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# Variability of Laboratory Procedures and Results in Geoarchaeology

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Many laboratory techniques are available to geoarchaeologists to determine physical or chemical attributes of sediment or soil samples, and several techniques are often available for analysis of a single attribute. This is illustrated by analyzing duplicate sediment and soil samples from the British Camp Site (Washington) and Lubbock Lake Site (Texas) using two methods for organic carbon content, two methods for organic matter content, and three methods for calcium carbonate content, and several pretreatment procedures for particle-size analysis. These comparisons illustrate that different methods of analysis for the same property of duplicate samples can yield different results. Therefore, when making comparisons with data from other sites or other investigators, the types of laboratory methods used in geoarchaeological analyses are important considerations. Choice of a particular technique will depend on a variety of factors, often conflicting, including the archaeological and geoarchaeological questions being asked, the physical and chemical nature of the samples, and the accuracy, efficiency, and cost of the method.

## INTRODUCTION

With the growth of interest and research in geoarchaeology has come increased application of laboratory techniques developed in the earth sciences. There are a great variety of methods (e.g., Shackley, 1975; Page et al., 1982; Klute, 1986) and a variety of geoarchaeological applications, including sedimentology (e.g., Stein and Farrand, 1985; Stein, 1987), petrology (e.g., Kempe and Harvey, 1983), pedology (e.g., Holliday, 1989), soil chemistry (e.g., Eide, 1985), geochemistry (e.g., Lambert, 1984), and geophysics (e.g., Wynn, 1986). Moreover, because there are often a variety of methods available for determination of a single attribute, such as phosphate or organic carbon content, the results usually vary. This variability can lead to considerable confusion and misunderstanding when trying to compare and interpret results. The selection of one method over another requires a number of considerations.

This paper is a review of several methods and results for some of the more frequently measured attributes of sediments and soils in geoarchaeological research. The purpose is not to recommend one method over another, but to

## VARIABILITY OF LABORATORY RESULTS

illustrate the possible variability among techniques and results, to caution geoarchaeologists concerning comparisons with data from different investigators, and to point out some of the factors that must be taken into consideration when selecting a particular technique.

The samples analyzed are from two different kinds of archaeological sites in different settings: the British Camp Site (45SJ24) on San Juan Island, Washington, and the Lubbock Lake Site (41LU1), Texas. 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. During the past 2000 years, marine and terrestrial fauna and flora, as well as abundant mineral material were deposited along the shoreline (Stein, 1984a). Samples used in this study were collected from individual layers within the midden, as well as from exposures of non-cultural deposits located near the site. Lubbock Lake is in a dry valley on the semiarid Southern High Plains (Johnson, 1987). The samples are from the profiles of several well-drained soils developed in sandy, eolian sediment comprising much of the late Holocene valley fill (Holliday, 1985a,b).

The techniques compared are those for organic matter, organic carbon, and calcium carbonate content. Particle-size analysis is also discussed, although not compared thoroughly. Organic carbon (OC), organic matter (OM), and calcium carbonate ( $\text{CaCO}_3$ ) are important components of sediments and soils and can yield important clues for interpreting prehistoric activity and site formation processes (Shackley, 1975; Stein, 1984b). Organic carbon is a component of organic matter, which includes plant, animal, and microbial residues, fresh and at all stages of decomposition, humus, and inert carbon forms such as charcoal, coal, and graphite (Nelson and Sommers, 1982; Stein, 1984b). Calcium carbonate occurs in soils and sediments as both a primary mineral derived from, for example, limestone or shell, and as a secondary precipitate. Particle-size analysis (PSA; also referred to as grain-size analysis or granulometry) is a measure of the distribution of gravel, sand, silt, and clay content and is a basic part of sedimentological (including site formation) and pedological studies (Shackley, 1975; Stein and Farrand, 1985; Stein, 1987; Holliday, 1989). For this paper various methods were applied to duplicate samples for OC, OM,  $\text{CaCO}_3$ , and PSA. Results were then compared for similarities in absolute values and trends.

## METHODS

In the following discussion a variety of analytical techniques are reviewed. Methods that are published are cited and if the procedures are not published or are modified from published accounts, then they are described.

### Organic Carbon and Organic Matter

Organic carbon and organic matter were determined on samples from British Camp following four different techniques; two of the techniques were

## VARIABILITY OF LABORATORY RESULTS

employed on samples from Lubbock Lake. The OC content for samples from both sites was measured using the Walkley-Black technique (Nelson and Sommers, 1982). The Lubbock Lake samples were processed in the soils laboratory of the University of Colorado and the British Camp samples were analyzed in the soils laboratory of Texas Tech University. In this method a known quantity of a strong oxidizing agent is used to oxidize the OC. The amount of OC present is then determined by measuring the remaining oxidizing agent using reduction titration. Samples from British Camp were also analyzed for OC by calculating the difference between total carbon and total inorganic carbon from carbonates; the analyses were performed in the soils laboratory of Texas A&M University. Total carbon was determined by dry combustion, a method based on the oxidation of OC, thermal decomposition of carbonate minerals in a total-carbon analyzer (a medium-temperature resistance furnace), and measurement of the liberated CO<sub>2</sub> (Soil Survey Staff, 1972, method 6A2b; Nelson and Sommers, 1982). Total inorganic carbon from carbonates was measured using a Chittick apparatus (see below). A variation on this method of determining OC by difference is described by Foscolos and Barefoot (1970).

The OM content of samples from Lubbock Lake and British Camp was determined using a loss-on-ignition (LOI) technique (Stein, 1984b) and the OM in samples from British Camp was also measured using a modified hydrogen peroxide weight-loss technique (Robinson, 1927; Soil Survey Staff, 1972, method 6A3a). The Lubbock Lake samples were processed using LOI in the soils laboratory of the University of Colorado and the British Camp samples were analyzed by LOI in the archaeological sediment laboratory of the University of Washington. The peroxide method was carried out in the geomorphology laboratory of the University of Wisconsin. In the LOI technique the sample was crushed to pass a two mm screen, then dried at 100°C for one hour and ignited at 500°C to burn off organic matter. The weight loss calculated from before and after the 500°C burn represents the organic matter content of the sample. In the peroxide method the samples were dried and crushed to pass a two mm screen. Carbonates were removed using 10% HCl and a carbonate-free weight was obtained after drying. The samples were placed in 500 ml beakers and warmed to medium temperatures on a hot plate. Organic matter was oxidized by adding progressively more concentrated H<sub>2</sub>O<sub>2</sub> (5%, 15%, 33%) over a period of three days. The samples were then dried and reweighed and the OM content was calculated as the percent weight difference before and after the peroxide treatment.

### Calcium Carbonate

The CaCO<sub>3</sub> content of the samples was measured using three methods. Samples from British Camp and Lubbock Lake were analyzed using the acid-neutralization method (U.S. Salinity Laboratory Staff, 1954) and the

## VARIABILITY OF LABORATORY RESULTS

Chittick apparatus (Machette, 1986). All acid-neutralization analyses were conducted in the soils laboratory of Texas Tech University. A Chittick apparatus in the soils laboratory of Texas A&M University was used for the British Camp samples, and one in the soils laboratory of the University of Colorado was used for the Lubbock Lake samples. In both of these methods the carbonates are destroyed using HCl; in the acid-neutralization method the carbonate content is determined by measuring the amount of remaining HCl using titration and in the Chittick-apparatus method the volume of CO<sub>2</sub> gas evolved from the reaction is measured. The carbonate content for the British Camp samples was also determined using LOI (Stein, 1984b), performed at the University of Washington archaeological sediment laboratory. In this method samples are dried for one hour and burned at 1000°C. The weight loss calculated from before and after the 1000°C burn represents the carbonate content of the sample.

### Particle-Size Analysis

Particle-size analysis is most often conducted by either sieving or settling or a combination of methods (e.g. Shackley, 1975; Folk, 1980; Soil Survey Staff, 1984; Gee and Bauder, 1986; Singer and Janitzky, 1986), including various pretreatments. Sieving is usually used for fractionation of coarser material (sand-size and larger) and settling is used for PSA of the finer fractions. The settling method relies on the relationship that exists between settling velocity of particles in water and the particle diameter. This relationship is expressed by Stoke's Law, which essentially states that smaller particles settle at slower rates. There are two common methods of PSA by settling, hydrometer and pipet, and both were used in this study. In the hydrometer method a hydrometer is used to measure changes in the density of the water-sediment suspension at specific time intervals as the particles settle out. With the pipet, small subsamples of the sediment-water mixture are taken at specified depths at specified time intervals, dried and weighed. Calculations of the particle-size distribution are made using Stoke's Law following both procedures. Samples for PSA are commonly treated for the removal of secondary constituents, especially organic matter and carbonates, prior to the analysis.

In this study, samples from Lubbock Lake were analyzed using a combination of sieve, hydrometer, and pipet techniques and pretreated with sodium acetate (NaOAc) and H<sub>2</sub>O<sub>2</sub>. Samples from British Camp were analyzed using sieve and pipet techniques and pretreated using sodium hypochlorite (NaOCl; e.g., commercial chlorox bleach) and HCl. No samples were analyzed using all combinations of PSA and pretreatments. Thus, the comparisons suffer accordingly. The differences in results, however, based on methods of pretreatment, draw attention to the importance of carefully considering procedures before comparisons are made between sample results.

The particle classification for samples from Lubbock Lake is based on the

## VARIABILITY OF LABORATORY RESULTS

U.S. Department of Agriculture system for soils (Soil Survey Staff, 1975) with particles divided into sand (2 mm–.05 mm or 50  $\mu\text{m}$ ), silt (50  $\mu\text{m}$ –2  $\mu\text{m}$ ), and clay ( $\leq 2 \mu\text{m}$ ). Although the sands can be further subdivided, they were not in this study. The particle classification system used for samples from British Camp follows the phi ( $\Phi$ ) scale based on the Wentworth logarithmic scale (Folk, 1980, p. 23), both of which are commonly used in sedimentology (Blatt et al., 1980). The particles are classified at one-phi intervals: -1 (2 mm), 0 (1 mm), 1 (.5 mm), 2 (.25 mm), 3 (.125 mm), 4 (.063 mm or 63  $\mu\text{m}$ ), 5 (32  $\mu\text{m}$ ), 6 (16  $\mu\text{m}$ ), 7 (8  $\mu\text{m}$ ), 8 (4  $\mu\text{m}$ ), 9 (2  $\mu\text{m}$ ), 10 (1  $\mu\text{m}$ ), 11 (.5  $\mu\text{m}$ ).

Two approaches to PSA were employed on portions of the same samples from Lubbock Lake (all analyses carried out by the same analyst in the soils laboratory of the University of Colorado). In the first, organic matter and calcium carbonate were not removed from the samples. The samples were placed in a beaker with water and sodium hexametaphosphate for dispersing. The sand, silt, and clay content of the samples was then determined using the hydrometer method. In the second approach the samples were treated for removal of  $\text{CaCO}_3$  and OM, using  $\text{NaOAc}$  and 30%  $\text{H}_2\text{O}_2$ , respectively (Gee and Bauder, 1986). The sand was then wet-screened, dried, and weighed. The silt and clay mixture was dispersed using buffered sodium pyrophosphate and size fractions were determined using the pipet method.

Samples for PSA from the British Camp site (analyzed in the archaeological sediment laboratory of the University of Washington) were pretreated for organic matter using  $\text{NaOCl}$ , following Jackson (1969). The samples were then split into halves, one half pretreated for removal of carbonates (using  $\text{HCl}$ ) and the other half left untreated. Samples were initially washed through a four phi screen. The sand fraction (greater than 63  $\mu\text{m}$ ) retained on the screen was dried, sieved, and weighed. The silt-clay fraction was dispersed in sodium hexametaphosphate and analyzed in one-phi size intervals using the pipet method.

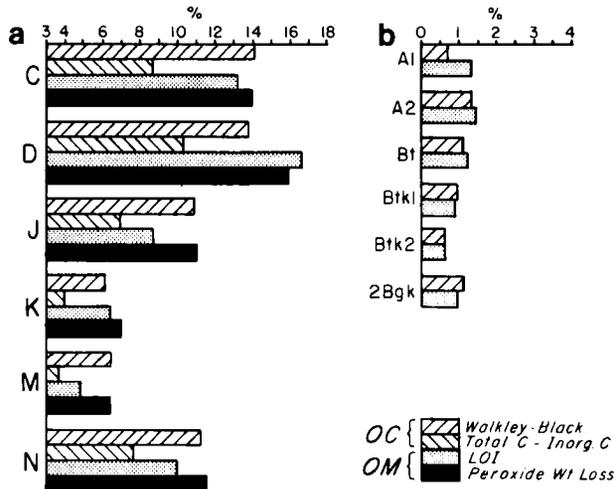
## RESULTS AND DISCUSSION

### Organic Carbon and Organic Matter

The four methods of OC and OM determination provide results with generally similar trends, but with some significant differences in absolute values (Figure 1). For the samples from British Camp the results from both methods of OM determination are generally similar. The OC content of the British Camp samples based on total carbon is consistently and significantly lower than those for OM. This relationship is to be expected because OM content should be 1.7 to 2.0 times that for OC (Nelson and Sommers, 1982). Considering this relationship, the levels of OC by Walkley-Black are surprising because the results are generally similar to those for OM.

In comparing the values of OM determined by loss-on-ignition with the values of OC determined by Walkley-Black, the OM is slightly lower than OC

## VARIABILITY OF LABORATORY RESULTS

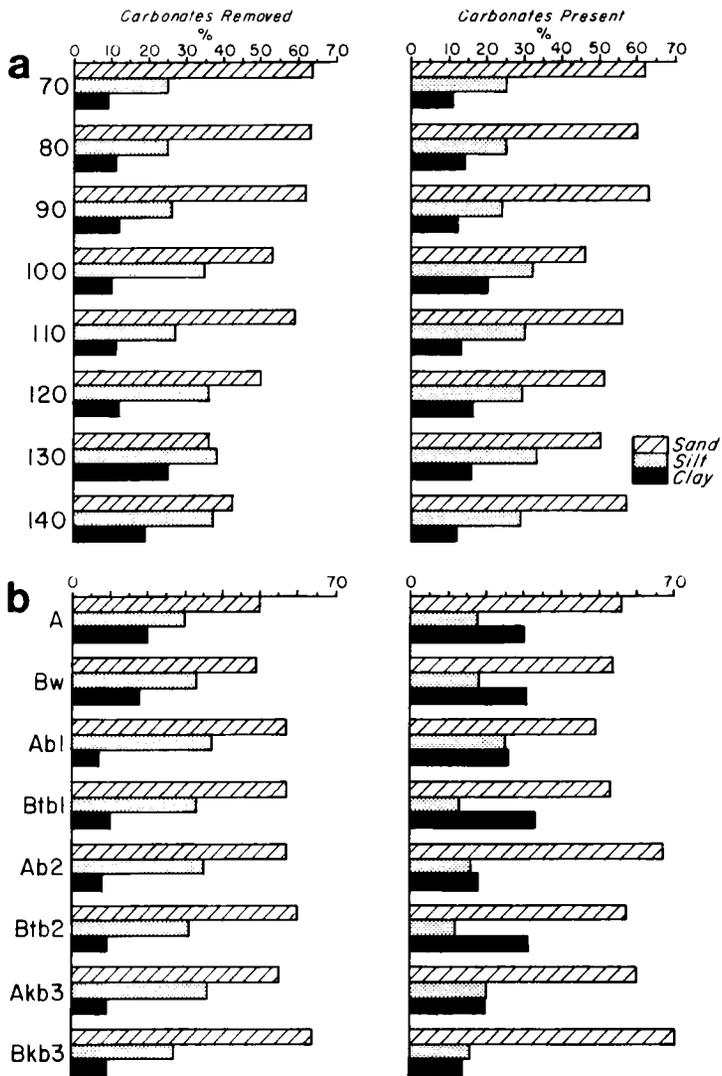


**Figure 1.** Comparisons of the results of analysis of samples from British Camp (a) and Lubbock Lake (b) for organic carbon (OC) content (Walkley-Black method and difference between total C and inorganic C) and organic matter (OM) content (loss-on-ignition method and weight-loss method using hydrogen peroxide). For British Camp samples the letters along the vertical axis are field designations. For Lubbock Lake samples the nomenclature along the vertical axis refers to soil horizons. Note that the scales for the horizontal axes differ for each site.

in four of the six samples from British Camp and the OM and OC are about the same in five of the six samples from Lubbock Lake (Figure 1). These data are in contrast to other published results that suggest that the LOI technique yields values that are consistently higher than Walkley-Black determined on calcareous and noncalcareous samples (Ball, 1964, ignition at 375°C and 850°C; Davies, 1974, ignition at 430°C). Clay and carbonate content can affect loss-on-ignition results (e.g., Davies, 1974) and these factors are accounted for in the results for the British Camp samples, although not those from Lubbock Lake. Such corrections in the Lubbock Lake samples would result in even lower loss-on-ignition values, although the Lubbock Lake samples contain generally less than 20% clay (on a carbonate-free basis) (Figure 2). The Lubbock Lake samples may simply have OC levels below the accurate resolution of LOI.

The mineralogy of the clay present in a sample can also affect LOI results (Ball, 1964; Dean, 1974). The samples from both sites are lithologically homogeneous; the Lubbock Lake sediments are quartzose with clays dominated by illite, mixed-layer illite-smectite, and minor amounts of smectite and kaolinite (Holliday, 1985a). No analysis has been done on the clay mineralogy of the British Camp samples. The British Camp samples analyzed in this study contain relatively small amounts of clay (generally less than 20% on a carbonate-free basis) (Figure 2).

## VARIABILITY OF LABORATORY RESULTS

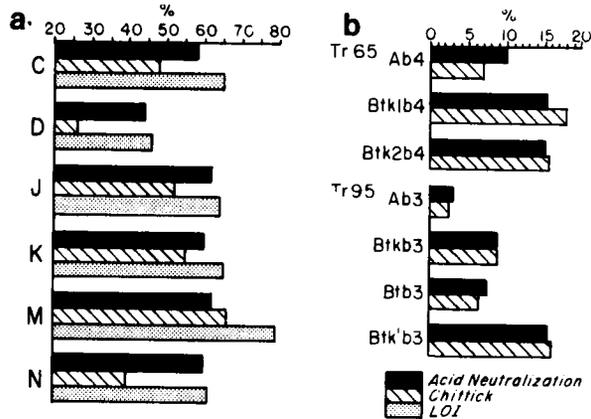


**Figure 2.** Comparisons of the results of particle-size analysis for samples pretreated for carbonate removal and samples not pretreated from British Camp (a) and Lubbock lake (b). Numbers and letters in left-most column refer to field designations of samples (depth below surface in cm for British Camp and soil horizons for Lubbock Lake).

### Calcium Carbonate

The results of the  $\text{CaCO}_3$  techniques are as variable as those for OC and OM. The acid-neutralization and Chittick methods yielded generally similar results for the samples from Lubbock Lake (Figure 3). For the British Camp

## VARIABILITY OF LABORATORY RESULTS



**Figure 3.** Comparisons of the results for three methods of analysis (acid-neutralization technique, use of the Chittick apparatus, and the loss-on-ignition technique) of samples from British Camp (a) and Lubbock Lake (b) for carbonate content. For British Camp samples the letters along the vertical axis are field designations. For Lubbock Lake samples the nomenclature along the vertical axis refers to soil horizons (the samples coming from soils in trenches 65 and 95). Note that the scales for the horizontal axes differ for each site.

samples the values from acid-neutralization are significantly higher than those from the Chittick method except for sample M (Figure 3). The results from loss-on-ignition are consistently 10–15% higher than those from Chittick and, except for sample M, consistently just a few percent higher than acid-neutralization. The significance of the difference between the values from the Lubbock Lake and British Camp samples is not known. A significant difference does exist in the nature of the carbonates from the two sites, however. The British Camp carbonate is from shell and limestone (primarily  $\text{CaCO}_3$ ), while the carbonate in the Lubbock Lake samples is pedogenically modified and derived from aerosolic dust and minor amounts of primary carbonate (also dominantly  $\text{CaCO}_3$ ) in the sediment.

### Particle-Size Analysis

A number of studies indicate that the results from hydrometer and pipet analyses are usually in agreement (Liu et al., 1966; Walter et al., 1978) with the pipet method, perhaps giving slightly better overall results (Sternberg and Creager, 1961). Because no one method of PSA was used on both the Lubbock Lake samples and the British Camp samples, the PSA results from the two sites cannot be compared. Within the Lubbock Lake samples different methods of PSA were also used before and after pretreatment. Even though this difference occurred, most of the variability in the Lubbock Lake results can probably be attributed to the effects of pretreatment, because the PSA methods have been tested and seen to give similar results. The PSA results from both

## VARIABILITY OF LABORATORY RESULTS

Lubbock Lake and British Camp show marked differences between samples that were pretreated and those that were not (Figure 2). The comparisons from both sites show higher clay contents in the untreated samples, although the differences are more pronounced in the Lubbock Lake values, and the untreated samples from British Camp also show higher fine silt content in the untreated samples. The presence of the carbonate could have flocculated the clay and produced higher coarse silt and sand content, but it appears that the addition of a dispersant to the samples prevented this. The presence of increased fines in the untreated samples is probably a measure of the carbonate particles, which at both sites are largely illuvial.

This study, although not thoroughly rigorous, suggests that secondary accumulations of organic matter, carbonates, and salts should be removed from samples for PSA because they can induce erroneous readings (Gee and Bauder, 1986). If, on the other hand, constituents such as carbonates are primary components of the sediments (e.g., the sediments are derived from limestone or contain shell), then pretreatments should not be carried out. In either case the methods of pretreatment and PSA should be clearly stated to avoid inaccurate comparisons between data sets.

## CONCLUSIONS

This study indicates that the results of analyses for organic carbon and organic matter content, and calcium carbonate content can vary significantly as the analytical methods vary. Variability in results between laboratories, due to operator variability, is another potential problem, but one not easily documented. The study also indicates that methods of pretreatment are important considerations in PSA. The purpose of this study, however, is not to recommend any particular method or to address the determination of any specific characteristic of geoarchaeological sediments. Rather it is to make several more general points concerning analytical methods in geoarchaeology.

There are a great variety of analytical techniques from which to choose in geoarchaeological studies and there are often a number of methods available for analysis of a single attribute. As the methods vary, however, so can the results. In choosing a particular technique a number of factors must be considered and these considerations are sometimes at odds. For example, in PSA a decision to forego sample pretreatments makes for a quicker procedure, but one that yields results considerably less accurate if secondary (post-depositional) components are present. The decision of whether to pretreat samples also depends on interests concerning analysis of microartifacts (sand-sized artifacts). Some pretreatments can damage or destroy sand-sized artifacts (e.g., shell-tempered sherds, bone, and charcoal). If such analysis is desirable, then pretreatment is ignored. The factors involved in deciding which techniques to use should be included in published discussions of methods.

## VARIABILITY OF LABORATORY RESULTS

Accuracy, efficiency, and cost are usually the primary considerations in selecting among laboratory techniques, including those described in this paper. All of the procedures discussed require accurate weighing to several decimal points and, therefore, an analytical balance is necessary. Otherwise the accuracy, speed, and cost, including equipment requirements, can vary considerably among the methods described. The methods of PSA are relatively inexpensive, requiring only moderate investment in glassware and reagents. The pretreatments are simple but can be time consuming and the hydrometer method, although significantly quicker than pipet, is also time consuming. As mentioned, however, there is evidence to suggest that PSA by pipet is somewhat more accurate.

Most of the OM and OC techniques yield similar trends, if not similar values. The Walkley-Black OC method can yield variable results (Nelson and Sommers, 1982), but is used widely because it is relatively simple and requires only a moderate investment in laboratory equipment. The analysis of OC by calculating the difference between total C and inorganic C is generally reliable, but requires a total carbon analyzer, which is expensive. The loss-on-ignition and peroxide methods for OM give variable results, especially the latter (Nelson and Sommers, 1982), but both are simple, quick, and inexpensive techniques.

For carbonate analyses, loss-on-ignition is the simplest method, but the Chittick method probably yields more reliable results. Both methods appear to produce very similar trends, however, and the Chittick method is also very simple, although requiring a moderate investment in the apparatus and greater operator skill.

In deciding on laboratory procedures the research questions involved are to be considered. In particular, if comparisons are to be made with the work of other investigators, then the same procedures must be used. This raises the final point. The methods used in any analytical study should be explicitly identified and referenced or described.

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