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Methods of soil P analysis in archaeology

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Abstract

Phosphorus (P) is unique among the elements in being a sensitive and persistent indicator of human activity. It has long been of interest to archaeologists because of its potential to inform them about the presence of past human occupation and to offer clues regarding the type and intensity of human activity. A wide variety of methods have been developed in both soil science and in archaeology to extract and measure soil P, resulting in a tremendous amount of data and a wide array of interpretations, but also considerable confusion over appropriateness of methods and terminology. The primary purpose of this paper is to address these issues by clarifying soil P analyses. Anthropogenic additions of phosphorus to the soil come from human refuse and waste, burials, the products of animal husbandry in barns, pens, and on livestock paths, or intentional enrichment from soil fertilizer. Once added to the soil, phosphorus in its common form as phosphate is stable and generally immobile in soils. Soil P comes in many forms, organized for the purposes of this paper on the basis of extraction and measurement procedures as (1) extraction for available P (Pav); (2) portable field techniques (the spot test or ring test); (3) chemical digestion of a soil sample for total P (Ptot); (4) extractions of inorganic P (Pin) for fractionation studies and extractions to look at individual compounds of P; (5) measurements of organic P (Porg); and (6) extractions for total elemental analysis. To compare the suitability of various extractants as the "best" indicator of human input and activity we subjected samples from three very different archaeological sites (Lubbock Lake, TX; Hulburt Creek, IA; British Camp, WA) to four methods of soil P extraction: perchloric acid digestion (Ptot), sulfuric–nitric acid extraction Ptot), hydrochloric acid extraction after ignition (Pin), and citric acid extraction (Pav). Further, methods of measurement were compared via colorimetry vs. Inductively Coupled Plasma (ICP) spectrometry, and the two methods of supposed "total P" were both measured via ICP. In general, the stronger extractants yielded more soil P, but the result are not clear-cut. Likely variables include the intensity of occupation, nature of the parent material, and postdepositional weathering (e.g., the addition of dust).

Keywords: Phosphate; Soil phosphorus; Lubbock Lake site; Hulburt Creek site; British Camp site

1. Introduction

Phosphorus (P) is an archaeologically significant indicator of human activity among agricultural and pre-agricultural societies. Many elements are left in the soil by humans (e.g. [65,150,235,245]); but few are as ubiquitous, as sensitive, and as persistent of an indicator of human activity as phosphorus. As a result, the analysis of phosphorus has long been of interest to archaeologists as a means of detecting and interpreting evidence for human activity. The literature on the topic of archaeological P is vast (e.g., Tables 1 and 2) and likely to be significantly larger than any other single aspect of soil science in archaeology. Moreover, phosphorus is important in plant growth and has been the topic of considerable research in soil science, resulting in another very large literature (e.g. [201]).

A wide variety of methods have been developed in both soil science and in archaeology to extract and measure soil P. The result is a tremendous amount of data and a wide array of interpretations, but also considerable confusion over the

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Table 1
Soil P, selected general references in soil chemistry and archaeology[a]

Type of study	References
Phosphorus chemistry in soils	[129 (ch9)[b],23,30,31,48,83[c],86,104,118, 147,148,172[b],192,204,205,217 (ch9)[b], 222,223,232,233,242]
Early investigations	[2–4,35,44,49,56,129–132,142,210]
Summaries and reviews	[18[b,c],28(ch4),35,58,59[b],60[c],61,80[c], 82[b],91 (ch9)[b],122,175[b],176,185,202[b], 224,238,243[c],244[b,c],245]
Methods for archaeology	[20,57–59[b],60[c],61,63–65,82[b],84,110[d], 123[e],127,176[b],179,187,224,229,238,243,244]

[a] Modified from Holliday [96, table 11.4].
[b] Good review discussion of soil P and/or archaeological P.
[c] Extensive list of references.
[d] Good historical review of methods.
[e] Based largely on the work of Eidt [57,58].

terminology, and the appropriateness and meaning of individual procedures. The primary purpose of this paper is to address these issues by clarifying soil P analyses. This will be done in several ways, following significant revision of a discussion by Holliday [96, pp. 304–314, 343–362]. First is a set of review discussions of the sources of anthropogenic P, the basic chemistry of soil P, a brief history of soil P research in archaeology, and soil P dynamics. This is followed by an in-depth review of the most common methods of soil P analysis in soil science and especially in archaeology. That section is followed by a review of some comparative studies of the various methods in archaeological contexts, and then a presentation and discussion of comparative data generated by the authors. Comparative studies of soil P analyses are important as a means of understanding the implications of soil P data. Most such studies are based on analyses of samples from one site using several different methods. We took the approach a step further by subjecting samples from very different archaeological sites to a battery of soil P analyses. Finally, throughout the discussion, we attempt to clarify the often bewildering array of terminology used in soil P studies.

Table 2
Selected case studies of soil P in Archaeology[a]

Type of study	References
North America	[1,17,20,28,32,35,39,52,56,74,76,77, 88–90,99,101,111,115,116,124,144, 153,182,185,186,189,203,220,221, 229,245,246]
Central America	[6,35,37,53,139,140,157,169,170, 207,224[b],237[c]]
South America	[59,61,110,118,126,128,154,180,181]
Europe	[5,29,34,38,39,42–46,50,51,63–65, 80–82,110,122,124,125,127,135,138, 160–162,164,174,176,177,179,183, 193–196,231]
Africa	[84,124]
Asia/Pacific	[24,141,166,219,239,240]

[a] Modified from Holliday [96, table 11.4].
[b] Good review of chemical analyses of anthrosols.
[c] Good review of P studies in Mesoamerican archaeology.

2. Anthropogenic phosphorus

General sources of anthropogenic phosphorus among pre-Industrial era peoples include: human waste; refuse, especially organic discard derived from bone, meat, fish, and plants; burials; and ash from fires [18,59 (pp. 29–30),175,176]. The development of agricultural economies necessitated fertilization in many areas, with techniques ranging from burning and the use of "green manures" to the application of guano, human waste, animal products, and chemical fertilizers (see Miller and Gleason [152] for a review discussion of fertilizer in archaeological contexts). The products of animal husbandry may also be added to fields as well as accumulate in barns, pens, and on livestock paths. While the above may add significant amounts of phosphorus to archaeological deposits, as detailed in a later section, many human activities either do not affect or may deplete soil phosphorus levels.

The most common chemical elements affected by human activity are carbon, nitrogen, sodium, phosphorus, and calcium, with lesser amounts of potassium, magnesium, sulphur, copper, zinc, and other metals [35 (pp. 1–3),59 (pp. 25–27),60,245 (pp. 1396–1399)]. These elements can be used as indicators of past human activity (e.g. [65,189,235,245]). Phosphorus in its common form as phosphate,[1] however, is often less susceptible than most of these elements to leaching, oxidation, reduction or plant uptake [28 (pp. 127–131), 58]; with the nature and rates of transformations or losses from the soil determined by local biological and pedological processes.

When people add P to the soil as organic products or inorganic compounds, the P quickly bonds with Fe, Al, or Ca ions (depending on local chemical conditions, particularly pH and microbial activity) to form relatively stable chemical compounds of inorganic phosphate minerals and organic phosphate esters [18,175]. Some forms of soil P are highly resistant to normal oxidation, reduction, or leaching processes [58–60,175]. When humans add P to the soil, therefore, it often accumulates at the site of the deposition. With prolonged occupation, the accumulation of anthropogenic P can become quite large (by orders of magnitude) in comparison to the content of natural P in the soil. This is because P is one element that is cycled mainly in geological time [58 (p. 1327), 233], while many other elements are often cycled much more rapidly.

Although some forms of soil phosphorus are relatively stable in terms of biochemical weathering, as detailed in a later section, soil P transformations and leaching have been documented in sandy soils, some redoximorphic soils, neutral pH soils, and in soils with certain organic amendments or regimes of land-use. Natural and cultural processes may redistribute or remove particles that host P compounds. There are also complex relationships between the forms of P, landscape position, time, and total soil phosphorus content [205,233]. Nevertheless, in many relatively undisturbed settings, people can add

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[1] Phosphorus exists in soils as the phosphate ion. Some organic P compounds are not phosphates, however, so the term "phosphorus" should be used when referring to total soil P [18, p. 5].

so much P to occupation surfaces that the above may not compromise P analysis.

The relationship between natural and anthropogenic P in the soil and the movement of P through natural and cultural environments is nicely encapsulated by Bethell and Máté [18, p. 9]:

> "Human activities can strongly redistribute P in soils. Plants take up P from the soil. They can be eaten by animals or harvested. The animals themselves can be moved or 'harvested'; they can be enfolded, concentrating P in a particular area. Dung residues can be collected and used as manures, respread over the fields; on the other hand they may be used as a fuel, as a walling material, or ignored…As part of the produce of an economic system, P is very mobile; it's importance lies in the strong fixative powers of the soil. When P enters the soil system it is relatively immobile compared to other elements concentrated by the activities of humans."

Another factor which makes P suitable for geoarchaeological study is that anthropogenic P can exist in the pH range of most soils. Under acidic condition, P combines with iron and aluminum, whereas under basic conditions, P combines with calcium. Consequently, soil P analysis can be used successfully in a wide variety of archaeological contexts. Indeed, as described below, where there is little or no surface evidence of human occupation, soil P analysis may be an appropriate tool for detecting traces of human activity, and for determining the particular form and function associated with that presence, though many caveats apply.

3. Soil P basics

Soil P chemistry is very complex and many aspects of it are poorly understood. Further, research by archaeologists, geoarchaeologists, and soil chemists since the middle of the 20th Century has resulted in a bewildering array of terms for referring to soil P. In part this is because of the various forms of phosphorus in the soil, and in part because of the different chemical fractions that can be extracted both in field tests and in lab analyses. The resulting nomenclature refers to P in terms of its chemistry (e.g. organic P, inorganic P, total P), its place in a biogeochemical cycle (e.g., labile P, available P, occluded P), or in terms of sequential extraction (e.g. Ca-P, recalcitrant P). Understanding the difference between the forms of P and the extractions or fractions of P is a key to understanding soil P in archaeological contexts. The discussions below sort through the basic chemistry of soil P and soil P dynamics. The following section then focuses on the laboratory methods for extracting P and the nature of the extractants.

Fig. 1 is an attempt to organize the different forms of soil P and their common linkages in the soil P cycle. There are many classifications of soil P and depictions of the soil P cycle. The nomenclature and even the categorization of P forms vary significantly [41, pp. 204–206]. Fig. 1 is thus highly selective, though we believe it highlights those components and pathways of the soil P cycle that are of primary interest to most archaeologists. We adopted the basic schema used by Stevenson and Cole [217, p. 292] and Bethell and Máté [18] as they are reasonably descriptive in terms of chemistry and the placement of P compounds within a soil. Iyamuremye and Dick [103, pp. 140–144] well summarize the different forms of organic P and the importance of microorganisms in soil P transformations. Modifications of the Hedley fractionation procedure (I, II, III in the left column of Fig. 1) ([86] discussed below) seem to differentiate between soil P "pools", or groupings of soil P types based on activity levels, though uncertainties remain. Lehmann et al. [118, pp. 115–118] offer a concise summary of the general links between the chemical extractants used in fractionation studies and soil P pools (see also Beuler et al. [25, p. 868]).

As shown in Fig. 1, both organic and inorganic phosphorus compounds can persist in soils for an extended period of time and occur in a variety of forms. Inorganic soil P (Pin) may be found dissolved in soil solution, as a chemical precipitate, as orthophosphate ions ($H_2PO_4^-$, HPO_4^{2-}, PO_4^{3-}) adsorped onto particle surfaces, or as orthophosphate ions occluded within particles. Sorption refers to the association of a chemical with soil solids, typically the surface of a particle, and is accomplished through chemical and biological mechanisms (see Scow and Johnson [188] for a thorough review of sorption dynamics). Occluded P refers to orthophosphate ions that have become physically incorporated or chemically entrapped within particles, generally clays composed of amorphous hydrated oxides of iron and aluminum or amorphous aluminosilicates. Organic phosphorus (Porg) often accounts for 20–80% of total P (Ptot) and, like Pin, is found dissolved in soil solution and sorped to soil particles. Phosphate esters, especially inositol phosphates, nucleic acid phosphorus, and phospholipids are the most common Porg compounds in soils. Porg esters strongly bound to clay minerals and organic matter are highly stable and may persist in soils for a long time, particularly if they are immobilized or aggregate protected [103, pp. 143–144; 164].

Precipitation, sorption, and occlusion processes are reversible by dissolution and desorption. The extent of P dissolution or desorption is controlled by biological and chemical mechanisms, the equilibria between dissolved P and particulate P, and the type and strength of P bonds to soil particles [103,217]. P adsorped to the surface of a soil particle is directly exposed to microorganisms and chemical compounds and is therefore more likely to dissolve or desorp than occluded P. Weakly sorped P is more likely to break free from a soil particle than strongly sorped P. Physical, chemical, and biological weathering of soil solids may affect P exposure to microorganisms and chemical compounds or the strength of the P bond to a particle. However, the dissolution or desorption of phosphorus does not necessarily result in the loss of soil P. Rather, P commonly "resorpts" to a particle, if P receptor sites are available, or precipitates out again from solution. Dissolution, desorption, and the transformation of soil P forms by microorganisms are also favored by certain soil conditions and human activities as detailed in the next section.

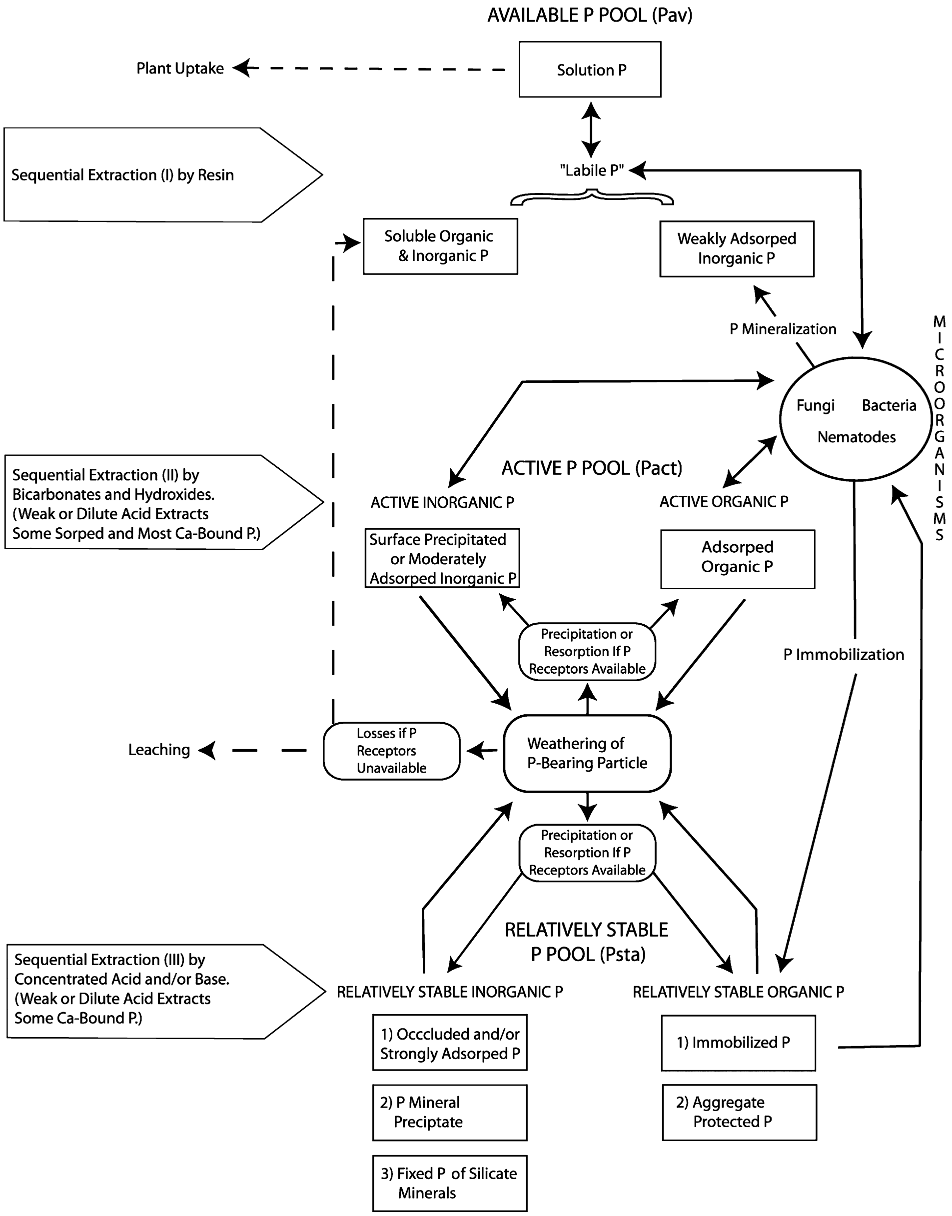

Fig. 1. Selected components and pathways of the soil P cycle. The different forms of soil P, *encased in rectangles*, are grouped into activity pools. Arrows show most major microbial and weathering pathways between soil P forms and/or pools. The left-hand side pentagons show the sequential chemical extraction (I, II, III) of soil P forms based on a highly generalized abstraction of the Hedley procedure [86] (and are not the same as Fractions I, II, and III of Eidt [59]). After Bethel and Máté [18], Iyamuremye and Dick [103], Stephenson and Cole [217], Lehmann et al. [118], and this paper.

Microorganisms are particularly important for the transformation of one soil P form into another. Two of the more important microbial transformations of soil P are mineralization, the breakdown or conversion of Porg forms to “weakly adsorped, inorganic P”, and immobilization, the formation of a relatively stable form of Porg P that is highly resistant to desorption and dissolution (Fig. 1). “Weakly adsorped, inorganic P” readily dissolves and is taken up by plants. Immobilized P can be a significant portion of the Ptot at an archaeological site.

Fig. 1 organizes soil P forms into “activity pools”, though once again uncertainties exist about assigning specific soil P forms to specific activity pools. Soluble and weakly adsorped forms of P, are shown near the top of Fig. 1. They are labeled “Available P Pool” (Pav) in Fig. 1 because they are highly mobile and very susceptible to dissolution, desorption, and transformation. Pav is easily extracted by mild reagents such as resin. Despite its mobility, Pav is often present at archaeological sites (e.g. [186]). We have grouped inorganic and organic forms of soil P with moderately strong bonds to the surface of soil particles into the “Active P Pool” (Pact). P molecules in the Active Pool are susceptible to dissolution, desorption, and transformation with prolonged exposure to microorganisms and weathering and in certain soil environments. Most Pact can be extracted by bicarbonates and hydroxides. The sequential extraction of soil P at archaeology sites (e.g., Fraction I in Lillios [125]) and from anthrosols (e.g., bicarbonate-Pi, bicarbonate-Po, hydroxide Pi, and hydroxide Po in

Lehmann et al. [118] indicate that the Active Pool may comprise the largest proportion of soil P at a site. The "Relatively Stable P Pool" (Psta) consists of inorganic P that is occluded, strongly adsorped, or part of mineral matrices as well as Porg that is immobilized or aggregate protected. Psta is not very susceptible to dissolution, desorption, and transformation. Nevertheless, in certain soil environments, or with prolonged exposure to weathering and microorganisms, Psta may still circulate within the soil P cycle. In comparison to the other soil P pools, concentrated reagents are needed to extract Psta from a sample during laboratory analysis. Psta may also constitute a relatively large percentage of soil P in archaeological sites (e.g., Fraction II from the sequential extraction procedure of Eidt [59], or solutions "C" and "D" from the sequential extraction procedure of Woods [244].

A further comment on terminology of soil P is in order. The terms "available P" and "labile P" are sometimes used interchangeably (e.g. [91, p. 182]) along with terms such as solution P, soluble P, non-fixed P, and non-occluded P. These various forms of P are related but are not necessarily the same thing. Solution P is the P already in solution, but soluble P is the soil P that is soluble but not yet in solution. Solution P is the direct source of P for plants. The soluble P comes from the much larger labile pool. The labile pool includes but is not necessarily the same thing as the non-fixed P or the non-occluded P. Some of these forms of P are more labile or more easily converted to soluble P than other forms. The available P includes the solution P and the labile P [217, pp. 292–293, 294–300].

4. Soil P in archaeology

The study of archaeological phosphorus evolved throughout the 20th century (well summarized by Bethell and Máté [18], Eidt [59], and Woods [243]). Most of the early research was by O. Arrehnius and W. Lorch (Table 1) working in northwestern Europe. After the Second World War, British and American investigators applied some of the evolving methods of soil P analysis (e.g., Solecki, Dauncy, Dietz, and Mattingly and Williams; Table 1). Following this pioneering work, there were several landmark studies that influenced most subsequent phosphorus work. Cook and Heizer [35] published what still remains one of the most comprehensive and extensive studies of soil P, and one of the first multi-element studies in archaeological contexts. They published data on sites in the western U.S. and Mexico. Unfortunately, they do not mention their methods nor do they discuss their results in terms of different forms or fractions of P. However, they provided the first systematic discussion of soil chemistry, including P chemistry, with an archaeological focus, and showed that P in archaeological sites must be considered relative to other elements and to the environments of deposition.

Archaeological P studies gained further attention in the 1970s with the rapid expansion of methods and applications, particularly the work of Eidt [57–60], Eidt and Woods [61], and Woods [243,244], and the review paper by Proudfoot [175]. Of particular significance in this work was the recognition and incorporation of the extensive research into soil P and P fractionation by soil scientists, especially the work of Chang, Jackson, Stevenson, Syers, Smeck, and Walker (Table 1). A more recent soil science development is the Hedley sequential fractionation procedure [86], which segregates both organic and inorganic forms of soil P into stability pools. A modified version of the Hedley fractionation technique [226] has promising, though so far limited, archaeological applications (e.g. [118]).

5. Soil characteristics and soil P dynamics

Our understanding of soil P dynamics at an archaeological site has improved markedly in recent years by: (1) the sequential extraction of various P forms from anthrosols as well as from soils under different land-use regimes; (2) the voluminous research devoted to the influence of environmental factors on P mobilization, which has significant implications for agricultural practices and aquatic ecosystem management; and (3) ethnoarchaeological studies of soil chemistry and human behavior. This research shows that organic amendments to soils, microbial activity, weathering, and land-use all affect the forms, interactions, and redistribution of P compounds. The susceptibility of soil P forms to dissolution, desorption, and transformation is affected by organic matter, pH, soil moisture, particle size, and mineral content. These variables can dramatically affect the interpretation of soil P trends at archaeological sites [42,43].

5.1. Organic matter

The addition of organic matter to a soil significantly affects the forms, interactions, and redistribution of P [103, figure 1]. Organic matter is a major biological and anthropogenic source of soil P. Moreover, the type of organic amendment seems to influence the forms of soil P [25] through biological and chemical cycling.

Organic matter decay stimulates microbial and biotic activity critical to the soil P cycle [103 (pp. 142–144),218]. Bacteria and fungi, for example, respond to particulate organic matter in different ways, with fungal colonization of particulate organic matter dramatically increasing P immobilization [178]. The decay of organic matter releases organic particulates and chemical compounds to the soil that influence the number and availability of P receptor sites [79,103 (pp. 148–154)] as well as the interactions between soil minerals, organic matter, and microorganisms that stabilize soil P [102 (pp. 426–431),218]. The release of organic compounds also affects desorption and dissolution of soil P through a number of chemical reactions [103, pp. 156–167].

Land-use and the type of organic amendment can strongly influence soil P forms and mobility. For example, both the Pav and Pact pools (Fig. 1) may be sensitive to crop rotations [249] and types of leaf litter in agroforestry systems [119]. The decomposition of organic matter may produce phosphate-mobilizing enzymes and chelating agents that replace phosphate with other salts of calcium, iron, or aluminum (sensu Huang [102, pp. 410–418]).

Bronze-Age peoples at Tofts Ness, located in the northeastern Orkney Islands, added grassy turf, human waste, ash, and organic discard to the calcareous wind-blown sand deposits there in order to improve cultivating conditions. These organic inputs significantly elevated phosphorus levels in the remnant cultivated soils there, which were preserved below mounds [195]. Although there is long history of cultivation in Scotland, plaggen-like anthrosols have, thus far, only been reported in some Bronze-Age [195] or in Medieval [194] field systems there. This suggests that only specific organic amendments and manuring strategies will result in a soil with long-term elevated phosphorus levels [33,168]. Indeed, ancient agriculture depleted soil P, and changed other soil properties across much of northwest Europe in ways that continue to affect modern forests [55,230].

Organic discard and microbial action play a significant role in the development of a family of anthrosols in the Amazon basin known as *terra preta* [71,120,143]. Among the more important additions to the soil is black carbon, which is produced by the incomplete combustion of organic matter. *Terra preta* often has an order of magnitude more black carbon than adjacent Oxisols. Black carbon is resistant to chemical and microbial breakdown, elevates soil cation exchange capacity, slowly oxidizes into carboxylic acid which readily forms esters important to organic P chemistry, and may forge organo-mineral complexes with silt and clay sized particles that retain P and other nutrients [72,73]. The importance of black carbon, fish and animal bone discard, and microbial action for the high soil P content of terra preta is documented by thin section and electron microprobe analysis [184].

However, many organic amendments to the soil do not result in elevated levels of soil P [103, pp. 148–154]. Dissolved organic carbon and phosphorus may compete for receptor sites in acidic forest soils [107,108] and in iron- and aluminum-rich humic soils [70]. Ash beds in slash and burn agroforestry may increase the amount of plant-available P near the ground surface over the short-term. Yet fire can destroy organic matter and disrupt illuviation in ways that affects P forms and cycling within deeper mineral horizons over the long-term [27]. These different outcomes show that the specific type of organic amendment [121], the entire suite of chemical compounds released during organic decomposition [79], and the local soil environment [41] determine the forms and amounts of soil P.

5.2. pH

pH has long been known to play a major role in P solubility and precipitation [103, p. 142]. An increase in pH decreases the P-binding capacity of Fe and Al compounds in acidic soils. Under these circumstances, P mobilization is primarily due to ligand exchange reactions where hydroxide ions replace phosphorus at receptor sites [103, pp. 156–159]. In neutral and alkaline soils, an increase in pH increases the number of soil P receptor sites by stimulating calcite and apatite formation. In sum, phosphorus solubility and mobilization is greatest in soils with a neutral pH.

Stable forms of P can be mobilized if there is a change in pH. Butzer [26, p. 156], for example, noted that phosphorus peaks were upwards of 80 cm below major occupation levels at Cueva Morin, Spain due to the mobilization of phosphorus in neutral pH cave sediments.

5.3. Soil moisture

Soil moisture conditions affect soil P solubility and mobilization through chemical and biological mechanisms, although the lack of consensus suggests that many variables are at play [85,103 (pp. 171–176)]. Moderately well-drained soils often retain more Pin than poorly-drained soils in adjacent riparian settings (e.g. [133]). This tendency is partially due to the release of P when Fe^{3+} is reduced to Fe^{2+} during saturation. Soil redox reactions may produce compounds that influence the number of available soil P receptors. Periodic saturation favors the sequestration of nitrogen and carbon in some settings, which in turn affects the forms and amounts of Porg [40]. Soil moisture conditions also affect vegetative decomposition, organic acid formation, and microbial activity [156], which in turn influence P levels, solubility, sorption, mobilization, and resorption [103].

Floodwater sediment was an important source of nutrients, including P, for the fields of many pre-Industrial era agriculturalists. Periodic saturation may have a "buffering effect" that minimizes soil P losses, as shown by the analysis of soils from experimental shifting cultivation plots located in the Amazon River floodplain and adjacent uplands [248]. However, the precise relationships between soil moisture, soil P forms, and soil P cycling there await further study. Soil moisture, along with organic inputs and microbial activity, limited soil degradation in traditional Zuni agricultural fields over long periods, as shown by the analysis of soils from upland and alluvial sites in New Mexico [100,159].

5.4. Particle size and mineralogy

Particle size is directly related to the number and efficacy of P receptors. Clay particles have more surface area per unit mass and are more highly charged than sand. Clay is thus highly reactive in soils. Finely-textured soils have a higher P sorption capacity since surface-precipitated and adsorped P are common in most soils. As aforementioned, clays readily combine with organic compounds to form organo-mineral complexes high in soil P.

Many, but not all clays, are composed of Fe- and Al-hydroxy oxides that readily bind with soil P. Clay mineralogy can be just as important as particle size for P retention, with lower rates of adsorption and desorption associated with kaolinite than with Fe- and Al-hydroxy oxides [171].

Solomon et al. [212] examined the covariation between particle size and soil P forms across a land-use transect of primary forest, degraded woodland, and cultivated fields in northern Tanzania. They sequentially extracted P from individual particle size classes at each site and found that forest clearance and agriculture depleted far more soil P from sands than silts or clays. Both organic and inorganic forms of soil P were depleted from coarsely textured particles. In addition, different forms of Porg exhibited differential rates of mineralization during cultivation [211].

The sequential extraction of P from anthrosols that have formed in calcium- and gypsum-rich sediments in the Maya lowlands well illustrates the influence of parent materials on soil P forms [54, pp. 259–260]. Organic additions of P to ground surfaces are quickly mineralized in limestone-derived soils with a high base saturation there (see also Weisbach et al. [234] for details on the relationship between calcareous parent materials, soil moisture, organic matter, and P sorption). The ancient Maya intensively added organic materials, and in some cases terraced, infields in the Petexbatun region to combat rapid rates of P mineralization. Parent materials may also influence laboratory results. The sequential extraction of soil P produced ambiguous results in samples with very high levels of Ca in the above study of Maya land-use. P can re-bond with free cations such as Ca in the sample extract, if they are not removed during sample pre-treatment. However, strong chemical pre-treatment to eliminate Ca from the sample can also remove Ca-bound phosphates [53, pp. 207].

5.5. Time

Soil P forms can change with the passage of time (e.g. [19, pp. 211–213]). More specifically, there is an overall decrease in Ptot and a general shift, in percentage terms, from Pact to Psta over time [233]. Several innovative P fractionation studies have used this relationship to distinguish between ancient and modern inputs of P [59, pp. 42–43]. Early Bronze-Age settlements in central Portugal had less soil P and a higher ratio of occluded P to "easily extractable P" than Middle Age settlements [125]. A similar soil P relationship differentiates ancient from modern Maya agriculture, although the diverse topographic settings around Chunchucmil are also a source of soil P variation there [13].

6. Human activities and soil P dynamics

Many archaeological studies have been concerned with documenting elevated levels of soil P. However, ethnographic observations, historical data, and experimental archaeology all show that some human activities increase levels of soil P while others decrease it or have no affect on soil P values. Human activity may affect soil P levels at an archaeological site through multiple indirect means. As detailed above, changes in land-use or discard may change the pH or organic matter content of soils and thus affect soil P forms, interactions, and redistribution. The variable distribution of P and other elements across a site, augmented by other data and an understanding of the local soil environment, have proven invaluable for interpreting activity areas at scales ranging from individual rooms (e.g. [231]) to intrasettlement land-use [6] and settlement hierarchies [225].

The cold climate and poor drainage of delta soils in the Arctic favors retention of P and other elements at modern Cupiit Eskimo fishing camps in Alaska [113]. P was an order of magnitude higher in soils below roof-covered fish drying racks at the Chevik site than in nearby control samples. Fish drippings also significantly elevated the levels of K, Mg, Ba, Ca, K, and Na there, while hearths had elevated levels of P, K, and Mg. Yet P was not elevated in the fish smokehouse, in part because of high pedestrian traffic and in part because the mud floor there was refurbished. The use of fire barrels and the voracity of dogs in devouring fish discard also affected P levels at modern Cupiit sites.

Elevated levels of organic matter and soil P have been documented in other Arctic archaeological sites with middens and house basins (e.g. [32]). Since human activities vary, we can expect soil P values to also vary across a site. Natural factors must be taken into account, however. The influence of topography on soil moisture, organic decomposition, and microbial populations has been shown to affect sodium bicarbonate-extractable phosphorus values across ancient Dorset and Thule sites in Arctic Canada [47]. Bird droppings around perch rocks there had soil P levels nearly as high as human habitation areas. The anthropogenic enrichment of soil P affects vegetation – an important component of the P cycle – for many centuries in the Canadian Arctic. Nevertheless, despite the influence of natural factors, villages had between 5 and 7 times more soil P than nearby control areas [47].

Several investigators combined the spatial distribution of organic carbon, Ca, Mg, and P from Iroquoian sites with historic documents to identify activity areas at the scale of the long house and also the village [77,88,89]. Hearths and middens containing ashes are particularly important in the above studies as are the relationships between hearths and families in the multi-family long house. Historic accounts of Iroquoian peoples using fire and other organic amendments to manage soil fertility were confirmed by excavation of the Creemore Corn Hill site, though the affect of such practices on soil P varied widely across the relict fields [87, p. 387].

Luis Barba and his colleagues have long carried out exemplary ethnographic and archaeological studies of soil chemistry and human behavior in different Mesoamerican environments including Tlaxcala [8], Chiapas [7], and the Yucatan [11]. Modern observations of human behavior are combined with maps of P concentrations and those of other inorganic elements such as Ca and Fe, carbonates, soil parameters such as pH and color, and organic compounds such as fatty acids, carbohydrates, and protein-rich albumin residues to chemically characterize activity areas. Despite variation between sites, there are several generalities with respect to P that emerge from this research. Hearths, cooking features, and middens with wood ash generally have elevated levels of P, K, and other elements. Organic residues in food preparation, consumption, and disposal areas also elevate soil P levels. Food preparation may affect many soil characteristics, particularly if *nixtamal* (dough made from lime, maize, and water) is prepared in one place for an extended period of time (see also Middleton and Price [150, pp. 678–679]). Yet, this work also shows that many human activities, both within the house and around the farmstead, do not influence soil P values.

Interdisciplinary research and the remarkable preservation of stucco floors at Teotihuacán, near modern day Mexico City, offer an unusual opportunity to apply the ethnographic observations to archaeological contexts [9,139]. Chemical compounds are readily fixed into the porous matrix of stucco floors, which are chemically "clean" after construction. The authors combined high-interval geophysical, geochemical, archaeological, floral and faunal analyses in each room of a multi-family apartment compound at Teotihuacán. They found high P levels in cooking and animal butchering areas, around patio drains, in most refuse areas, and in certain household cult zones with archaeological evidence for burials, organic offerings, and perhaps the burning of incense. In contrast, a significant portion of the apartment compound, including storage facilities, craft production areas, large sections of house floors, parts of the cult zone, and pathways generally exhibited little or no change in P levels.

The relationships between daily life and soil chemistry in the Maya region are somewhat variable. An ethnoarchaeology and soil chemistry study at the Q'eqchi' Maya village of Las Pozas, Guatemala readily identified food preparation and consumption areas, as well as middens, by their high soil P values [66]. Ash and discard elevated levels of K, Mg, and pH in the kitchen area, while organic table droppings lowered pH in the eating area. High traffic areas exhibited low P and trace element levels. Ethnographic observations and soil analyses of a modern Maya farmstead near San Pedro, Belize, also document elevated soil P levels with food preparation, consumption, and disposal areas [247, pp. 4–8]. At San Pedro, however, the kitchen was the center of social life and there was little spatial segregation of activity areas. The kitchen was the only activity area at San Pedro identifiable by elevated levels of phosphorus, as well as other elements. Paths and courtyards were either phosphate neutral or exhibited slightly lower levels of soil P. Middens did not have a consistent chemical signature, though only a small proportion of the modern trash there was biodegradable over short time periods. Soil P levels were not helpful in identifying structures at San Pedro, unlike Teotihuacán, though a low ratio of Al to Ca consistently predicted structures with marl floors [247, pp. 4–8].

Archaeological research at the Classic Maya site of Piedras Negras shows that variation in soil P levels at site is due to many cultural factors as well as soil P dynamics [169,237]. The "C group" is an elite residential cluster of house-mounds at Pedras Negras. High levels of soil P were associated with organic discard here, with moderate levels in a storage area, and low levels elsewhere. The "J group" is a residential group that also exhibited elevated levels of soil P in midden areas. However, the highest soil P concentrations did not correlate with artifact density there. "J group" middens exhibiting post-occupation soil development, rather than those covered by post-occupation building collapse, had the highest soil P levels [169, p. 865]. The "O & N" residential group is associated with a royal acropolis. The highest soil P levels correlated with the kitchen middens there, while the lowest levels were found near the entrance to the acropolis. Presumably, the low levels are related to courtyard sweeping and the presence of a path.

Interdisciplinary research that includes soil chemistry also refined our understanding of ceremonial life in ancient Mesoamerica [170]. Some ritual activity may deplete soil P due to prescriptions on social behavior and the vigorous cleaning and sweeping of sacred surfaces. For example, an elaborately constructed patio and a painted building (U-17) associated with the royal acropolis at Piedras Negras exhibited comparatively low soil P levels. The painted building also exhibited high levels of heavy metals such as mercury [237, pp. 456–458]; the Maya used mercury sulfide [HgS] to make red pigments. Low levels of soil P and elevated levels of heavy metals have been documented in other painted buildings, such as at the Classic Period Maya site of Cerén.

Ritual activities may significantly elevate soil P levels. Analyses of artifacts and soil elements at the Maya site of El Coyote [235], plus ethnoarchaeological studies [236], suggest that ritual feasts account for the high values of soil P in the southeastern part of the plaza. The low values of K and Ca values in the southeastern portion of the plaza indicate that the food was prepared elsewhere on the site [235, pp. 75–77].

Historic documents indicate that plant and animal products, food and beverage offerings, and human sacrifices were all part of the Aztec rites performed in the *Hall of the Eagle Warriors,* located in the Aztec capital of Tenochtitlán. Barba et al. [10] used a suite of analytical techniques, including P analysis, to map the chemical signatures across stuccoed floors. They found high P values around the stairs and columns of Room 1, around the main altar and a charcoal and ash pit in Room 2, and around the braziers and offerings box in Room 4. P values were also elevated in front of representations of *Mictlantecuhtli* (the Death God) and Eagle Warriors. Room 3 generally had low P values, and P values around the altar there were only slightly elevated. Some corners in rooms throughout the temple and short segments of pedestrian paths were also low in P. Room 2 was a major locus for many Aztec rites described in historic documents, given the high concentration of fatty acids, carbohydrates, and albumin as well as the comparatively low levels of carbonate there.

Historic documents also suggest that relationships between soil P levels and activity areas are not always straightforward. Entwistle et al. [65] found that enrichments of K, Th, Rb, and Cs were often associated with habitation areas on an 18th century Hebridean estate. Elevated levels of Ca and Sr were found in the fields. Soil P levels did not vary in a consistent fashion in either set of activity areas, perhaps because the existence of a croft kailyard and the conversion of byres led to superimposed P signatures from domestic and agricultural activities on the same soil surface.

Experimental archaeology in Britain clearly demonstrates the importance of the local soil environment for P studies. In 1960, researchers built two earthworks in different British environmental settings to investigate the formation of the archaeological record [14]. The earthwork constructed at

Wareham Heath, Dorset, was built with extremely acidic, coarsely textured, Fe-deficient podzols that were generally low in soil P. After 33 years, 200 features with known quantities of bone and other materials were excavated from the pre-mound surface and also the earthwork proper. Only one feature within the earthwork proper had elevated levels of soil P, due to the lack of suitable soil P receptors and the high rates of leaching in the sandy soils of the mound [43]. Earthwork construction had no appreciable affect on soil P levels from the buried pre-mound soil, despite mound construction creating favorable conditions for soil moisture retention, microbial and nematode activity, and the formation of organo-mineral complexes there [136].

Analyses at Butser Farm, a reconstructed Iron Age farmstead near Hampshire, suggest that microscopic crust formation is an import characteristic for P retention in animal stables [137]. Animal waste promotes localized and short-lived anaerobic conditions on those portions of the stable floor which promotes crust formation. P compounds, particularly hydroxyl-apatite, become fixed in a crust consisting of interbedded plant fragments, silt particles, and secondary calcite. Crusts have nearly twice the P values of other portions of the stable floor. Analyses of domestic floors at Butser Farm suggest that the degree of floor compaction and its mineralogical content largely explain the variability in P depletion from surfaces exposed to pedestrian traffic and house cleaning.

7. Soil P methods in archaeological contexts

Macphail et al. [138, p. 72] note that there ‘‘appears to be as many methods of extracting P from the soil as there have been workers in the field.’’ They exaggerate, of course, but a grain of truth lies at the heart of their comment. Over 50 methods are published in general soil chemistry [96, tA2.1], and of these over 30 have been applied in archaeology (Table 3). No comprehensive review is available for soil P methods in archaeology, but very useful summaries of a wide variety of them are provided by Keeley [110], Hamond [82], Eidt [59], Gurney [80], Bethell and Máté [18], and Macphail et al. [138]. In soil science, several very useful compendia of laboratory methods are available, and all include discussion of P analyses [21,167,172,208,214]. The following discussion is a review commentary on the methods used in soil P analysis, focusing on methods most commonly employed in archaeological contexts. A following section summarizes some comparison studies of these methods. An important point of this discussion, and an important aspect of all geoarchaeological P research and publication, are to clearly spell out and reference the procedures used in order to understand what fraction is extracted and for comparison with the work of others.

There are two basic components to P analyses: the extraction of P from the soil and the measurement of P in the extractant. The basic idea for extracting P from the sample is to break the bonds between P molecules and their hosts with one or more reagents. The capacity for all reagents to liberate P molecules depends on concentration, or the relative amount of reagent in solution, and strength, which is the relative ease that reagents ionize, or disassociate, in solution. Most research on soil P has focused on the extraction procedures because they help to obtain the amount of P added to the soil or P available for plant uptake or both. The various archaeological approaches to extracting P can be grouped into four to six basic categories, depending on how the methods are segregated (following Gurney [80, pp. 2–3], Bethell and Máté [18, pp. 10–13], and Terry et al. [224, p. 153]). The following groupings, which are a mix of extraction and measurement procedures, are made for this discussion based on the most widely used methods in the literature: (1) extraction for available P; (2) portable field techniques (the spot test or ring test); (3) chemical digestion of a soil sample for total P (Ptot); (4) extractions of inorganic P (Pin) for fractionation studies and extractions to look at individual compounds of P; (5) measurements of organic P (Porg); and (6) extractions for total elemental analysis by ICP. For the most part, the chemical extracts represent some portion of Pin, but ‘‘most commonly used extractants do not yield clean separations nor discrete groupings of P forms in soils’’ [205, p. 186]. This is not always understood and misstatements regarding the nature of soil P in archaeological contexts are all too common. As discussed below, some chemical fractions measured in the laboratory appear to approximate specific forms of P (e.g. [30,59,86], but others clearly do not. Further, all methods are presumed to extract some or all anthropogenic P, yet one of the greatest challenges of soil P analysis is understanding how a specific method targets a specific form of soil P and its source.

Approaches to measurement of most P extractions fall into two basic categories: colorimetry and ICP. In addition, Marwick [141] reports use of X-ray fluorescence for samples with very low levels of P, and Direct Coupled Plasma spectrometry (DCP) apparently was used in some archaeological analyses of soil P, but details are sketchy. Most colorimetry is based on the technique of Murphy and Riley [158] (see discussion in Kuo [117, pp. 906–910]). The basic idea is to reduce molybdophosphoric compounds in an acidic environment. Chemical reduction of molybdophosphoric compounds creates a blue-colored sample solution, the shade of which is proportional to the P content of the particular fraction that was extracted (e.g., Pav, Ptot). A spectrophotometer measures the capacity of each sample solution to either absorb or transmit a specific wavelength of light. The P content of each sample solution is calculated by means of a standard curve. A standard curve for each trial is calculated through an ordinary least squares regression line, the points of which are determined by spectrophotometer readings on samples of a known P concentration. The parameters of the regression equation are then used to plot the archaeological samples on the standard curve.

The ICP (Inductively Coupled Plasma) spectrometer measures the light spectra emitted by elements super-heated in a plasma torch [213]. As with a colorimeter, a sample solution is extracted with an acid digestion procedure. It is then sprayed into an argon gas, which is heated to10,000 K (the argon is heated by means of ohmic resistance when it is introduced into a high frequency electromagnetic field). The sample is instantly atomized at this temperature and forms plasma

Table 3
Some methods for analysis of soil phosphorous used in archaeology[a]

Method[b]	References and comments[c,d]	Archaeological application
Total P (Colorimetry)		
Sodium carbonate (Na_2CO_3) fusion	[15,117,163 (24-2.2)]	[45,52,194]
Sodium hydroxide (NaOH) fusion	[206]	[46,195,196]
Digest with perchloric acid ($HClO_4$)	[15,117,163 (24-2.30)]	[1,34,37,142,182,203,219,220]
Digest with sulfuric acid (H_2SO_4)–hydrogen peroxide (H_2O_2)–hydroflouric acid (HF)	[22,117]	[122,126]
Oxidize with sodium hypobromite (NaOBr), dissolve in dilute sulfuric acid (H_2SO_4)	[15,48,117]	[42,80,110,180,181]
Total P? (Colorimetry)[e]		
Ignition at 240 °C, extract w/hydrochloric acid (HCl)	[162]; can be part of Porg procedure	[76,190[f]]
Ignition at 550 °C, extract w/hydrochloric acid (HCl)	[32[g],82[g],149]; Bethell and Máté [18] use 550 °C for noncalcareous soils, 400 °C for any soil	[32,82,231]
Ignition at 550 °C, extract w/2% citric acid ($C_6H_8O_7$) and HCl in calcareous soils; R. Macphail, pers. comm., 2000)	[138]	[62,134 (table 1), 138]
Extract by boiling in H_2SO_4	[36 (pp. 174–176)]; Shackley [191, (pp. 69)] describes this method as a measure of total P	
Extract with concentrated H_2SO_4 and concentrated HNO_3	[114]; adapted from Greenberg et al. [75, pp. 4–108 – 4–117)] (see also Pote and Daniel [173])	[99]
Total P (ICP)		
Digest with HCl + HNO_3		[124]
Digest with HNO_3 + HF		[144]
Total P? (ICP)		
Digest with hydroflouric acid (HF)	[127]	[127]
Extract with nitric acid (HNO_3)	[127]; developed in lieu of HF extraction for ICP–AES; results approach the total dissolution of HF	[127]
Digest with nitric acid–perchloric acid (HNO_3–$HClO_3$)	"Double acid" extraction [63–65]	[63–65,124,170]
Total Inorganic P by Fractionation (Colorimetry)		
"Chang & Jackson Fractionation"[h]	[30] (subsequently modified by Williams et al. [242], Bender and Wood [15] and others; see Kuo [117])[h]	[111,122]
1. Extract with ammonium chloride	Water soluble/labile phosphate (easily soluble P)	
2. Extract with ammonium fluoride	Al-phosphate; exclude this step in calcareous soils (NH_4F-P)	
3. Extract with sodium hydroxide	Fe-phosphate (1st NaOH-P)	
(1 + 2 + 3)	(non-occluded P)	
4. Extract with sodium citrate + sodium dithionite (extract with sodium citrate + sodium dithionite + sodium bicarbonate "CDB")	Reductant-soluble P (reductant-soluble P)	
5. (Extract with sodium hydroxide)	(2nd NaOH-P)	
6. Extract with 0.5 N sulfuric acid (H_2SO_4) (extract with 0.5 N HCl, then 1 N HCl)	Ca-phosphate (acid-extractable Ca-P or Pca)	
7. (Ignition at 550 °C then extract with HCl)	(residual organic P)	
8. (Digest with Na_2CO_3 fusion)	(residual inorganic P after 1–7; total P of original sample)	
(4 + 5 + 8)	(occluded P)	
(1 + 2 + 3 + 4 + 5 + 6 + 8)	(organic P)	
(total P − inorganic P)		
"Eidt Fractionation"	[59]	[24,53,59,99,125,153,166,182[i],186,207]
Eidt I or NaOH + CB fraction: extract with NaOH and sodium citrate–sodium bicarbonate	Easily extractable P; mainly loosely bound Al-phosphate and Fe-phosphate and that resorbed by $CaCO_3$, as well as the minute amount in solution; Pav	

Table 3 (*continued*)

Method[b]	References and comments[c,d]	Archaeological application
Eidt II or CBD fraction: extract w/NaOH and sodium citrate–sodium bicarbonate–sodium dithionite	Tightly bound or occluded P; absorbed by diffusive penetration or by incorporation with Al and Fe oxides.	
Eidt III or HCl fraction: extract w/HCl	Fixed P within apatite or tightly bound to Ca-phosphate.	
"Hedley Fractionation"	Chang & Jackson/Williams/Syers technique modified by Hedley et al. [86] (in turn modified by Tiessen and Moir [226])	[118]
Anion exchange resin	Extractable Pin	
Extract with $NaHCO_3$	Labile Porg and Pin, some microbial Pin	
Chloroform ($CHCl_3$) + $NaHCO_3$	microbial P	
Extract with NaOH	Porg and Pin adsorbed to Fe and Al minerals	
Ultrasonification and extract w/NaOH	Porg and Pin from internal surfaces of soil aggregates	
Extract with HCl	P from apatite; occluded P in weathered soils	
Digest with H_2SO_4 and oxidize w/H_2O_2	stable Porg and highly insoluble mineral P	
Organic P (Colorimetry)		
H_2SO_4 extract for Pinorg; ignite at 550°; add H_2SO_4 for Pt; Porg = Pt-Pin	[151,163 (24-3.3)]	[38[j],81,185]
Extract with concentrated sulfuric acid (H_2SO_4) and dilute sodium hydroxide (NaOH) for Pin; digest extract in perchloric acid for Ptot; difference is Porg	[69]	[122]
Available or extractable P (Colorimetry)		
Extract with hydrochloric acid (HCl)	[32[k]]; described as Pin, used with ignition Ptot for Porg	[32]
Extract by boiling in hydrochloric acid (HCl) (total Pin?)	[39,176,193]	[29,39,51,50,80,176,193,239,240]
Extract with HCl in ultrasonic bath		[179]
Extract by boiling in H_2SO_4	[36, pp. 174–176]	[90[l], 175]
Extract with 0.002 N H_2SO_4 buffered at pH 3 with $(NH_4)_2SO_4$	"Truog P" [109,227]; removes Ca-P; used in a widely available and popular field kit	[203]
Extract with sodium hydroxide (NaOH) and hydrochloric acid (HCl)	unpublished except for Woods [246, pp. 69], W. Woods, pers. comm., 2002	[246]
Extract with acetic acid (CH_3COOH)	[34]	[34]
Extract with acetic acid (CH_3COOH) + sodium acetate ($NaC_2H_3O_2$); measure by comparison with color chips	Morgan "double acid" extract [109,155]; Used in LaMotte STH series soil test kits; from www.lamotte.com; tends to extract less Pav than Olsen P or Bray-1.	
Extract with sulfuric acid (H_2SO_4) + hydrochloric acid (HCl)	Mehlich-1 or "double acid" or "North Carolina" soil test [109,117,147,163 (24-5.2),197]; removes Ca-P and strongly fixed P in acid soils; extracts much more P than Bray-1; also used in LaMotte AST and DCL series soil test kits; www.lamotte.com	[84,126]
Extract with acetic acid (CH_3COOH) + NH_4F + NH_4Cl + HCl ammonium fluoride + ammonium chloride	Mehlich-2, dilute acid solution [147] "soluble and readily-labile P" of Terry et al. [224, pp. 155]	[169,170,224,237]
Extract with acetic acid (CH_3COOH) + NH_4F + NH_4NO_3 + HNO_3 ammonium fluoride + ammonium nitrate + nitric acid	Mehlich-3 [148,198]; results comparable to Mehlich-1, Bray-1 and Olsen P	[37,126]
Extract with water or dilute salt solution $CaCl_2$	P soluble in water (soil-solution P) [117,163 (24-5.3),190]; very small fraction of Pav	[56[m]]
Extract with sodium bicarbonate ($NaHCO_3$)	"Olsen P" [109,117,163 (24-5.40),199]; measures Al-P and Ca-P in calcareous, alkaline, or neutral soils; comparable to Bray-1	[52,122,181]
Extract with 0.025 N HCl + 0.03 N ammonium fluoride (NH_4F) in 1:10 soil solution	"Bray & Kurtz P-1" or "Bray-1" (absorbed P) [145 (4.45),163 (24-5.1),200,208 (6S3[n])]; easily acid-soluble P, largely Ca-phosphates and a portion of the Al- and Fe-phosphates; comparable to Olsen P	[1,157,220,221]
Extract with 0.1 N HCl + 0.03 N NH_4F in 1:17 soil/solution	"Bray-2" or "medium strength" Bray [145 (4.43)]; easily acid-soluble P, largely Ca-phosphates and a portion of the Al- and Fe-phosphates	[221,229]

(*continued on next page*)

Table 3 (*continued*)

Method[b]	References and comments[c,d]	Archaeological application
Extract with 0.1 N HCl + 0.05 N NH_4F	"Strong Bray" [145 (4.460)]; easily acid-soluble P, largely Ca-phosphates and a portion of the Al- and Fe-phosphates	[153]
Extract with citric acid ($C_6H_8O_7$)	[208 (6S5)]	[4,62,74,126,134,138]
Extract with nitric acid (HNO_3) + ammonium molybdate; reduce with ascorbic acid ($C_6H_8O_6$)[o]	Spot test or Ring test or "Gundlach method" [57,78,187,243]	[5,20,57,58,80,82,110,128,139,165,187,207,243,246]
Extract with sulfuric acid (H_2SO_4) + ammonium molybdate; reduce with ascorbic acid	The original extraction and measurement method of Murphy and Riley [158]	[183]
Extractable P (ICP)		
Extract with HCl	[150]; probably yields some form of extractable P or Pav.	[101,150,225]

[a] Modified from Holliday [96, table A2.1].

[b] Categories are subdivided on the basis of measurement technique (mostly colorimetry or ICP). A variety of methods for colorimetry are available [117,163,208] most based on Murphy and Riley [158]. ICP extractions are for simultaneous measurement of a wide array of trace elements. Reagents: acetic acid (CH_3COOH); ammonium bicarbonate (NH_4HCO_3); ammonium chloride (NH_4Cl); ammonium fluoride (NH_4F); ammonium nitrate (NH_4NO_3); ascorbic acid ($C_6H_8O_6$); boric acid ($B(OH_3)$); chloroform ($CHCl_3$) ($C_6H_8O_7$); citric acid ($C_6H_8O_7$); hydrochloric acid (HCl); hydrogen fluoride (hydroflouric acid) (HF); hydrogen peroxide (H_2O_2); nitric acid (HNO_3); perchloric acid ($HClO_4$); sodium acetate ($NaC_2H_3O_2$); sodium bicarbonate ($NaHCO_3$); sodium carbonate (Na_2CO_3); sodium citrate ($Na_3C_6H_5O_7$); sodium dithionite ($Na_2S_2O_4$); sodium hydroxide (NaOH); sulfuric acid (H_2SO_4).

[c] Number following [163] is the identification system they used to refer to specific methods. Kuo [117] does not follow this system.

[d] Number following [208] is the identification system used by the Soil Survey Laboratory Staff.

[e] These methods are described as measuring total P, but they probably do not measure true total P.

[f] Heidenerich et al. [90] do not indicate ignition temperature.

[g] Chaya [32] and Hamond [82] each used different colorimetry.

[h] Methods and terms in parenthesis represent significant modifications of original Chang and Jackson [30] procedure by Williams et al. [242].

[i] Sandor et al. [182] used Eidt I only for Pin or "moderately available P."

[j] Courty and Nørnberg [38] do not indicate ignition temperature.

[k] Method not referenced, but possibly following Olsen and Sommers [163, 24-3.2.3].

[l] Heidenrich et al. [90] note that their method is a modification of Cornwall's method but do not indicate the nature of the modification.

[m] Eddy and Dregne [56] do not describe specific procedures for extraction or measurement; they may not be equivalent to the Soil Survey Laboratory Staff [208] procedure.

[n] 6S3 Bray P-1 can be measured with a spectrophotometer or with a flow-injection automated ion analyzer.

[o] The original Gundlach [78] procedure extracted with nitric acid. Eidt [57] and Woods [243] discuss substitution of nitric with hydrochloric acid for recovery of a greater variety and quantity of P compounds. Note also the following method which uses sulfuric acid.

consisting of "excited" ionized atoms. Each "excited" ionized atom emits a distinctive light spectrum as it relaxes back to its base state and is then introduced to the mass spectrometer. The mass spectrometer separates and collects ions according to their mass to charge ratios. It then counts the number of individual ions. Three approaches are used in ICP work: Direct Coupled Plasma–Optical Emission Spectroscopy (DCP–OES), Inductively Coupled Plasma–Mass Spectrometry (ICP–MS) and Inductively Coupled Plasma–Atomic Emission Spectrometry (ICP–AES). DCP–OES uses an optical spectrometer to measure wavelengths emitted by atoms as they relax to their base state, with certain measurements proportional to the concentration of particular elements in the sample solution. In ICP–AES the plasma heats the atom and a phototube measures the intensity of the color of the element. ICP–MS uses a plasma only to ionize the atoms and then sucks them into a magnet at high voltage, which bends them around a curve to an ion detector, sorting them by mass (the paths of light atoms bend more than the paths of heavier ones). They do the same thing in different ways. DCP–OES is an older method, though it is still used today. In practice ICP–AES is best for elements that easily glow when hot; generally the top and left part of the periodic table. ICP–MS is more sensitive and can measure less abundant elements and works well with heavy ions; generally the bottom and right side of the table [213, J. Burton, personal communication, 2002]. Although the ICP measurements are highly precise, an accurate measure of P concentration is partially dependent on the capacity of the reagent to liberate P molecules from the sample during digestion. Sample digestion thus affects methods employing the colorimeter and the ICP. In sum, an accurate measure of Ptot during ICP analysis requires that the sample be completely obliterated prior to atomization. There are sources of error unique to ICP analysis – elemental spectra can overlap and doubly-charged ions can complicate element identification. However, these problems are uncommon.

The following discussion is organized around the sixfold categorization of extraction and measurement procedures presented above. The categorization is not a mutually exclusive list of approaches, but rather summarizes the extraction and measurement methods as they are most commonly presented in the literature.

7.1. Available P

Much of the archaeological interest in soil P has focused on available P (Pav). This is because of the wide variety of relatively easy techniques for identifying or measuring Pav and because of the long interest of agricultural scientists in Pav. Essentially all of the early work on P in archaeology focused on Pav (though some erroneously described the measurements as total P) (e.g., Arrhenius, Lorch, Dauncy, Solecki, Eddy and Dregne, Cook and Heizer in Tables 1 and 2). Measurement of Pav was developed in the agricultural sciences to measure plant-nutrient availability (i.e., it is an estimate of the small amount of Pav for plant growth) [18 (p. 6),59 (p. 35)]. Soil scientists asked to analyze the P content of archaeological soils but not otherwise familiar with geoarchaeological techniques or questions typically provide data on the easily Pav. Kamprath and Watson [109] present a useful review discussion of testing soils for Pav.

There are several significant problems in measuring and interpreting Pav, and it may not be the best archaeological indicator (summarized by Hamond [82, pp. 61–62]). Available P measures soil-solution P and labile Pin, which is not a single simple chemical, compound or mineral, and represents only a minute portion (~1–3%) of Ptot P [18, p. 6]. Moreover, availability of P to plants depends on soil chemistry, water, texture, and structure, and varies from soil to soil [12,217]. Different plants also extract different amounts of P from the same soil. Determination of Pav, therefore, may indicate roughly the P status of a soil, but does not correlate with any particular P fraction that exists in nature [18 (p. 6),59 (p. 35)] nor does it necessarily measure anthropogenic inputs of P. Estimates of easily extractable Pav are "extremely difficult to make because they attempt artificially to recreate chemical conditions around plant roots" [59, p. 35]. Moreover, the variety of methods for extracting Pav yields different amounts of P. This is largely because the different extractants get at different forms of Pav, depending on the strength of the extraction reagent and on the degree of solubility of the P [117, p. 890]. For example, a simple water wash will get at the most easily extractable P, and progressively more vigorous techniques (e.g., citric acid extraction, boiling in HCl for 10 min, boiling in HCl for 2 h) will yield progressively more P. The stronger reagents are probably extracting surface-precipitated or moderately sorped Pin in addition to soluble and labile P.

The early geoarchaeological work on soil P dealt with citrate-soluble P, which is one of the more easily extractable forms. Some investigators question whether easily extractable Pav data have any utility in geoarchaeological research (summarized by Bethell and Máté [18, p. 11]). In spite of the reservations about the utility or meaning of the easily extractable P, it does seem to be broadly indicative of human activity. In one of the earlier applications, Eddy and Dregne [56] measured the Pav extracted by a simple water wash (water-extractable P; Table 3). Their data correlate well with occupation zones in late prehistoric sites in southwestern Colorado and northwestern New Mexico. Further, the requirement for elevated levels of citrate-soluble phosphate in the "Anthropic epipedon" of the U.S.D.A. soil classification system [209, p. 22] is a measure of easily extractable Pav [18 (p. 11),138 (p. 72)] and these can be quite high in archaeological contexts though exactly why is unclear. Macphail et al. [138, p. 72] illustrate this for several sites in Britain. Further, at the Wilson-Leonard site in central Texas, levels of citrate-soluble Pav from occupation zones were significantly higher than in non-occupation zones [74,96 (table 11.5)]. But these zones high in Pav were also buried A horizons likely naturally higher in Pav. No off-site control samples were analyzed. In summary, measurement of Pav as an indicator of human activity seems to work well in drier environments, such as the southwestern U.S. (e.g. [99]), but may be more problematic in wetter, leaching environments (e.g. [82]).

7.2. Portable P techniques

A significant amount of soil P research in archaeology has been devoted to analytical methods that can be applied in the field. The attraction of these approaches is the potential ability to use P levels to prospect for sites and to map activity areas. An important step toward these ends was development of the spot test or ring test (or "Gundlach method," Table 3) for quick field evaluation of P levels on archaeological sites [18, p. 12]. The method tests for easily extractable Pav. As a result, the meaning of the spot test can be ambiguous (summarized by Hamond [82, pp. 55–61]), given the vagaries of Pav interpretation noted above. Further, the results are qualitative and not always reproducible [58,59 (pp. 36–38),80 (p. 2)]. The original spot test described by Gundlach [78] used HNO_3; but Eidt [57] showed that HCl gives better results, while Hassan [84] states that either H_2SO_4 or HCl is appropriate for a rapid field test. The problems of using the spot test are well illustrated by Keeley [110]. In 20 applications of the spot test there were10 successful attempts at locating or defining occupation zones (in the UK and Sicily), four "partial successes" (i.e., slight enhancement of P allowed some inferences about occupation zones) (in the UK and Peru), and six failures where no relationship between soil P and occupations (in the UK) could be identified.

These drawbacks have lead to outright rejection of the spot test by some archaeologists (e.g. [202, p. 451]). Others take a more realistic approach and recognize the utility of the method given its simplicity and portability, but also its limitations, and view the method as an important component of field investigations (e.g. [18 (p. 12),80 (p. 2),82 (p. 61),165 (pp. 268–269)]). Lippi [128], for example, applied the test at the remote site of Nambillo in Ecuador. A systematic coring strategy was used to establish the site stratigraphy and identify buried land forms. Soil samples recovered during coring were subjected to in-field phosphate analysis, and the results were used to identify areas of human activity. These data were then used to design an excavation strategy for the site. Bjelajac et al. [20] also showed how the spot test could be calibrated at known sites to determine a minimum "site value" in a given region and then be used locally to aid in identifying or delimiting other sites. Thurston [225], in an analysis of settlement hierarchies and sociopolitical integration, used a rapid P test to determine the areal extent of Iron Age settlements on the Jutland Peninsula of Denmark.

Another significant improvement in field-based, portable P analysis was development of a semi-quantitative procedure based on the extraction of P and measurement using colorimetry [18, p. 12] (a fully quantitative technique requires sample drying and pre-treatment which defeats the purpose of having a quick and easy field test). Like the spot test, the method is relatively portable, quick, and easy, but it is also semi-quantitative, so it is a popular technique for on-site analyses (e.g. [39,80,84,169,176,224,237]). A wide variety of procedures for extractions and measurement are now available (Table 3 "Available or Extractable P-Colorimetry"). As a result, the forms of P extracted can vary considerably. Many of the methods measure Pav [18, p. 12], well-described by Terry et al. [224, p. 153] as "soluble and readily labile P." Terry et al. [224] further note that their extraction "is not always proportional to the total P of the soil; however, for archaeological prospection and activity area research, the spatial patterns of phosphate levels are important, rather than the absolute concentration." That is probably a fair comment, given the many variables that affect P levels in soil (e.g. [12,18,175,217,238]).

7.3. Total P

Once geoarchaeologists began following the P research from soil science, work on soil P in archaeology began to focus on Ptot [18]. Until the late 1980s and 1990s, however, Ptot analysis in archaeology was limited because the procedures were tedious and involved strong and dangerous reagents such as perchloric acid and hydrofluoric acid [18 (pp. 12–13),32,34,48,67 (p. 88),80 (p. 3),110,117 (p. 874),138]. Further, some methods described as extracting Ptot probably do not.

Widely used methods for determination of Ptot include digestion in perchloric acid ($HClO_4$), fusion with sodium carbonate (Na_2CO_3), and sequential digestion in sulfuric acid (H_2SO_4), hydrogen peroxide (H_2O_2), and hydrofluoric acid (HF) (Table 3). The perchloric acid digestion does not extract all P however, unless HF is added to the digestion solution to fully destroy minerals containing phosphate [48]. Dick and Tabatabai [48] developed an alkali oxidation method that is simpler and safer than the others: boil with sodium hypobromite (NaOBr–NaOH), extract P with H_2SO_4, and determine P colorimetrically (Table 3). They compared the results from this method to those from the other procedures and they compared favorably to $HClO_4$ digestion. The advent of ICP technology also provides a means of relatively simple and safe measurement of Ptot and is discussed in a separate section below.

Measurements of Ptot, along with total Pin (discussed below) are used in determination of Porg. The difference between Ptot and Pin is taken as Porg (discussed below). The Ptot is measured by colorimetry following ignition and then acid extraction. This procedure alone is rarely mentioned as a method of Ptot determination, probably because it is not a true measure of Ptot. Chaya [32], however, used ignition and HCl extraction (Table 3) to determine Ptot. A comparison with "total P" determined by electron microprobe showed generally similar results, but the microprobe method was not explained.

A variety of analytical methods for soil P have been described as measuring Ptot, but clearly they do not. Most of these methods involve a strong acid such as hydrochloric (HCl) or sulfuric (H_2SO_4). Cornwall [36, pp. 174–176] discusses a method of phosphate analysis using 3 M H_2SO_4 and colorimetry; described by Shackley [191, p. 69] as measuring "total P". And Meixner [149] presents a method using HCl extraction described as measuring Ptot (similar to Chaya [32], noted above). The reagents employed in these methods are incapable of fully digesting all P-bearing minerals and compounds in the soil, however. This point is further elaborated upon in our own research, described and discussed below.

Measurement of Ptot produces quantitative, comparable results, in contrast to many measures of Pav or the spot test, and may be the best indicator of human inputs of P when comparisons are made with natural soils [18, p. 20]. For example, following a comparison of methods, Skinner [203] concluded that Ptot produced the highest correlation with anthrosols, but positive only 60% of the time. Alternatively, Terry et al. [224] present data that suggest that Pin extractions may be more sensitive to human inputs than Ptot. In a related study, Parnell et al. [170] argue that extractable P is indeed more sensitive than Ptot. This may be because Ptot includes all mineral P, which can be significantly higher than anthropogenic P. Soil parent materials high in P (e.g., high in apatite) will yield Ptot levels that overwhelm any signatures of human activity. This can be a significant drawback to soil-geoarchaeological work in areas with high natural P.

7.4. Fractionation of inorganic P

There are many procedures for extracting various forms or fractions of Pin (Table 3). Each method probably gets at different forms of P. In comparing results with that of other investigators, considerable care must be taken to reproduce the methods used. For example, Heidenreich et al. [90] compared an extraction from a non-ignited H_2SO_4 treatment (based on Cornwall [36, pp. 174–176, Table 3]) with an ignited HCl extraction. The ignited HCl extraction yielded higher P; in part because of the oxidation of Porg during ignition.

A well-known approach to measuring Pin fractions in both soil science and pedology is the sequential extraction procedure developed by Chang and Jackson [30], modified by a number of investigators (e.g.[86,226 (Table 3),241,242]). The best known of the fractionation methods in geoarchaeology is that of Eidt [58,59], based on Williams et al. [241] [166, p. 123]. The basic fractionation scheme involves extraction of: Fraction I, or solution P, P resorped by $CaCO_3$, and loosely bound Al and Fe phosphates (Inorganic P from the Available and Active pools in Fig. 1); Fraction II, or tightly bound or occluded forms of Al and Fe oxides and hydrous oxides (Inorganic P from the Relatively Stable Pool in Fig. 1); and Fraction III, or occluded Ca phosphates (Inorganic P from the Relatively Stable Pool in Fig. 1)[2] [59, p. 42]. Eidt [59, pp. 41, 43] further proposed that sequential extraction of various forms of Pin provide a measure of total inorganic P. According to Sandor et al. [182, p. 178] Eidt's inorganic P probably does not extract all inorganic phosphorus.

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[2] Fractions I, II, and III of Eidt [59] are not the same as extractions I, II, and III in Fig. 1.

The fractionation method developed by Eidt [58,59] probably generated more interest and controversy than any other single P procedure in archaeology (e.g. [18,82,112,122]). The fractionation method also has had relatively minimal application, probably because it is labor-intensive, time consuming, and expensive (e.g. [80 (p. 3),99 (p. 142),122 (p. 352)]) and because of questions concerning the meaning of the results. Eidt's [58 (p. 1328),59 (pp. 40−42)] approach is based on the idea that total Pin is the best indicator of anthropogenic activity. The basic theory is probably sound: human inputs of P-bearing materials are probably quickly converted to Pin. High levels of Pin are reported from archaeological sites in comparison to local natural soils [111,125,186].

Eidt [59, pp. 41, 43] further proposed that sequential extraction of various forms of Pin may reveal clues to human activity based on a purported close correlation between land-use and inorganic P levels. This issue is problematic, however. Eidt [59, p. 43], building on the work of Lorch (Table 1) asserted that low levels of Pto (10−220 ppm) corresponded to ranching and farming; moderate levels of Pto (200−2000 ppm) from more intense activities as would be found around dwellings, gardens, and manufacturing areas; and very high levels (>2000 ppm) from burials, garbage pits, slaughter areas, and urbanized zones. Eidt [59, p. 43] also noted that the P data could be used to identify crop and forest types. All of these correlations were asserted, not demonstrated. Yet in field studies Eidt [59, pp. 55−72, 87−106] presented data on Pto for less than a dozen samples of contemporary gardens and residences in order to infer crop or plant types (e.g., manioc, yucca, rice) from archaeological zones. Given the small sample size, and the great diversity of agricultural techniques, additional research is needed to justify his conclusions. Much more information is needed on: (1) the range of variation of Pto for different types of activities and for natural soils in any given study area; and (2) the nature of the soils associated with the contemporary and archaeological activity areas, e.g., mineralogy and pH. Lillios [125], for example, gathered a sizeable data set on Pto for contemporary vegetation before trying to interpret her archaeological Pto data.

7.5. Organic P

Few studies focus on the relationship of Porg to human occupation. Porg represents a large part of the Ptot pool and human activity can produce Porg. Of the archaeological studies that have determined both Porg and Ptot, Porg (as a percentage of Ptot) was low in the soils with archaeological contexts (summarized by Bethell and Máté [18, p. 18]). This is attributed to the high content of P derived from bone (hence high Pin) at most archaeological sites. Porg can mineralize relatively rapidly depending on microbial activity. Higher Porg to Pin ratios do seem to be associated with crop residues. Courty and Nørnberg [38] and Engelmark and Linderholm [62] provide two of the few studies of Porg. In comparisons with uncultivated soil they found elevated levels of Porg in abandoned agricultural fields.

Most measures of Porg are indirect and based on ignition or acid−base extraction. Both approaches are described by Olsen and Sommers [163], but the acid−base extractions were later modified significantly based on the discussion by Kuo [117]. In the ignition method, P is extracted with HCl following ignition (at 240 °C [162] or 550 °C [32,149]) or with H_2SO_4 following ignition at 550 °C [163]; a variant of the method for archaeological applications is described by Mikkelsen [151] and then measured by colorimetry. Porg was calculated as the difference between Pin in the ignited and unignited samples, while residual P, assumed to represent Porg, was calculated as the difference between Ptot and Pin in the ignited sample. The extraction following ignition is assumed to represent Ptot. A non-ignited sample is also extracted for Pin. Porg is the difference between the Ptot and Pin. As noted above, the ignition and acid extraction probably is not a true measure of Ptot. Indeed, Walker [232] uses the difference between H_2SO_4 extraction following ignition and without ignition as a mean of estimating Porg, but uses the $HF-HNO_3$ digestion for Ptot. The ignition process probably does oxidize most Porg and the difference between the ignited and non-ignited sample probably provides a rough estimate of Porg. All of the methods vary in their efficiency and accuracy. A decision on choice of methods probably will depend on availability of lab facilities and desired accuracy.

7.6. ICP approaches to soil P

The advent of ICP (Inductively Coupled Plasma) spectrometry in the 1980s and 1990s, provides a relatively rapid method of measuring all elements, including P, in a sample. As a result, measurement of Ptot in archaeological soils has become common (e.g. [63,65,127,144]). The ICP studies of archaeological soils are almost always done in the context of multi-element analyses and, unfortunately, most of the method discussions do not specify or otherwise deal with the form of P being analyzed, though some inferences are possible based on the extraction procedure. Linderholm and Lundberg [127] argue that trends in elemental concentration of P using total digestion of samples by hydroflouric acid (HF) (i.e., probably yielding Ptot) can be reproduced more quickly by analyzing extracts made from nitric acid (HNO_3) (i.e., some form of extractable P, rather than Ptot). Subsequent research by others [63−65] involved a nitric acid−perchloric acid (HNO_3-HClO_3) digestion for the extract, which may approximate Ptot given that perchloric extraction is a common method for Ptot. Entwistle et al. [65] recovered low levels of P, however, due to the lack of manure and the superpositioning of activity areas (see Section 6). A simple HCl extract was used by Middleton and Price [150], the concentration of which should liberate all P from the Pav, and most Pin from the Pact and Psta pools (Fig. 1).

Several investigators have noted some problems or potential problems with P analysis by means of ICP spectrometry. One is that anthropogenic P may be present in too high a concentration to be determined along with trace elements [63]. The P can be analyzed as a single element, but this may be difficult to justify economically. Entwistle and Abrahams [63, p. 415], as part of their ICP research, determined P by one of the standard colorimetric methods. Another issue is chemical interaction among elements [144]. Phosphorus, for example, has a tendency to strongly bind with other elements such as Ca to form insoluble compounds. Depending on extraction procedures, therefore, elevated levels of P may or may not be detected.

ICP analysis is relatively straightforward and efficient and, with the growing availability of ICP spectrometers, an important component of soil chemical analysis. Those interested in using the ICP for anthropogenic P analyses should be aware of potential problems, however.

8. P comparisons − previous studies

There are surprisingly few comparative studies that explore the influence of laboratory methods on soil P trends in archaeological contexts. Despite the many methods available to extract and measure soil P, it is still uncertain: (1) which methods produce more or less similar values; (2) which methods produce the highest levels of P; (3) which methods yield results that best correlate with anthropogenically altered soils; and (4) which methods yield sound results and also are the most efficient and suitable for the field. The following discussion summarizes some of the handfull of studies that are available.

The earliest method of P extraction used in archaeological context, and one still used today, is based on 2% citric acid. Though citric acid is traditionally viewed as an extractant for Pav, Macphail et al. [138] argue that it liberates Pin found in acid soils. Their summary discussion (p. 72) certainly makes a good case that the method removes large amounts of P, but they do not clearly indicate which forms of soil P are extracted by citric acid. Bakkevig [5, p. 86] notes that 2 N HCl extracts 10 times as much P as 2% citric acid.

Ahler [1] presents one of the earlier studies comparing P methods in an archaeological context. Samples were taken from fill at Rodgers Shelter, Missouri. The study compared the results of Ptot by perchloric acid digestion to Pav by the Bray-1 acid extraction (Table 3). He then compared both results to the density of occupation debris. Ptot varied as a function of artifact density through the stratigraphic sequence. Elevated Pav correlated with artifact density only in the lower levels, but not the upper deposits. The upper levels accumulated much more slowly than the lower ones, however, and the relatively low levels of Pav in the upper deposits were attributed to weathering.

Skinner [203] compared a wide variety of P methods to determine which was most reliable for identification of anthropogenicaly modified soils in Ohio. The methods included the spot test, a Hellige-Truog kit (a commercially-available kit for field analysis of soils; the P method is the Truog H_2SO_4 extraction for Pav, Table 3), perchloric acid extraction for Ptot, and HCl extraction for Pin. The perchloric acid extraction produced the highest correlation with anthrosols, but was positive only 60% of the time.

Leonardi et al. [122] compared different forms and extractions of P to determine which best supported the interpretation of ancient agricultural use of buried soils. They looked at Porg vs. Ptot as well as the fractionation of P. The results of both the Porg and Ptot analyses supported the interpretation of agricultural use of the soils, but the latter method is quicker and less expensive than the fractionation scheme [122, p. 352].

Terry et al. [224] evaluated a soil test kit for use in an archaeological field laboratory. The P method in the kit was based on the Mehlich-2 dilute acid procedure (Table 3) and was compared to a bicarbonate extraction (for a Pin fraction) [163, procedure 24-5.4, Table 3], perchloric acid digestion (for Ptot), and the ring test (rated on a 1−5 scale) of Eidt [57]. The trends in both the Mehlich and bicarbonate procedures are similar, but the Mehlich procedure produced more extractable P than the bicarbonate method. The Ptot digestion produced roughly 100 times the P of the Mehlich procedure, which was expected because the method dissolves all P in the parent material. The values from the Mehlich method could not be used to estimate Ptot. However, both of the Pin fractions (Mehlich and bicarbonate) better reflected anthropogenic alterations of the soil than did the Ptot. The ring test results had only a moderate correlation with the Mehlich results. In the end, Terry et al. [224] decided to use the Mehlich-2 kit because of its apparent sensitivity to human inputs of P, its efficiency and cost, and its portability.

Several studies have compared methods of extracting and measuring Pav. Kamprath and Watson [109] show that the “Bray-1”, Olsen P, and North Carolina (or “Mehlich-1”) tests (Table 3) produce generally comparable results. In a geoarchaeological context, Proudfoot [175, p. 95] compared Pav levels based on a variety of extractants (using calcium lactate, hydrochloric acid, acetic acid, citric acid, and distilled water). The values generally ranged significantly, due to variable P levels as well as chemical reactions during the extraction processes.

In a comparison that is not in an archaeological context, Kuo [117, pp. 870, 874] summarizes the results of the various methods of Ptot measurement. The perchloric acid and sodium hypobromite digestions yield comparable results, but both methods may underestimate Ptot in proportion to P embedded in the matrix of silicate minerals such as quartz. Sodium carbonate fusion and the H_2SO_4−H_2O_2−HF method both tend to extract more P than the perchloric acid and sodium hypobromite digestions. The difference between Ptot determined by fusion and that determined by perchloric acid digestion was greatest in samples with a high sand content (see also Section 5.4).

9. P comparisons − this paper

To further comparative studies of soil P analyses in archaeological contexts, we subjected samples from three very different archaeological sites to a variety of soil P extractions and measurements. The samples are from: the Lubbock Lake site (41LU1), Texas; the Hulburt Creek site (47SK292), Wisconsin; and the British Camp site (45SJ24) on San Juan Island, Washington.

Lubbock Lake is in a dry valley on the semiarid Southern High Plains of northwestern Texas [105]. The samples are from a stratified sequence of well-drained buried soils developed in calcareous, quartzose, and sandy eolian (and some slopewash) sediment (Table 4) that comprise much of the late Holocene valley fill there [92–94]. Geologic and anthropogenic sources of P are minor. The late Holocene pre-European occupation was largely by hunter–gatherers who produced temporary campsites, some including bison kill/butchery locales.

The Hulburt Creek site consists of remnant agricultural fields (ridges and ditches) and earth ovens across the modern ground surface. They were built ∼1000 RCYBP (radiocarbon years B.P.) in coarse-textured, nutrient-poor, shallow soils on uplands (Table 4). The parent material is sand and re-worked sandy sediment exfoliated from exposed Mt. Simon Formation sandstone. The samples include a buried pre-agricultural soil, the initial construction of the ridges in eroded sediments and pre-agricultural deposits, and a relict planting surface which native peoples periodically refurbished with ditch deposits and repeated burning [69, pp. 673–675]. Anthropogenic sources of soil P were primarily from the construction and

Table 4
Soil characteristics of the study sites (see text for references)

Stratum	Horizon	Soil	Texture[b]	Diagnostic horizon[c]	Classification[c]
Lubbock Lake site					
Tr 95					
5B	A	Singer	SCL	ochric	Ustochrept
	Bw		SCL	cambic	
5A	Ab1	Apache	SCL	ochric	
	Bkb1		SCL	cambic; calcic	
4B	A1b2	Lubbock Lake	L	ochric[a]	
	A2b2		fSL	ochric[a]	
Tr 104					
5B	A	Singer	SCL	ochric	Ustochrept
	A&Bw		SCL	cambic	
	Ab1		fSL	ochric	
	ABwb1		SCL	cambic	
5A	Ab2	Apache	SCL	ochric	
	Bw1b2		SCL	cambic	
	Bw2b2		SCL	cambic	
	Bw3b2		SCL	cambic	
	Ab3		SCL	ochric	
	Cb3		SCL		
4B	A1b4	Lubbock Lake	SCL	ochric[a]	
	A2b4		SCL	ochric[a]	

Horizon	Texture[b]	Diagnostic horizon[c]	Classification[c]
Hulburt Creek site			
Raised field profile			
OA	SiL	ochric	Hapludalf
Bw	SiL	cambic	
Ab	SiL	ochric	
Bwb	SL	cambic	
2BCb	S		
2Cb	S		
Control profile			
OA	SL		
Bw	SL		
2C	S		
3R	sandstone		

Stratum	Texture[d]	Diagnostic horizon[c]	Classification[c]
British Camp site			
C	L/SiL	anthropic	Udoll?
D	L	anthropic	Udoll?
J	L	anthropic	Udoll?
K	SiL	anthropic	Udoll?
M	L/SiL	anthropic	Udoll?
N	CL	anthropic	Udoll?

[a] Probably mollic before burial.

[b] Following the U.S. Soil Taxonomy [209]; classification for surface soil only.

[c] S = sand; SL = sandy loam, fSL = fine sandy loam; SCL = sandy clay loam; L = loam; SiL = silty loam; clay loam.

[d] Based on <2 mm fraction free of shell fragments.

maintenance of ridged fields during the 11th century AD. Geologic sources of soil P were minor here.

The British Camp site is a large, prehistoric, Northwest Coast shell midden underlying the historic site of British Camp, within the San Juan Island National Historic Park [215]. The island is in the Gulf of Georgia near the mouth of Puget Sound. Beginning about 500 AD, Marpole and San Juan phase peoples created a midden composed of dense, stratified layers of shell, along with bone, and other marine and terrestrial organic debris atop a wave-cut bank (Table 4). Though never described or classified as soils, the midden debris can be considered some type of cumulic anthrosol. Samples used in this study were collected from individual layers within the midden. Natural sources of soil P are difficult to assess without comprehensive off-site sampling, but only human activity can account for the high soil P values at the British Camp site.

We examined four procedures that extract some proportion of Ptot. Sequential extraction of soil P may aid in interpreting past human activity and environmental history, but methods that extract some proportion of Ptot are more common in archaeological studies because sequential fractionation is a relatively costly and lengthy process. Samples from Lubbock Lake, San Juan Island, and Hulburt Creek were all subjected to four methods of acid extraction: perchloric acid digestion for Ptot [117,163]; sulfuric–nitric acid extraction (“double acid”) for Ptot [114]; hydrochloric acid extraction after ignition for extractable P [149]; and citric acid for extractable P [208, method 6S5]. The perchloric acid extractant was measured on an ICP. The remaining extractants were measured on a spectrophotometer. In addition, for comparative purposes, selected samples from the perchloric acid trials were measured on a colorimeter and selected samples from the HCl trials were measured on an ICP. The Soil and Plant Analysis Laboratory of the University of Wisconsin-Madison carried out the perchloric acid extractions and all ICP measurements. We conducted all remaining extractions and performed all of the spectrophotometer measurements at the University of Wisconsin-Milwaukee Soils Laboratory. The values reported in this paper supercede those presented by Holliday et al. [98, pp. 367–373].

9.1. Sample pre-treatment and P curve calculations for the colorimeter

All samples were air-dried, ground, and sieved through a 2 mm mesh. In order to volatilize organic matter, all samples were placed overnight in a muffle furnace at 440 °C. Most, but not all, organic P compounds will convert to inorganic forms of P during this pretreatment procedure. Any additional pre-treatments – for example, to remove free calcium carbonate – are technique- or case-specific (see citations above). For purposes of quality control, we also ran duplicate archaeological samples and blanks during each trial.

Colorimetry only provides an indirect measure of P concentration. Thus, standards with a known concentration of phosphorus were analyzed at the same time as the archaeological samples in order to calculate a standard curve (see Section 7 above). We used a Spectronic 20 spectrometer to analyze color development. In general, we found that %transmittance was more sensitive to P variability than absorbance. The two measures are related by the following formula: absorbance = 2 – (LOG (%transmittance)).

9.2. Potential sources of variability

Storage of air-dried soils does affect soil P levels, primarily from the Pav pool, with a general decrease in Pin and an increase in Porg in as little as three years. Acidic soils are particularly prone to storage-induced changes, which are related to the disruption of coatings on mineral surfaces, the solid-phase diffusion of P molecules, and the decay of microbial cells [228]. The sites discussed below were excavated at different times and each site samples stored in different places prior to this study. The soil P values of some samples may have been significantly affected by long-term soil storage. We suspect, however, that overall site trends may not have changed too much since samples from individual sites were collected at the same time and stored together.

There are many forms of soil P and many reactions that accompany its chemical extraction from a soil sample. The use of strong acids, or bases for that matter, to extract Pin from soils may cause the oxidation or hydrolytic breakdown of Porg forms (well summarized by McKelvie [146, pp. 2–3 and ff]). In addition, certain elements and chemical compounds are known to affect color development and thus potentially affect spectrophotometric determinations of P concentration. Jackson [104] termed these elements and compounds interfering substances and developed many permutations for each of his P fractionation techniques to combat the problem. Of course, one must know the chemical composition of a soil before neutralizing an interfering substance.

The chemical precipitation and resorption of P molecules during sequential extraction is well-documented (e.g. [16,53]). Soils are a complex mix of chemical compounds; sample-specific reactions that interfere with P measurement and P resorption are possible while extracting some proportion of Ptot. Since the ICP directly measures P concentration, the problem of interfering substances is minimal. Nevertheless, the problem of incomplete sample digestion and P resorption can compromise P analyses – regardless of the method to measure P concentration.

9.3. Site specific trends: the Lubbock Lake site

All soil P samples from Lubbock Lake were collected from exposures near or around excavated activity areas (Trench 104 in Area 8 and Trench 95 near Area 19) representing short-term occupations [95, fig. 1,106, fig. 1], but none are known to be directly from archaeological features. Trench 95 soil horizons (Table 4) correspond to the Apache and Singer Soils [92,93,95], which formed over the past 800 years in sandy eolian, and gravelly slopewash deposits of strata 5A and 5B, respectively. Trench 95 is ~50 m south of excavation Area 19 and ~80 m northwest of excavation Areas 5 and 6. Area 19 yielded a small camp/hearth feature in stratum 5A [97]. Areas 5 and 6 contain multiple late prehistoric bison and historic bison and horse bone beds representing kill/butchery locales in stratum 5 m, a muddy lowland facies of both 5A and 5B [94,105]. The most likely sources of anthropogenic P in Trench 95 are from bone and burning of woody plant material, but no obvious archaeological features were found in the immediate sampling area. Trench 104 exposed the Lubbock Lake Soil (Table 4), which formed in loamy eolian sediments (stratum 4B) deposited 5000-4500 RCYBP, in addition to the Apache and Singer Soils [92–94]. The parent materials here are slightly coarser than the Trench 95 deposits (Table 4). Trench 104 represents the walls of excavation Area 8, which yielded multiple occupation zones including camping features in all three soils and bison bone beds in strata 5A and 5B [105,106].

9.3.1. Lubbock Lake Tr 95

The magnitude of soil P at Lubbock Lake Trench 95 is generally proportional to the capacity of each acid to obliterate the bonds between P molecules and parent materials (Table 5; Fig. 2). Perchloric acid is a stronger and more concentrated reagent than the other acids in our study and, not surprisingly, it extracted more soil P than the sulfuric–nitric acid, HCl, and citric acid techniques. Nevertheless, despite considerable variation in the magnitude of soil P, each technique produced broadly similar trends.

At a finer scale of analysis, however, there is some variability that reflects the complexity of soil P dynamics and chemistry. For example, the citric acid trial indicates that soil P decreases from the A2b2 to the A1b2 horizon of the Lubbock Lake Soil. However, the three remaining methods, particularly perchloric acid digestion, document an increase in soil P over this transition (Table 5; Fig. 2). The A1b2 horizon has a higher clay and organic matter content, as well as significantly more iron and aluminum, than the A2b2 horizon (Table 4). These conditions favor the sequestration of P in the Psta pool (Fig. 1), particularly for Porg (see Sections 3, 5.1, and 5.4 above). The citric acid technique has a relatively low capacity to break the comparatively resilient bonds between P compounds and parent materials in the Psta pool, particularly in a basic soil setting (sensu Macphail et al. [138]).

There are also divergent trends between the top of the Apache Soil (Ab1) and the base of the overlying Singer soil (Bw) (Table 5; Fig. 2). Soil P values from the sulfuric–nitric acid and, especially, the citric acid trials decrease from the Ab1 horizon to the overlying Bw horizon, whereas the HCl and perchloric acid trials document a trend of increasing soil P values. These divergent trends cannot be explained entirely in terms of acid capacity and the type of P receptors, given the high capacity of sulfuric–nitric acid to liberate occluded and strongly adsorped P elsewhere in our analysis. Both horizons are very similar in terms of particle size and are identical in pH (Table 4). The buried A horizon of the Apache Soil has more than three times the organic carbon than the overlying Bw horizon of the surface soil. It is likely, therefore, that

Table 5
Lubbock Lake, physical and chemical characteristics

Horizon	Sand (%)	Silt (%)	Clay (%)	pH	OC (%)	Ca (ppm)	Fe (ppm)	Al (ppm)	P (citric) (ppm)	P (HCl) (ppm)	P (H_2SO_4–HNO_3) (ppm)	P ($HClO_4$ & ICP) (ppm)	P ($HClO_4$ & color) (ppm)	P (HCl & ICP) (ppm)
Trench 95														
A	61	22	17	8.0	1.1	56,318	7725	14,173	75	146	229	261	211	155
Bw	60	23	17	8.0	0.2	57,678	6644	12,238	23	134	141	192	157	90
Ab1	60	23	17	8.0	0.7	87,959	8708	16,178	45	112	152	184	158	109
Bkb1	42	29	29	8.0	0.5	39,583	8026	14,125	60	166	229	300	423	237
A1b2	65	23	12	8.0	0.6	36,492	6290	11,310	8	126	168	224	172	165
A2b2	72	20	8	8.0	0.4	21,192	5527	9751	23	118	157	164	154	122
Trench 104														
A	61	26	13	8.0	0.5	26,481	7136	12,485	23	140	240	217	205	–
A & Bw	74	16	10	8.0	0.4	37,052	8892	15,327	23	142	233	213	184	–
Ab1	75	16	9	8.0	0.4	57,965	6510	11,865	8	90	126	134	152	–
ABwb1	73	15	12	8.0	0.3	39,081	6586	11,710	45	130	181	194	171	–
Ab2	65	20	15	8.0	0.5	51,910	6976	12,524	23	116	175	164	145	–
Bw1b2	69	18	13	8.0	0.3	57,753	7005	12,808	8	102	143	155	153	–
Bw2b2	78	11	11	8.0	0.3	9061	8329	14,149	45	182	179	193	218	–
Bw3b2	66	24	10	8.0	0.2	59,256	5509	10,166	8	60	89	92	96	–
Ab3	67	20	13	8.0	0.4	53,337	8323	14,944	45	116	164	185	168	–
Cb3	81	11	8	8.0	0.1	44,203	6530	11,920	75	94	124	217	147	–
A1b4	76	22	2	8.0	0.2	44,405	7118	12,721	45	110	131	192	170	–
A2b4	74	23	3	8.0	0.2	47,103	8686	15,234	60	172	209	264	213	–

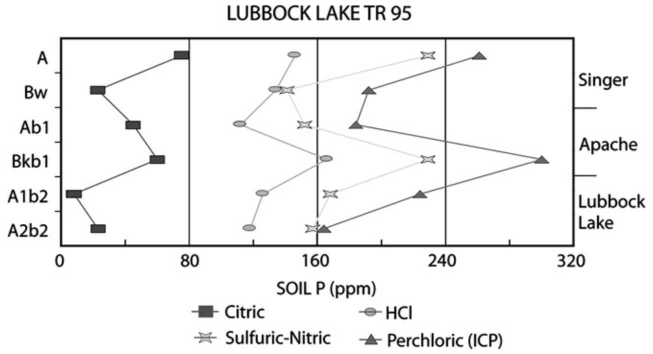

Fig. 2. Soil P trends at Lubbock Lake, Trench 95.

each acid is differentially reacting to the specific Psta- and Porg-derived compounds present in the Ab1 horizon.

All four techniques document an increase of soil P from the Bw to the A horizon in the Singer Soil (Table 5; Fig. 2). Yet, this increase is minimal for sample digestion in HCl. Given the comparatively high organic carbon content of the A horizon, it seems likely that HCl did not extract as much Psta or Porg derived compounds here as the other techniques. This interpretation is bolstered by the soil P trends at Trench 104 discussed in the next sub-section (see also Section 5.1 above).

Although geologic sources of soil P are minor at Lubbock Lake, they do influence soil P trends here. All four acid extraction techniques documented relatively high soil P values in the Apache Soil Bkb1 horizon (Table 5; Fig. 2).

We generally expect A horizons to have high soil P values since, by definition, A horizons represent stable ground surfaces and as such are the primary loci of human and biologic sources of soil P. Two major sources of clay at Lubbock Lake, dust and localized slopewash along valley margins (both originating from Pleistocene soils on the surrounding High Plains surface), have strongly influenced local soil morphology [95]. Inputs of aerosolic clay accelerated both calcic and argillic horizon formation here, with stage I carbonate morphology forming in as little as 200 years and illuvial clay horizons forming in as little as 450 years [95, 602–607]. Since the Bkb1 horizon has the highest clay content in Trench 95 (Table 4), it seems reasonable to conclude that inputs of aerosolic clays and translocation, the latter facilitated by shrink–swell, have influenced the Trench 95 soil P values as well. In sum, P-bearing particles and compounds have migrated down-profile at Trench 95 due to soil development over the last few centuries (see also Section 5.5 above).

9.3.2. Lubbock Lake Tr 104

Pedogenesis and geologic sources of P also influenced soil P trends from the Trench 104 sequence. As with the Trench 95 deposits, the magnitude of soil P at Trench 104 is roughly correlated with acid capacity (Table 5; Fig. 3). All four techniques produced broadly similar soil P trends at Trench 104. Yet, as with Trench 95, there are several deviations from these generalizations that illustrate the complexity of soil P chemistry and soil P dynamics.

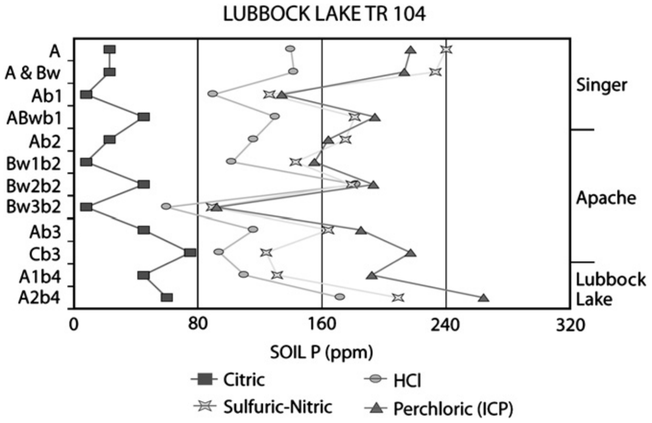

Fig. 3. Soil P trends at Lubbock Lake, Trench 104.

We expected, *a priori*, that the magnitude of soil P would be correlated with acid strength and concentration. At issue is the capacity of an acid to obliterate soil parent materials and liberate P molecules. Perchloric acid is the strongest and most concentrated acid in our analysis, yet the sulfuric–nitric acid and perchloric acid trials produced nearly identical numbers for each horizon of the Apache and Singer Soils (Table 5; Fig. 3). Indeed, sample digestion in sulfuric–nitric acid produced slightly higher soil P numbers than did perchloric acid in the organic-rich horizons of the Singer Soil and the Ab2 horizon of the Apache Soil (Table 5; Fig. 3). These horizons are relatively high in organic carbon, clay, and either Fe or Al (Table 4), suggesting that sulfuric–nitric acid digestion was particularly effective in extracting soil P compounds from organo-mineral complexes present at the site (see Section 5.4 above).

All four acid extraction techniques show a peak in the Bw2b2 horizon of the Apache Soil in Trench 104 (Table 5; Fig. 3). As with the Trench 95 sequence, the soil P peak in Trench 104 reflects the influence of aerosolic clays and translocation. Yet, with the exception of the HCl trial, the magnitude of soil P from the Bw2b2 horizon in Trench 104 is less than its pedostratigraphic equivalent in Trench 95. Parent materials for the Apache Soil vary spatially, with slopewash and gravel lenses present in some profiles [92 (pp. 943, 945), 93 (p. 1489)]. Carbonate clasts are also present in the B horizon of Trench 95, but not at Trench 104 [92, table 1]. Soil P variation across the Apache Soil is minor. Nevertheless, a comparison of soil P values from Apache Soil profiles shows that geology and pedology can influence soil P trends at an archaeological site.

The differential capacity of each acid to extract P, and sample-specific reactions, explain the additional variability in soil P trends at Trench 104. For example, all techniques document that the Singer Soil has more soil P than the Ab1 horizon of the Apache Soil. The difference is minimal for citric acid digestion and greatest for sulfuric–nitric acid digestion. HCl extraction suggests a slight decrease in soil P from the bottom to the top of the Singer Soil, while the remaining techniques suggest no change or a slight increase. As at Trench 95, HCl was not particularly effective for extracting Psta- and Porg-derived compounds at Trench 104.

All four extraction methods rank the cumulic horizons comprising the Lubbock Lake Soil, particularly the A2b4 horizon, as being high in soil P. Indeed, the A2b4 horizon had the highest levels of soil P of any horizon at Trench 104 – except for the citric acid trial where it ranked second to the Cb3 horizon (Table 5; Fig. 3). The relatively high soil P content of the Lubbock Lake Soil is not directly related to the organic carbon content of the soil or the number of P receptors (Table 4). Rather, the major difference between the A horizons of the Lubbock Lake Soil and the other soil surfaces is time. The A horizon of the Lubbock Lake Soil developed for several thousand years, in contrast to those for the Apache and Singer Soils, both of which formed over several centuries [95]. The Lubbock Lake Soil is also the oldest of the three soils. The net result is the sequestration of P, particularly Pin, in forms readily extracted by the acids used in this study (see Section 5.5 above). P mineralization is probably an important soil P process in the Lubbock Lake Soil, given the extended length of time for biologic inputs of P and the results of the citric acid trials.

The most striking anomaly to emerge from the Trench 104 analyses is the divergent soil P trends between the A1b4 horizon of the Lubbock Lake Soil and the overlying Cb3 horizon at the base in the Apache Soil (Fig. 3). Both the perchloric acid and the citric acid techniques document a trend of increasing soil P content during this transition while the HCl and sulfuric–nitric acid trials indicate decreasing P content. The physical and chemical attributes of the A1b4 and Cb3 horizons are not unusual (Table 4). We suspect that interfering substances enhanced color development for the A1b4 horizon during the citric and perchloric acid trials (see Section 9.2 above), particularly as these two acids often produced similar trends during our study.

More generally, another unusual trend among many of the soil horizons sampled at Lubbock Lake is the low level of P measured by the perchloric acid digestion (Table 5; Figs. 2 and 3). Indeed, some of the extractions by sulfuric–nitric acid are higher than the perchloric digestion. In theory, perchloric acid extracts all soil P while sulfuric and nitric acid extracts only some soil forms. This incongruity would suggest some procedural problems, but the trends between the two sets of extractions are generally similar (e.g., highest levels of P are in the A horizons). A combination of factors may explain the unexpected similarities between the two methods. The capacity of perchloric acid and sulfuric–nitric acid to extract P compounds is evidently similar, with the exception of the most resilient P bonds in the Relatively Stable Pool. These resilient Psta compounds may not be present in the A2b2 horizon of Trench 95 or in the Singer and Apache Soils at Trench 104. In other words, perchloric Ptot is roughly equivalent to sulfuric–nitric Pav plus sulfuric–nitric Pact plus some proportion of sulfuric–nitric Psta in the horizons noted above. Trace amounts of an interfering substance that enhanced color development during the sulfuric–nitric acid trials could then explain those few cases where it yielded higher soil P values than perchloric acid. Another possibility is that sulfuric–nitric acid extracts a form of soil P that perchloric acid does not. Whatever the reason, a similar trend is apparent at the Hulburt Creek Archaeological District, where the perchloric acid and sulfuric–nitric acid trials produced nearly identical soil P numbers below the planting surfaces.

9.4. Site specific trends: the Hulburt Creek archaeological district

Late Woodland peoples cleared and burned a large area to construct the Hulburt Creek ridged fields. Silts, clays, charcoal, and ash all eroded down gentle slopes (Bw2 horizon), burying the original surface of the ground (Ab horizon). Native peoples ditched this newly formed surface and incorporated excavated materials into the fields (the OA and Bw1 horizons). Yet, large-scale land clearance did not significantly change soil P values below the planting surfaces (Fig. 4), even though the accumulated materials at the base of the ridge (Bw2 horizon) are a full texture class finer than the buried soil (Table 4). Intensive human activities do not always affect soil P values (see Section 6 above).

Except for the planting surfaces, the capacity of an acid to obliterate P-bearing materials was not as important at the Hulburt Creek site as it was for many samples at the Lubbock Lake or English Camp sites. There is a general convergence of soil P values with increasing depth below the planting surface for all techniques. This suggests that Ptot at the Hulburt Creek site is dominated by Pav and Pact forms below the planting surfaces. It also suggests that native North American agricultural practices and biological activity can contribute many different P compounds to all soil P pools.

The perchloric acid extraction produced the highest values of soil P in the OA and Bw1 horizons. This was expected because perchloric acid is the strongest and most concentrated reagent in our study. However, the sulfuric–nitric acid digestion produced the highest values of soil P in the remaining deposits below the planting surfaces, which either formed in slopewash associated with aboriginal land clearance (Bw2) or represent the pre-agricultural soil (Ab, Bwb1, Bwb2, 2BCb, 2Cb). As noted at the Lubbock Lake site, the capacities of acids to extract P compounds become similar when Ptot is dominated by Pav and Pact. However, this cannot explain why sulfuric–nitric acid extracted significantly more soil P than perchloric acid in the Bwb1 horizon.

Drainage is one difference between the buried soil and the ridged-field deposits. Perhaps sulfuric–nitric acid obliterated all P-bearing parent materials in the redoximorphic horizons of the buried soil while perchloric acid did not. (Sulfuric–nitric acid also extracted a large amount of P from inundated strata at the British Camp site discussed below.) Another possibility is that an interfering substance enhanced color development during the sulfuric–nitric acid trial.

Analyses of the Hulburt Creek ridged fields did, however, produce broadly similar soil P trends, with one exception. Extraction with sulfuric–nitric acid produced a slightly higher soil P value for the Bw1b horizon than the overlying 2Ab horizon (Fig. 4). The difference is minor; 12 ppm, which is

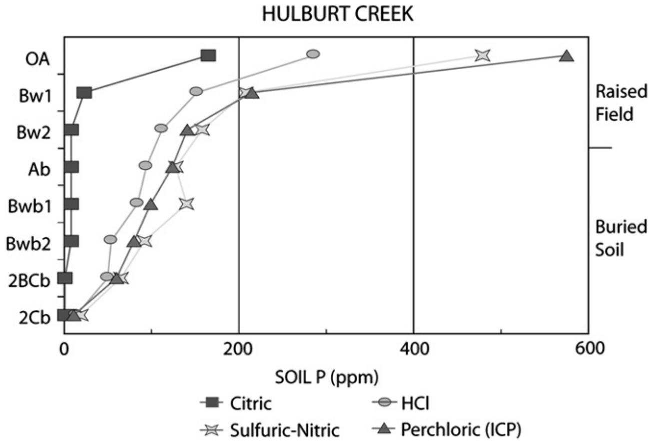

Fig. 4. Soil P trends at Hulburt Creek raised field locality.

slightly higher than the percent error established by our quality control protocols.

All four acid extraction techniques document the highest soil P values in the ridged-field planting surfaces (OA and Bw1 horizons in Table 6 and Fig. 4). This was expected, as the ridged-field planting surfaces were the primary loci for human activity and, following site abandonment, for biological inputs of P. Although the concentration of soil P within the ridged fields seems modest compared to many archaeological deposits, soil P levels in the planting surfaces are five times that of the pre-agricultural soil and nearly twice that of a nearby control profile [69, pp. 457, 459–460, table 6]. Planting surface maintenance, rather than ridged-field construction is primarily responsible for the elevated levels of soil P in the OA and Bw1 horizons (although ridged-field construction improved cultivating conditions, including soil fertility, in other ways [68 (pp. 673–675),69 (pp. 450–453, 459–467)].

Native peoples maintained soil fertility at the Hulburt Creek site through the burning of crop stubble and possibly woody materials gathered from nearby areas as well as the periodic refurbishment of planting surfaces with organic-rich ditch deposits. Thin section analysis identified badly degraded ash crystals as a major source of P and Ca compounds within the Hulburt Creek ridged fields [69, pp. 460–464, fig. 161]. The anthropogenic enrichment of P in the ridged-field planting surfaces is particularly evident in the citric acid trial, where the soil P level in the OA horizon is a full order of magnitude greater than the pre-agricultural soil (Fig. 4). Citric acid readily extracts P in the Pav pool, most Ca-bound P, and other weak to moderately sorped P in the Pact pool. Ash contributes P to these very sources. Badly degraded ash crystals were less common in soil strata associated with aboriginal land clearance (Bw2 and Ab horizons). Perchloric acid extracted far more soil P from the planting surface than did the other reagents, suggesting the presence of Psta compounds there. Evidently, the addition of organic-rich ditch deposits to planting surfaces not only contributed soil P but also influenced the redistribution of P into different pools (see Section 5.1 above).

Table 6
Hulburt Creek, physical and chemical characteristics of a raised field profile

Horizon	Sand (%)	Silt (%)	Clay (%)	pH	OC (%)	Ca (ppm)	Fe (ppm)	Al (ppm)	P (Citric) (ppm)	P (HCl) (ppm)	P (H_2SO_4– HNO_3) (ppm)	P ($HClO_4$ & ICP) (ppm)	P ($HClO_4$ & color) (ppm)	P (HCl & ICP) (ppm)
OA	32	62	6	4.1	5.0	356	15,113	19,149	165	286	479	575	432	201
Bw1	33	57	10	4.5	1.5	167	16,357	20,313	23	152	208	215	164	102
Bw2	33	49	18	4.7	0.7	157	17,798	24,781	8	112	158	141	99	67
Ab	33	52	15	4.6	0.8	142	16,962	22,746	8	94	128	124	135	41
Bw1b	34	47	19	4.6	0.5	134	21,287	24,636	8	84	140	99	–	27
Bw2b	54	35	11	4.8	0.5	136	14,938	17,076	8	54	92	80	–	19
2BCb	91	7	2	4.7	0.1	29	5968	4852	1	50	65	60	–	39
2Cb	99+	00	00	5.0	0.1	10	315	718	1	14	19	11	–	8

A general trend of decreasing soil P with depth at the Hulburt Creek site may not only be due to human activity at the surface. The near surface position of bedrock at the Hulburt Creek site restricts drainage, while the coarsely textured soils there limit soil water-holding capacity. The net result is a wildly fluctuating moisture regime in the buried soil, as reflected by the formation of prominent mottles in the 2BCb horizon and distinct mottles in the Bwb horizon [69, pp. 443, 452–453, figs. 142–145]. Redox conditions often, but not always, favor P desorption and mobilization (see Section 5.3 above).

Gartner [69, pp. 457, 459–460, table 6] analyzed the Hulburt Creek deposits and a control profile by the Bray-1 method (Table 3). In general terms, the Bray-1 procedure adds an additional reagent, ammonium fluoride, to the HCl extraction procedure. The addition of ammonium fluoride to the procedure doubled the amount of soil P extracted from the sample over extraction with HCl alone (the method used in this study). Fluoride ions form aluminum fluoride complexes in the extractant solution that limit P resorption by aluminum (see Section 9.2 above). The marked difference in soil P shows that resorption can be significant in studies that extract some proportion of Ptot.

9.5. Site specific trends: the British Camp site

Associations between human occupation and elevated levels of soil P are particularly dramatic at the British Camp site. The large midden analyzed here ("Operation A" in Stein [216]), is composed of bone, shell, fish remains and other organic discard. The midden often produced soil P values that are an order of magnitude greater than those from the other sites (Table 7; Fig. 5). Soil P values at the British Camp site are generally related to acid strength and concentration, with several notable exceptions. The perchloric acid trial produced soil P values that are a full order of magnitude greater than the sulfuric–nitric acid trial and two orders of magnitude greater than the HCl and citric acid techniques. Sample digestion with sulfuric–nitric acid produced higher soil P values than the HCl trials (Fig. 5).

We expected that HCl would extract more soil P than citric acid. This was certainly the case for strata J and M. However, the citric acid trials produced higher soil P values than the HCl trials in stratum C. Both techniques yielded comparable soil P values in strata D, K, and N. Why this is so is not readily obvious from the physical or chemical attributes of the deposits. Strata C, D, K, and N exhibit marked variation in terms of particle size, pH, and the concentration of Ca, Fe, and Al cations. Conversely, HCl extracted more P from strata J and M than did citric acid, even though both are significantly different in terms of their physical and chemical attributes (Table 7). As argued by MacPhail et al. [138], and as shown by the analysis of the Hulburt Creek planting surfaces, citric acid is very effective in extracting anthropogenic inputs from the Pav and Pact pools. The capacity of HCl to extract P becomes greater than that of citric acid as the P bonds become stronger.

Table 7
British Camp, physical and chemical characteristics of a shell midden

Stratum	Shell fragments (%)	Sand (%)	Silt (%)	Clay (%)	pH	OC (%)	Ca (ppm)	Fe (ppm)	Al (ppm)	P (citric) (ppm)	P (HCl) (ppm)	P (H_2SO_4–HNO_3) (ppm)	P ($HClO_4$ & ICP) (ppm)	P ($HClO_4$ & color) (ppm)	P (HCl & ICP) (ppm)
C	5	26	48	21	7.6	14.1	43,560	7437	7917	690	576	1776	17,705	–	–
D	5	39	34	22	7.8	13.8	43,461	7681	8784	585	646	1689	13,683	–	–
J	4	40	43	13	7.8	10.9	46,249	7035	8838	375	604	2148	14,332	–	–
K	8	53	33	6	7.8	6.1	37,610	9340	10,388	570	590	2039	37,056	–	–
M	7	51	35	7	8.0	6.5	56,414	4249	5012	338	576	1709	9051	–	–
N	20	48	22	10	7.7	11.2	32,804	10,609	11,869	570	590	1296	15,422	–	–

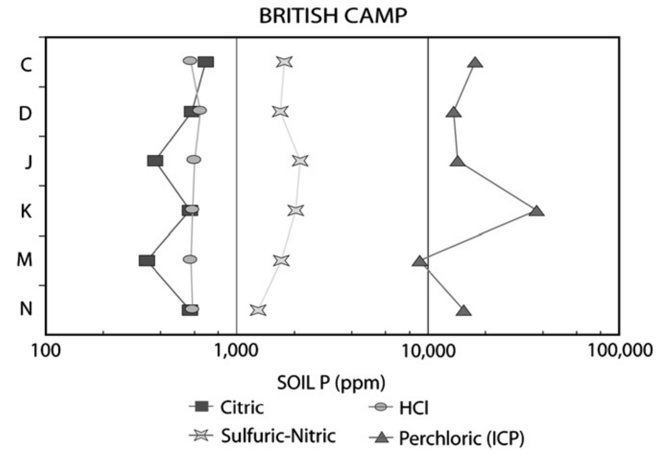

Fig. 5. Soil P trends at British Camp.

All of the extractants identified stratum M as the one with the least soil P, except for the sulfuric–nitric acid trial where stratum M had nearly 400 ppm more P than stratum N. In general, preservation of herring and other small bones in stratum M was less than that of the other strata due to groundwater inundation [215]. In addition to preservation issues, certain redox conditions also favor P mobilization (see Section 5.3 above).

The soil P trends generated by the citric acid and perchloric acid trials are broadly similar. So, too, are the HCl and sulfuric–nitric acid trends. For example, both the perchloric and citric acid trials indicate decreasing soil P values from strata K to J, while the HCl and sulfuric–nitric acid trials document an increasing trend over this transition. In addition, there is comparatively little variation in the down-profile trends for the HCl and sulfuric–nitric acid trials compared to that of the citric acid and perchloric acid trials (Fig. 5).

Each acid extraction technique documented a different P peak at the English Camp site; stratum C for citric acid, stratum D for HCl, stratum J for sulfuric–nitric acid, and stratum K for perchloric acid. Variable correlations between P peaks and P methods have also been documented in other comparative studies (see Section 8 above). Obviously, there are sample-specific reactions for each acid extraction technique. The question is why? We suspect, based on archaeological analyses [216], that the answer lies with variation in the type of organic discard through time. Organic discard significantly affects the forms, interactions, and redistribution of P (see Section 5.1 above). The variability noted above does not invalidate the value of soil P analysis, but it does offer a cautionary tale for the use of specific P values to infer specific human activities (e.g. [59]).

9.6. Soil P trends: acid strength and ICP spectrometry

The primary purpose of our study is to document variability in soil P trends as a function of acid strength and concentration. However, we used two different methods for measuring P concentrations: a colorimeter for the citric acid, HCl, and sulfuric–nitric acid trials; and an ICP spectrometer for the perchloric acid trial. Differences in analytical measurement introduce another variable to our study because the ICP spectrometer directly measures the number of P atoms in a sample solution while the colorimeter measures a property associated with P compounds (see Section 7 above). The subsequent sections explore the potential for a measurement bias in our study through additional analyses of selected samples from Lubbock Lake and the Hulburt Creek ridged fields. More specifically, we compare duplicate samples extracted with the perchloric acid on an ICP spectrometer and a colorimeter, then duplicate samples extracted with HCl and perchloric acid and measured on an ICP. This latter comparison shows the degree to which acid strength and concentration affect soil P values during ICP measurement. The colorimetry readings were conducted at Rock River Laboratories. The UW-Madison Laboratory for Archaeological Chemistry conducted the HCl extractions and ICP analyses.

9.6.1. Soil P trends: colorimetry and ICP spectrometry

Pre-treated samples from Trench 104 at Lubbock Lake and from the Hulburt Creek ridged fields were digested in perchloric acid and then measured for absorbance on a colorimeter. The results are graphed with the perchloric acid trial that was measured on an ICP (Figs. 6 and 7). The ICP and colorimeter produced broadly similar soil P trends, with several notable exceptions, indicating that any measurement biases were generally minor.

Measurements of soil P on the ICP and colorimeter produced nearly identical values for the Bw1b2 and Bw3b2 horizons at Lubbock Lake Trench 104 (Fig. 6). In general, with the exceptions of the Ab1 and Bw2b2 horizons, the ICP measurements often produced slightly higher soil P values than the colorimeter. There is a discrepancy of over 30% between the two forms of soil P measurement in the Cb3 horizon. Chemical compounds that interfere with color development and absorbance can significantly affect soil P values (see Section 9.2 and Jackson [104]).

The ICP generally produced higher soil P values than the colorimeter at the Hulburt Creek raised field site as well

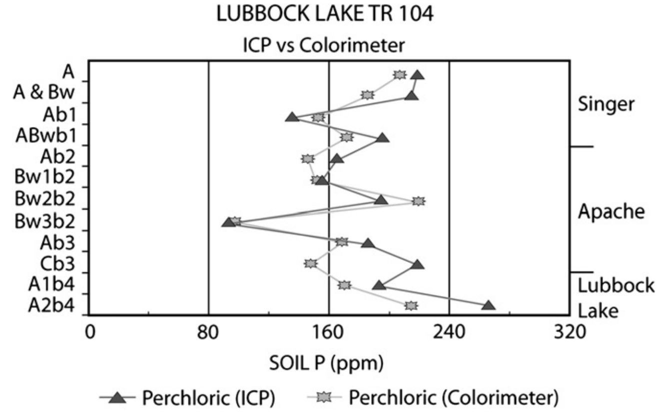

Fig. 6. Comparison of perchloric acid extractions from Lubbock Lake, Trench 104 samples measured by ICP vs. colorimetry.

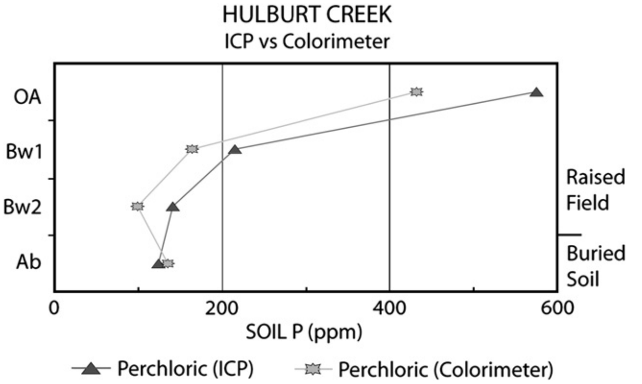

Fig. 7. Comparison of perchloric acid extractions from Hulburt Creek samples measured by ICP vs. colorimetry.

(Fig. 7). Although the trends are nearly identical within the raised field, the relative differences in percentage terms increase down-profile and approach 30% in the Bw horizon. The greatest absolute difference in soil P values is at the planting surface (OA horizon), and decreases down-profile. The colorimeter produced slightly higher soil P values than the ICP in the deepest (Ab) horizon. Perhaps aboriginal land clearance introduced organic compounds that survived pretreatment and enhanced color development of the Ab horizon during laboratory analysis.

9.6.2. Soil P trends: acid strength and ICP spectrometry

Selected pre-treated samples from Lubbock Lake Trench 95 and the Hulburt Creek ridged fields were measured on an ICP, but digested using both the perchloric acid treatment and HCl (Figs. 8 and 9). As expected, the magnitude of soil P is generally related to the capacity of the acid to obliterate parent materials in samples from both sites. Despite differences in the magnitude of soil P extracted by HCl and perchloric acid, both techniques produced very similar soil P trends at both sites. We note slight discrepancies in the 2BCb horizon at the Hulburt Creek site (Fig. 8) and the Bw horizon at Lubbock Lake Trench 95 (Fig. 9). These deviations may be related to the forms or amounts of Psta present in those horizons. Nevertheless, the soil P trends are remarkably similar.

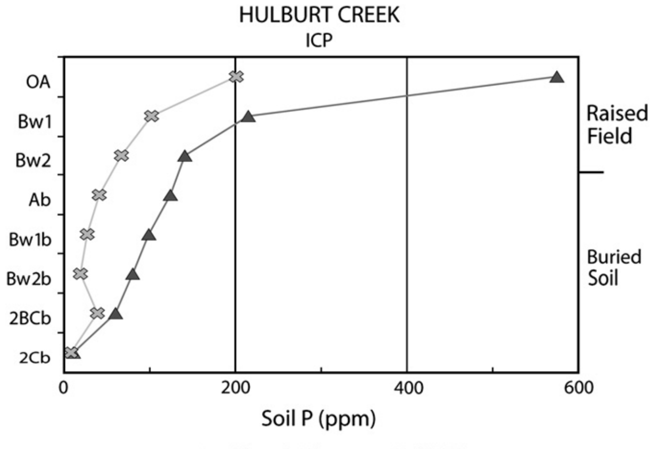

Fig. 8. Comparison of ICP measurement of perchloric acid vs. HCl extractions from Hulburt Creek samples.

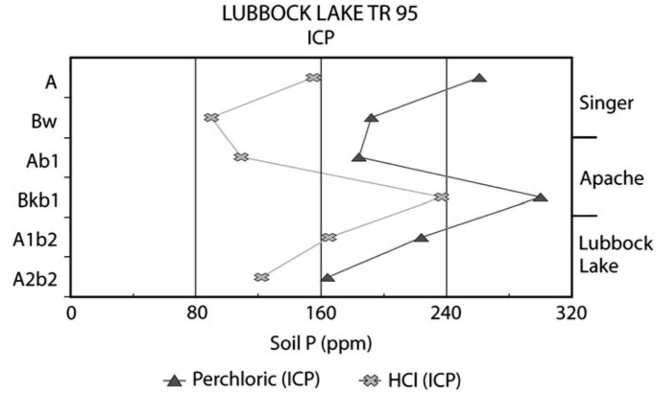

Fig. 9. Comparison of ICP measurement of perchloric acid vs. HCl extractions from Lubbock Lake, Trench 95 samples.

The perchloric acid trial extracted more than twice the soil P from the Hulburt Creek planting surface than the HCl trial (Fig. 8). Yet, the difference between the two sample digestion techniques is substantially lower in the pre-agricultural buried soil there. Since perchloric acid has a greater capacity to liberate P molecules than HCl, it seems likely that native practices of soil management and perhaps post-abandonment biological activity contributed Psta to the planting surfaces.

10. Conclusions

Phosphorus in soils has long been of interest to archaeologists because of its potential to inform them about the presence of past human occupation and to offer clues regarding the type and intensity of human activity. Soil P dynamics are quite complex, however. Phosphorus is a common allotropic element with many possible natural and cultural sources. It enters the soil through a variety of direct and indirect pathways. Phosphorus forms bonds with soil particles, primarily through biologic immobilization, chemical precipitation, and sorption. The precise form of Porg and Pin present in the soil, the strength of P bonds, and the loci of fixation within a soil horizon, depend largely on-site environmental factors.

Sandy soils, certain parent materials and organic amendments, redoxymorphic conditions, a neutral pH, and time all favor soil P transformation and mobilization. Nevertheless, P may form long-lasting bonds with soil particles. Although fixed soil P is stable for thousands of years, soil particles and compounds may be physically rearranged by geomorphic forces, soil formation, and disturbance. These processes can lead to the transfer and loss of both dissolved and particle-bound forms of soil P.

Human activity and the introduction of anthropogenic P further complicates the cycling of phosphorus through soil systems. Some human activities increase soil P levels, while other activities are phosphate neutral or deplete the soil of phosphorus compounds. Environmental conditions and the number of Pav receptors can also influence soil P trends.

Thus, the same activity in two different environments may result in two very different soil P signatures.

Data on soil P have been widely used by archaeologists and geoarchaeologists for a variety of purposes. Bethell and Máté [18, pp. 14, 16, 17] present a rather negative assessment of much of this work, noting that it has tended to support conclusions already drawn, has been used for a kind of fishing expedition (application of soil P analysis just to see what would turn up), and has rarely been used to find unseen and unknown sites. These are not unreasonable statements, but soil P analyses are useful. They help to direct excavations by locating activity areas, for example, and for delimiting site areas. Levels of Ptot and Pin seem to be the best indicators of human activity. Quantitative field extractions are best for fieldwork, but rapid lab analyses (e.g., various ICP techniques) are now commonly available as well. Specific fractions of P may also be indicative of specific kinds of human activity, but the complexities here are just emerging. There is probably much more complexity in soil systems and the P in them than we can explain at this point. Much more empirical data need to be gathered before specific relationships can be confidently offered. In particular, a variety of factors must be taken into consideration, including the chemistry of the original soils and sediments, the duration of pedogenesis, and landscape position. The specific laboratory procedures used to extract and measure soil P must also be considered when assessing soil P data. Interpretations of soil P generally seem strongest when supported by other information such as elemental and SOM data, and soil magnetism.

Comparative studies are a useful way to understand the utility, meaning, and complications of soil P measurements in archaeological contexts and for making archaeological interpretations. We measured duplicate samples of soil P from three very different archaeological sites with four separate acid extraction procedures. Selected duplicate samples were also measured on an ICP and a colorimeter.

Some variability is to be expected when conducting multiple trials on the same samples using the same methods. Nevertheless, our data (Figs. 2–9 and Tables 5–7) show that the results of soil P analyses can vary significantly with the type of analytical method. Indeed, the same sample can produce soil P values that vary over an order of magnitude or more simply as a function of laboratory method. Such variability indicates that inter-site comparisons of individual soil P values are inappropriate – if researchers have used different methods to extract soil P. Further, the amount of soil P in individual samples did not always match expectations based on our fieldwork.

Strong and concentrated acids have a greater capacity to obliterate soil particles and liberate soil P molecules than weak or dilute ones. Not surprisingly, sample digestion in perchloric acid produced the highest soil P values for 23 of the 32 samples in our study. However, sulfuric and nitric acid extracted more soil P than the other techniques, including perchloric acid digestion, for nine soil horizons. It is tempting to suggest that various acid extraction techniques discriminate among the different sources of soil P. Yet, both the sources of soil P and the deposits are very different and there are no common extenuating conditions for these nine horizons. Some were subjected to redoxymorphic conditions (e.g., the pre-agricultural soil at the Hulburt Creek site). Others were exposed to human activity (e.g., the Bw2 and Ab horizon at the Hulburt Creek site), and/or biologic activity (the A horizons for the Singer and Apache Soils at the Lubbock Lake Tr 104 and the Ab horizon at the Hulburt Creek site). The capacities of the two methods to extract soil P likely overlap when levels of Pav plus Pact plus some proportion of Psta approximate Ptot. Sulfuric–nitric acid generally extracted more soil P than HCl with the possible exception of the Bw2b2 horizon at Lubbock Lake where the difference is slight. HCl extraction produced higher soil P values than sample digestion with citric acid, except for stratum C at the British Camp site. The capacities of HCl and citric acid converge when Ptot is dominated by soil P forms from the Pav and Pact pools and if soil P is bound to Ca.

The association between human occupation and soil P is particularly dramatic at the British Camp site (Table 6; Fig. 6). The soil P values there are often a full order of magnitude greater than those from the other sites. These very high levels of soil P are not surprising given the nature of the site, a midden with abundant shell and bone.

Some human activities can, in certain environments, leave large quantities of Psta in the soil that are best extracted with a strong or concentrated reagent. Perchloric acid appears to be far more effective at extracting anthropogenic sources of soil P than the other techniques, at least for the sites in our study. Perchloric acid extracted significantly more soil P from the planting surfaces of the Hulburt Creek site, and an order of magnitude more soil P from the British Camp shell midden, than did the sulfuric–nitric acid. At the British Camp site, the differences in soil P levels between the perchloric acid and the citric acid trials are two orders of magnitude. This large difference is significant as one characteristic of an anthropic epipedon, according to the Soil Survey Staff [209, p. 13], is a minimum soil P level of 1500 ppm extractable by 1% citric acid. P levels from all midden strata easily exceeded this threshold during the perchloric acid trials. Yet, not a single stratum crossed the 1500 ppm soil P threshold during the citric acid trials, even though the deposits are demonstrably anthropogenic in origin.

Citric acid does extract many forms of soil P. In some cases, it extracted the same soil P forms as the more vigorous HCl and the sulfuric–nitric acid extraction techniques. However, human activity may produce soil P forms that are not easily extracted by citric acid and this may complicate efforts to identify and map anthropic epipedons.

This and other comparative laboratory studies show that correlations between P levels and human activity are partially a function of laboratory method. At the British Camp site, each acid extraction technique documented a different P peak; stratum C for citric acid, stratum D for HCl, stratum J for sulfuric–nitric acid, and stratum K for perchloric acid. This variability should be expected in a simple P test given the complexities of human behavior, site formation, soil P dynamics, and soil chemistry. More sophisticated analyses of multiple soil elements and compounds have the potential to explain the specific chemical reactions noted above.

Duplicate samples were extracted by the same acid and measured on an ICP and a colorimeter. The ICP generally yielded slightly higher numbers than the colorimeter, due to the presence of interfering compounds that affected either light transmittance or absorbance. For the most part, however, the two types of measurement produced similar soil P values.

Although the magnitudes of soil P values in our study are largely dependent on acid strength and concentration, the different acid extraction methods often, but not always, yielded similar trends. This is critically important for it underscores the rigor and analytical value of conducting soil P analyses during archaeological investigations. All techniques identified elevated levels of soil P at the British Camp shell midden and the Hulburt Creek planting surfaces. A horizons, the primary surface for human and biological activity, often had higher soil P values than the other horizons. There is some trend variability between the techniques to be sure. And, the results did not always match our expectations based on fieldwork. Yet, the discrepancies noted in previous sections also have significant archaeological value as they force attention to the dynamic nature of site formation and the complex qualities of archaeological site chemistry.

The importance of understanding site geoarchaeology and soil P dynamics is crucial in interpreting the results of soil P studies. The Lubbock Lake site well illustrates the importance of understanding all sources of soil P as well as soil formation at an archaeological site. Parent material variability influenced soil P values for certain analytical methods. Inputs of aerosolic clays and translocation influenced soil P trends at Trench 104 and Trench 95. Soil formation can rearrange soil particles and distort soil P trends as shown by the influence of shrink–swell and translocation on soil P values from the Trench 95 Bkb1 horizon. At the Hulburt Creek site, redoxymorphic conditions and coarsely textured deposits affected soil P trends in the pre-agricultural soil, while specific organic amendments influenced soil P trends in the ridged-field planting surfaces. Soil moisture conditions also affected soil P values for stratum M at the British Camp site.

A wide variety of methods for P analyses are available to archaeologists and geoarchaeologists. Only a few were examined here, yet our study clearly shows the importance of the acid extraction method on soil P results. Which method or methods are best for a particular project will depend on the research questions and the availability of equipment, time, and funds. This raises three points in closing. The interpretation of soil P trends is dependent on an understanding of site geoarchaeology and soil P dynamics. The comparison of soil P values, even Ptot values, are suspect if the analytical methods are different. Further, all soil P methods (or any lab methods, for that matter) should be thoroughly described or referenced to facilitate interpretations.

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