight and inexpensive; all of the necessary chemicals are available in ready-to-use packages. Samples can be analyzed in a short time period giving archaeologists almost immediate results at a low cost. Conversely, the total P procedure generally requires harsh chemicals that need careful handling, limiting the application of this procedure to laboratory conditions. The digestion procedures for total P are also time and labor intensive. Our experience shows that the extractable phosphate procedure is not only better because of more interpretive detail in the resulting chemical patterns, but it is also more cost effective and functional in both laboratory and field settings.

The chemical signatures resulting from DTPA and total metal procedures are slightly different (Table 4). Several of the areas of high concentration corresponded between the two methods, but there were several areas that one method or the other detected (see table 3). Thus, it appears that total heavy metal analysis and DTPA heavy metal analysis are both valid procedures. The main difference between these two methods involves the safety, cost and specialized equipment necessary. Soil digestion for total metal analysis involves concentrated acids and digestion must be performed under a perchloric acid hood. The turn around time of this procedure is about three times that of the DTPA method. The DTPA extractable heavy metal does not involve hazardous acids or expensive digestion and safety equipment. We also found that the signature of the DTPA extractable metals is more closely related to cultural deposits, thus making interpretation much easier. Furthermore, total copper and mercury concentrations were sometimes inconsistent. Although DTPA extractable barium and cadmium were also inconsistent, these elements are not considered as vital for interpretation of activity areas. As the DTPA method corresponded more with the activities of the group and is more conducive in time, health risks, and resources, we argue this to be the superior method of the two when applied to the highly calcareous soils of Guatemala.

With the refinement of the relationship of individual chemical signatures with ancient activities performed in a site, a new dimension of information for activity analysis is opened. The refinement of chemical analysis in the context of ancient activity areas conveys staggering interpretive potential. Chemical analysis is useful in identifying areas of food preparation and craft production, and invaluable in showing a relationship between cultural deposits and their corresponding areas of activity through sweeping patterns and similar chemical signatures.

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Table 1. Correlation matrix of the DTPA extractable metals

	Cd	Cu	Fe	Hg	Mn	Pb	Zn
Ba	.396*	.290*	.026	027	.652*	011	.237*
Cd		.836*	.025	.066	.700*	.289*	062
Cu			.138	.295*	.629*	.395*	096
Fe				.836*	011	.472*	.606*
Hg					009	.494*	.490*
Mn						.093	.004
Pb	Comolotion is						.228*

# **DTPA Correlation Matrix**

\* Correlation is significant at the 0.05 level (2-tailed) as determined by bivariate correlation.

Table 2. Correlation matrix of the total metal analysis

	Cd	Cu	Fe	Hg	Mn	Pb	Zn
Ba	385*	.129	.834*	562*	.841*	455*	.775*
Cd		.770*	098	.807*	135	.961*	254*
Cu			.309*	.444*	.437*	.731*	.288*
Fe				287*	.724*	188	.575*
Hg					317*	.890*	373*
Mn						165	.891*
Pb					1. 11		274*

# **Total Correlation Matrix**

\* Correlation is significant at the 0.05 level (2-tailed) as determined by bivariate correlation.

# SOIL CHEMICAL INVESTIGATIONS OF MAYA ACTIVITY AREAS: A COMPARISON OF AN ETHNOGRAPHIC SITE WITH RAPIDLY AND SLOWLY ABANDONED SITES

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# ABSTRACT

Domestic activities performed in certain areas over long periods of time tend to leave chemical residues as evidence of those activities. Some of the questions studied in this paper deal with the interpretive capabilities provided by chemical patterns. Soil samples from three areas were analyzed to provide information on specific chemical patterns associated with known activities and the persistence of chemical signatures in the soil. Chemical signatures of a modern Kekchi Maya household at Las Pozas, Guatemala, are related to current documented activities and compared with chemical patterns from Cerén, El Salvador, a rapidly abandoned site, and Piedras Negras, Guatemala, a slowly abandoned site. Cluster analysis was used to compare the chemical patterns of the three sites. We found a strong relationship between modern activities and chemical signatures, particularly phosphate associated with food preparation, consumption, and disposal. Some chemical signatures found in the ethnographic case were not applicable to the ancient sites due to contamination from modern activities as well as loss of the chemical signature over a short period of time. In examining the heavy metals and phosphate, however, we generally found similar chemical signatures between the ethnographic site and the ancient sites that suggest comparable activities.

Keywords: Soil Chemical Analysis, Ethnoarchaeology, Activity Areas, Maya

#### **INTRODUCTION**

With the development of studies directed toward Maya household groups over the past few decades, the demand for more efficient means of interpreting archaeological evidence has grown (Bawden 1982; Bermann 1994; Deetz 1982; Drennan 1988; Manzanilla 1987; Santley and Hirth 1993; Smith 1987; Tringham 1991; Wilk and Ashmore 1988; Wilk and Rathje 1982). The driving force behind the development of household investigations stems from the usage of the household group as a socio-economic unit to answer more complex social questions regarding ancient behavior

(Hammel 1980; Hammel and Laslett 1974; Laslett 1969; Laslett and Wall 1972; Nakane 1970; Netting 1965, 1993; Netting, et al. 1984; Wilk 1983, 1989, 1991; Yanagisako 1979). There are, however, several difficulties that often hamper the efficient analysis of space use patterns. Conventional interpretation based exclusively on artifact distribution can often be misleading (Manzanilla and Barba 1990). However, unlike traditional artifacts that are easily transported or removed from the actual activity areas, chemical signatures are evidence of specific activity and usually become fixed in the soil where the activity takes place. Thus, the development of soil chemical analysis provides an essential facet to the analysis of activity areas and space use, particularly in the field of Maya household archaeology (Carr 1984; Kent 1984, 1987, 1990; Kroll and Price 1991). In order to increase the role of chemical analysis in household archaeology, our aims for this study were: (1) to examine the relationship between soil chemical residues and activities through ethnographic studies to aid in establishing specific signatures associated with domestic activities, and (2) to incorporate soil chemical analyses in the study of households by combining soil chemistry with traditional artifact- and architecture-based research to gain a more complete understanding of household organization and domestic activities in the study area.

#### Theoretical Framework

The underlying premise of soil chemical analyses is that activities performed in the same place over a long period of time leave behind distinct chemical signatures, or residues that are trapped in the soil where they remain relatively unaffected over time (Parnell, et al., n.d.). Unlike traditional artifacts, the spatial pattern of many chemicals fixed in the floor or soil remains relatively unaffected through artificial or natural processes and closely reflects the use of space in the past.

Past studies involving soil chemical analyses have demonstrated significant interpretive potential in the study of prehistoric land-use and activity patterns (e.g. Cavanagh et al. 1988; Coultas et al. 1993; Dunning 1993; Ball and Kelsay 1992; Lippi 1998; Manzanilla and Barba 1990). Ethnographic studies aimed at the association of specific activities with chemical signatures demonstrate the interpretive value of chemical analysis (Barba and Ortiz 1992; Hayden and Cannon 1983; Manzanilla et al.1996; Smyth 1990). In those studies, chemical data from floor and soil samples collected and analyzed from modern houses were compared with the ethnographic information on space use and activity patterns.

Once the correlation between human activities and chemical concentrations is refined through ethnographic studies, chemical analysis can be applied to the interpretation of ancient activities (Middleton and Price 1996). Where artifact assemblage in its original locus of use is often dependent upon the rate of abandonment as mentioned previously, chemical information persists in the soil and is unaffected by abandonment. By looking at an ethnographic site, the chemical signatures related to specific activities can be determined. Theoretically, these chemical signatures can then be applied to ancient sites regardless of rate of abandonment.

#### *Phosphate Analysis*

The analysis of soil phosphate concentrations has a long tradition in archaeological research, and its utility in the study of domestic activities and land use is well established (Dauncey 1952; Proudfoot 1976; Sánchez et al. 1996; see Bethell and Máté, 1989; Craddock, et al. 1986; Gurney 1985; Hammond 1983; Scudder, et al. 1996 for reviews). Since phosphates are rapidly fixed by naturally-occurring compounds in the soil, they tend to remain stable in soils for very long periods of time.

The association of phosphate with human activities lies in the organic remains of food waste. Soil phosphorus (P) exists in a complex equilibrium of different forms, including inorganic P fixed by aluminum, calcium, and iron compounds; soluble and labile inorganic P; and organic P. Plants obtain their essential phosphate from the soluble and labile inorganic P forms. When the plants are harvested and transported, the phosphate is relocated with them in the form of membranes and other cellular structures. As the plants in the form of food waste or fecal materials decompose, the mineralized phosphate is readily sorbed or fixed on the surface of the soil particles where they remain for centuries. Eventually the outfield soils, where crops were grown are depleted of soil phosphate while the soil phosphate concentrations of the areas of food preparation, consumption, and waste deposition are augmented. This process of phosphate transport implies that household gardens fertilized with organic waste would contain increased concentrations of phosphates while areas of intensive agriculture which did not benefit from the enrichment of decomposing plants or remains would have lower concentrations (Eidt, 1984; McManamon, 1984; Woods, 1977).

Ethnoarchaeological work by Barba and Ortiz (1992) demonstrated that phosphate values indeed correlate with known activities. They reported that high concentrations of phosphates were found in the floors of kitchen and eating areas, while soils of the discard area for maize soaking water showed moderate levels. Walkway soils exhibited low P concentrations. However, they informed that further refinement of the methods and interpretation is necessary.

# Exchangeable Cations

Ethnoarchaeological studies have indicated that several domestic activities leave behind residues rich in exchangeable cations, calcium (Ca), sodium (Na), magnesium (Mg), and potassium (K) (Barba and Ortiz 1992; Hayden and Cannon 1983; Manzanilla, et al. 1996; Smyth 1990). Some of the daily activities that would enhance the concentration of some of these elements include soaking and preparing maize for grinding and burning wood (Hayden and Cannon 1983; Smyth 1990). The lime used to soak maize, which is later discarded with the water, is composed mostly of calcium. Ashes from the fire hearth increase the concentration of potassium and magnesium. The analysis of exchangeable cations in archaeological sites has not been widely used as an interpretative tool.

# Trace Metals

The past decade has witnessed a growing interest in the detection of trace elements, particularly trace metals, e.g., copper (Cu), iron (Fe), mercury (Hg), manganese (Mn), lead (Pb), zinc (Zn) (Bintliff et al. 1990; Entwistle et al. 1998; Lewis et al. 1993; Linderholm and Lundberg 1994; Scudder et al. 1996; Lambert et al. 1984). Metals are readily sorbed or precipitated on the mineral surfaces of calcareous soils and stuccos commonly found at Maya archaeological sites.

Activities of the ancient Maya involved the use of a variety of metal-containing substances. High Fe concentrations in soils could be found in areas associated with ancient *Agave* processing or animal butchering (Manzanilla 1996). Hematite (iron oxide, Fe<sub>2</sub>O<sub>3</sub>) and iron ochre (hydrated ferric oxide, Fe<sub>2</sub>O<sub>3</sub>·H<sub>2</sub>O) were used in pigments. Cinnabar (mercuric sulfide, HgS) is a bright red mineral that was often used by the Maya as a decorative paint or dye for ritual purposes and is found in ceremonial or sacred areas, such as burials or caches. Additional minerals used as pigments included pyrolusite (manganese dioxide, MnO<sub>2</sub>) for blacks, malachite (copper carbonate, CuCO<sub>3</sub>·Cu(OH)<sub>2</sub>) for greens, and azurite (copper carbonate, 2CuCO<sub>3</sub>·Cu(OH)<sub>2</sub>) for blues (Goffer 1980; see also Vásquez and Velázquez 1996a, 1996b for examples). Thus, trace metal analysis of soils in and around residential and ceremonial architecture will prove useful in identifying areas of pigment processing and ritual activities. The use of trace metal data in archaeology, however, is still in an infant stage, and the interpretation of data from the highly calcareous soils and stuccos of Maya sites requires further

refinement.

# LOCATION

#### Las Pozas, Guatemala

The ethnographic portion of this study took place at the residential compound of the Xe family at the Kekchi Maya village of Las Pozas located 10 km to the southeast of the ancient Maya city of Aguateca (Fig. 1). The Xe family, as well as most of the village inhabitants, moved to Las Pozas from the northern Guatemalan Highlands roughly 15 years ago. Because of the relative short period of occupation, the pattern of space use in this compound has not changed significantly, making the correlation between soil chemistry and activities relatively straightforward. They maintain an essentially traditional lifestyle, subsistence, and diet. Thus, this site provides a reference case of a traditional Maya household. The compound includes the present house of the Xe family, a garden area, the site of the previous house (completely dismantled), and walkways.

#### Cerén, El Salvador

The Pre-Columbian village of Cerén is located near San Salvador, El Salvador (Fig. 1). The Cerén site was suddenly entombed by a deep volcanic ash deposit in approximately 600 A.D. Because the initial ash was moist and fine-grained, it preserved even organic materials such as food stored in vessels, thatched roofs, and plants in gardens. The essentially complete preservation of architecture and artifacts in their original loci of storage and use allows for a higher degree of confidence in the reconstruction of activity areas than is usually possible (Sheets 1992, 1994).

Cerén is important because of the degree of artifact preservation and accessibility of samples, providing the unusual opportunity to study the behavioral processes that link them (Schiffer, 1987; Webster, et al. 1997). It is rare that archaeologists have the opportunity to analyze soils associated with known ancient activities. These conditions allowed us to test the efficacy of soil chemical data in the analyses of activity areas and space use.

#### Piedras Negras, Guatemala

Piedras Negras lies on the eastern bank of the Usumacinta River in the Northwest portion of the Petén rainforest of Guatemala (Fig. 1). It is a Classic Maya city of central importance to Mesoamerican archaeology. Recent archaeological investigations have included expansive excavations in modest, non-elite structures at the site. These buildings tended to cluster in sectors or 'barrios' compacted between ceremonial plazas, monumental buildings, and the river bank. They have distinctive chronology and architectural patterns, suggesting variable functions during their occupation. While most sectors at this site were deserted gradually, some buildings in the Acropolis may have been abandoned rapidly (Holley, 1983). To date, excavations in these locations have produced data on household size and composition and on production activities revealed by artifact distributions and soil chemistry (Escobedo and Houston, 1998; Houston et al., 1998, 1999; Terry et al. 2000; Wells et al. 2000).

#### **Sample Collection**

#### Las Pozas

We mapped the study area, including areas of domestic activity, and collected soil samples on a grid system established with the aid of a compass and measuring tape. Activity areas of the site were documented on the map and with a photographic record of the inside and outside of the houses. Through interviews, members of the family provided a detailed description of the different activities and spacial usage of the site.

Soil and floor samples were collected from the first 5 cm of the surface at the grid intersections. The grid sizes depended on the area of study, activities performed and desired detail. Floor samples were collected from the kitchen in a 0.5-meter grid and of a 1-meter grid in the entrance room. In the proximity of the house a 1-m grid system was used. A 2-meter grid system was employed for the rest of the lot, except for the location of the old abandoned house on the property. A total of 268 samples were collected covering an area of about of 520 square meters.

#### Cerén

At Cerén, the availability of soil samples is more limited, because of the concern for the preservation of unique earthen structures. Yet, over the past 10 years, soil and floor samples have been collected during excavation for future pollen analysis. These soil samples were collected from areas of confirmed ancient human activity. The samples were obtained from milpas (maize fields), walkways between buildings, a midden, and from the floor of Str. 10, a religious structure for ceremonial feasting in the Pre-Columbian village. With the cooperation of the Salvadoran National Museum, 27 soil samples were divided into two portions: one for pollen analysis as originally planned and the other for phosphate and trace element analyses.

# Piedras Negras

Soil samples were collected from a sub elite residential group in the "U" sector of the University of Pennsylvania map of Piedras Negras (Operation 33). Surface samples (0-10 cm below leaf layer) were collected using a 2 meter grid formed with tape and compass. The structure was later strip excavated and additional samples were taken from areas of suspected activities. A total of 91 samples were collected and analyzed from this group.

#### **Chemical Analyses**

#### *Extractable phosphate procedure*

The method of extractable phosphate analysis we used is based on the Mehlich II extraction solution and Hach reagents (Hach, Co., Loveland, CO)(Terry, et al. 2000). The key advantage of this method for extractable phosphate analysis is that it is a quantitative field test that can easily be taken to a remote area, thus allowing us to perform the chemical analysis during the field season. Two grams of air dried, sieved (< 2 mm) floor or soil sample were placed in one of six 50 ml jars attached to a board for facilitation of simultaneous processing of multiple samples. Each soil sample was extracted with twenty ml of the Mehlich II solution for 5 minutes. The samples were then filtered and the filtrate collected in clean 50 ml jars. One ml of the extract was dispensed to a vial, diluted to 10 ml, and the contents of a PhosVer 3 powder pillow was added to the vial. The sample was shaken by hand for one minute and allowed to stand an additional 4 minutes for color development. The phosphorus in the extract reacts with the contents of the chemical pillow added giving a blue color. The more phosphorus in the solution, the darker the color. The concentration of phosphate in the samples is determined on a Hach DR 700 spectrophotometer at a wavelength of 880 nm and comparing the transmittance with a standard curve. A more detailed description of the procedure can be found in Terry, et al. (2000).

#### Exchangeable Cation procedure

The exchangeable cations Ca, Mg, K, and Na were determined by ammonium acetate extraction buffered at pH 8.5. Five grams of air dried, sieved (< 2 mm) floor or soil sample was placed in a 50 ml centrifuge tube. Each sample was extracted with 25 ml of ammonium acetate solution for 5 minutes. The samples were then centrifuged until the supernatant liquid was clear, separating the soil from the extracted cations. Each sample was filtered through a 11-cm qualitative filter paper and the filtrate was collected in clean

125 ml plastic bottle. The procedure was repeated three more times to collect the remaining cations. An aliquot from the 4 extracts of each sample was then used to determine the exchangeable cations using a Thermo Jarrell Ash ICP spectrometer.

#### *Extractable trace metal analysis*

Samples were analyzed for extractable trace metal concentrations using the DTPA (diethylenetriaminepentaacetic acid) extraction procedure developed by Lindsay and Norvell (1978). A chelate (DTPA) extraction of the highly calcareous soils and floors of the study areas was appropriate for trace metal analysis because the procedure avoids the complete dissolution of CaCO<sub>3</sub>, therefore only the trace elements that were deposited in the soil and adsorbed on the surfaces of soil particles were removed. In this procedure, 10 g of air dried, sieved (< 2mm) soil is mixed with 20 ml of 0.005 *M* DTPA solution buffered at pH 7.3 to extract the metals from the soil. The samples are then shaken for 2 hours, after which the extracting solution is separated from the soil by centrifugation and filtration. For the present analysis, the concentrations of barium (Ba), cadmium (Cd), copper (Cu), iron (Fe), mercury (Hg), manganese (Mn), lead (Pb), and zinc (Zn) were determined simultaneously on a Thermo Jarrell Ash ICP spectrometer (see Linderholm and Lundberg 1994).

# **Statistical Analysis**

Data from chemical analyses were subjected to geostatistical analysis to examine

spatial patterns of soil chemicals and to deduce the configuration of activity areas. Soil physical and chemical properties and ancient settlements, home sites, and artifacts are distributed over geographic regions with some degree of interdependence. Geostatistical analysis of the data allowed interpolation of concentrations of soil chemicals from one sampling location to the next. Thus, the patterns of ancient activities become more apparent. The geostatistical analysis was performed on the data resulting from the chemical analysis using SPSS software and a map was interpolated using Surfer software (Golden Software, Golden, CO).

#### **RESULTS AND DISCUSSION**

#### *Phosphate*

The spatial distribution of extractable phosphate concentrations in the house lot belonging to Francisco Xe is shown in Figure 2. The isopleth lines outline areas of highest phosphate concentrations corresponding to food preparation, consumption, and waste disposal areas. The highest concentrations of phosphate were found in the kitchen. As a result of the food preparation, food materials that are rich in phosphate fall to the ground and are eventually incorporated into the dirt floor. Moderate concentrations of phosphate were located in the living room of the house, possibly associated with maize storage and food consumption. The moderate levels of phosphate in the doorway are probably the result of daily sweeping from inside the house. As food remains are swept from the kitchen to the outside, smaller pieces are likely trapped in the soil. Elevated concentrations of phosphate to the north of the house indicate the location of a previous outhouse. High concentrations of phosphate in the southeast part of the study area correlated with the disposal of organic debris from the kitchen and other residues into the garden area as fertilizer. In front of the house, beyond the path areas, phosphate levels were slightly elevated due to the accumulation of residues (garbage) and the swept materials from the house into that area. Phosphate concentrations associated with pathways and roof drip lines around the house were low. Pathway areas were heavily traveled and swept often. Drainage from the roof may have washed organic debris away from the house. High levels of phosphate in the southwest corner of the study area indicate the location of the former house. Phosphate levels in the area of the razed house proved to be very similar to those of the present house. The highest concentrations of phosphate corresponded to the old kitchen and former waste disposal areas. Two areas near the pathway located between the previous home and the current home were enriched in phosphate. This area was probably used previously for waste disposal connected with the old house. This finding illustrates that the soil is a valuable interpretive resource (Entwistle et al. 1998). The soil holds a chemical residue memory from each successive human activity.

Much like the ethnographic site, the sampled area of highest phosphate concentration in the Cerén structure was associated with refuse disposal (Fig. 3). The area to the southeast of the structure was used for temporary disposal of refuse and sweepings until the waste could be removed (Sheets 1994). The ceramic assemblage was

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greater in this area than any others tested (Sheets, personal communication). The samples analyzed from the front of the structure and the floor surfaces inside the rooms were the lowest in phosphate concentration. These samples were collected from areas that would have been well swept of any organic wastes that would have increased the phosphate concentration, similar to patterns found in the ethnographic study.

Operation 33 at Piedras Negras also showed phosphate patterns similar to those found at Las Pozas. The highest concentrations of phosphate are associated with the northern room and the center room with three benches (Fig. 4). The northern room (kitchen area) contained a higher concentration of artifacts associated with food preparation such as metates and obsidian blades, and chert. The center room was most likely an area of food consumption. There is another area of high phosphate concentration to the east of the kitchen area. This is most likely evidence of ancient sweeping patters and the removal of kitchen remains. Like the ethnographic site, there was moderate phosphate concentration associated with the entrances to both the food preparation and consumption areas. These patterns indicate sweeping activities and the accumulation of small particles of food waste near the steps or doorways. Low concentrations of phosphate, indicative of heavy traffic, sweeping, or a drip line surrounded the entire structure.

# Exchangeable Cations

Exchangeable cation concentrations represent activities generally associated with

burning. Potassium is a major component of wood ash, therefore, the highest levels were found in the kitchen near the cooking hearth (Fig. 5). Background levels of  $K^+$  in the soils of the pathways and patios ranged from 300 to 800 mg/kg. Elevated  $K^+$  levels in excess of 1000 mg/kg were found where refuse was disposed and at the location of the hearth at the previous house. The distribution of exchangeable Mg<sup>++</sup> in the soils of both house lots was very similar to that of  $K^+$ . It is important to note that the exchangeable cations are not very stable in this soil. A simple comparison of the present house with the former indicates a significant decline in the elemental concentrations due to leaching (Fig. 5). Within five years since the old house had been razed, sodium levels were depleted to levels similar to the surrounding areas that were exposed to rainfall and leaching.

#### Trace Metals

Levels of DTPA extractable iron were in the range of 10 to 30 mg/kg in the pathways and patio areas of the Xe home (Fig. 6). Increased concentrations found inside the home could be related to the use of metallic cooking utensils. The household uses a cast-iron range top as opposed to the traditional stone hearth located on the floor. Similar patterns of spatial distribution were noted for Cu, Hg, and Pb. There were relatively low levels of these heavy metals outside the new and old homes with as much as double the concentrations inside. Zinc was also found in high concentrations at certain locations outside the home. The greatest concentrations of Zn were found at localized spots inside the garden area and other areas of refuse accumulation (Fig. 7). During a follow-up visit to the Xe family in Spring 2000, we observed old flashlight batteries, Zn-coated metal cans and rubber tires disposed in these areas of Zn accumulation. This is an example of the differences in chemical signatures of modern and ancient activities.

The highest concentrations of metals from the samples collected from Cerén were found in the center room of the structure (Fig. 8). All three samples from this room had elevated concentrations of metals with known association with pigments. Artifacts recovered from this room include a small cylinder used to store cinnabar and several vessels including a painted gourd and a deer headdress used in religious ceremonies (Sheets, 1994, personal communication). Clearly, the chemical analysis of samples and the ceremonial artifacts found in the center room indicate the heavy use of pigments.

There were four interesting concentration peaks of metals from analysis of the Piedras Negras operation 33 samples. Three of the peaks corresponded with areas of high phosphate concentration, including the northern room, the center room, and the area east of the northern room (Fig. 9). The fourth area of high metals was east of the southern room. These areas are most likely areas associated with craft production and the use of pigments. The high concentrations in the northern room and east of the northern room correspond to the kitchen area and the sweeping pattern of kitchen remains (also high in metals in the ethnographic study). The high concentrations in the center room and east of the southern room were very similar. The center room is a focal point for reception of guests to the ancient site and most likely involved the use of paints or pigments that were possibly processed or discarded east of the southern room.

#### Statistical Analysis

The cluster analysis of the soil chemical data illustrates the relationship of the various elemental residues to activity areas. This technique groups together the different soil samples according to chemical composition. This analysis can help to determined the different groups of chemicals that are associated with specific activities. Soil samples in cluster group 1 were highest in DTPA extractable metals (see table 1). This group was associated with modern waste such as old batteries, and pigments at ancient sites. Group 2 samples contained average to moderate concentration of metals. This chemical signature relates to areas of low activity away from the sweeping areas and pathways (Figs. 10, 11, & 12). Samples labeled as group 3 had the lowest chemical composition, below average in all elements analyzed. These samples are generally located in areas of pathways that are swept clean or roof drip lines. Group 4 samples were very high in mercury, copper, lead, iron, and phosphate. This group was associated only with the interior of the ethnographic structure. Group 5 samples were extremely elevated in zinc, and slightly elevated in phosphate. These samples are associated with areas of disposal of batteries and tires (ethnographic case) and pigments. Group 6 contained samples with extremely high levels of manganese and phosphate. These levels may be a result of a combination of pigment and kitchen disposal.

#### **Conclusions and Future Research**

Soil chemical analyses of anthropogenic soils and stucco floors served to positively identify areas of modern and ancient activity. Coupled with the analysis of artifacts related to distinct activities, chemical analysis magnifies the interpretive capabilities of household archaeologists. Most Maya sites were well maintained prior to abandonment and contain few in situ artifacts. In these situations, soil chemical analyses provide a powerful tool for archaeologists in the clarification of space use and the types of activity performed. The interpretive potential of ancient activity areas through the use of soil chemical analyses is limited only by our understanding of specific ancient activities. The combination of quantitative soil chemistry data and unusually rich floor assemblages is expected to lead to more detailed views of Classic Maya households than have previously been possible. The development of soil chemical analyses will have significant implications for the future study of Maya households.

Although many of the activities performed in modern as well as ancient times leave distinct chemical signatures, some chemicals may not persist in the soil long enough to serve as chemical markers for specific activities, such as the exchangeable cations in this case study. Another difficulty with the relationship between modern activities, chemical signatures, and ancient activities is that some study areas may be contaminated by modern wastes, in which association and comparison of activity areas with chemical signatures may be problematic.

Soil analyses have been widely used in pre-excavation detection of archaeological

sites and specific activity areas. We expect that soil chemical residue analysis will also be useful in the study of ceremonial areas, where pigments may have been used and sacrificial blood may have been spilled. In addition, trace metal analysis can indicate whether some structures, though now eroded, were originally painted, possibly with designs and symbols that served as public expression of local identities, such as status, rank, or lineage (Wells, et al. 2000).

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Extractable Element	Clusters								
	1	2	3	4	5	6			
DTPA Cd	2.14	.52	47	24	73	.33			
DTPA Cu	2.71	.35	56	4.52	17	.45			
DTPA Fe	.97	.40	50	3.98	.30	.01			
DTPA Hg	2.90	.32	59	3.50	.29	1.07			
DTPA Mn	.82	.08	35	.46	31	3.54			

Table 1. Cluster analysis comparing the ethnoarchaeological site (Las Pozas), rapidly abandoned site (Cerén), and gradually abandoned site (Piedras Negras)\*.

Mehlich P	.36	.04	26	2.22	.82	1.45		
DTPA Zn	.49	06	35	.07	3.25	.76		
DTPA Pb	2.87	.38	55	3.89	02	.05		

\*Chemical data is reported in standard deviations from the mean.



Figure 1. Location of sites used in the study.

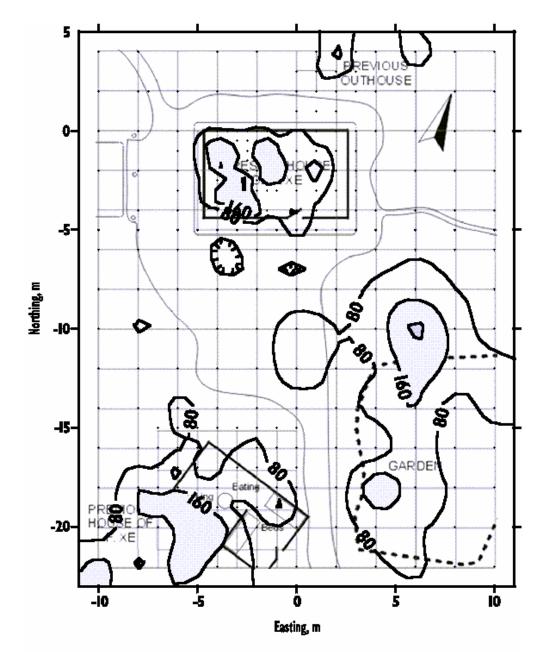


Figure 2. Mehlich extractable P concentration (mg/kg) from the houselot of the Francisco Xe family, Las Pozas.

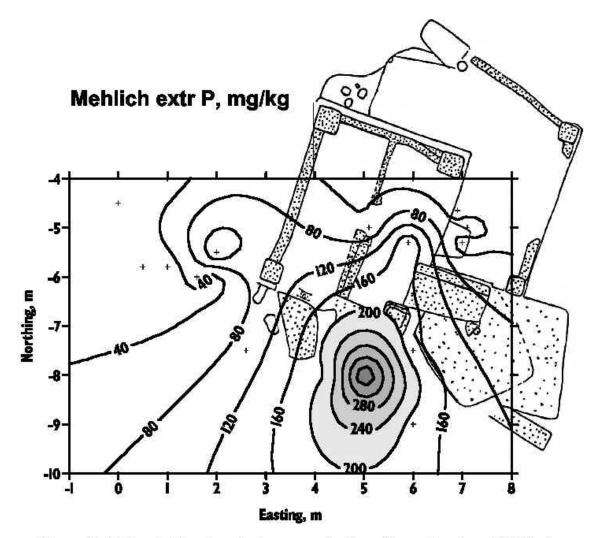


Figure 3. Extractable phosphate concentrations from structure 10, Cerén.

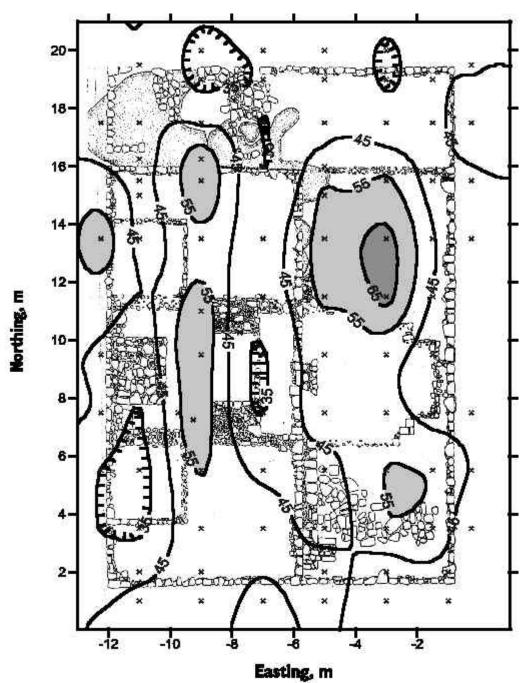


Figure 4. Mehlich extractable phosphate concentration (mg/kg) of Operation 33, Piedras Negras.

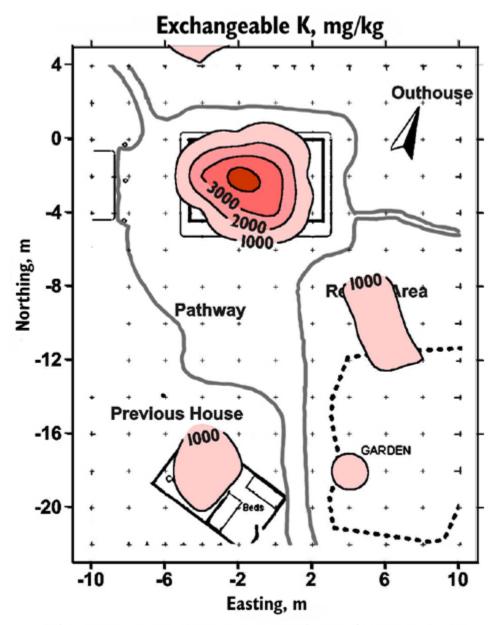


Figure 5. Exchangeable potassium of the houselot of the Francisco Xe family, Las Pozas.

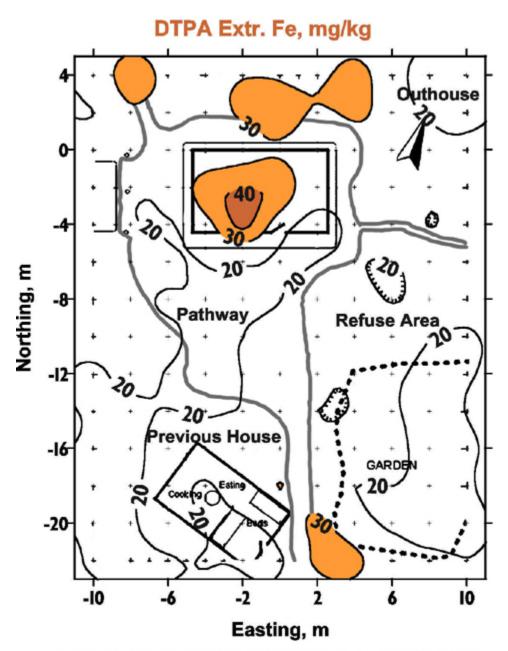


Figure 6. DTPA extractable iron concentrations from the houselot of the Francisco Xe family, Las Pozas.

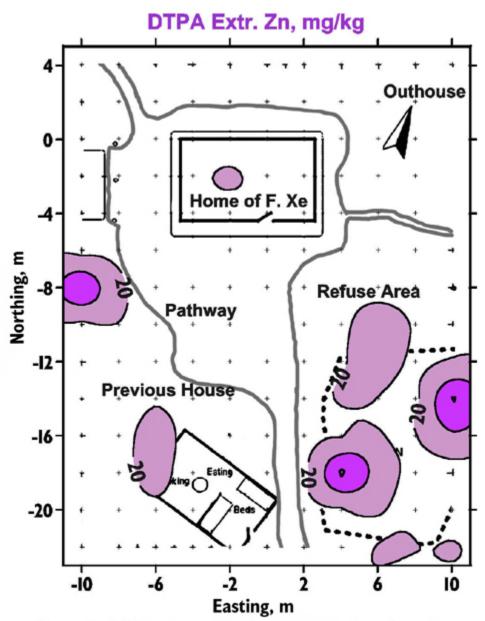


Figure 7. DTPA extractable zinc concentrations from the houselot of the Francisco Xe family, Las Pozas.

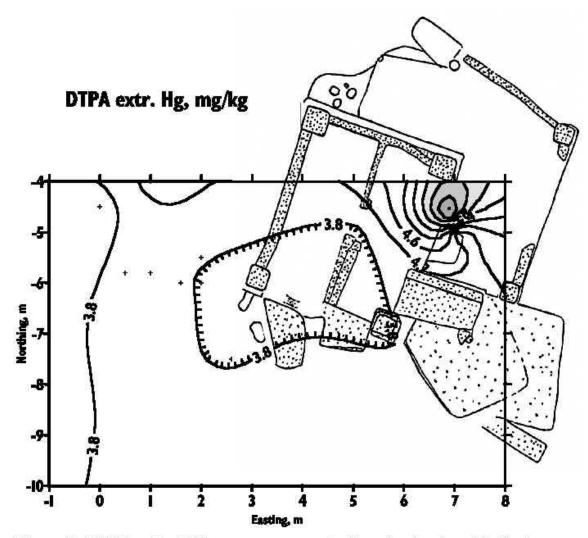


Figure 8. DTPA extractable mercury concentrations in structure 10, Cerén.

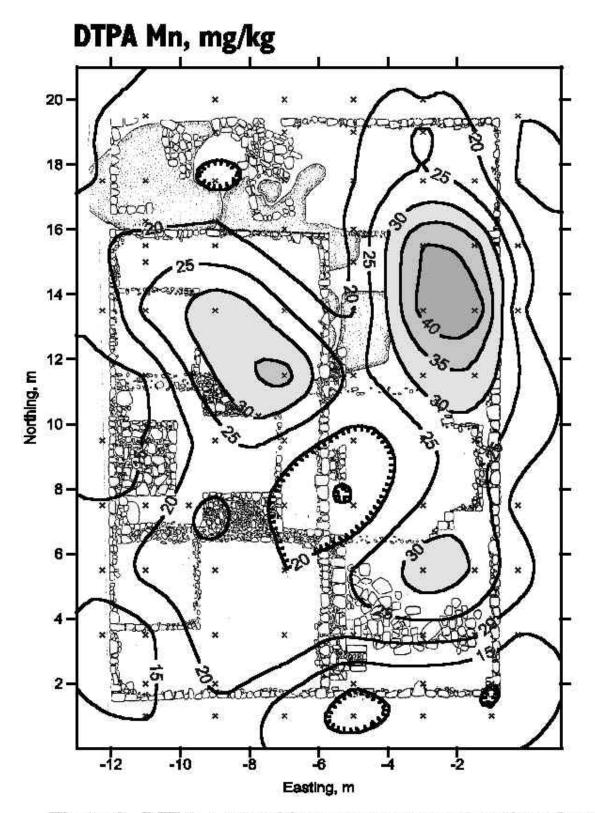


Figure 9. DTPA extractable manganese concentrations from Operation 33, Piedras Negras.

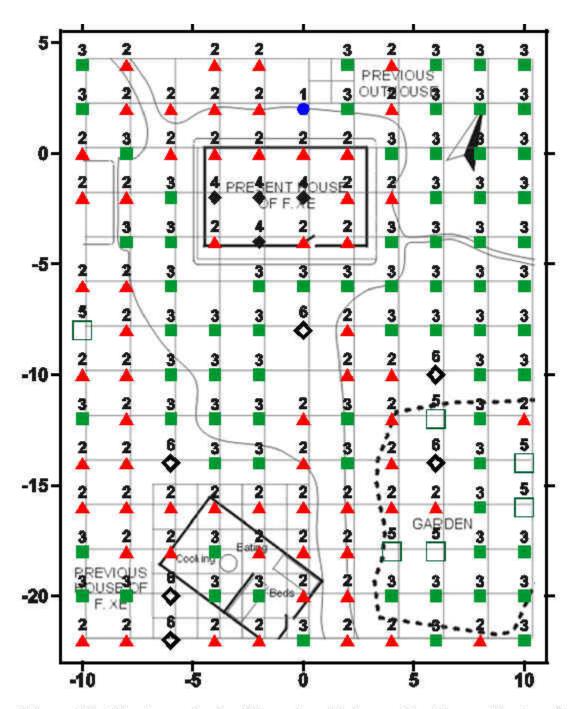
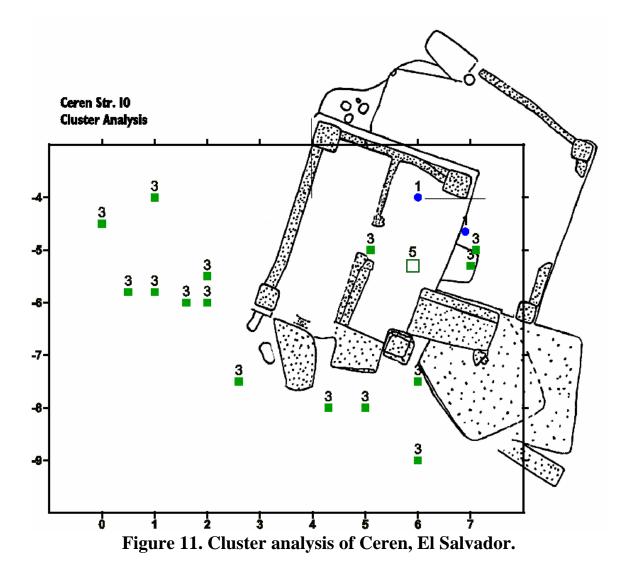


Figure 10. Cluster analysis of Francisco Xe home, Las Pozas, Guatemala.



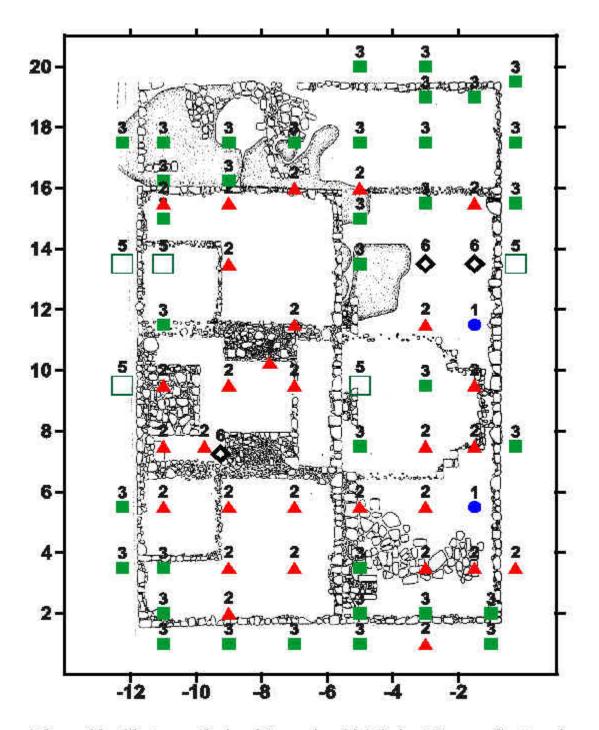


Figure 12. Cluster analysis of Operation 33, Piedras Negras, Guatemala.

### APPENDIX A

#### INTRODUCTION

Over the past few decades, means of exploring some of the more pressing questions involving the social dynamics of the Maya civilization have been presented. Many of these questions concerning the social aspect of the Maya arise from the lack of knowledge or information surrounding the household group and areas of activity (Bawden 1982; Bermann 1994; Deetz 1982; Drennan 1988; Manzanilla 1987; Santley and Hirth 1993; Smith 1987; Tringham 1991; Wilk and Ashmore 1988; Wilk and Rathje 1982). Due to the fact that the household is the most fundamental socio-economic unit, the information gathered from these household groups can often give researchers insights to larger questions (see Houston, et al. 1998 and Wells, et al. 2000). The complete analysis and better interpretation of such household groups as socio-economic units is considered critical for understanding various facets of human societies and behavior, including; gender relation, division of labor, social inequality, and demography (Hammel 1980; Hammel and Laslett 1974; Laslett 1969; Laslett and Wall 1972; Nakane 1970; Netting 1965, 1993; Netting et al. 1984; Wilk 1983, 1989, 1991; Yanagisako 1979).

Mesoamerican archaeologists have been playing an important role in developing household archaeology. Field investigations at various sites have generated a large amount of data on the Classic Maya household (Haviland 1985; Hendon 1996; McAnany

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1995; Tourtellot 1988; Webster and Gonlin 1988; Wilk and Ashmore 1988). Our understanding of the Classic Maya household, however, is still limited. Although breakthroughs have been made, there is still substantial mystery behind the daily activities of the ancient Maya.

Interpretation of household groups and their surrounding areas of activity has generally been established through the distribution of artifacts typical of definite activities; cooking utensils are found where cooking activities were accomplished. Unfortunately, most Maya settlements were abandoned gradually. When a site is deserted progressively, inhabitants have time to transport a large portion of their possessions to the next residence and leave few artifacts behind (Cameron and Tomka 1993; Schiffer 1987; Stevenson 1982). Another problem with the interpretation of artifact distribution alone is post habitational disturbance by later incidental inhabitants (Houston, et al. 1999). Due to the transplantable nature of traditional artifacts, the end location of an artifact may not reflect the areas of utilization of that artifact (Manzanilla and Barba, 1990).

The study of residual chemical signatures sheds light on many of the daily activities of the ancient Maya such as the cultivation, storage and preparation of food, as well as other activities. In fact, the only way that areas of social activity can be defined in archaeological units is through combining chemical analysis with other areas of archaeology, such as artifact distribution and architectural analysis (Manzanilla and Barba, 1990). Without the integration of chemical signatures to activity analysis, our

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understanding of the ancient human activities– resulting in the chemical adaptation of the soil– cannot be nearly as successful (Dunning, 1993).

Historically, the role of soil chemistry in archaeology has been relatively passive. The chemical analysis of soils has generally been used to compliment other results achieved through more active tools. Bethell and Máté (1989) point out that chemical data is usually used to support conclusions already drawn from excavation and rarely used to develop more detailed interpretations or to refine research strategies.

#### **OBJECTIVES**

The overall objective of this study is to perform an analysis of the relationship of soil chemical signatures to the activity areas of the ancient Maya. This objective is comprised of three components that will be independently publishable in journals applicable to Maya archaeology. The importance of each component in accomplishing this objective will be discussed in more detail later. The specific objectives of this thesis were:

- (1) to investigate the use of soil chemical patterns to prospect for residential middens and perform a statistical analysis of the relationship of soil phosphate signatures to the distribution of domestic activities as evidenced in the artifact assemblage recovered in the O/N, J, and C residential groups of Piedras Negras, Guatemala,
- (2) to investigate the use of soil chemical patterns to locate activity areas involved in food preparation and disposal, sweeping patterns, and workshop activities and to identify specific chemical patterns related to detailed activity verified through the distribution of activity-diagnostic artifacts in the U strip excavated residential group of Piedras Negras, Guatemala, using factor analysis. A statistical analysis will also be used to compare and contrast two current methods of heavy metal determination used in archaeology (Total vs. extractable) with respect to

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activity areas in a strip-excavated residential group (operation 33) in Piedras Negras, Guatemala,

(3) Use Ward's nonhierarchical cluster analysis in the comparison of patterns of soil signatures to related known activities in an ethnographic context in Las Pozas, Guatemala, a site of known activity areas at Cerén, El Salvador, and compare with chemical signatures from a strip excavated site in Piedras Negras, Guatemala,

These objectives follow a succession that will expand the role of soil chemical analysis in archaeology. The first objective uses soils chemical analysis, particularly phosphate analysis, as a prospection tool to look solely at the activity of residential refuse disposal. This objective provides researchers with a tool for the pre-excavation indication of residential middens, thus allowing the archaeologists to see a pattern of disposal in order to devise more efficient excavation strategies. The second objective incorporates soil chemical analysis directly into the excavation of residential areas. This objective takes a detailed look at the specific chemical patterns in order to amplify the interpretive capabilities of the researchers. This component focuses on an area poor in artifacts to determine the quality of activity area interpretations based solely on chemical analyses. The third objective not only establishes the relationship between specific chemical signatures and activity areas, but also connects that relationship to ancient sites.

the relationship of chemical signatures using the methods of this study with modern-day activities, we can show that these methods do detect the chemical residues from speicific activities.

These components expand the use of soil chemical analysis as a prospection tool, a tool for detailed interpretation of activity areas, and as a detector of ritual activity areas. By doing so, these analyses can be used to investigate areas where domestic or ritual activities may have taken place, including the application of statistical analyses of artifacts to these areas. It is hoped that this study will show that specific chemical signatures can be used to identify areas of distinct activities.

### LITERATURE REVIEW

#### The Analysis of Chemical Residues in Anthrosols

Chemical analyses of anthrosols, soils that have been chemically modified by human activity, have become powerful analytical tools in the study of prehistoric landuse patterns. The artifact records of many archaeological sites, including those in Mesoamerica, are frequently restricted by poor preservation due to erosional processes, biological processes and disturbance by people in both ancient and modern times (Wells, et al. 2000). In sites where these traditional archaeological records, such as artifacts or architecture, are lacking, the chemical enrichment of occupation areas is of great use to archaeological research (e.g., Cavanagh, et al.1988; Coultas, et al. 1993; Dunning 1993; Ball and Kelsay 1992; Lippi 1988).

### Soil Phosphate Analysis

Soil phosphate is the most widely used and developed soil chemical test in archaeology. In the 1930's, while testing soil fertility for a sugar beet company in Sweden, O. Arrhenius (1931) realized a peculiar occurrence associated with phosphorus levels and an area of ancient habitation. He noted that an old archaeological site that had long been abandoned showed elevated concentrations of phosphates. He found that phosphate concentration in the soil is related to past habitation. Through this discovery

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came the development of a new tool in the archaeological repertoire. The application of phosphate analysis in archaeology was first developed in Europe, where pioneering efforts by Arrhenius (1931) and Lorch (1940) were followed by work of numerous scholars, including Dauncey (1952), Sieveking et al. (1973), and Provan (1973).

The general acceptance and application of chemical methods in archaeology was relatively slow, particularly in America, until the influential work of Eidt (1973). Since that time, more studies have been completed on the relationship between the chemical signatures left in the soil and their relationship to ancient inhabitants. (e.g., Ball and Kelsay 1992; Barba and Manzanilla 1987; Coultas et al. 1993; Dunning 1994; Jacob 1995; Manzanilla and Barba 1990; Manzanilla 1983) as well as contemporary inhabitants (e.g., Barba and Bello 1978; Barba and Ortiz 1992).

The chemical analysis of phosphate has proven successful in applications to soils formed under a wide variety of geological conditions and subject to a range of erosional and depositional processes. Soil phosphate testing in the Maya region is particularly promising given the highly calcareous nature of the soils derived from the karst topography of the region, as calcium serves to fix phosphate compounds in the soil for long periods. Soil chemical data provides important clues as to the activity of a given area when it has been difficult to judge from artifact data alone (e.g., Ball and Kelsay 1992; Cavanagh et al. 1988; Coultas et al. 1993; Dunning 1993; Lippi 1988).

The logic of the association of phosphate and areas of human activity lies in the phosphorus cycle. Soil phosphorus (P) exists in a complex equilibrium of different

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forms, including fixed inorganic P absorbed to aluminum, calcium, and iron compounds; soluble and labile inorganic P; and organic P. Plants obtain their required phosphates from the soluble and labile inorganic P fraction of the phosphate gamut. When the plants are harvested and transported, the phosphates become relocated with them in the form of nucleic acids, membranes and other molecular structures. As the plants, in the form of organic garbage (fecal residues or food waste), decompose, the phosphates become readily sorbed or fixed on the surface of the soil particles where they remain for centuries. This process depletes the outfield areas where plants were grown of soil phosphate while enriching the soil phosphate concentration of the areas of compost, consumption, and deposition. This principle indicates that household gardens fertilized with organic waste would contain increased concentrations of phosphates while areas of intensive agriculture which did not benefit from the phosphorus buildup of decomposing plants or remains would have lower concentrations when compared with background levels (Eidt 1984; McManamon 1984; Woods 1977). These differences in the chemical concentration can then be read, mapped, and interpreted according to their spatial pattern.

Soil phosphate chemical analyses in archaeology have been used primarily in three areas of study. First, soil samples are analyzed in pre-excavation prospection to locate or delimit archaeological sites to help orient future excavation. This has been the most common use of phosphate analysis in archaeology (e.g., Ball and Kelsay 1992; Dauncey 1952; Eidt 1984; Hammond 1983; Provan 1973; Sieveking et al. 1973). Second, phosphate analysis is used as a tool in landscape archaeology to examine past agricultural practice (Coultas et al. 1993; Dunning 1993; Dunning et al. 1997, 1998). The third area is the delineation of features and activity areas through phosphate analysis during and after excavation (Manzanilla and Barba 1990; Solecki 1951; Terry, et al. 2000; Wells, et al. 2000; Weston 1995).

There are four principle methods of soil phosphorus (P) analysis. The first method is the chemical digestion of soil for total P analysis. The soil sample is digested using a strong acid that releases all forms of phosphate (Olson and Sommers 1982; Hassan 1981). Drawbacks of this method are that it involves the digestion of soil, is quite time and labor consuming, and it is not feasible to perform total P analysis in a field laboratory. The second method is P fractionation, which separates and measures different compounds of soil P (Eidt 1973, 1977, 1984; Lillios 1992). This method is very detailed and has significant potential, however, it involves intensive lab work, making it impractical for field application. Also, the interpretation of data based on the distribution of inorganic P among three fractions (calcium, aluminum and iron phosphates), remains problematic. Bethell and Máté (1989: 18-19) criticize that the interpretation of P fractionation data (Eidt 1973, 1977, 1984) does not adequately take into account complex soil processes and the widely varying properties of different soil types (White 1978). The third method is generally called the spot test or ring test. A small amount of soil is placed on filter paper, to which drops of an acid extractant are applied, followed by the color development reagent. The concentration of P is estimated by the intensity of

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coloring reaction on the filter paper. The merit of this method is that it can be easily conducted in the field and the result is obtained instantaneously. With the refinement of the method by Eidt (1973, 1977), soil phosphate testing became quite popular among field archaeologists. However, the evaluation of ring formation and coloring is subjective, and it is not a truly quantitative method.

In 1997, Terry, et al. (2000) introduced to archaeology a field procedure for a quantitative determination of phosphate concentrations (Mehlich 1978). This method is more quantitative than Eidt's ring test method and more simple and economic than previous methodological advancements (e.g., Eidt 1973, 1977, 1984; Hassan 1981; Mehlich 1978; Olsen and Sommers 1982; Woods 1977). The analytical method adapted by Terry provides quantitative data that are far more reliable and detailed than ring-test results. In preliminary studies, soil chemistry data correlated relatively well with artifact distribution, indicating the validity of these analyses for the study of past behavior (Terry el al. 2000). The advent of this procedure permits archaeologists to achieve phosphate results in the field at low cost. It consists of extracting the soluble and readily labile soil phosphorus and determining the concentrations quantitatively with the aid of a colorimeter. Generally this type of method has been conducted in laboratories, but has recently been developed with Mehlich II extraction (Mehlich 1978) that can be carried out in the field. An observation related to the analysis of the extractable phosphorus is that it is not always proportional to the total phosphorus of the soil. However, for the household and activity analysis research, the relative spatial patterns of phosphate levels

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are important, rather than the absolute concentration (Terry, et al., 2000). Acquisition of soil phosphorus analysis data in the field facilitates direction of excavation and soil sampling strategies. The field lab method that Terry has developed offers this advantage, as well as highly reliable, quantitative data. It is essential to use quantitative methods for the further refinement of activity feature analysis (Bethell and Máté 1989:17).

## Trace Element Analysis

Once the analysis of phosphate became an accepted technique used in archaeology, methods for analysis of other activity-diagnostic chemicals were gradually implemented. Particular interest has grown within the last decade in the analysis of heavy metals, e.g., copper (Cu), iron(Fe), mercury (Hg), manganese (Mn), lead (Pb), zinc (Zn). Trace metals have demonstrated interpretable results in soils associated with ancient settlements (Blintiff, et al. 1990; Entwistle, et al. 1998; Lewis, et al. 1993; Linderholm and Lundberg 1994; Lambert, et al. 1984; Middleton and Price 1996). Metals are readily adsorbed or precipitated on the mineral surfaces of calcareous soils and stuccos commonly found at Maya archaeological sites. Metallic ions remain stable in alkaline soils for long periods in the form of adsorbed and complexed ions on clay surfaces, and as insoluble oxides, sulfides, or carbonates (Alloway 1995; Lindsay 1979; see also Wells, et al. 2000). Researchers have used a variety of extractions and total elemental analysis procedures. Lewis et al. (1993) and Scudder et al. (1996) used a dilute acid extraction of trace elements from soils of native American sites in the Eastern U.S. and from ancient Roman sites in Italy and Tunisia. Entwistle et al. (1998) performed total elemental analysis of anthrosols from Scottish historical sites by digesting soil samples in nitric and perchloric acid. Bintliff et al. (1990) analyzed trace metal accumulation in soils near ancient Greek settlements. They concluded that trace metals accumulate at very significant excess levels on and around ancient sites, and that these patterns can be useful for archaeological prospecting and site survey, as well as for reconstructions of land-use histories. They caution however, that many close-spaced soil samples need to be taken in order to evaluate the usefulness of trace metal assays for past land-use intensity. Bintliff et al. (1990) did not list their method of soil treatment for trace element analysis, but one of the coauthors (Davies 1978) has used EDTA chelate extractions to determine the extent of trace metal contamination of garden soils in both urban and rural areas in England and Wales. Lambert et al. (1984) used a chelate extraction procedure to determine trace metals in soils of the New River flood plain of Belize.

Middleton and Price (1996) observed that the properties of greatest interest in anthropogenic soils are the concentrations of mobile or extractable elements. Total analysis procedures, such as the nitric-perchloric (double acid) digestion, would measure the chemicals naturally found in the mineral forms within the soil particles. The high concentration of metals from a digested sample would overwhelm the comparably minute concentration of chelate extractable metals. The total analysis measures the total chemical concentration, including chemicals that are naturally fixed within the lattice of

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clays, whereas anthropogenic chemicals would most likely be found adsorbed to the surface of mineral soils. The anthropogenic fraction of the chemical would theoretically give us better interpretive capabilities. This study will be able to compare the extractable metals and the total digested metals to artifact distribution and activity areas.

The use of trace metal analysis in Mesoamerican archaeology has been quite limited, yet this analysis represents a significant tool for archaeologists. Activities of the ancient Maya involved the use of a variety of metallic substances. High iron (Fe) concentration in soils has been found in areas associated with ancient Agave processing or animal butchering (Manzanilla 1996). Hematite (iron oxide, Fe<sub>2</sub>O<sub>3</sub>) and iron ochre (hydrated ferric oxide,  $Fe_2O_3 \cdot H_2O$ ) were used in pigments. Cinnabar (mercuric sulfide, HgS) is a bright red mineral often used by the Maya as a decorative paint or dye for ceremonial purposes, such as burials or caches. Additional minerals used as pigments included pyrolusite (manganese dioxide,  $MnO_2$ ) for blacks, malachite (copper carbonate,  $CuCO_3 \cdot Cu(OH)_2$ ) for greens, and azurite (copper carbonate,  $2CuCO_3 \cdot Cu(OH)_2$ ) for blues (see Goffer 1980:167-173 for a review of pigment chemistry; see also Vásquez and Velázquez 1996a, 1996b for examples). Thus, trace metal analysis of soils in and around residential and ceremonial architecture will prove useful in identifying the chemical signatures of pigment processing and other activities. Of particular interest to the research in this study, heavy metals analysis of soils close to residential architecture has proven useful for Wells et al, (2000) in identifying possible signatures of paint residues from polychromatic facades of ancient Maya dwellings at Piedras Negras, Guatemala,

that may have served to publicly mark local identities such as status, rank, or lineage affiliation.

### Ethnoarchaeology

Ethnoarchaeology is the application of archaeological techniques to a modern setting. Over the past few decades, researchers have been able to piece together some valuable information concerning archaeological techniques, and even possible patterns of ancestors. The best cases of ethnoarchaeology look at groups or individuals that have somehow remained isolated from many of the modern-day technological advances. The assumption involved in many of these cases is that the activities and social structure of these groups will have remained somewhat unchanged from the time of their ancestors (Kent, 1984, 1987, 1990). Several ethnoarchaeological studies have been realized to correlate areas of activity with artifacts and chemical signatures left behind. Hayden and Cannon (1983) analyzed traditional villages in the Maya highland; Chanal, Aguacatenango and San Mateo. They observed some of the refuse disposal patterns of modern-day inhabitants. In 1990, Smyth complimented their research with an examination of food storage, production, and disposal among Highland Maya of the Puuc region of Guatemala. His research revealed evidence of distinct chemical patterns from his analysis on the soils of the activity areas, particularly phosphorus and calcium in areas of maize treatment. Barba and Ortiz (1992) examined the activity patterns of contemporary inhabitants of Tlaxcala, Mexico. They correlated each activity with the

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different chemical patterns found at that site. Their research focused on food preparation, food consumption and cultivation. One important aspect of the research of Barba and Ortiz is that their results were applied to Oztoyahualco, Coba, Mexico. Manzanilla et al. (1992, 1996) collected several samples from rooms and house groups at Oztoyahualco and Coba for analysis. Through chemical analysis, they were able to locate food processing, consumption, storage, and butchering areas. These areas of activity were confirmed through archaeological evidence.

Within the past few years, more objective, detailed and reliable data provided by quantitative methods have allowed archaeologists to compare soil data with known activities in a rigorous, standardized manner. The history of these soil chemical analyses has demonstrated that interpretable concentrations of phosphorus are be associated with soil enhancement in these archaeological areas (e.g., Dauncy 1952; Griffith 1981; Proudfood 1976; Sánchez et al. 1996; Soleki 1951; Houston et al. 1999; Weston 1995; for a detailed overview, see Bethell and Máté 1989; Craddock et al. 1986; Burney 1985; Hammond 1983; Scudder et al. 1996; Wells et al. 2000). Several ethnoarchaeological studies have correlated areas of activity with artifacts and the chemical signatures left behind. In these studies, isolated households were observed for daily activities. These activities included sweepings, which pushed organic materials to the patio peripheries, gardening, waste handling and the storage and preparation of food (Fernandez, et al., 2000; Hayden, et al. 1982; Smyth, 1990 ). Smyth found distinct patterns of pH levels and calcium levels from his analysis on the soils of the activity sites. The occupants used lime in maize preparation. As the maize would be washed, calcium levels where the water was discarded would be elevated. Barba and Bello (1978) analyzed the soil phosphorus of a modern house in Tlaxcala, Mexico. They found that highly traveled areas and rest areas were low in phosphates, whereas areas dealing with food consumption and disposal were very high. Barba and Ortiz (1992) returned to the same area to do more extensive organic analyses arriving at the same essential conclusions. Barba and Ortiz correlated each activity with the different chemical patterns found at that site. These investigations, and others carried out by Barba et al. (1995), in Muxucuxcab, Mexico, established general chemical patterns of various activities. The following observations emerged from these studies:

- 1. Rest or sleeping areas are found in inner rooms or, if there is only one room, opposite the fireplace. Generally these are poor in chemical compounds.
- Areas where meals were prepared and cooked normally include hearths, which correlate with low phosphate values, color changes and high pH. Such areas frequently contain carbonized seeds.
- Areas where food was consumed are rich in phosphates. Food consumption areas are located near preparation areas and contain low pH values. Anomalous calcium concentrations are attributable in some cases to corn bread preparation (*nixtamal* for tortillas).
- 4. Areas where animals were confined show concentrations of phosphates.

5. Areas of heavy foot traffic suffer much wear and are thus poor in chemical contents.

(Barba and Lazos 1999; Manzanilla 1990)

One key aspect to the development of chemical analyses in archaeology is the application and comparison of results from ethnographic settings to archaeological sites, such as Oztoyahualco, and Coba, Mexico (Barba and Manzanilla 1987; Manzanilla, et al. 1992, 1996; Manzanilla 1993; Ortiz and Barba 1993). Through chemical analysis, archaeologists in these studies were able to locate areas of food processing, food consumption, food storage and butchering areas. These areas of activity were confirmed through traditional artifactual evidence.

Over the past few decades, investigators have found chemical signatures that are related to specific human activity. Several studies have shown that there is a relationship of chemical concentrations found in the soil with activities of the area (Barba et al. 1987; Cavanagh et al. 1988; Craddock et al. 1986; Konrad et al. 1983; Manzanilla et al. 1990; Ortiz and Barba, 1993; Proudfoot 1976; Sanchez 1998; Terry et al. 2000; Wells et al. 2000). Ethnographic studies have proven an invaluable tool to study the relationship of chemical concentrations with human activities, due to the fact that definite identification of domestic activities using chemical patterns in archaeological sites can only be realized through comparison of similar sites where chemical analyses of soils have been confirmed through artifact data or ethnographic observation (Barba and Ortiz 1992; Manzanilla and Barba 1990). Soil samples collected as part of an excavation strategy can be analyzed and chemical patterns can then be determined, mapped, and compared with the known or suspected activities.

### SAMPLING AND METHOD SELECTION

Although soil chemical analysis has become largely accepted, there are still several issues that concern archaeologists, geographers and soil scientists regarding several aspects of the application of results in archaeological interpretation. Crowther (1997) points out that the precision of the analysis of soil chemicals as tool for archaeological interpretation in many sites depends on the depth at which the sample is collected. Deposition and soil formation can often bury the chemically altered occupational level. There are, however, some sites, as mentioned by Craddock et al. (1985), where deposition or soil formation has not been a significant factor, where settlement patterns and activity areas can be revealed in the topsoil. Such is the case with Piedras Negras, where maximum soil development has been estimated as about fifteen to twenty centimeters since the time of ancient occupation (Fernandez, et al., 2001).

Another concern specifically mentioned by Eidt (1977), Conway (1983), and others, is the form of the chemical measured by different methods of analysis. As mentioned above, there are many forms of each chemical of interest in the soil. Soil phosphorus is a good example of this quandary. There is currently no established method for measuring anthropogenic phosphorus. The phosphorus method originally used by Arrhenius and others was an adaptation of an agronomic method for determining available phosphorus for plants. The most common methods among archaeologists today are Eidt's ring test (Eidt 1977), an extractable (plant available) phosphorus test (Terry, et al. 2000), and measuring total phosphorus (Conway 1983).

It should be noted that this same concern over the ideal method also exists in the analysis of trace elements and heavy metals. Middleton and Price (1996) and Conway (1983) have pointed out an important observation; that it isn't the form in which we find the chemical of interest, but the spatial pattern with relation to the site that will determine the usefulness of chemical analysis at any given site. The natural levels of chemicals often vary greatly between soils, limiting our ability to place absolute values of chemical concentration with known activities. From the pattern of multiple soil samples analyzed from any site we can find likely areas of activity. In other words, the form of the chemicals we measure isn't as important as the spatial distribution, because all forms will have been altered by past human activity.

As mentioned by White (1978), our comprehension and interpretation of the chemical signatures found in activity areas lies in our ability to view the soils as a complex medium and our ability to remain flexible in our method selection for each individual site. Collins and Shapiro (1987) compared anthropogenically modified soils and natural soils in Florida and found that total phosphorus was lower in archaeological soils where extractable phosphorus was high. They concluded that the use of total

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phosphorus as an indication of activity areas would not work in areas with naturally high phosphorus. This confirms a point implied by Terry et al., (2000) and mentioned by Middleton and Price (1996) that often the extractable portion is too low compared with the total element; that the natural variations of the soil can minimize the effect of the phosphorus test as an interpretive tool.

### LOCATION

#### Piedras Negras

Piedras Negras is a Classic period (c. A.D. 250-850) Maya center located in the western reaches of the bank of the Petén jungle on the eastern bands of the Usumacinta River in present-day Guatemala. The site lies on a Cretaceous carbonate rock plateau in a region of tropical semi-evergreen rainforest characterized by a tall broadleaf canopy made up of hardwoods, such as mahogany and cedar and an understory primarily composed of a variety of palm species (Breedlove 1973; Lundell 1937; Wells, et al. 2000; see also Beach and Dunning 1995). The area around Piedras Negras falls in the humid tropical climate with annual rainfall up to 200 cm and falls in the Afwg classification of the Köeppen system (Lee and Hayden 1988). Soils of the area are generally well drained and fertile and consist principally of Lithic and Cumulic Ustirendolls and Vertic Argiustolls which formed from the underlying Cretaceous carbonate rock plateau (Aliphat 1996).

The site was first recorded and explored by Teobert Maler (Maler 1901: 40). Throughout the 1930s, Mason and Satterthwaite (Mason 1933; Satterthwaite 1936, 1943)

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of the University Museum of the University of Pennsylvania recorded extraordinary monumental sculpture and layout of the site, but their excavations brought little light on settlement patterns, modest structures or ceramics and other artifacts of the site (Satterthwaite 1936, 1943, 1952; cf. Houston, et al. 1997). From the rich epigraphic record throughout Piedras Negras came the stunning detection of the historical content of Maya writing by Tatiana Proskouriakoff (1960, cf. Houston, et al. 1998) which led to breakthroughs in Maya writing. More recent research, that improves our knowledge of ceramics and caching practices (Coe 1959; Holley 1983), has brought these earlier studies into sharper focus (Wells, et al. 2000).

Following a series of heavy warfare between Piedras Negras, having suffered losses to their rival, Yaxchilan, was slowly abandoned leaving a succession of squatters inhabiting the once glorious city (Houston, et al. 1999). The gradual abandonment of Piedras Negras brought about a displacement of artifacts from their original location, thus making the interpretation of activity areas difficult.

In 1997, Brigham Young University and the Universidad del Valle began a planned five-year study of this Classic Maya city. Archaeological investigations have included expansive excavations in more modest, non-elite structures at the site. These buildings tended to cluster in sectors or 'barrios' compacted between ceremonial plazas, monumental buildings, and the river bank. They have distinctive chronology and architectural patterns, suggesting variable functions during their occupation. Some, such as the N/O group (the residential group in the O and N quadrants of the Piedras Negras

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map created by the University of Pennsylvania in the 1930's), are hypothesized to have served as service areas for the royal palace of Piedras Negras (Houston, et al. 1999). The project has focused its excavation efforts on numerous residential and ceremonial areas within the city. To date, excavations in these locations have produced data on household size and composition through the excavation of human burials, production activities revealed by artifact distribution, and residential growth documented by the construction stages of domestic buildings.

The location of Piedras Negras was very beneficial to the success of the studies performed. Because it has been set apart as a biological preserve, there has been little human occupation since the time of abandonment. Another advantage of the location of Piedras Negras lies in its geography. Since the time of ancient occupation, there has been very little erosion, deposition, or soil development. Soil development from a patio where erosion and deposition are unlikely was measured at approximately 11 cm over the past millennium. These two advantages imply little chemical contamination of the soil and the accessibility of the occupational soil horizons.

#### Joya de Cerén

Joya de Cerén, often described as a Mesoamerican Pompeii, was buried by several meters of volcanic ash approximately 600 A.D. From the artifact data, we know that they had enough warning that all of the inhabitants were able to escape. Fortunately, the former inhabitants left everything as it was as they fled. The coating of hot wet ash

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and the quick burial of the site preserved many artifacts that would normally have decomposed hundreds of years ago. Over the past 10 years, soil and floor samples have been collected during excavation for future analysis. These soil samples were collected from areas of confirmed ancient human activity. There are samples of the maize-planted agricultural areas, walkways between buildings, a midden and other areas associated with known activities. This near complete preservation of architecture and artifacts in situ allows a greater capacity in the reconstruction of activity areas than is generally plausible. Cerén is invaluable because of the extent of artifact preservation and accessibility of samples, providing the rare opportunity to study the behavioral processes that link them (Schiffer 1987; Webster, et al. 1997). It is atypical that the archaeologist has the opportunity to analyze soils associated with known ancient activities. These circumstances allow us to test the strength of soil chemical data in the analyses of activity areas and space use.

#### Aguateca

Aguateca is located in the Petexbatún region of the Southwestern Petén, Guatemala. After the pioneering studies by Graham (1967) and Houston (1987, 1993; Houston and Mathews 1985), Takeshi Inomata of Yale University conducted systematic research from 1990 to 1993 as part of the Petexbatún Regional Archaeological Project under the direction of Arthur Demarest. Their research demonstrated that a series of defensive walls were constructed at Aguateca at the end of the Classic period as a response to the intensification of warfare, and the center was finally attacked and brought down by enemies at the beginning of the ninth century. The central part of Aguateca was burned, and the residents fled or were taken away leaving most of the belongings behind (Demarest et al. 1991, 1992, 1997; Inomata 1995, 1997, n.d.a, n.d.b; Inomata and Stiver 1994, 1998; Inomata et al. 1993; Valdés 1993).

Inomata started the Aguateca Archaeological Project in 1996 with the specific objective of studying household organization and domestic activities, taking advantage of rich floor assemblages resulted from rapid abandonment. Project members excavated several residential structures. The results of excavation show that the occupants carried out a wide range of activities, including food preparation, storage and consumption, as well as craft production and administrative work (Inomata n.d.c; Inomata and Triadan n.d.a, n.d.b; Inomata et al. 1996, 1997a, 1997b, 1998; Ponciano et al. 1998; Webster and Inomata n.d.).

This study is focused on three residential areas that were thoroughly excavated during the first three years of the archaeological project. Operation 33 is an elite residential area located in the southeast of Piedras Negras. Operation 23 is an elite residential area found near several large temples. This structure is thought to have been the residence of one of the early governors of Piedras Negras (Houston, et al. 1997). The O/N group is a residential group in an area that was thought to have housed the King's servants in the later years of Piedras Negras' glory (Houston, et al. 1998).

## ANALYTICAL PROCEDURES

A broad range of chemical signatures was analyzed from the soil samples collected. The chemical procedures tested each sample for extractable phosphate, total phosphate, organic matter, pH, extractable metals, and total metals.

### *Extractable phosphate procedure*

The primary method of extractable phosphate analysis we used is based on the Mehlich II extraction solution and Hach reagents (Hach, Co., Loveland, CO)(for a methodological comparison see Terry et al. 1999). The key advantage of this method for this study is that it is a quantitative field test that can easily be taken to a remote area. One of the obstacles is the source of pure water for field analysis. Water from the river is purified by passing it through a series of activated charcoal (Barnstead #D0813) an deionization (Barnstead #D0803) columns. This provides water that is sufficiently pure for phosphate analysis. Another observation by early researchers of this method (Terry, et al. 2000) is that the high water temperature can affect the phosphate analysis. The water temperature of the water in the storage bottles and purification columns reached as high as 35° C in the afternoon. The most effective laboratory work was conducted before 10 A.M. where the water temperature ranged between 24° and 29° C.

Two grams of air dried, sieved (<2 mm) floor or soil sample were placed in one of six 50 ml jars attached to a board for facilitation of simultaneous processing of multiple samples. Each soil sample was extracted with twenty ml of the Mehlich II solution for 5 minutes. The samples are then filtered and the filtrate collected in clean 50 ml jars. One ml of the extract is dispensed to a vial, diluted to 10 ml, and the contents of a PhosVer 3 powder pillow are added to the vial. The sample is shaken by hand for exactly one minute and allowed to stand an additional 4 minutes for color development. The concentration of phosphate in the samples is determined on a Hach DR 700 spectrophotometer at a wavelength of 880 nm. A control sample was selected and analyzed with each run. All glassware was washed with Liqui-Nox<sup>®</sup> cleaning solution and rinsed with HCl and distilled water following each run. A more detailed description of the procedure can be found in Terry et al. (2000).

A standard curve was performed frequently throughout the testing to calibrate the spectrophotometer and ensure accuracy of the results. A solution of 100 ppm P was made mixing 0.4394 g of  $KH_2PO_4$  to a total of 1 liter with distilled water. This solution was further diluted to 1.5 ppm P for standard curve concentrations. An example of one of the standard curves with P concentrations and dilutions is found in the appendix (Table X).

### Extractable trace metal analysis

Samples were analyzed for extractable trace metal concentrations using the DTPA (diethylenetriaminepentaacetic acid) extraction procedure developed by Lindsay and Norvell (1978). A chelate (DTPA) extraction of the highly calcareous soils and floors of Piedras Negras was appropriate for trace metal analysis because the procedure avoids the complete dissolution of CaCO<sub>3</sub>, therefore only the trace elements that were deposited in the soil and adsorbed on the surfaces of soil particles were removed. In this procedure, 10 g of air dried, sieved (<2mm) soil is mixed with 20 ml of 0.005 M DTPA solution buffered at pH 7.3 to extract the metals from the soil. The samples are then shaken for 2 hours, after which the extracting solution is separated from the soil by centrifugation and filtration. For the present analysis, the concentrations of barium (Ba), cadmium (Cd), copper (Cu), iron (Fe), mercury (Hg), manganese (Mn), lead (Pb), and zinc (Zn) were determined simultaneously on a Thermo Jarrell Ash ICP spectrometer (see Linderholm and Lundberg 1994). A standard for the ICP was made using VWR brand 1000 ppm atomic absorption standards for each element diluted in DTPA extracting solution. We used DTPA solution as a blank. A control sample was selected and analyzed with each run. All glassware was washed with Liqui-Nox<sup>®</sup> cleaning solution and rinsed with HCl and distilled water following each run.

### Total trace metal analysis

A total trace metal analysis of the soil samples was also performed for

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comparison using the double acid procedure (see Entwistle, et al. 1997, 1998). Soil samples were ground to 40 mesh (425 micrometers) in a mortar and pestle. Five ml of concentrated nitric acid was used to digest a 0.5 g portion of the soil sample. Due to the high calcium carbonate of the samples, nitric acid was added in two ml and then three ml aliquots to reduce overflow of sample from the exceptional effervescence. The sample was allowed to sit overnight and then heated in a ventilating hood digestion block at approximately 200° C to burn off the organic material. Once the organic material was burnt off, 3 ml of concentrated perchloric acid was added to the cooled solution. The mixture was then heated in the digestion block at approximately 200° C for approximately 1 h. The mixture was diluted, agitated and allowed to settle prior to ICP analysis. A standard for the ICP was made using VWR brand 1000 ppm atomic absorption standards for each element diluted in distilled water. Distilled water was used as a blank. Total phosphorus was measured colorimetrically from this extract (see Sommers and Nelson 1972). A control sample was selected and analyzed with each run to test the reproducibility of the analysis. All glassware was washed with Liqui-Nox® cleaning solution and rinsed with HCl and distilled water following each run.

### Soil Organic Matter

Soil organic matter was determined using the Walkley and Black (1934) method (see Nelson and Sommers 1996). Due to the high organic content of the soil samples, the amount of soil analyzed was reduced. Soil samples were finely ground to 40 mesh (425 micormeters) to ensure good representation of the sample in a mortar and pestle. Ten ml of  $1.0 \text{ N K}_2\text{Cr}_2\text{O}_7$  was added to 0.5 g of each sample in a 500 ml Erlenmeyer flask. A blank was prepared by adding 10 ml of  $1.0 \text{ N K}_2\text{Cr}_2\text{O}_7$  to an empty 500 ml Erlenmeyer flask. Twenty ml of concentrated H<sub>2</sub>SO<sub>4</sub> was added to each of the samples and blanks using a Brinkman dispenser under a ventilation hood. The mixture was immediately swirled for one minute. The flasks were allowed to cool for 30 min and diluted with 100 ml of distilled water. An indicator, 0.5 ml of 1,10-phenanthroline ferrous sulfate complex, was added to each flask. The samples and blank were titrated using 1.0 N ferrous sulfate.

Note: The 1.0 N ferrous sulfate is sensitive to light and was stored in a foil covered flask. Potassium dichromate is a hazardous waste and was disposed of properly.

A control sample was selected and analyzed with each run. All glassware was washed with Liqui-Nox<sup>®</sup> cleaning solution and rinsed with HCl and distilled water following each run.

#### pH Analysis

Five grams of air dried, sieved (<2mm) soil was weighed. Five ml of distilled water was added to each sample and allowed to sit for 1 h following vigorous mixing. After 1 h, the pH of each sample was measured using glass electrode. A control sample was selected and analyzed with each set of samples to ensure reproducibility.

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### *Controls*

In order to ensure the accuracy and reproducibility of the results obtained, several control measures were taken. With every run of the above analyses, a pre-selected sample was included with other samples. These control samples were generally selected from samples collected from the sites to ensure uniform soil properties between the test samples and the controls. Three standardized controls were also used to help determine the accuracy of the methods used. NIST certified standard samples 'Montana 2710' and 'Montana 2711' were purchased to fine-tune our methods and procedures. Sample ARDFA 97104 was obtained from the BYU Soil and Plant Analysis Laboratory and used as a standard in running some of the analysis. When switching control samples, there was always overlap of the successor sample with the previously used control.

For the phosphate analysis, we used three different samples, 33C 4-3 was a sample collected from a residential site within Piedras Negras in the 'U' sector of the Penn map. Due to limited sample, a soil sample (labeled 'Timp Loam') collected at the BYU farm was used as a control for remaining analysis. The control used for the analysis of pH levels in the soil samples was the Timp Loam sample collected from the BYU Farm. The control samples used with the DTPA extraction of heavy metals were samples 25 C-2 from the 'R' sector, and sample 33b 22-3 from the 'U' sector of Piedras Negras.

## **METHODS**

**Objective 1**: Investigate the use of soil chemical patterns to prospect for residential middens and perform a statistical analysis of the relationship of soil phosphate signatures to the distribution of domestic activities as evidenced in the artifact assemblage recovered in the O/N, J, and C residential groups of Piedras Negras, Guatemala.

Surface (0-5 cm) Soil samples were collected targeted from the residential housemound groups in quadrants "O", "N", "C", and "J" within Piedras Negras. Grid size varied depending on the desired detail of each particular area and the size of the individual site, but did not exceed 5 m. Grids were established using a compass and measuring tape. Over four hundred soil samples were collected from these groups and analyzed for extractable phosphate signatures indicative of known activities dealing with food consumption, preparation, storage, and disposal. The "C" group was most likely a non-royal elite residential group located in the northern valley upon entering Piedras Negras. A total of 62 soil samples were collected on a 5-m grid for the purpose of testing the phosphate midden prospecting method. The Northwest Court (those structures associated with J-24, -25 and -26) of the Acropolis appears to have been an elite residential group, though such an interpretation is tentative. It was sampled in a 2-m grid. A total of 92 samples were taken from the platforms and patios of this area. The "O/N" residential group was sampled using a 5-m grid. Over 250 soil samples were collected from this area.

Subsequent to the analysis of soil phosphate content, test pits were placed in areas of varying phosphate concentration throughout the "C" and Northwest Court groups. Phosphate concentration was plotted against ceramic density recovered from each test pit to determine effectiveness of extractable phosphate as a residential midden prospecting tool. Some of the test pits in the O/N residential group were placed in areas of high phosphate concentration for the determination of refuse middens while other test pits were placed according to structural and archaeological interest. Archaeological collaboration on this objective was gratefully received from Charles Golden of the University of Pennsylvania.

Phosphate concentration was plotted against ceramic density as an indicator of a residential midden. The dimensions of each test pit was used along with ceramic weight to determine the ceramic density per cubic meter. The data was then analyzed using SPSS software to determine efficacy of extractable phosphate method in locating residential middens.

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**Objective 2**: Investigate the use of soil chemical patterns to locate activity areas involved in food preparation and disposal, sweeping patterns, and workshop activities and to identify specific chemical patterns related to detailed activity verified through the distribution of activity-diagnostic artifacts in the "U" strip excavated residential group of Piedras Negras, Guatemala, through factor analysis. A statistical analysis was also used to compare and contrast two current methods of heavy metal determination used in archaeology (total vs. extractable) with respect to activity areas in a strip-excavated residential group (operation 33).

Soil samples for this objective were collected from the residential site in Piedras Negras on a grid following archaeological excavations. Grid size varied depending on the desired detail of each particular area (from suspected activities) and the excavation methods used. Surface soil samples (0-10 cm) from Operation 33 (N=91) ("U" residential group) were collected at intervals of 2 m with additional samples collected in areas of suspected activity. The archaeological site was subject to extensive horizontal strip excavation with detailed record of artifact data recovered. Soil samples from this site was air dried and sieved to 2 mm and transported to the Brigham Young University soil and plant analysis lab for chemical analysis. Archaeological collaboration for this objective was gratefully received from Zachary Nelson, of Brigham Young University.

A geostatistical analysis was performed on the patterns resulting from the

chemical analysis and a map was interpolated using [Golden (Surfer) software]. A correlation matrix was analyzed between chemical signatures using SPSS software. Chemical data that was significantly correlated was reduced using factor analysis (principal components analysis) to generate a three dimensional map of chemical trends. By using regression analysis to compare each individual element with the group of metals to which it corresponds from factor analysis, we were able to determine areas where variation of individual chemical signatures was inconsistent. The assumption is that these areas were chemically enhanced through activities. Similar chemical signatures throughout the site might indicate specific activities. We plotted the standardized residual of the regression of the single variable with the reduced variable to find areas of enhanced chemical concentration. These areas were compared to the artifact distribution and architecture for the interpretation of archaeological activities.

**Objective 3**: Analyze the patterns of soil signatures related to known activities in an ethnographic context of several residential areas near Aguateca, and Las Pozas, Guatemala, and compare these signatures with chemical patterns found in the archaeological sites of Cerén, El Salvador, and Piedras Negras, Guatemala.

Las Pozas is comprised of indigenous Kekchi Maya that emigrated from the northeast of Guatemala (Beach and Dunning, 1995). The house in the village of Las Pozas selected for this study was that of Francisco Xe (N=268). Soil samples were taken from inside the structures as well as from the surrounding patio and garden areas of these two houses. The pattern of soil collection was generally a two meter grid with exception in areas of expected activity where the grid density was increased. Specific activities areas such as food preparation, disposal, consumption, etc... were carefully mapped. Fabian Fernandez, of Brigham Young University, was one of the main proponents of this objective.

The significance of this portion of the study is that it has the potential to carry the results of the ethnographic study to applications in the interpretation of ancient activity areas. Due to the astonishing preservation of the archaeological site of Cerén, there is a lot of information regarding activity areas that would normally be lost. The hot volcanic ash conserved many items and made casts of others that normally would have decomposed centuries ago. Due to the rapid abandonment of the site, the artifacts were left in their loci of utilization. This site serves as a link between a modern site where activities are known and an ancient site. Using this information, we can compare the chemical patterns of these two sites to a site where artifact information regarding is scarce, such as Operation 33 of Piedras Negras.

A geostatistical analysis was performed on the patterns resulting from the chemical analysis and a map was interpolated using [Golden (Surfer) software]. This map was used to indicate areas of possible activity. A nonhierarchical cluster analysis was performed on the data of the three sites to determine areas of specific activity.

### RESULTS

One control sample was analyzed for every set of soil samples analyzed for Mehlich extractable P. Control sample PN 33C 4-3 was analyzed 106 times with test samples and had an average P concentration of 44 mg/kg over the course of the study. The standard deviation for this sample was 7.39. There was a 17 % relative standard deviation with this sample. Control sample "Timp loam" was analyzed 17 times with the test samples of this study. The mean concentration of P was 82 mg/kg with a standard deviation of 5.43. There was a 6.5 % relative standard deviation with this sample.

Similar to phosphate analysis, a control sample was analyzed for each run of DTPA extractable metals for the test samples. Sample PN 25 C-2 was analyzed 28 times along with the test samples for this study.

The pH analysis was done on a "Timp loam" sample collected from the BYU

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farm in Spanish Fork, Utah. The pH of sample was measured 26 times with an average pH of 7.84. The standard deviation of the pH of this sample was 0.048 with a relative standard deviation of 0.6 percent.

The discussion of the results of the analyses of the soil samples involved in each of the specific objectives mentioned above are contained in the individual papers addressing each one of the major objectives. There are, however, several general trends that were observed throughout the course of the fulfillment of these objectives. One observation that is important for facilitating this research is the amount of soil development that has occurred over the past 1300 years in this area. Soil development in areas of little to no erosion from the surface leaf layer to the occupational level ran between 10 to 15 centimeters, or about one cm per century (Fernandez, et al., 2001). The proximity of the level of occupation to the surface made it easier to access the chemically altered soils. The minimal development of topsoil also promoted strip excavations of several residential sites, allowing deeper insight into possible activity areas.

Approximately 1500 soil samples were collected from areas within Piedras Negras as well as some of the outlying rural groups. Throughout the area of Piedras Negras, we found a general trend of high background levels of phosphate within the city and low background levels in the rural areas. Residential sites outside of Piedras Negras (N = 200) tended to have a mean phosphorus concentration of approximately 13 ppm, while residential areas tested within the city (N = 662) had an average background level of 61 ppm. The ritual structures that were sampled had a lower average background

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concentration of phosphorus than the residential structures within Piedras Negras. The average background level of extractable soil phosphorus among the sweatbaths (N = 601) was approximately 24 ppm. This would indicate that the increased concentration of phosphate may be a result of intense use of resources within the city. The background level of phosphate in the residential areas, where food resources would be most used was the highest. Residential rural sites in the valley to the north as well as the valley to the south had lower concentrations of phosphate. There are several possible explanations for this lower concentration of phosphate. One likely reason is the sparsity of these rural sites allowed easier removal of refuse outside the proximity of the house group. The disposal of this refuse may have served as organic fertilizer for crops grown in these areas. Another is the possibility of these being temporary or structures that were briefly inhabited, thus lacking the accumulation found in the city.

We found that kitchen areas tended to have higher concentrations of phosphorus, iron, and a few other heavy metals associated with them. Kitchen areas of archaeological sites were interpreted through the spacial association of useware artifacts. Craft production areas were generally high in heavy metals while generally low in phosphorus. We also observed that the method used to measure the amount of phosphate in the soil was sensitive enough to determine areas of sweeping patterns.

### CONCLUSIONS

Piedras Negras was an ideal geographic setting for this study for several reasons. First, due to the lack of post occupational anthropogenic disturbance, there was little concern with contamination or misrepresentation of sample results. There has been little to no agricultural contamination of the soils due to fertilizers often applied. Second, the high levels of calcium carbonate from the parent material underlying the area served to fix much of the chemicals of interest, such as phosphorus. Finally, the low levels of soil development since the time of habitation provided easy access to the occupational level for sample collection.

Soil phosphate analysis proved invaluable to archaeological research in that it enhanced the interpretive ability of the researchers. Not only are major areas of activity, such as refuse disposal and food preparation, detectable through chemical analysis, but also minor areas of activity such as sweeping patterns and ritual activities. Chemical analysis may also serve to indicate long vanished structural components such as drip lines. Another possibility of phosphate analysis in archaeology is the indication of length of habitation or intensity of habitation of an area. From the differences in the background levels of residential sites inside the city and those outside the city, we found that the background level increased with long-term occupation or intense occupation. The duration of occupation based on the difference between background and midden samples may be a future application of phosphate analysis.

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