Phytoliths as an interpretive device of paleoenvironments in archaeological sites

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PHYTOLITHS AS AN INTERPRETIVE DEVICE OF
PALEOENVIRONMENTS IN ARCHAEOLOGICAL SITES

By
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B.A., University of Montana, 1967

Presented in partial fulfillment of the
requirements for the degree of
Master of Arts
UNIVERSITY OF MONTANA
1972

Approved by:

[Signatures and dates]
ACKNOWLEDGMENTS

The patience and guidance of my committee members, Dee C. Taylor, Floyd Sharrock of the Department of Anthropology and Gary Crosby, Department of Geology are appreciated as are the courtesy and assistance of Dale Fredland, University of Montana, who so graciously permitted sampling of site 24RB1012. I thank Mary Van Gilder for field and laboratory assistance. Laboratory facilities used at the University of Montana were made available by Leland M. Yates of the Department of Chemistry.

At Washington State University, I am indebted for assistance from the Department of Botany, especially to Rexford F. Daubenmire; Henry W. Smith and Rose Okazaki, Department of Agronomy and Soils, are thanked for their guidance throughout this study. Arthur L. Cohen and Edward Steever, Jr., Electron Microscope Center, provided generous assistance and instruction regarding use of the transmission electron microscope. G. A. Crosby, Department of Chemistry, assisted with chemical processing when the project first began.

This study was supported by the Quaternary Studies Option in Anthropology at Washington State University (funded by NSF grant #G2 1353), by a Northwest Scientific Association summer grant, and by a grant-in-aid for summer research through the Department of Anthropology at Washington State University.
The Department of Anthropology has given much additional support to this study by extending the use of faculty, equipment, and laboratory facilities. Carl E. Gustafson, Peter J. Mehringer, Jr., Henry T. Irwin, and Richard D. Daugherty gave their assistance and encouragement which has been sincerely appreciated. Most of all I would like to thank Roald Fryxell and William B. Hall, University of Idaho, for their cooperation, patience, and editing. Geneva Burkart, Jan Smart, and Sue Fletcher, Washington State University, typed early drafts of this thesis, and Alice L. Campbell typed the final copy.
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CHAPTER I

INTRODUCTION

Phytoliths are minute bodies of isotropic opaline silica which has been precipitated within the cells of grasses, sedges, reeds, and some woody plants. Baker (1959a) suggests that phytoliths may have been deposited as unwanted material, or as reinforcement of cell structures.

Silica in plants was first observed in 1804 (de Saussure) and it was noted then that graminaceous plants contained more silica than leguminous types. It also has been noted that the percentage of silica present in the plants was affected by the amount of monosilicic acid in the soil solution (Jones and Handreck, 1965a).

Opaline silica has certain advantages over pollen for paleoenvironment interpretations: (1) phytoliths are not easily destroyed by variations in soil chemistry; (2) they are less susceptible to decomposition by weathering; (3) they normally are deposited in situ by decomposition of plants rather than transported by wind; and (4) they may be more readily identifiable for grass family and genus than pollen has proven to be.

Verma and Rust (1969, p. 749) discuss the paleoclimatic utility of phytoliths and conclude that,
Opal phytoliths are released from different plants (Jones and Milne, 1963; Tyruin, 1937) containing them and are incorporated into the soil during the decomposition or destruction of grasses and forests or sometimes via the dung of animals. Identification of such opal phytoliths separated from the soil can be a useful tool in the interpretation of the past vegetation.

This thesis has three distinct parts dealing with phytolith analysis: (1) the study of phytoliths from a bog in north central Washington; (2) the comparison of phytolith analysis with pollen analysis from an archaeological site; and (3) development and description of laboratory procedures for isolation and study of phytoliths.

The bog used in the first analysis was Creston Bog. Since Creston Bog was analyzed earlier for pollen by Hansen (1944, 1947) with published results, the bog is an ideal location for phytolith recovery and study.

The purpose of the first part of this research was threefold:

1. To discover if phytoliths were present, and to what depth. The preservation of phytoliths has been questioned by many researchers in the field. Creston Bog, with its volcanic time-stratigraphic markers, Mazama Ash and Glacial Peak Ash, is an excellent locality to check for the presence of phytoliths at a known age (Powers and Wilcox, 1964; Fryxell, 1965),

2. To record the variety of phytoliths isolated and;

3. To see whether phytoliths vary in type with depth and stratigraphy. Previous studies have primarily measured the numbers of phytoliths present in the soil, rather than exploring stratigraphic differentiation.
The second part of this study utilized soil samples from an archaeological site near Colstrip, Montana, excavated by members of the University of Montana Department of Anthropology. The purpose of this section of the study was also threefold:

1. To determine if phytoliths are preserved in a steppe environment,

2. To compare phytoliths from living plants to those recovered from the soil in one specific area, and,

3. To compare phytolith analysis with pollen analysis of an archaeological site.

The main purpose of this thesis, as a whole, was to explore further the extent to which phytolith analysis may be useful archaeologically in attempts to reconstruct paleoenvironments.
CHAPTER II

REVIEW OF LITERATURE

Previous studies of phytoliths have followed several lines of investigation: physical and chemical description, morphology, physiological and environmental significance, and some aspects of the effect of weathering processes on opal. The least known aspects of phytoliths involve their specific identification and significance, especially environmentally. The term phytolith has been defined as follows:

The term phytolith, implying the stone part of a plant, appears to be used only for bodies which are minute parts of the plants which secrete them. It is not applied to diatom skeletons where the secretion is the full size of the organism. Though the term might logically be used for any mineral substance secreted by a plant (e.g., calcium carbonate in Chara), in all the cases dealt with in the present paper the material is opal, SiO$_2$.nH$_2$O (Smithson, 1958).

Physical and Chemical Description

Silicon dioxide (SiO$_2$) can be found in three crystalline and one amorphous forms in nature: tridymite, cristobalite, quartz, and opal. The first two of these are rare high temperature and/or high pressure forms; they are of no concern to this problem. The third, quartz, may be found in both macrocrystalline and crypto-crystalline forms. It also may combine with water and form the fourth, amorphous form, opal.
The following table comparing specific gravity, refractive index, and water content is taken from Smithson (1956b).

Table 1.—Comparison of specific gravity, refractive index, and water content in various siliceous minerals.

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Specific Gravity</th>
<th>Mean Refractive Index</th>
<th>Water Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>2.65</td>
<td>1.55</td>
<td>nil</td>
</tr>
<tr>
<td>Chert and flint</td>
<td>2.62</td>
<td>1.55</td>
<td>trace</td>
</tr>
<tr>
<td>Opaline silica</td>
<td>2.15</td>
<td>1.43</td>
<td>up to 10%</td>
</tr>
</tbody>
</table>

Opaline silica occurs in many environments in nature. According to Seiver (1957), opal occurs as an alteration product of volcanic ash and as "a deposit from thermal spring water" (Stevens, 1967). It is also found in diatoms, sponges, and some plants (opal phytoliths).

Of the amorphous forms, opal phytoliths, presumed to have formed in living plants, make up the major portion of this material in the soils of the world. The names given to the various forms of amorphous silica change as the degree of hydration of the SiO₂·nH₂O changes, with silica gel representing the most highly hydrated and chalcedonite, one of the least hydrated. Opaline material is considered the least hydrated form found in plants. Because the name given to a silica material changes with dehydration, different researchers refer to opal phytoliths as different materials (Stevens, 1967).

Opal phytoliths are not pure SiO₂·nH₂O; they contain many impurities as shown by chemical analysis in Table 2 compiled by Kanno and Arimura (1958).
### Table 2.—Impurities found in opal phytoliths

<table>
<thead>
<tr>
<th>Impurity</th>
<th>%</th>
<th>Impurity</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>84.93</td>
<td>CaO</td>
<td>2.04</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>1.12</td>
<td>Na₂O</td>
<td>3.44</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>.87</td>
<td>K₂O</td>
<td>.97</td>
</tr>
<tr>
<td>TiO₂</td>
<td>.00</td>
<td>H₂O(−)</td>
<td>1.28</td>
</tr>
<tr>
<td>MnO</td>
<td>.02</td>
<td>H₂O(+)</td>
<td>4.93</td>
</tr>
<tr>
<td>MgO</td>
<td>.55</td>
<td>P₂O₅</td>
<td>.02</td>
</tr>
</tbody>
</table>

The actual nature of silica contained in plants has been discussed at length by many authors. Lanning (1958) states that the silica in the culm epidermis of papyrus consists of both quartz and opal. Jones and Handreck (1967) refuted Lanning's statement by explaining that the "dry" ashing method used by Lanning caused some of the opal to become tridymite and cristobalite, the high-temperature forms of quartz. This conclusion was verified through experiments conducted by Jones and Milne (unpublished).

Opaline phytoliths contained in plants can be categorized as: (1) cell lining, (2) filling plant cells, (3) actual cell wall replacements, (4) mineralized structures resembling the internal cuticular ribs, (5) mineralized plant hairs, spines, and hooks, (6) other microscopic bodies secreted by a plant (Baker, 1959b; Pease, 1967).

There are contrasting views on the utility of opal contained in plants. According to Richardson (1920):
There can be no doubt that plants acquired the silica habit early in their evolutionary history and it may be found to function physiologically, osmotically, or structurally. It is difficult to think of an active surviving plant organism absorbing and storing up such a substance, which has and can have no real and positive use in its life cycle.

Other writers have agreed with Richardson and have conducted further research into the possible usefulness of silica structures in plants. Iler (1955), especially, reports that Raleigh, Okawa, and Lipman all consider silica important in plant growth.

Lundie (1913), German (1934), Wagner (1940), and Yosli (1941), as reported by Iler (1955) demonstrated that the deposition of silica in the epidermis of plants increases their resistance to such fungus diseases as rust, mildew, and rice-blasts. The structural support, created by the deposition of silica, has been shown in many plants, such as scouring rush.

Because no specific essential function for silica has been found in plants containing large amounts of silica, many researchers, like Iler, are following the idea expressed by Frey-Wyssling (1930). Frey-Wyssling believed that the secretion of silica in plants was merely a separation of non-assimilable material taken in with the transpiration stream. Frey-Wyssling pointed out that in most plants, silica is deposited in peripheral tissues and along conducting vessels similar to the separation of calcium salts in some plants. This can be considered as increasing evidence supporting the assumption that silica is deposited as a surplus from transpiration. This deposition in peripheral tissues also helps explain the highly silicified elements of plant structure, such as the stinging hairs of nettles (Stevens, 1967).

According to Parry and Smithson (1958), Esau says the bulliform cells in grass have the function of unrolling leaves during development, and hygroscopic opening and closing of mature leaves. Parry and Smithson also think
these deposits would upset the plants' hygroscopic and water storage functions and cause dessication of the leaf. A general theory for the utility of silica in plants thus has not been generally accepted.

Phytolithic silica was identified as opal SiO_2·nH_2O by its index of refraction and isotropic character (Parry and Smithson, 1957; Smithson, 1958). Jones and Milne (1963) identify the index of refraction as ranging from 1.42 to 1.44 and the specific gravity as 2.04. More variation has been observed, however, in the index of refraction and specific gravity than this, as shown by Jones and Beavers (1963); refractive index ranges from 1.41 to 1.465, and specific gravity from 1.50 to 2.30. Kanno and Arimura (1958) and Brydon (1963) found similar results for specific gravity as well as for index of refraction. Jones et al. (1966) confirmed by X-ray that phytolithic silica was amorphous (i.e., not crystalline and thus not quartz). Phytoliths vary in size from 2 to 1,000 microns, most occurring between 20 to 200 microns.

Morphological Studies

The morphology of phytoliths has been studied intermittently since Ehrenberg's (1845,1847) classification of ten genera and 90 species of phytolitheria. Folger et al. (1967) continued this classification by listing two additional genera and eight species.
In 1969, Twiss, Suess, and Smith, seeking to treat phytoliths not as independent biologic entities but rather as specific plant parts, developed four classes and related three of these to grass subfamilies. In refining that study, Lutwick (personal communication) has made a preliminary identification of phytoliths distinctive of one particular genus of Gramineae with subdivisions representing six species.

Parry and Smithson (1964) have described phytoliths from living plants and related them to different areas found in the plant, and note that phytoliths may vary in plants of the same species. In 1957 they discussed the possibility that phytoliths can be misinterpreted owing to improper laboratory or preparation methods. Pease (1967) described and illustrated phytoliths from root tissues of *Bouteloua eriopoda*, black grama. He described them as being rectangular in shape. It will later be illustrated in this paper that these phytoliths are indistinguishable from some discovered in sediments of Creston Bog. Black grama is not presently found in Washington; the rectangular shape, furthermore, is not diagnostic of this plant (see Chapter VIII). Other complications are illustrated by studies of woody plants. Garber (1966), for example, processed many wood species of plants from northern Idaho and discovered phytoliths in only Douglas-fir and western larch. It may be that he used the non-accreting portions of the plants, since Pease (1967) found phytoliths in various trees, such as ponderosa pine, pinyon pine, and juniper.
Phytoliths as Interpretative Devices

The utility of phytoliths as interpretative devices is best summed up by Dormaar and Lutwick (1968) when they say:

Those (phytoliths) deposited in the leaf epidermal cells assume shapes characteristic of the grass in which they are found. These opal phytoliths, when found in soils, can be used as indicators of the vegetative history of the site (p. 29-30).

Many soil scientists have recovered phytoliths from various soil types; Pease (1967) used them in an attempt to distinguish A horizons in paleosols, and Garber (1966) found a correlation between phytoliths and spodosols. Lutwich and Johnson (1968) and Dormaar and Lutwick (1968) have used the presence of various grass phytoliths to delineate grassland movements in transitional areas. Witty and Knox (1964) use phytoliths similarly in north central Oregon, as did Verma and Rust (1969) in southeastern Minnesota. They have also identified some of the phytoliths as to various species of plants now growing in the area. Jones and Beavers (1963) discuss the distribution of phytoliths with depth in some Illinois soils and conclude that the variations are mainly due to the time gradient in the deposition of the loess.

Rovner (1971) most recently morphologically studied phytoliths from 30 live plant specimens and typed 16. He recorded differences in the phytoliths from a few major plant groups, but made no further interpretations.
Stability of Opal Phytoliths in Soil Environments

Opinion differs regarding the stability of phytoliths in soils. Gill (1967) called attention to this diversity of opinion, pointing out that Baker believed that phytoliths usually last less than a 1,000 years, and Wilding (1967) stating he has a date of 13,000±450 years. Gill himself noted preservation of fossil phytoliths in sediments of Tertiary and Quaternary ages.

It seems evident that phytoliths may indeed persist for 13,000 years as suggested by Wilding, or even longer perhaps, if they have been preserved by a reducing environment, such as at Creston Bog.
CHAPTER III

METHOD

Techniques for the extraction and concentration of phytoliths from soil, peat, and/or fresh samples have been the subject of numerous investigations. Both "wet" and "dry" ashing techniques have been developed for extraction.

"Wet" ashing involves digesting the plant material with mixtures of either sulfuric and chromic acids or nitric and perchloric acids." Following this procedure the material should be washed "successively with hydrochloric acid and distilled water" (Jones and Handreck, 1967, p. 125).

The "wet" ashing method is preferred to "dry" ashing (which involves igniting the plant materials at temperatures between 450 and 900°C) because the latter tends to change the opal to cristobalite and tridymite and also fuses many of the phytoliths. Unfortunately, many investigators have continued to use the "dry" ashing technique regardless of its disadvantages. Incorrect physical and morphological interpretations of the opaline silica bodies may thus have resulted unnoticed.

Methods for extraction of phytoliths described below are modifications of those discussed by Moody (1971) and Rovner (1971). Figure 1 is a flowsheet showing these methods.
Extraction from Soil

Pease (1967) and others have used mesh screens for extraction of phytoliths from the clay and silt fraction of soil. This method was found unsatisfactory because larger phytoliths are lost in the coarse fraction. Consequently, heavy density separation was used in this study and found to be more effective.

Experimentation with many different heavy density liquids was performed, resulting in the use of zinc bromide, or bromoform—somewhat less efficiently. In peat samples it was discovered also that the plant and other organic material should be removed prior to heavy density separation if a complete assemblage of phytoliths is to be obtained.

The extraction method for soil samples containing little organic matter is:

1. Place approximately 25 ml of soil material in a beaker with calgon; mix well.
2. Rinse thoroughly with distilled water.
3. Add 10% HCl. Mix to make certain that HCl comes into contact with all phytoliths which may be cemented together with carbonates. The suspension may be heated to accelerate the process.
4. Rinse thoroughly with distilled water.
5. Mix soil and water until a vortex forms.
6. Let particles settle to the bottom until only clay remains floating, following Stokes law and formula of sedimentation (Krumbien and Pettijohn, 1938).
7. Decant clays (check to see whether phytoliths are discarding accidentally).
8. Stir rapidly again forming a vortex and pour immediately into another container, separating the finer fractions from the sand.

9. Transfer the decanted silt and phytolith material to a 15 ml centrifuge tube (preferably glass).

10. Rinse, centrifuge, and decant material.


12. Centrifuge, decant.

13. Let sample sit undisturbed until most of acetone is evaporated.

14. Add heavy density liquid, and agitate completely. Prepare the zinc bromide by dissolving the crystals in water until the mixture has a density of about 2.3 (determined by weight). If a heavy liquid density hydrometer is available, make the solution a little heavier than 2.3 to account for the small amount of acetone that will be present. If no hydrometer is available, make the solution to about 2.4. Determine the exact weight by weighing it. Then, dilute the mixture with water until it reaches a specific gravity of 2.3. The amount of water needed can be determined by the formula:

\[ Z(Sp.Z) + H_2O = 2.3 (Z + H_2O) \]

where \( Z \) = amount in ml of zinc bromide and water,
\( Sp.Z \) = specific gravity of above liquid,
\( H_2O \) = amount of ml of water,
\( Sp.H_2O \) = specific gravity of water, and
2.3 = specific gravity needed in final form.

The only unknown in the above formula is the amount of water needed, so one needs only to solve for \( H_2O \). Add this amount of water to the liquid already made and the result will be a mixture of zinc bromide and water with specific gravity of 2.3.

15. Spin sample in centrifuge for 20 to 30 minutes.

16. Decant into 50 ml centrifuge tube (preferably polypropylene), and dilute with distilled water and rapid fix to clean out bromides.
17. Centrifuge, decant. Repeat steps 16 and 17 until bromides are no longer present.

18. Rinse, centrifuge, decant until the sample is clean.

19. If sample contains organic material, transfer it to a 15 ml centrifuge tube and add the chromic-sulfuric acid solution.

20. Rinse, centrifuge, and decant until sample is clean. (The wash by centrifuging is accomplished in 3 minutes at 2,000 rpm).

21. Wash with different strengths of ethyl alcohol or acetone, depending on type of oil used for mounting.

Extraction from Plant Samples

The following procedure for removal and isolation of phytoliths from plant samples is adapted from the procedures of Moody (1971) and Rovner (1971):

1. On fresh plant materials, wash either whole or shredded samples with detergents and HCl successively to remove any extraneous materials.

2. Rinse with distilled water thoroughly.

3. Prepare sulfuric-chromic acid solution by adding 800 cc of concentrated sulfuric acid to 500 cc of a saturated solution of potassium dichromate and water. (If the potassium dichromate is supersaturated, crystals will form; they do not interfere with the rest of the reactions.)

4. Add about 10 ml of acid mixture to sample, stir with glass rod and agitate. Heat tubes for 2-4 hours under exhaust hood. In samples of peat or muck, the sample may be left in the solution overnight. The acid will turn green when the reaction is completed.

5. Centrifuge and decant liquid into large beaker of water. If organic material is still present, do not decant; instead remove by pipetting the middle portion of acid, add fresh acid, and repeat steps 4 and 5.
6. Wash with distilled water, centrifuge, and decant; repeat as necessary for removal of all the acid.

7. For fresh plant samples, use step 21 of Extraction Technique for Soil Samples.

8. Sample is ready for slide mounting in oil, Canada Fir Balsam or Thermoplastic Transparent Cement.

In transmission electron microscope work, the phytoliths are best observed if left in distilled water.

**Extraction from Peat**

Extraction of phytoliths from peat (Moody, 1971) has been accomplished successfully with the following procedure:

1. Place 2 grams of peat in small (15 ml) centrifuge tubes with dilute HCl. This must be agitated to make certain the HCl comes into contact with all phytoliths that may be cemented together with carbonates. The tubes may be heated slightly to accelerate the process.

2. Centrifuge for three to five minutes; decant off liquid. Repeat steps 1 and 2, if necessary.

3. Rinse thoroughly with water; agitate, centrifuge, and decant liquid at least three times, or more, if necessary.

4. Follow steps 4 to 6 of the fresh plant phytolith extraction method.

5. Water should be removed from the sample so that the heavy density separation will work. This may be done by evaporating slowly, by allowing it to dry overnight, or by using acetone.

6. Add about 10 ml of zinc bromide solution to the dried samples, agitate, centrifuge for at least 10 minutes (some samples take longer).

7. Pipette the phytoliths from the top of the mixture and place in clean centrifuge tube.

8. Dilute with water, agitate, centrifuge, and decant off liquid.
9. Wash thoroughly with water, agitating, centrifuging, and decanting.

10. Wash a minimum of three times with increasing strengths of ethyl alcohol or constant full strength acetone, depending on type of oil used for mounting.
Fig. 1.—Flowsheet showing steps involved for extraction of opal phytoliths from plant, peat, and soil materials.
CHAPTER IV

MATERIAL ANALYZED FROM CRESTON BOG, WASHINGTON

Material analyzed for study of phytoliths in peat was recovered from core samples collected at Creston Bog, two miles east of Creston, Washington, and a few hundred feet south of U.S. Highway 2. It is located in Section 13, Township 26N, Range 34E. Both Rigg (1958) and Hansen (1944) describe this bog.

Hansen says:

... A swamp formed in a scabland channel about 10 miles east of Wilbur, Washington. This swamp is situated near the edge of the timbered zone, and an occasional western yellow pine (Pinus ponderosa) occurs on favorable local sites. The surface is covered with a swamp associes of plants, with standing water in the center. The latter contains both floating and submerged seres. The thickness of the organic sediments in the area of the sampling is 2.6 meters, and they rest directly upon basalt. Silt is the principal component of the lowest decimeter of sediments, followed upward by 2 meters of limnic peat and about 0.5 meter of fibrous peat composed of sedges, cattails, bulrushes, and water smartweed. The volcanic ash stratum occurs at 1.2 meters, while a sharply defined layer of diatomite is present at 0.4 meters. To the naked eye, these two strata look very much alike, while palpably they are also similar.

Samples used in this study were taken at 10 centimeter intervals from a core measuring 2.8 meters long. Ten of these samples then were processed for the phytoliths they contained to show differentiation through depth.

The stratigraphic profile is shown on Fig. 2. This core illustrates different details of stratigraphy than
$x = \text{Sample Point}$

- **Existing Surface**
  - Peat and Silt
  - Ash and Diatoms
  - Ash and Silt

- **Reworked**
  - Mazama Ash
  - and Loess

- **Mazama Ash (6,700 years B.P.)**

- **Peat**
  - Diatoms and Silt

- **Peat**

- Depth to Bedrock
  - Undetermined

*Fig. 2.—Sketch of monolith showing stratigraphy at Creston Bog.*
either of those studied by Hansen or Rigg. The core did not reach the bottom of the bog. It was collected by Dr. Henry W. Smith, Department of Soil Science, Washington State University, in the area designated on Fig. 3 after Wildesen (1971).

The samples were collected following natural stratigraphy; where there was no variation in the stratigraphy, the core was sampled at 10 centimeter levels, for a total of 30 samples. Ten of these samples were processed for this thesis.
Fig. 3.—Map of Creston Bog showing area of sampling.
CHAPTER V

OBSERVATIONS OF PHYTOLITHS COLLECTED
FROM CRESTON BOG, WASHINGTON

Observations of the phytoliths recovered from sediments collected at Creston Bog were made by the use of a Nikon petrographic microscope and a Phillips 100 Transmission Electron Microscope; descriptions were made with the aid of the Nikon.

The phytoliths made a natural separation when centrifuged in distilled water, with the larger phytoliths (100-200 microns) on the bottom, and the smaller (2-100 microns) on the top. The separation is recognizable by eye because the organic material that escaped disintegration in the treatment process remains with the smaller phytoliths, thus giving the deposit a darker color. To prepare slides of the phytoliths, one must be certain to get samples from both size categories.

Since a key to phytoloth identification has not been developed, the phytoliths from Creston Bog were classed morphologically, according to external form. Twenty-four morphologic types of phytoliths were observed and sketched. These forms are shown as Figs. 4-9. The samples were counted in the same manner as reconnaissance pollen studies (200 phytoliths in each sample), recording the numbers present of each form for each sample.
Fig. 4.—Morphologic types of phytoliths found at Creston Bog.
Fig. 5.—Morphologic types of phytoliths found at Creston Bog.
Fig. 6.—Morphologic types of phytoliths found at Creston Bog.
Fig. 7.—Morphologic types of phytoliths found at Creston Bog.
Fig. 8.—Morphologic types of phytoliths found at Creston Bog.
Fig. 9.—Morphologic types of phytoliths found at Creston Bog.
Some of these types (i,r,s) showed variation through time, represented by stratigraphic prevalence and relative depth; these are shown in Fig. 10. The third type (s) is thought to be from a conifer tree, while the two others (i,r) are found mainly in grasses. When Fig. 10 and Fig. 2 are correlated, some interesting phenomena appear. The conifer type is present in sediments beneath Mazama Ash, then declines and the grass types dominate. Another high frequency of the conifer type occurs midway between the Mazama Ash and modern flora. In Hansen's "Postglacial Vegetation of Eastern Washington" (1944), all his pollen diagrams from the area, including Creston Bog (Wilbur) appear to have an increase in conifers at approximately the same period (Fig. 11).

When the electron microscope was used, far more detail of the phytolith morphology was evident than was originally visible by light microscope. Figures 12-14 were taken on the electron microscope. From these pictures, Lazelle (1971) has identified the rectangular phytoliths (Figs. 12b,12d,13a,13c) with holes along one side as being derived from conifers. Scalloped types (Figs. 12a,12c) are from graminaceous species. It was noticed that there were many nonopaline crystals present with the conifer phytoliths. These may eventually help in identification of the latter.

When observing phytoliths from organic sediments such as peat material, there is a great problem with diatoms. Many of the diatoms have similar density as the phytoliths
Fig. 10—Three morphologic types of phytoliths found at Creston Bog, showing percentage of total phytoliths per level each one constitutes.
Fig. 1.—Pollen diagram of Harrington sedimentary column.

Fig. 2.—Pollen diagram of Wilbur sedimentary column.

Fig. 3.—Pollen diagram of Liberty Lake sedimentary column.

Fig. 4.—Pollen diagram of Eloika Lake sedimentary column.

Fig. 11.—Pollen diagrams of bogs in Eastern Washington (Hansen, 1944).
Fig. 12.—Transmission electron photomicrographs of phytoliths from Creston Bog. Magnifications are: a. 1600x, b. 6000x, c. 2100x, d. 2400x.
Fig. 13.—Transmission electron photomicrographs of phytoliths from Creston Bog. Magnifications are: a. 3000x, b. 12,100x, c. 4800x, d. 3000x.
Fig. 14.—Transmission electron photomicrographs of phytoliths from Creston Bog. Magnifications are: a. 1800x, b. 2500x.
and float with them. Fragmented diatoms are difficult to distinguish from phytoliths, but may be separated by optical means (see Chapter VI).
CHAPTER VI

DISCUSSION OF
CRESTON BOG MATERIAL

After using both petrographic and electron microscopes for examination of phytoliths, it seems apparent that only the electron microscopes provide sufficient morphological detail to be recorded to provide a reasonable basis for reliable identification and classification of all but the most distinctive phytoliths. It further seems probable that a scanning electron microscope (SEM) would provide a more detailed and three dimensional-appearing view of these features (Wilding and Drees, 1971) than is possible with the transmission electron microscope (TEM); stereoscopic SEM photography of phytoliths clearly is both feasible and desirable.

Other mechanical devices also have proven useful in making easier the extraction and study of phytoliths from organic sediments. They are:

1. A water pump device to reclaim the zinc bromide.

2. The use of an oil with a refractive index matching diatoms (thus optically eliminating the diatoms by making them invisible, and avoiding confusion of them with phytoliths).

3. Use of a bent plastic tube during heavy density separation to pour off the phytoliths rather than removing them by pipette, as has been done previously.
Phytoliths, as well as pollen, present many difficulties when used as interpretative devices of paleoenvironments. Some of the most evident problems include the following:

1. Phytoliths are subject to alluvial and colluvial transport, and, therefore, may be mixed, broken, worn, or partly corroded.

2. In porous material, such as coarse sand or gravel, phytoliths are subject to vertical transport by pedologic eluviation/illuviation processes, and, therefore, may not always appear in their proper stratigraphic context.

3. Plants of different species do not contain and, therefore, do not deposit equal amounts of phytoliths, and, therefore, may not provide accurate quantitative representation relative to one another.

4. Plants must be identified by suites of phytoliths in some cases; many types are found consistently in generically different plants, although many types seem to be specific.

5. It may be difficult to recognize climax communities as opposed to seral communities, and, therefore, the microenvironment recorded by phytoliths does not necessarily reflect a whole habitat type.

6. Similarly, topographic, edaphic, and climatic climaxes cannot be distinguished.

7. Finally, although phytoliths are direct indicators of the plants which produce them, they obviously are only indirect indicators of the nature of the environment.

Regardless of these obvious problems in phytolith analysis, some positive conclusions are warranted:

1. Phytoliths have been proven to be present for more than 6,700 years because they are found below the volcanic ash marker horizon of the Mazama eruption (Powers and Wilcox, 1964; Fryxell, 1965).
2. Many of the phytoliths at Creston Bog vary in quantity and proportion with stratigraphy, and thus eventually may be correlated with pollen diagrams (see p. 30).

3. Some of the phytoliths have been described as conifer types; others are in the process of identification.

4. Finally, it became obvious during this study that phytoliths are too small for proper study with the petrographic microscope. A transmission or scanning electron microscope must be used for clear identification. At the present time, the use of the TEM has many drawbacks: they are expensive machines, the grids are actually too small for counting, and many of the phytoliths are too large to be photographed effectively. Because of these reasons, the petrographic microscope was used on the remainder of this study in spite of its own limitations.
CHAPTER VII

MATERIAL ANALYZED FROM SITE 24RB1012

The materials analyzed for the second part of this thesis were taken in the field from Site 24RB1012 or Colt 45 Shelter by Mary Van Gilder and the author. In this case, terrestrial sediments low in organic matter, and associated with an archaeological site, were sampled for comparison and contrast with Creston Bog. At the time of sampling, the site was being excavated by a crew from the University of Montana under the supervision of Dale Fredland. The site is located about seven miles southwest of Colstrip, Montana.

Phytoliths were extracted from a series of 13 soil samples, which were taken adjacent to a preserved stratigraphic section, or monolith (for technique used, see Fryxell and Daugherty, 1964; Smith and Moodie, 1947; Smith, McDreery, and Moodie, 1952). The monolith, which serves as a documentary record of the sediments, is described and sketched on Fig. 15 and Appendix I. Since the monolith and samples are not exactly the same, due to some lateral variation, the soil sample description is found in Appendix II; pH values from the samples are shown on Table 3.

Soil samples were taken at each discernable stratigraphic level, determined by texture, structure, and color.
Fig. 15.—Sketch of monolith showing stratigraphy at 24RB1012. Carbon-14 dates were supplied by Dale Fredland (personal communication).
The site, 24RB1012, is located in the Bouteloua gracilis vegetation province (Daubenmire, personal communication). Because of the predominately sandy soil on which it is growing, the vegetation present is considered an edaphic climax vegetation. Plants were collected in the immediate area of the site and were identified later by Dr. Robert Turner of the University of Texas. The plants selected are: Helianthus petiolaria, Stipa comata, Andropogon hallii, Eurotia lanata, Yucca glauca, Cirsium, and Pinus ponderosa. These plants were processed by the method described earlier for fresh plant phytolith extraction.
CHAPTER VIII

OBSERVATIONS FROM MATERIAL COLLECTED
AT SITE 24RB1012

Seven plants were processed for the extraction of phytoliths because of their present abundance at the site and their probable representation among phytoliths extracted from stratigraphic samples. Descriptions and sketches of morphologic types of phytoliths for each plant follow; each is numbered arbitrarily so that plants identified and phytoliths extracted from them have identical numbers.

Phytolith Descriptions

1. Helianthus petiolaria

One distinctive type of phytolith was found in Helianthus petiolaria (sunflower). This phytolith, a circular cell filling one (Fig. 16, lc), is called flower type in the rest of this thesis. The rest of the phytoliths distinguishable for this plant (square, rectangular, and bar, Fig. 16, la and lb) are found in all of the other plants examined and thus are not distinctive although the square one (la) has an extension on one side.
Fig. 16.—Sketches of phytoliths from fresh plant samples. The plants are: 1 = Helianthus petiolaria; 2 = Stipa comata; 3 = Andropogon hallii; 4 = Eurotia lanata.
2. *Stipa comata*

Three fairly distinctive phytolith forms (Fig. 16, 2a,b,c) were found in *Stipa comata* (needle and thread grass). The first is a serrated wide rectangular form, called serrate; the second is a capsule shape, thus called capsule; the third is triangular with concave sides.

3. *Andropogon hallii*

Both *Andropogon hallii* (big sand bluestem grass) and its phytoliths superficially resemble *Stipa comata* and its phytolith assemblage. One phytolith form (Fig. 16, 3c) that is very nearly the same as 2a, the difference being mainly the width of the phytolith, in that type 3c is a micron or more narrower. The other two types, 3a and 3b (Fig. 16), are variations of the dumbbell type described by Rovner (1971). These dumbbell types are quite distinguishable from the capsule type above.

4. *Eurotia lanata*

Phytoliths with distinguishable shapes encountered in *Eurotia lanata* (winterfat) were all less than two microns. Both square and lenticular types (Fig. 16, 4a,b,c) were found.

5. *Yucca glauca*

Phytoliths found in this plant, *Yucca glauca* (yucca), are less than two microns. One of the small rod forms
appears to have a projection on one end perpendicular to the body (Fig. 17, 5a,5b).

6. Cirsium

Four distinctive types of phytoliths (Fig. 17, 6a, b,c,d) were extracted from Cirsium (common thistle). The first of these is a lenticular type much larger than that described for Eurotia lanata; the second is a consolidation of many of the rectangular ones into a brick laying form; and the third is a rod that is partitioned off every two microns. The fourth phytolith type is rectangular and is more clearly three dimensional than most, with one of the dimensions grooved.

7. Pinus ponderosa

Many amorphous phytoliths were extracted from Pinus ponderosa but only the one, 7c, described in Chapter VI (Fig. 5) is distinctive. Figure 17, 7a, shows a wedge shape that also is found in Pinus ponderosa (ponderosa pine); 7b is one of the amorphous types.

General Comments

Phytolith types described from living plants were sought in the 13 soil samples also collected from Site 24RB-1012. Equal volume of each soil sample was processed for the extraction of pollen and phytoliths, using the method described in this thesis for the latter. When slides were
Fig. 17.—Sketches of phytoliths from fresh plant samples. The plants are: $5 = \text{Yucca glauca}$; $6 = \text{Circium}$; $7 = \text{Pinus ponderosa}$. 
made of the materials extracted, and examined with a light microscope, data on the following tables (Tables 4-5) were collected.

**Table 4.--Phytolith distribution through depth at site 24RB1012**

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<th>Sample</th>
<th>Depth (in)</th>
<th>Phytolith Plant No.</th>
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<tr>
<td>1</td>
<td>1</td>
<td>X</td>
</tr>
<tr>
<td>2</td>
<td>1-1/2</td>
<td>X</td>
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<tr>
<td>3</td>
<td>1-1/2</td>
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</tr>
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<td>5-1/2</td>
<td>X</td>
</tr>
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<td>7</td>
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<td>X</td>
</tr>
<tr>
<td>8</td>
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<td>X</td>
</tr>
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<td>9</td>
<td>9-3/4</td>
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<td>X</td>
</tr>
<tr>
<td>13</td>
<td>21-1/2</td>
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Table 5.— Pollen analysis from site 24RB1012

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<td></td>
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<td>7-11</td>
<td>12-13</td>
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<tr>
<td>Helianthus</td>
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<td>...</td>
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<td>c</td>
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<td></td>
<td>b</td>
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<td></td>
<td></td>
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<td>b</td>
<td>c</td>
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<td>Cirsium</td>
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<td>c</td>
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<td>10</td>
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<td>c</td>
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<td>c</td>
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<td>4</td>
<td>...</td>
<td>b</td>
<td>c</td>
</tr>
</tbody>
</table>

^aDepths of the samples are indicated in Appendix II.

^bNot enough pollen to count; mostly *Artemisia*.

^cStill not enough pollen but similar to Sample 4.
CHAPTER IX

DISCUSSION OF
MATERIAL FROM SITE 24RB1012

Most phytolith types, such as 1c and 6d discussed in the previous chapter, were not found in the processed soil samples. Probably the delicate nature of these types resulted in crushing after burial and before processing. Even though these types are distinctive for the particular type of plant in which they are found, they are not useful in paleoreconstruction of archaeological sites with this particular type of sediment. These particular types of phytoliths may preserve well in lake sediments or in bogs. Phytoliths from other plants not processed are present.

Phytolith occurrence plotted as a graph in Table 4 record Type 1 to be present in abundance throughout the whole stratigraphic section. This relative frequency is misleading, because the types described as 1a and 1b (and 5a also) are common phytoliths found in nearly all plants.

Occurrence of the phytoliths from plants 2 and 3 is noteworthy also; only the phytoliths distinctive to the given species were listed. The upper two stratigraphic levels which are mixed with manure contain Andropogon hallii and Stipa comata; thus the presence of the former was not found else-
where except in level 8. Phytoliths from *Stipa* in level 8 may indicate either a change in environment or a change in feeding habits of animals grazing in the area; it also may indicate a change in the type of animal inhabiting the site.

Although there is an abundance of *Pinus ponderosa* in the area, phytoliths of this type are scarce in the sediments. This scarcity may be due to the fact that the tree has not been present at this exact location, for even at present, no needles have collected here from a tree only 100 feet away. *Pinus ponderosa* phytoliths thus illustrate one problem in the use of phytoliths in the interpretation of paleoenvironments: phytoliths present in the soil will be mainly from plants either growing on the surface at a particular time or phytoliths brought in from plants elsewhere by man or other animals. Since man's activities often kill off plants in the immediate area of occupation, the sample of phytoliths would be extremely biased. On the other hand, this same localization of phytolith preservation may be advantageous in recording introduction and concentration of plants used as food.

The pollen record from Site 24RB1012, Colt 45 Shelter, is incomplete in the central part of the stratigraphic section (Table 5). The area in which the pollen count diminishes abruptly is in the B2 horizon (zone of eluviation or position of maximum clay accumulation in the soil profile). This relationship does not appear coincidental; probably it is due to the combination of two factors: (1) the continual
wetting and drying of this portion of the profile and (2) the accumulation of carbonates and other alkaline substances in this level due to illuviation/eluviation processes.

The pollen and the phytolith analyses obviously are not directly comparable; some interpretations may be acceptable and useful, particularly if both tools are used simultaneously. There is a definite drop in Pinus pollen after the third level and an increase in Artemisia with depth (time, see Fig. 15). If this information is coupled with the knowledge of changes in grass, recorded by phytoliths, a change in vegetation may be inferred to have occurred sometime after the third stratigraphic level was deposited. Interpretation is dependent on more extensive data.

Other interesting phenomena in this data consist of noting that neither pollen or phytoliths distinctive of Helianthus are present even in surface layers despite its presence at the site. Another point is that Gramineae pollen is indistinguishable between the genuses discussed in this study and was also extremely scarce in the samples. Grass phytoliths were the only ones that were identifiable in the phytolith analysis.
CHAPTER X

SUMMARY

Conclusions reached in the present study may be listed as follows:

1. Phytoliths are present in sediments older than 6,700 BP.

2. Phytoliths may change in relative frequency of one morphologic type to another through time.

3. Phytoliths are preserved in a dry steppe environment, in this case better than pollen.

4. Pollen from the same site is diminished from weathering and the effect of bases when the B2 horizon is reached.

5. Most phytolith types distinctive enough to identify are those from grasses; the minute size of some phytoliths and the fragility of others from various plants reduce their usefulness.

6. At the present time, phytoliths may be used in determining the kinds of grasses used by man but not as an interpretative device for past environments; in some cases it may be used to supplement the pollen analysis of a site.

Eventually it may be possible to utilize phytoliths for interpretation of paleoenvironments in areas in which man has not influenced, such as bogs and lakes. Also, if one samples in numerous places around an archaeological site, a more complete sample of the phytoliths may be recovered. A comprehensive collection of phytoliths should be made in all areas, on all types of terrain.
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APPENDIX I

DESCRIPTION OF A STRATIGRAPHIC SECTION AT SITE

24RB1012, COLT 45 SHELTER

<table>
<thead>
<tr>
<th>Horizon</th>
<th>Depth below Designation Surface (ft.)</th>
<th>Horizon Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>02</td>
<td>+1/8 - 0</td>
<td>Manure</td>
</tr>
<tr>
<td>A11</td>
<td>0 - 1-1/2</td>
<td>Yellowish brown (10YR5/4 dry, 10YR3/4 moist) loamy sand; moderate, thin platy structure breaking to very fine to fine granular structure; weakly coherent when dry, slightly sticky and nonplastic when wet; very slightly effervescent in dilute HCl; very abrupt, smooth boundary; average thickness 1/2 inches (1/2 - 1-1/2 inches). Other observations: horizon is separated from horizon below by difference in structural grade and class, and by increased amount of carbonate present downward.</td>
</tr>
<tr>
<td>A12</td>
<td>1-1/2 - 2-3/4</td>
<td>Yellowish brown (10YR5/4 dry, 10YR-4/4 moist) loamy sand; moderately strong, medium platy; slightly hard when dry, slightly sticky, non-plastic when wet; slightly effervescent in dilute HCl; abrupt smooth boundary; average thickness 1-3/8 inches (1-1/4 - 1-1/2 inches). Other observations: horizon has noticeably more carbonate than the previous horizon but much less than the following one; a few pieces of charcoal are present.</td>
</tr>
<tr>
<td>IIA3ca</td>
<td>2-3/4 - 5-1/2</td>
<td>Light yellowish brown (10YR6/4 dry, 10YR4/4 moist) sand; moderate, very fine angular blocky; weakly coherent when dry, nonsticky, non-plastic when wet; violently</td>
</tr>
</tbody>
</table>
Horizon Description

**Horizon**  **Depth below Designation Surface(ft.)**  

IIB21  5-1/2 - 11  
Pale brown (10YR6/3 dry, 10YR4/3 moist) loamy sand; dark strong medium columnar structure breaking to strong coarse blocky; hard when dry, slightly sticky, slightly plastic when wet; violently effervescent in dilute HCl; irregular, clear, boundary; average thickness 5 inches (4 - 6 inches). Other observations: distinct white particles present above are absent in this horizon; very little charcoal is present but stone flakes are interspersed through the horizon.

IIB22  11 - 17  
Pale brown (10YR6/3 dry, 10YR4/3 moist) matrix with grayish brown (10YR5/2 dry, 10YR4/2 moist) mottles loamy sand; common medium mottles; moderate, fine to medium, angular to subangular blocky; slightly hard when dry, slightly sticky, nonplastic matrix with slightly sticky slightly plastic mottles when wet; irregular, clear boundary, violently effervescent in dilute HCl. Average thickness 5 inches (4 - 6 inches). Other observations: large pieces of charcoal are present, mainly in mottled areas; increased plasticity in mottled areas seems due to humus.

IIB23  17 - 21-1/4  
Light brownish gray (10YR6/2 dry, 10YR4/2 moist) matrix with grayish brown (10YR5/2 dry, 10YR4/2 moist) mottles; loamy sand; weak, fine angular blocky breaking to fine granular, weakly coherent when dry; slightly sticky, nonplastic when
Horizon Depth below Description Surface(ft.) Horizon Description

wet; violently effervescent in dilute HCl; irregular gradual boundary; average thickness 2-1/8 inches (variation: 2 - 4-1/4 inches). Other observations: horizon is distinguished from that above mainly by structural grade and class; some charcoal present, but less than in preceding horizon.

IIB3 21-1/4 - 24 Pale brown (10YR6/3 dry, 10YR4/3 moist) loamy fine sand; weak, thin platy structure; weakly coherent when dry, slightly sticky, non-plastic when wet; violently effervescent; clear, wavy boundary; average thickness 2-1/4 inches (1-3/4 - 2-3/4 inches).

IIC 24 - Depth undetermined Yellowish brown (10YR5/4 dry, 10YR4/4 moist) loamy sand; weak very fine blocky breaking to moderate, very fine and fine granular; noncoherent when dry, slightly sticky, slightly plastic when wet; violently effervescent in dilute HCl.

Many general observations about this stratigraphic section are noted.

For example, the violent effervescent in dilute HCl is due, at least in part, to the calcareous composition of the surrounding sandstone bedrock. Consequently, the symbol "ca" was not used except for the one horizon which has an accumulation of white carbonate particles. The rest of the carbonate reaction is considered to be natural for the type of parent material present.

The delineation of separate parent materials is based on two factors:
1. The upper two horizons (All, A12) have much less carbonate material present;

2. The addition of domestic grazing animals to the area has added more organic matter to the sand.

Another way of handling this problem would be to add a "p" to the levels (Allp, A12p) and dispense with II. The other method is used because it is felt that there is a significant difference in the materials.

It becomes obvious upon inspection of the monolith that nearly all horizons were an A1 at one time, and that humic material and charcoal present are not due to the illuviation/eluviation process but are actually artifacts "in situ." For this reason, the letter "h" was not used with the horizon designation.

Since the site was in the process of excavation, this monolith does not extend to the bottom of the site; it includes only the upper 27 inches.
APPENDIX II

DESCRIPTION OF SEDIMENT SAMPLES FROM

24RB1012, COLT 45 SHELTER

Sample 1 1 - 1-1/2 inches below surface; dark yellowish brown (10YR4/4 dry, 10YR3/4 moist) loamy sand; slightly sticky, nonplastic when wet; very slightly effervescent in dilute HCl; pH = 8.22.

Sample 2 1-1/2 - 2-1/2 inches below surface; yellowish brown (10YR5/4 dry, 10YR4/4 moist) loamy sand; slightly sticky, nonplastic when wet; slightly effervescent in dilute HCl; pH = 8.14.

Sample 3 Small lense between 1-1/2 - 2-1/2 inches depth; light yellowish brown (10YR6/4 dry, 10YR4/4 moist) loamy sand; slightly sticky, nonplastic when wet; slightly effervescent in dilute HCl; pH = 8.27.

Sample 4 2-1/2 - 4-1/2 inches below surface; light yellowish brown (10YR6/4 dry, 10YR4/3 moist) sand; slightly sticky, nonplastic when wet; slightly effervescent in dilute HCl; pH = 8.16.

Sample 5 4-1/2 - 5-1/2 inches below surface; light yellowish brown (10YR6/4 dry, 10YR5/4 moist) loamy sand, sticky, slightly plastic when wet; slightly effervescent in dilute HCl; pH = 8.24.

Sample 6 5-1/2 - 7 inches below surface; pale brown (10YR 6/3 dry, 10YR4/4 moist) loamy sand; sticky, slightly plastic when wet; effervescent in dilute HCl; pH = 8.2.

Sample 7 7 - 9-1/2 inches below surface; pale brown (10YR 6/3 dry, 10YR4/3 moist) loamy sand; sticky, plastic when wet; effervescent in dilute HCl; pH = 8.45.

Sample 8 9-1/2 - 9-3/4 inches below surface; from sandstone block; very pale brown (10YR7/4 dry, 2.5YR 4/4 moist); slightly sticky, slightly plastic when wet; effervescent in dilute HCl; pH = 8.33.
Sample 9 9-3/4 - 10-1/2 inches below surface; pale brown (10YR6/3 dry, 10YR3/4 moist) loamy sand; slightly sticky, slightly plastic when wet; effervescent in dilute HCl; pH = 8.4.

Sample 10 10-1/2 - 12-1/2 inches below surface; light yellowish brown (10YR6/4 dry, 10YR4/4 moist) loamy sand; slightly sticky, slightly plastic when wet; charcoal present; highly effervescent in dilute HCl; pH = 8.49.

Sample 11 12-1/2 - 14-1/2 inches below surface; brown (10YR5/3 dry, 10YR3/3 moist) loamy sand; slightly sticky, very slightly plastic when wet; charcoal present; effervescent in dilute HCl; pH = 8.4.

Sample 12 14-1/2 - 21-1/2 inches below surface; pale brown (10YR6/3 dry, 10YR3/3 moist) loamy sand; slightly sticky, slightly plastic when wet; charcoal present; highly effervescent; pH = 8.52.

Sample 13 21-1/2 - 25 inches below surface; grayish brown (10YR5/2 dry, 10YR3/3 moist) loamy sand; slightly sticky, slightly plastic when wet; charcoal present; highly effervescent in dilute HCl; pH = 8.7.