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SELECTED CHARACTERISTICS OF OPALINE PHYTOLITHS  
BETWEEN SOME UDOLLS AND USTOLLS

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## CHAPTER I

### INTRODUCTION

Opaline phytoliths are of the same chemical composition,  $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ , as the geologically derived mineral opal. They are generally considered to be amorphous silica (Smithson, 1956a) but may be submicrocrystalline (Fronde1, 1962). Opaline phytoliths are deposited in and around plant cells from soluble silica taken up by plants, and subsequently accumulated in soils upon the decay of plant parts in which they form. Although opaline phytoliths<sup>1</sup> (also referred to as plant opal, grass opal, opaline silica, and biogenetic opal) are found in most plants, they are more abundant in grasses. Since they are composed primarily of silica, opaline phytoliths are relatively resistant to weathering, and thus serve as at least a semi-permanent record of the vegetative history of soils as well as implying climatic conditions of soil formation. Earth scientists and botanists inherit the responsibility of properly interpreting this phytolith record. Phytolith shapes depend somewhat on the species in which they form (Twiss et al., 1969) and vary within plants from one part to another (Parry and Smithson, 1966). The variation of shapes among plants allows some analyses of vegetative history and soil formation.

Since no previous opaline phytolith studies had been conducted in

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<sup>1</sup> In this paper the term opaline phytolith will be shortened to phytolith for brevity.

Oklahoma, a preliminary study was necessary to broadly characterize the phytoliths found in the state and to gain familiarity with phytolith separation and analysis techniques. From the preliminary study, opaline phytoliths in Oklahoma appeared to be smaller than those found by Jones and Beavers (1964a) in Illinois. This difference was considered to be significant because of its possible relationship to climate, especially moisture conditions, under which opaline depositing plants grew. A second study was then initiated to test the hypothesis that opaline phytoliths vary in size with variation in annual rainfall. Three soils, an Udoll, an Udoll-Ustoll, and an Ustoll, were sampled along an east-west transect in North Central Oklahoma for comparison of phytolith sizes. An additional soil containing buried horizons was sampled in order to observe opaline phytolith variation with depth in such a soil.

The objectives of the study were as follows:

1. To study phytolith size as a function of variation in mean annual precipitation in Oklahoma.
2. Establish the value of opaline phytoliths as indicators of the lower boundary of a soil pedon.
3. Characterize phytoliths in the major soil horizons on the basis of root or top origin.
4. Note phytolith distribution with soil depth.
5. Determine the value of phytoliths as indicators of buried surface horizons.



## CHAPTER II

### LITERATURE REVIEW

#### Silica Uptake and Phytolith Formation by Plants

Jones and Handreck (1963) established that the form of silica in the soil solution was monosilicic acid,  $\text{Si(OH)}_4$ , between pH values of 2 and 9, and found that lowering the pH within this range increased the amount of monosilicic acid in solution. Later they pointed out (Jones and Handreck, 1967) that Krauskopf also found silica in the soil solution to be monosilicic acid. This work indicated that above pH 9, silica is available for plant in the ionic form. They also reported that the soluble silica level, at a given pH, is influenced by the kind and crystallinity of free sesquioxides present.

Silica absorbed by grasses and other plants is precipitated in the cells to form phytolith objects especially within the epidermal and vascular portions of the plant (Witty and Knox, 1964). Baker (1959b) characterized these phytolith bodies as isotrophic silica precipitated as unwanted materials or re-inforcements of cell structures. He and others (Witty and Knox, 1964) listed the plants most commonly precipitating silica to include grasses, sedges, reeds and some woods. McKeague and Cline (1963) stated that the range of silica uptake is from very little by temperate zone trees to large quantities producing almost pure silica gel in the hollow stems of bamboo (Bambusa spp.). Suggested mechanisms of deposition include penetration of silicic acid

through the pectate primary cell walls, simultaneous deposition with cellulose (Jones et al., 1963), and silicic acid filling of the apertures of cellulose micelles, forming a silica-cellulose membrane (Yoshida et al., 1959).

### The Nature of Opaline Phytoliths

All silica secreted by living organisms appears to be of the opaline variety (Smithson, 1956a). Opaline silica so deposited has the same properties as geologically produced opal ( $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ ) and most workers (Smithson, 1956a,b; Jones and Beavers, 1963a) have considered it to be amorphous, but Frondel (1962) regards it as submicrocrystalline aggregates of cristobalite, containing a large amount of non-essential water. The silica is not rigid when it is first laid down since it does not prevent extension of mature cells (Jones et al., 1963b). In the mature oat (*Avena sativa*) plant practically all the silica is in the form of solid opaline phytoliths (Jones et al., 1963b). Opaline phytoliths have a low specific gravity ranging from 1.5 - 2.3 (Jones and Beavers, 1963a) compared to quartz (sp gr, 2.65) and contain, in addition to silica, small quantities of aluminum, iron, magnesium, potassium, manganese, phosphorus, titanium, sodium, and carbon (Jones and Beavers, 1963a; Kanno and Arimura, 1958). The water content in Victorian soil phytoliths ranged from 3.5 - 5% (Baker, 1959b) while phytoliths from oats ranged from 9.8 - 19% (Jones et al., 1963). Phytoliths tend to lose their water in a relatively short time, geologically speaking (Smithson, 1956a). Beavers and Stephen (1958) noted replacement of opal by chalcedonic silica. Jones et al. (1965) found that upon heating tabashir and opaline phytoliths both were converted

to cristobalite, but that the opaline phytoliths were changed with 200 C less heat (1150 C). It seems evident that opaline phytoliths may lose their identity through dessication and this may partially explain why phytolith levels in arid soils are not as high as one would expect from the contribution of vegetation. The data of Pease (1967), from New Mexico, indicate that phytolith levels are lower than expected based on vegetative growth.

Carbon was found to be occluded in opaline phytoliths by Jones and Beavers (1963a). They found a high incidence of dark brown and black phytolith grains in the lower specific gravity fractions ( $< 2.1$ ), and found these fractions to be greater than average in carbon content. Wilding et al. (1967) found three times as much carbon in the lower specific gravity, darker phytoliths.

The size of opaline phytoliths varies from clay size to sand size according to Jones and Beavers (1964a). They observed approximately a 1:1 ratio of phytoliths in the soil from the 5-20 $\mu$  and 20-50 $\mu$  fractions. However, the clay and fine silt (5-20 $\mu$ ) fractions represented a larger fraction of the phytoliths obtained from plants. They explained the absence of large quantities of clay size phytoliths in the soil on the basis of more rapid weathering and re-entry into the silica cycle. They considered the phytoliths in the 20-50 $\mu$  size fraction to be one-third to one-ninth of the total phytolith content, and assumed this relationship to be constant among the Illinois soils studied (Jones and Beavers, 1964a). Witty and Knox (1964) worked with the 15-100 $\mu$  size fraction, and Beavers and Stephen (1958) found the fine sand phytoliths to commonly exceed those of the silt fraction. Jones et al. (1963b), working with oats, found wide variation in size depending on the plant

part observed. Phytolith size seems significant for at least two reasons. Phytoliths of one size may be more susceptible to weathering than another. Jones and Beavers (1964a) noted that this was important with clay size phytoliths. Further Daubenmire (1948) points out that under less desirable moisture levels plants have smaller cells; it therefore seems that phytoliths would be smaller in arid climates. In fact, Twiss et al. (1969) show phytoliths from the Chloridoid grasses (short grasses) to be smaller than that of the Panicoid grasses (tall grasses). Maximov (1929) also discussed the decreased size of plant cells under less favorable moisture conditions.

Dry ashing of plant materials may also alter the phytolith size and shape (Jones and Milne, 1963; Jones and Beavers, 1964a). Wet ashing of plant materials is therefore preferred for phytolith analysis.

#### Phytolith Accumulation in Soils

Opaline phytoliths are released from plants by natural decay of plant tissue, by grass or forest fires (Baker, 1959b), or they may be disseminated in the dung of herbivorous animals (Smithson, 1956; Baker, 1959b). They may also be disseminated by wind as evidenced by the collection of plant opal by Folger et al. (1967) in a dust storm at sea off the coast of Africa. The source of phytoliths in soils was previously thought to be primarily from the aerial portions of the plants (Jones and Beavers, 1964a). Other workers (Lanning et al., 1958) even state that silica usually does not deposit in underground parts. On the other hand, Jones et al. (1963b) described the shapes of phytoliths observed in oat roots and Pease and Anderson (1969) observed a larger accumulation of phytoliths in the roots than in the tops from black

grama (Bouteloua eriopida).

Kanno and Arimura (1958) reported a sharp decrease in phytolith content of soils below the A horizon, as did Beavers and Stephens (1958), who found very low phytolith levels in the C horizon; however, the distribution may be more uniform with depth in areas of relatively slow deposition, as in the case of loess (Jones and Beavers, 1964b). McKeague and Cline (1963) noted that Mollisols are likely to contain large amounts of plant opal from grasses which tend to segregate such particles. Jones and Beavers (1964a) estimated a yearly deposition of 15 pounds per acre of the 20-50 $\mu$  size and Witty and Knox (1964) estimated 20-30 pounds per acre of the 15-100 $\mu$  size fraction. It seems that such calculations are subject to gross error considering the possibility of climatic variation during the development of these soils.

The stability of opaline phytoliths presently appears to be open to conjecture. As previously noted (Pease, 1967; Jones and Beavers, 1964a), some workers suggest loss of phytolith identity through dessication or loss into solution of smaller phytoliths. Baker and Leeper (1958) estimated that the mean annual life of phytoliths was 1000 years or less. However Gill (1967) observed phytoliths from materials of Pliocene age which had been potassium-argon dated at 4.35 million years before present and Wilding (1967)  $C^{14}$  dated phytoliths from Ohio soils at 13,300 years before present. This appears to be an area where additional work is needed.

Jones and Handreck (1967) summarized the phytolith cycle in soils. Initially the soluble silica occurs as silicic acid which is taken up by the plant and deposited in and around plant cells. Upon death and decay of the plant, phytoliths are collected in the soil, or they may

pass, essentially unchanged, through the digestive tract of a herbivore to the soil. Once back in the soil, phytoliths are slowly weathered to the soluble silicic acid form, and the cycle is repeated.

### Morphology of Opaline Phytoliths

The resultant shapes and sizes of secreted bodies of mineral matter are generally controlled by the shapes of the various units of plant structure (Baker, 1959a). The characterization of phytoliths has involved in situ observation with plants as well as plant residue observations. The lengths and widths of phytoliths vary with the plant part (Jones et al., 1963; Baker, 1960; Parry and Smithson, 1966). Beavers and Stephen (1958) observed elongated, squat, flattened, rod-like, acircular, and serrated grains. Witty and Knox (1964) found netted and rod shaped phytoliths in pine needles and Baker (1960) reported hook-shaped phytoliths from oats. Parry and Smithson (1966), working with grass leaves, noted that phytoliths of costal cells were long while intercostal phytoliths were short. Parry and Smithson (1966) observed dumbbell shapes in grass inflorescence and had previously (1957) observed them in grass leaves, while Lanning et al. (1958) observed this shape in sorghum. Kanno and Arimura (1958) observed dumbbells in Japanese soils, however they noted that dumbbells were much more common in the silt than in the sand fraction.

Jones et al. (1963b) observed greatly pitted cells in oat roots while Pease and Anderson (1969) were able to associate rectangular phytoliths with the roots of black grama.

Sponge spicules, identified on the basis of their axial canal, are of the same mineralogy as opaline phytoliths, and have been observed

in soils by Jones and Beavers (1963b). Smithson (1959) also observed them in the A horizons of a Spodosol and cautioned against their confusion with phytoliths.

Smithson (1956b) concluded that it was not possible to name specific grasses from which phytoliths came, but that the phytolith characteristics provided an indication of the groups of grasses from which they originated. Recently, Twiss et al. (1969) were able to classify phytolith shapes; they related them to three subfamilies of grasses. The families included Festucoid (brome-grasses, fescue), Chloridoid (includes short grass species), and Panicoid (includes tall grass species). An Elongate group had shapes distributed in all species.

#### Uses of Opaline Phytoliths in Soil Genesis

Beavers and Stephens (1958) advocated the use of opaline phytoliths as an "index mineral" for confirming the presence and location of buried A horizons of Prairie soils since phytoliths have a fairly high stability. Kanno and Arimura found buried A horizons to be high in phytoliths. They also correlated the phytoliths in A horizons with maturity of soils. Jones and Beavers (1964b) compared Brunizem and Gray-Brown Podzolic soils on the basis of phytolith content and found the forest soil to be a relic from an xothermic time (4000-5000 years before present). They also studied a catena and found maximum phytolith content in the intermediate drainage class and interpreted that to be a result of vegetative production. They also were able to apply the distribution of phytoliths with depth to an interpretation of rate of loess deposition.

Witty and Knox (1964) studied the stability of a grassland-forest boundary based on the phytolith content of the two areas.

Wilding (1967) dated occluded carbon from an Ohio soil and obtained a much older date (13,300 before present) than was anticipated (1000-1500 years) on the basis of phytolith accumulations. Carbon dating of phytoliths coupled with the ability to identify plant groups which were influential in the genesis of a soil promises to be an important soil genesis tool.



## CHAPTER III

### METHODS OF ANALYSIS

#### Plant Sampling

For the preliminary study, plant samples were collected in order to observe the sizes and shapes of opaline phytoliths present in various types of native vegetation. Plants were big bluestem (Andropogon gerardi), little bluestem (A. scoparius), Indiangrass (Sorghastrum nutans), buffalograss (Buchloe dactyloides), post oak (Quercus stellata), and blackjack oak (Q. marilandica). Tree leaves were mature and the grasses were in full growth but had not produced seeds. The top and root plant portions were separated for individual phytolith analysis and washed in distilled water and detergent to remove adhering soil particles. The samples were dried at 75 C in a forced air oven. Prior to analysis, leaves were crushed by hand and roots were cut in approximately 1 inch lengths with scissors. Grinding was avoided to prevent destruction of the phytoliths as much as possible.

#### Plant Analysis for Phytoliths

The roots and tops of the plants were separately wet ashed by a method modified from the method of Gieseking et al. (1935). Two-gram samples of plant material were weighed into dry pre-weighed 400 ml beakers. Approximately 50 ml of a 3:1 mixture of nitric to perchloric acid were added to each sample. The samples were placed on a hot plate

under a perchloric acid hood and allowed to boil vigorously until the solution became clear and about 10 ml of solution remained. An additional 25 ml of the acid mixture were then added by a pipette, running the tip along the beaker walls in order to wash down any material adhering to the sides. The samples were again boiled down to about 10 ml cooled, and the beaker walls washed with distilled water.

The plant residue was then quantitatively transferred to pre-weighed pyrex crucibles with fine porosity fritted glass bottoms. The crucibles were fitted onto a filtering flask and the residue washed with approximately 150 ml of hot distilled water to remove the acid and soluble materials. The silica (phytolith) residue was dried in the crucible at 105 C, weighed, and the phytolith content expressed as a percentage of the dry plant weight.

Samples of phytoliths were then mounted in Cadex on glass slides and observed under a petrographic microscope.

### Soil Selection and Sampling

#### Preliminary Study

In the preliminary study, the A horizons of three Payne County soils were studied for variation in the amounts and types of opaline phytoliths. Site 1 was a Zaneis loam, a Udic Argiustoll on a 2% convex slope 1950 ft W and 132 ft S of the NE corner of Sec 10, T19N, R2E. The native vegetation was that of a tall grass prairie comprised primarily of big bluestem (Andropogon gerardi) and little bluestem (A. scoparius) along with other grasses and various forbs. Site 2 was a Kirkland loam, an Abruptic Paleustoll on a nearly level convex position 2130 ft W and 495 ft S of the NE corner of Sec 10, T19N, R2E.

The vegetation was dominated by buffalograss (Buchloe dactyloides). Site 3 was a Dougherty soil, an Arenic Haplustalf, on a 1-3% slope 897 ft E and 212 ft S of the center of Sec 36, T18N, R2E. The vegetation was comprised of oak forest. Post oak (Quercus stellata) was the dominant species occurring with other oaks and understory vegetation. Very few grasses were present on the forest floor. Soils were air dried and hand crushed prior to analysis.

### Transect Study

Three upland soils, similar in textural profile which occur on the upper, more stable landforms, were selected for this study. The soils occurred along an east-west transect in North Central Oklahoma, and occurred in areas varying in annual rainfall. Figure 1 shows the location of the soils sampled. The St. Paul is a Typic Argiustoll occurring in an area of approximately 24 inches of annual rainfall. The Zaneis is an Udic Argiustoll and occurs in an area of approximately 33 inches of annual rainfall. The Fitzhugh occurs in an area of approximately 40 inches of annual rainfall and is a Typic Argiudoll.

In addition to the transect soils, a Meno-like soil was sampled in an attempt to identify buried soil horizons on the basis of phytolith composition. The Meno is classified as an Aquic Arenic Haplustalf.

All soils were sampled by genetic horizon through the parent material. The samples were air dried and crushed prior to analysis.

## Soil Analyses

### Fractionation and Particle Size Analysis of Soils

Duplicate 40 gm samples of each horizon were treated with 0.2N HCl for carbonate destruction and 30% H<sub>2</sub>O<sub>2</sub> to destroy the organic matter.



Soluble salts were removed by repeated centrifuging until no chloride remained as evidenced by testing with  $\text{AgNO}_3$ . The samples were then shaken overnight with 100 ml of 5% calgon and transferred to 1000 ml cylinders through a 270 mesh sieve. The sand retained on the sieve was dried, weighed, and further sieved to determine the percent very fine sand (0.1-0.05 mm). The samples in the 1000 ml cylinders were then shaken up and siphoned at 30 cm depths at the appropriate time considering the temperature and the particle size to be removed (Jackson, 1956). Siphonings were repeated until the material siphoned was clear. The less than  $5\mu$  material was discarded. In the preliminary study, the silt was fractionated into 5-20 $\mu$  and 20-50 $\mu$  fractions. In the transect study, the silt was separated into 5-20 $\mu$ , 20-35 $\mu$ , and 35-50 $\mu$  size fractions.

In the preliminary study, particle size analyses were made by the pipette method (Kilmer and Alexander, 1949). In the transect study, each silt and sand fraction was weighed upon separation, and expressed as a percentage of the oven dry weight of the original sample. For each fraction, the average of the two duplicates was reported.

#### Separation of Phytoliths

Duplicated 1.5 gm samples of the fractionated silt were weighed into centrifuge tubes. In the preliminary study, opaline phytoliths were separated from the 5-20 $\mu$  and 20-50 $\mu$  soil fractions by the method of Jones and Beavers (1964a), centrifuging samples in a specific gravity liquid of 2.3 (composed of 1,1,2,2-tetrabromoethane and nitrobenzene). The surface of the centrifuged sample was poured off from the lower portion which was frozen in acetone cooled with dry ice. Samples were collected on #3 Whatman filter paper and washed with acetone. Samples

were then transferred to weighed aluminum weighing pans for drying and weight determination. After drying, phytolith grains were mounted in Cadex on glass slides and observed under a petrographic microscope.

The above method proved to be time consuming and was difficult to keep the lower portion frozen while the phytoliths were being decanted. Several alternate methods were tried, none of which were completely satisfactory.

In the transect study, the 2.3 specific gravity liquid mentioned above was again used, but the decanting procedure was altered. Constricted test tubes and plugs similar to those described by Schoen and Lee (1964) were used to effect separation of the phytoliths from the heavier minerals. This method had the advantage of being rapid, but the disadvantage of introducing impurities into the supernatant liquid by holding non-opaline materials on the constriction shoulder of the tube. The non-opaline particles were removed with a rubber policeman after five minutes of centrifuging, after which centrifuging was continued for 10 minutes. It was still not possible to obtain uncontaminated phytoliths in the supernatant liquid. The supernatant liquid was poured into weighed porcelain crucibles with fine porosity fritted glass bottoms. The crucibles had previously been placed in Walter's crucible holders on filtering flasks. Centrifuge tube sides were rinsed with the 2.3 specific gravity liquid from a wash bottle, allowing the particles washed from the tube side to be collected in the crucibles.

After each centrifuging, and decanting, silt materials were re-suspended by shaking the centrifuge tubes, which were stoppered with polyethylene stoppers. The suspension was evacuated between each

centrifuging to remove air introduced by re-suspension. Centrifuging was repeated until phytoliths no longer appeared in the supernatant.

When phytoliths were no longer obtained from centrifuging, the residue in the crucibles was washed thoroughly with acetone. Crucibles were then dried at 105 C, weighed, and the phytolith percentage from the silt fraction calculated. Residue samples were mounted in Cadex on glass slides and observed under a petrographic microscope. At least 200 grains were counted from each slide and the percent purity of phytoliths in the fraction was adjusted by the purity factor.<sup>1</sup> The adjusted percentage was multiplied by the percentage of the silt fraction in the soil. The phytolith content was reported as milligrams per gram of soil in each silt fraction in each soil horizon. The average of two duplicates was reported. The total phytolith content in each horizon from the three fractions was determined and that percentage represented by each size fraction was recorded. The relative percentage in the three fractions was used as a measure of phytolith size. Phytolith sizes fall essentially within the size parameters of the fraction from which they are separated. The phytolith percentage in each size fraction was compared in the three transect soils as an indicator of size variation along the transect. In comparing the phytolith sizes of the three transect soils, the phytolith level in the upper 25 cm of each soil was used since the major part of the phytoliths occur within this depth.

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<sup>1</sup> The impurity grains were usually smaller than the phytoliths, reducing the phytolith levels below their actual value, but not altering relative values.

### Organic Matter Determination

Organic matter determinations were made using the method outlined by the U. S. Salinity Laboratory Staff (1964) in Agriculture Handbook No. 60.

### Phytolith Shape Analysis

At least 200 phytolith grains of soil and plant origin were observed under the microscope and classified according to the system of Twiss et al. (1969). They were grouped into Festucoid, Chloridoid, Panicoid, and Elongate classes without regard to types within classes. In addition, the non descript group includes all phytolith shapes not falling into the above groups as well as fragmentary parts of the four groups listed. These data should be considered more qualitative than quantitative since larger numbers of grain counts would be required for highly reliable quantitative data (Brewer, 1964).



## CHAPTER IV

### RESULTS AND DISCUSSION

#### Preliminary Study

##### Phytoliths in Plants

The phytolith percentage in the vegetative parts of the plants sampled is shown in Table I. In both tops and roots the phytolith residue is considerably higher from grasses than from trees. This corroborates with the statement of McKeague and Cline (1963) that temperate region trees take up very little silica whereas grasses take up large quantities. Big bluestem contained the smallest percent of phytoliths of the grasses while buffalograss was highest in phytolith content. It is interesting to note that the phytolith level in the grasses studied appears to be inversely related to the known preferences of these grasses by livestock (Dwyer, 1961; Tomanek et al., 1958).

The phytolith content of the grass roots studied was not strikingly less than that of the tops, except in buffalograss. These results, as well as those of Pease (1967), show that the deposition of phytoliths in roots is variable depending on the type of plant.

##### Phytolith Content of Soils

The phytolith content of the 5-20 $\mu$  and 20-50 $\mu$  fractions from the A horizons in the preliminary study soils is shown in Table II. It is higher in the surfaces of Zaneis and Kirkland, the Mollisols, than in

TABLE I  
PHYTOLITH PERCENT OF VEGETATIVE PARTS

Plant	% Phytoliths	
	Tops	Roots
Big bluestem	2.22	2.79
Little bluestem	5.71	3.80
Indiangrass	3.96	3.64
Buffalograss	6.27	1.26
Post Oak	1.20	0.60
Blackjack	0.34	0.58

TABLE II  
PHYTOLITHS IN THE A HORIZONS OF THE PRELIMINARY STUDY SOILS

Soil and Horizon	Phytoliths, mg/g of Soil		$\frac{5-20u}{20-50u}$
	5-20u	20-50u	
Dougherty, A1	1.90	0.70	2.71
Dougherty, A2	1.08	0.57	1.89
Zaneis, A1	4.36	1.53	2.85
Kirkland, A1	3.15	1.47	2.14

the two horizons of Dougherty, an Alfisol, since grasses are the native vegetation under which Mollisols form. The phytolith content of the Zaneis is probably higher than that of the Kirkland due to the higher productivity of the Zaneis which is a moderately permeable soil, whereas the Kirkland is very slowly permeable, due to the presence of a claypan. Jones and Beavers (1964a) reported higher phytolith contents in soils of the medium drainage class in a catena.

Although the Dougherty, which now is under forest vegetation, is lower in phytoliths than the Mollisols, it is not as low as might be expected. Witty and Knox (1964) found the phytolith content of Prairie soils to be approximately 20 times that of forest soils.

The relative amounts of phytoliths in the 5-20 $\mu$  and 20-50 $\mu$  size fractions were of particular interest. While the data show over twice as much phytolith material in the 5-20 $\mu$  fraction compared to the 20-50 $\mu$  size fraction in the grass soils, Jones and Beavers (1964a) reported a 1:1 ratio of phytolith content for these two size fractions in Illinois soils. Since phytoliths are deposited in plant cells, which vary in size depending on availability of moisture (Daubenmire, 1948), a phytolith size-moisture relation may be a possibility in the regulation of phytolith accumulation in soils. If this were in fact a valid relationship, and could be shown to exist, it would be a valuable tool in elucidating previous moisture regimes, especially in the interpretation of buried A horizons of Paleosols.

#### Transect Study

##### Variation of Phytolith Content with Depth in Transect Soils

The depth to which phytoliths were still measurable in all size

groups was greatest (78 cm) in the Udoll, Fitzhugh (Figure 2). Phytoliths were detectable in all fractions to 46 cm in the Ustoll, St. Paul (Figure 3), while phytoliths in all fractions were not detectable below 40 cm in the Udoll-Ustoll, Zaneis (Figure 4). The most apparent explanation for the variation appears to be related to soil texture. In both the Fitzhugh and Zaneis, significant phytolith quantities were absent below horizons of clay loam texture, and in the St. Paul significant quantities were absent after the texture increase to a "heavy" silt loam with depth. The sharpest declines in phytolith levels occur below the surface horizon in all soils. This sharp decline below the surface suggests that phytoliths accumulated from plant tops, which are returned to the soil, is greatest in the surface, with perhaps little influence below the surface horizon. The pattern of phytolith distribution below the surface horizon appears to be related to clay content in the same manner that clay content regulates root growth. It seems logical that the contribution of phytoliths below surface horizons is primarily from roots. This further emphasizes that phytoliths are contributed not only by plant tops, but also by roots. Phytolith levels decreased below measurable quantities before reaching the C horizons. Therefore maximum depth of phytolith occurrence is unsatisfactory for determining the lower limit of a soil pedon (Soil Survey Staff, 1960) or where the soil-non soil boundary occurs based on depth of penetration of roots of native plants.

#### Phytolith Content of Transect Soils

Phytolith quantities in each horizon of each size fraction of the three transect soils are shown in Figures 2, 3 and 4. Fitzhugh, an Udoll, contained the largest phytolith levels in all soil fractions at

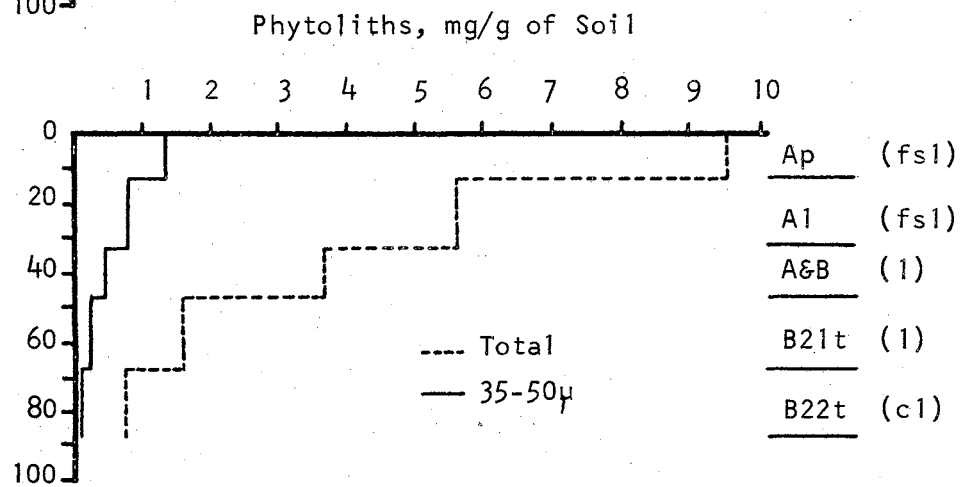
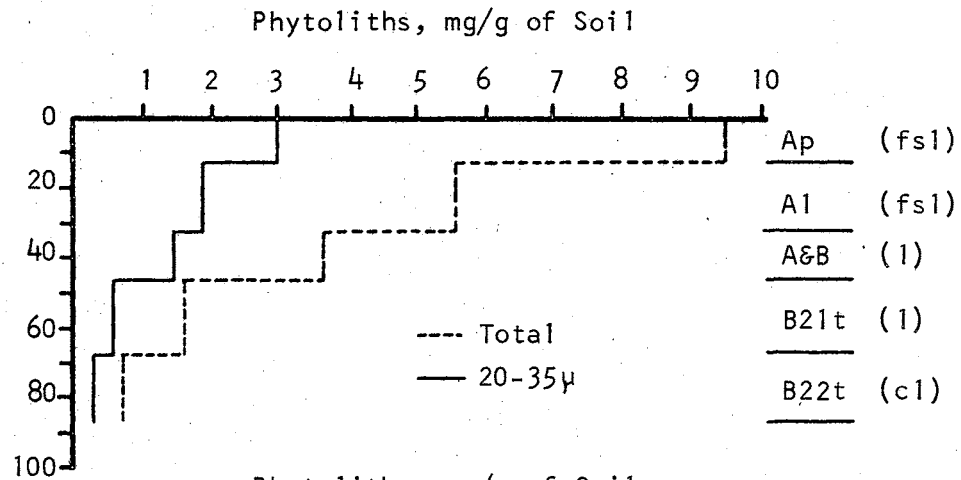
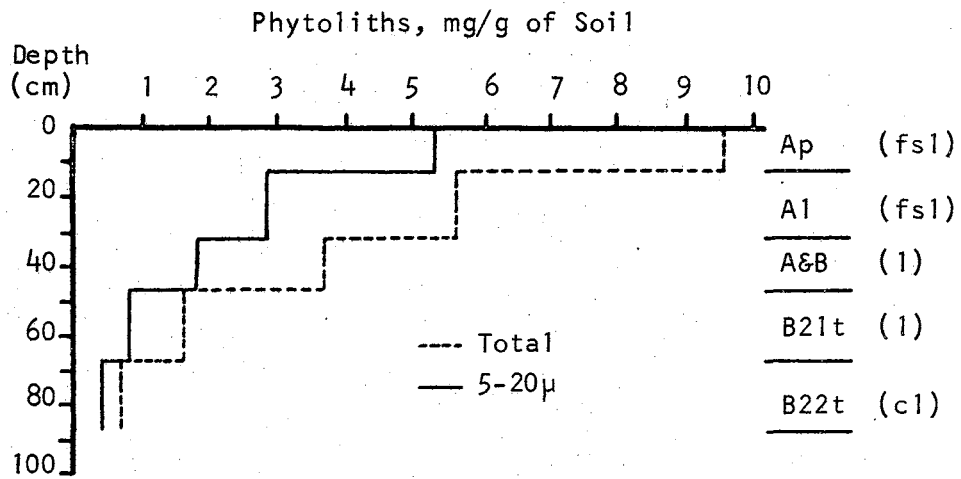


Figure 2. Phytolith Variation in the Fitzhugh Soil, an Udoll.

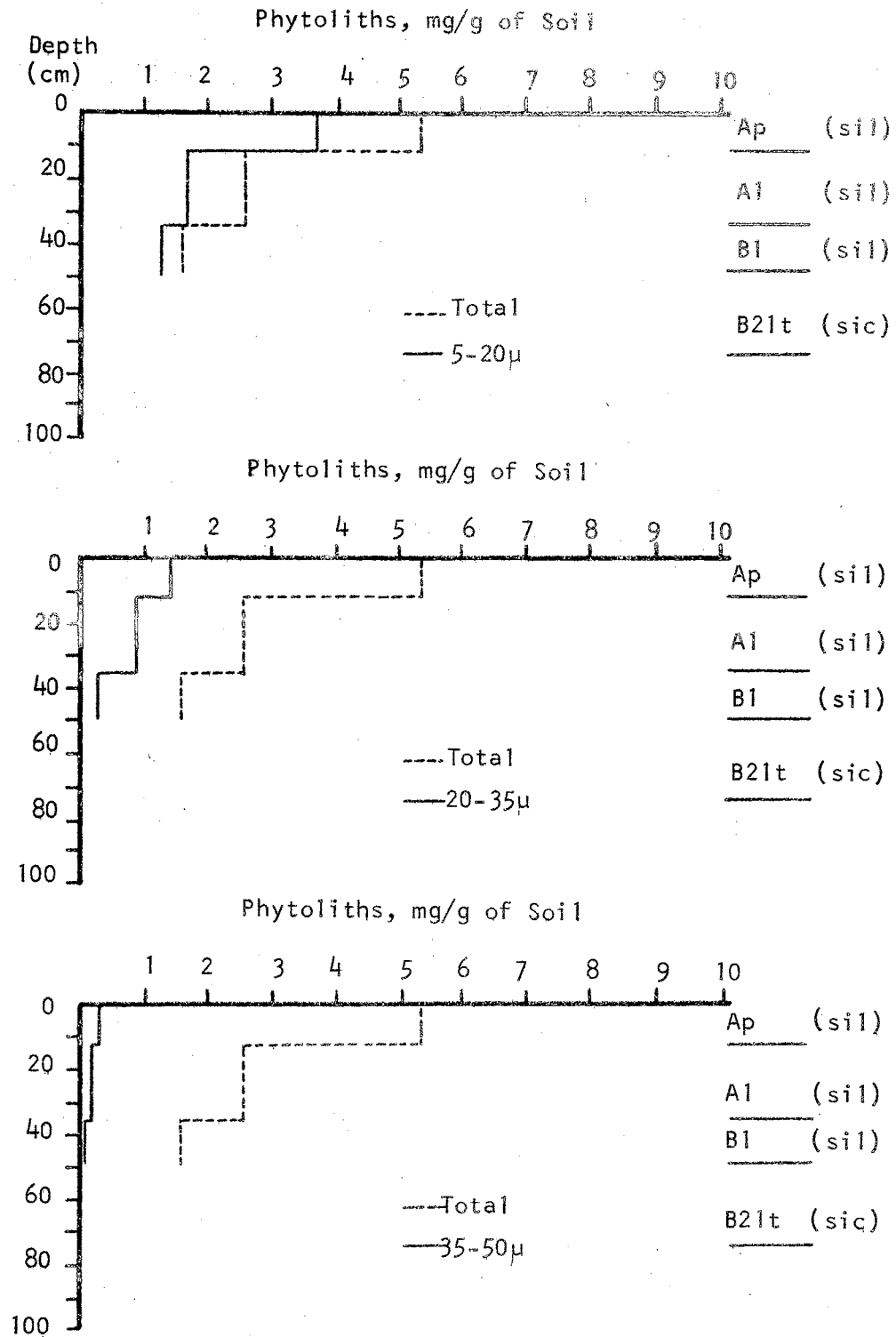


Figure 3. Phytolith Variation in the St. Paul Soil, an Ustoll.

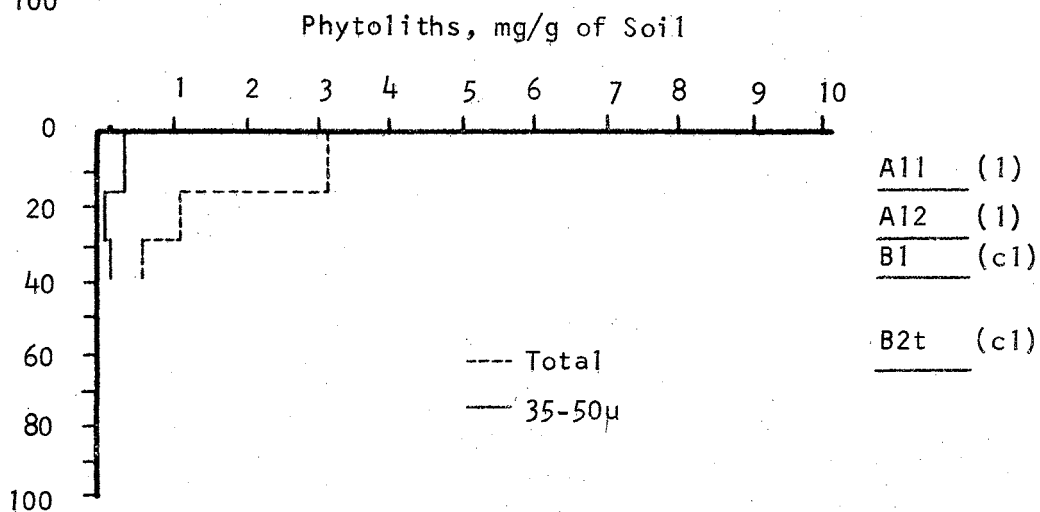
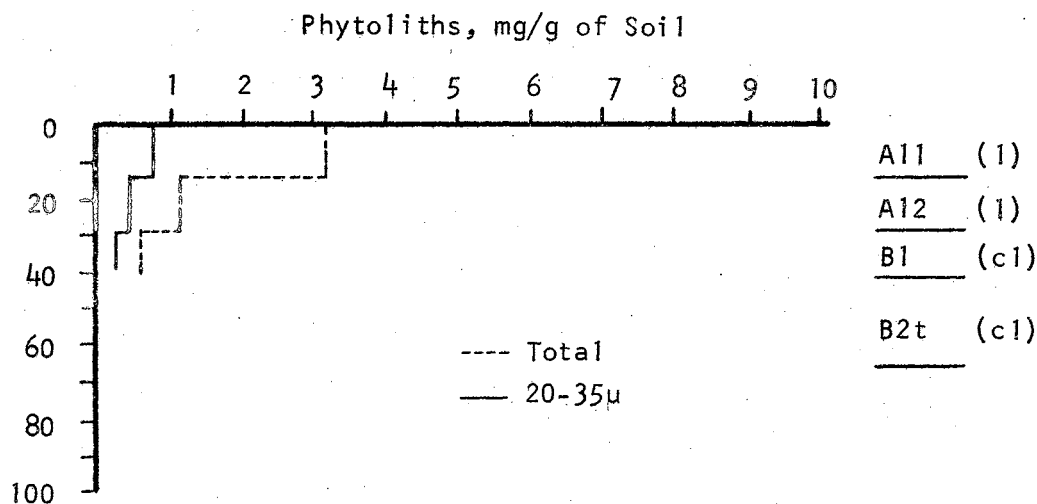
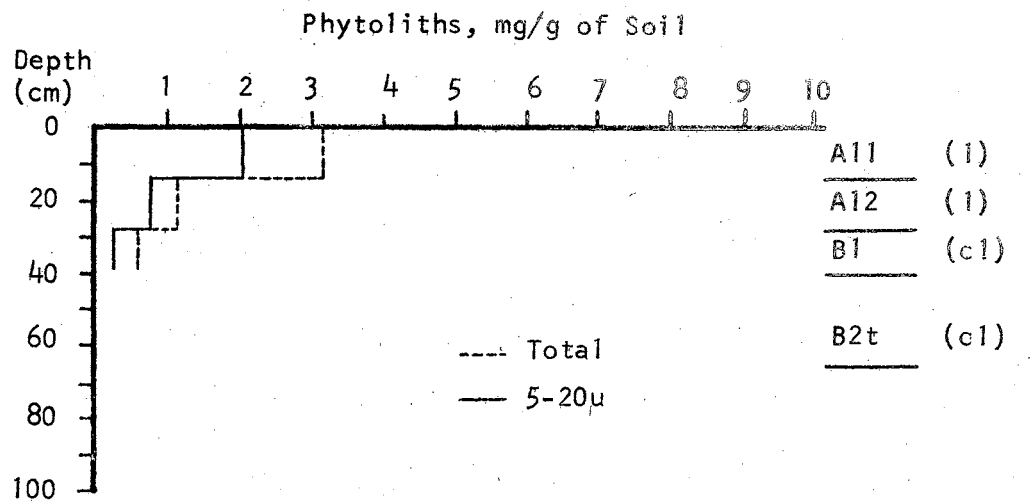


Figure 4. Phytolith Variation in the Zaneis Soil, an Udoll-Ustoll.

all depths and the Zaneis, an Udoll-Ustoll, contained the least in all horizons in all fractions at comparable depths, with one exception, the 35-50 $\mu$  fraction of the surface horizon was the same as the Ap of St. Paul in that fraction. The data show that in the St. Paul, an Ustoll, lower phytolith quantities accumulated, probably due to less production of vegetation than in the Udoll. The Udoll-Ustoll, which lies between the two soils, however, had the lowest phytolith content, and would be expected to be intermediate in phytolith content.

The present data afford no basis for explaining the low phytolith content in the Udoll-Ustoll profile.

#### Size Distribution of Phytoliths in Transect Soils

Table III shows the percent of phytoliths in each of the three size fractions for the upper 25 cm of the three transect soils. More phytoliths occur in the 5-20 $\mu$  fraction of the Ustoll than the 5-20 $\mu$  fraction of the Udoll. The phytoliths are more evenly distributed in the Udoll than in the Ustoll. The 35-50 $\mu$  fractions contain 14.8% in the Fitzhugh, the Udoll, and only 4.4% in the St. Paul, the Ustoll. Thus, phytoliths occurring in the St. Paul are dominantly smaller than those in the Fitzhugh. From a comparison of these two soils, an annual rainfall-phytolith size relationship is suggested. However the phytolith size distribution for the Zaneis soil, an Udic Argiustoll, was essentially the same as that for the Ustoll. Since the Udic Argiustoll occurs between the Ustoll and Udoll sites with relation to annual rainfall and in classification, it might be expected to contain intermediate phytolith sizes. However, the similarity of phytolith sizes of the two Ustolls, and the dissimilarity of these sizes to those found in the Udoll suggest phytolith size as a criteria for distinguishing between



Ustolls and Udolls. To fully establish this relationship, additional transect points would be necessary, especially between the Ustolls and the Udolls. A greater range in annual rainfall may also be beneficial.

TABLE III  
PERCENT OF PHYTOLITHS BY SIZE FRACTION IN THE  
UPPER 25 cm OF THE PROFILE

Soil	5-20 $\mu$	20-35 $\mu$	35-50 $\mu$
St. Paul, Ustoll	66.9	28.6	4.4
Zaneis, Ustoll-Udoll	66.6	27.4	5.6
Fitzhugh, Udoll	53.2	32.6	14.8

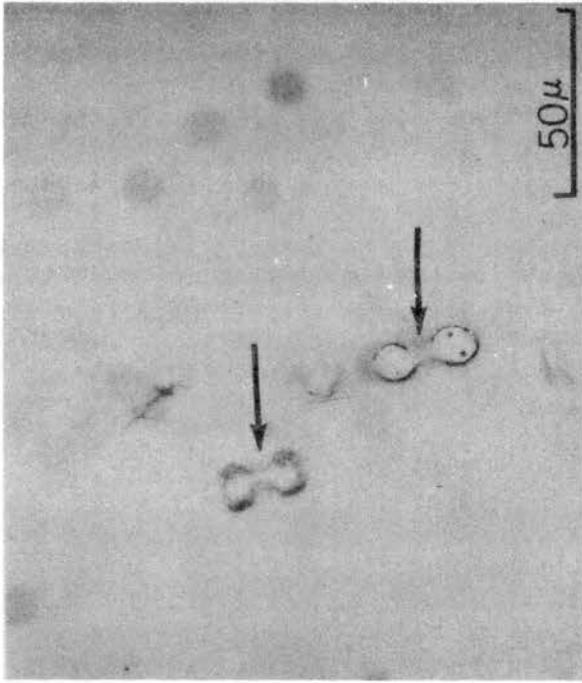
#### Phytolith Shape Characterization

##### Plant Residue

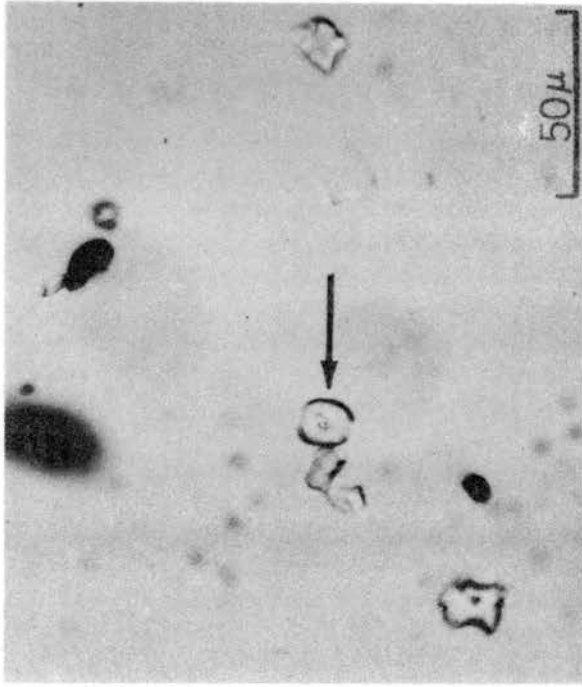
The occurrence of the classes of phytolith shapes described by Twiss et al. (1969) is shown in Table IV for the grass species studied in the preliminary study. The data were found to agree with that of Twiss et al. in that Elongate and Panicoid classes were observed in residue from the tops of the tall grass species (big bluestem, little bluestem, and Indiangrass) whereas Elongate and Chloridoid classes occurred in the tops of buffalograss, the only short grass species studied. The occurrence of the shapes is expressed as a percent. It was observed that those shapes characteristic of the Panicoid class were most abundant in the residue from big bluestem (44%) and lower in Indiangrass and little bluestem tops (17%). Typical Panicoid phytoliths are shown in Figure 5.

##### Soil Phytoliths

The phytolith shapes which were encountered in the surface horizons of the soils studied are given in Table IV. Festucoid,



A Panicoid Phytoliths



B Chloridoid Phytolith

Figure 5. Typical Panicoid and Chloridoid Opaline Phytoliths.

TABLE IV

PERCENT OF OPALINE PHYTOLITH CLASSES OCCURRING IN PLANT TOPS  
AND 5-20 $\mu$  FRACTIONS OF SELECTED SOIL HORIZONS

Sample	Festucoid	Chloridoid	Panicoid	Elongate	Non descript
<u>Plants</u>					
Big bluestem			44.1	6.6	49.2
Little bluestem			17.0	29.1	64.5
Indiangrass			17.8	32.2	50.0
Buffalograss		32.3		9.8	61.6
<u>Soils</u>					
St. Paul Ap	3.3	6.6		10.8	79.2
Zaneis A11	4.3	5.2	6.1	9.2	75.0
Fitzhugh Ap	1.8	3.5	4.7	11.7	78.4
Meno Ap	3.6	5.7	2.1	13.6	75.0
Meno A1	1.1	2.2	1.1	4.4	91.2
Meno B2	1.8	1.8	1.8	9.7	85.0
Meno C1		4.3		12.1	83.6
Meno II21tb				12.7	87.3

Chloridoid, and Elongate classes were encountered in the Ustoll, St. Paul, surface indicating a dominance of short grass species as well as introduced grass species. Mixed grasses are the climax vegetation of this region, although short grasses have probably been more abundant than tall grasses. The Udoll-Ustoll, Zaneis, surface contained Festucoid (Figure 5), Chloridoid, and Panicoid classes indicating the influence of introduced grasses as well as both short and tall grass species, which would be expected since the Zaneis is in an area where mixed grasses are considered the native vegetation (Harlan, 1958). The Udoll, Fitzhugh, contained the same shape classes as did the Udoll-Ustoll, Zaneis. The Fitzhugh occurs in a tall grass region of the state, however management and grazing practices may well account for the indication of the presence of grasses of the Chloridoid (short grass) group. The Fitzhugh contained a lower (4.7%) percentage of Panicoid grains than the Zaneis (6.1%); however, these data are considered to be mainly qualitative (Brewer, 1964).

It was observed that only the phytolith grains in the 5-20 $\mu$  fraction possessed the shapes characterized by Twiss et al. (1969) except for the Elongate class. The 20-35 $\mu$  and 35-50 $\mu$  fractions contained phytolith grains of somewhat more regular shape plus Elongate and teardrop shapes. Also the shapes characterized above were seldom encountered below the surface horizon, even in the 5-20 $\mu$  fraction. Therefore in the Oklahoma soils studied, it appears necessary to use the 5-20 $\mu$  size range from surface horizons to characterize phytolith grains on the basis of shape. The absence below the surface horizon of the shapes characterized from grass tops re-emphasizes that phytoliths below the surface horizon are primarily of root origin.

### Origin of the 20-50 $\mu$ Phytoliths

It was observed in this study that the percentage of phytoliths encountered in the plant residue seldom was larger than that observed in the 5-20 $\mu$  soil fractions. This poses the question of origin of the phytolith grains encountered in the soil within the 20-50 $\mu$  range. Plant samples studied were taken in Payne County in the same area in which the Zaneis transect soil was sampled. While the surface horizon of the Zaneis contained 33% phytoliths of the 20-50 $\mu$  size range, sizes of this magnitude were not obvious in the phytolith residue from plants of the same area.

The possibility exists that a part of the phytoliths of the 20-50 $\mu$  fraction is of geologic rather than plant origin, a factor usually not considered significant in opaline studies. Non-biogenetic opal could be present as residual opal or as a result of soil formation. Since opaline phytoliths are known to contain occluded carbon, perhaps a comparison of carbon content between the 5-20 $\mu$  and 20-50 $\mu$  fractions would serve to differentiate between opaline phytoliths and geologic opal. On the other hand, Twiss et al. (1969) showed Elongate forms of plant phytoliths that were in excess of 100 $\mu$ . Another possibility in light of the latter work is that the majority of the 20-50 $\mu$  phytolith grains were broken by the processing used in obtaining the residue. Since many broken phytolith grains are observed in plant residue, this may well account for the absence of significant numbers of large phytoliths in plant residue. From the data in this study, a solution to this problem cannot be obtained. This appears to be an area where additional study is needed.

### Interpretation of the Meno-Like Bisequum Soil

The phytoliths from the combined silt fraction of the Meno-like soil ranged from 0.96 mg/g of soil in the surface down to 0.46 mg/g of soil in the IIB21tb horizon at a depth of 48 cm (Figure 6). These phytolith levels, even though more uniformly distributed with depth than those of the transect soils, are considerably lower. The lower phytolith levels may be due to the lack of maturity and lack of continued grass influence in the formation of the upper profile. The slight increase of phytoliths in the 38-48 cm horizon or C1 was suggestive of a buried A horizon, however other soil properties discussed below fail to support this. The discontinuity appears to be between the 38-48 cm horizon and the 48-73 cm horizon based on the change of the percent of the sand that is very fine sand, and on organic matter content (Table V).

The upper soil appears to have a thickness of 48 cm which is underlain by B horizons of a buried soil.

Since some Panicoid and Chloridoid shapes are found throughout the modern soil (Table IV), it suggests that deposition of the transported material forming the soil was slow or intermittent allowing some growth of vegetation throughout deposition. Another explanation for the presence of plant-top phytoliths throughout the profile would be the presence of phytoliths in the material when it was deposited. The latter explanation is somewhat preferred because the 38-48 cm horizon has morphological properties characteristic of a C horizon including massive structure and an abrupt boundary (See Appendix A). If this is in fact a C horizon, then materials overlying it would be expected to contain phytoliths of similar shapes and similar phytolith quantities, as is the case. The types of phytoliths present throughout the upper

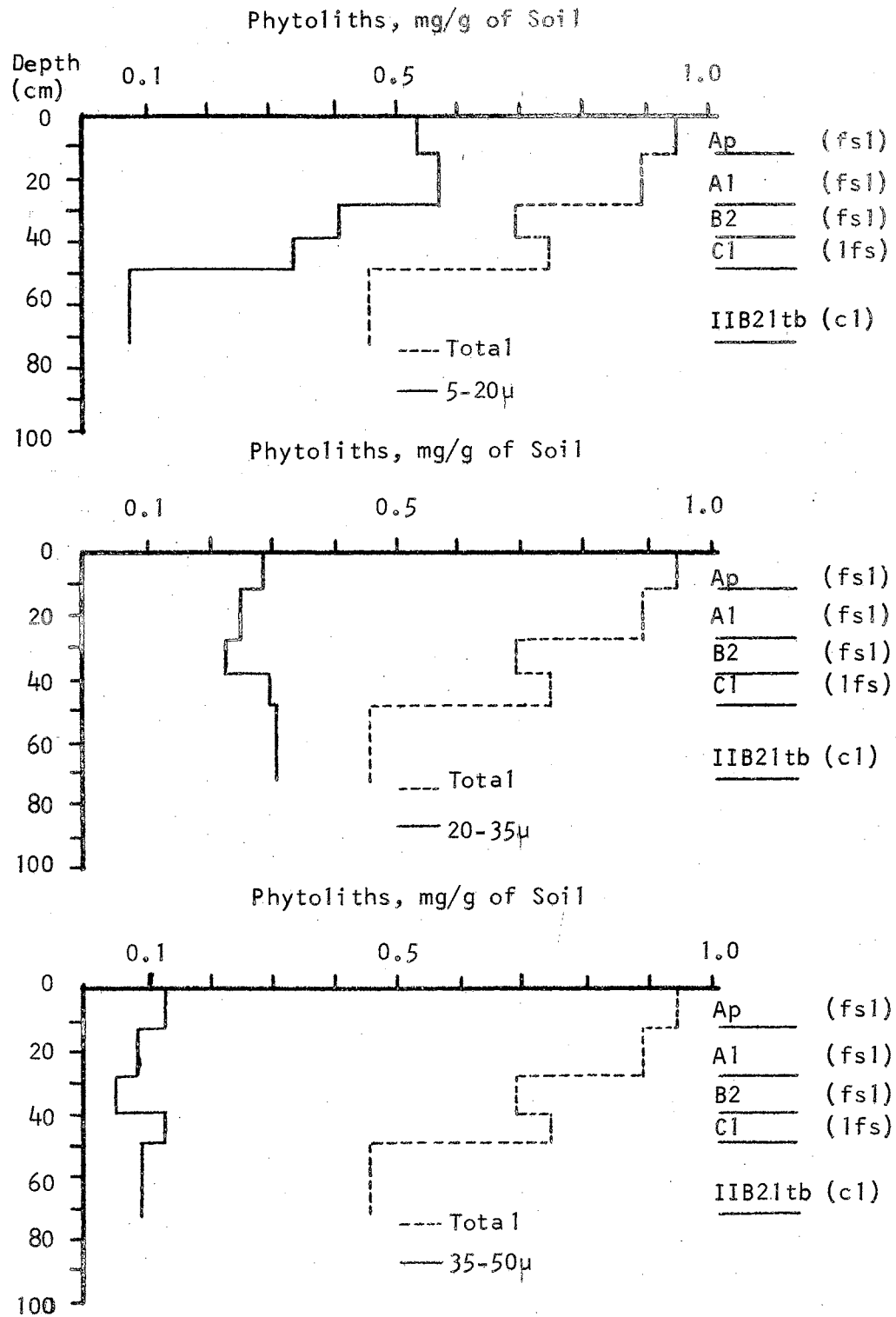


Figure 6. Phytolith Variation in the Meno-Like Bisequum Soil.

TABLE V  
SOIL FRACTIONATION AND ORGANIC MATTER PERCENTAGES IN THE MENO-LIKE SOIL

Horizon	Depth (cm)	% Silt				% Sand		% Organic Matter
		5-20 $\mu$	20-35 $\mu$	35-50 $\mu$	Total	vfs	Total	
Ap	0-13	4.85	5.88	7.37	18.10	36.09	68.00	1.10
A1	13-28	4.79	5.95	7.37	18.11	35.86	68.70	1.12
B2	28-38	5.20	6.11	7.50	18.81	34.02	69.70	0.88
C1	38-48	3.75	4.28	7.13	15.16	37.98	73.42	0.67
IIB21tb	48-73	15.61	11.42	10.54	37.57	20.34	31.58	1.20
IIB22tb	73-98	14.53	10.05	9.05	33.63	20.87	33.32	1.00



horizons of the soil profile imply that the soil parent material is of localized sediments since the soil occurs in a 30 inch rainfall area where mixed Chloridoid and Panicoid grasses grow. The origin of the dark IIB2b horizons is not definite, but they may have been surfaces below a shallow playa lake which collected organic materials, silts, and clays from adjoining grassland areas. The upper soil would have been deposited later by erosion of adjacent uplands. The time lapse since the deposition of the younger soil has probably been insufficient for the formation of a Mollisol, and deposition of phytoliths characteristic of them.

## CHAPTER V

### SUMMARY AND CONCLUSIONS

#### Summary

A preliminary study of opaline phytoliths from soils and plants was conducted in order to generally characterize phytoliths found in Oklahoma. A more specific study was then conducted to observe phytolith variations among three soils, a Typic Argiustoll, an Udic Argiustoll, and a Typic Argiudoll, occurring along an east-west transect. A fourth soil (a Meno-like soil) containing buried horizons was studied to observe phytolith variation with depth in such a soil.

From the preliminary study, phytoliths were found to be much more abundant in grasses than in trees. The phytolith content of grass roots was as high or higher than that of the aerial portions in most cases. Phytoliths in the soil were more highly concentrated in the 5-20 $\mu$  fraction than in the 20-50 $\mu$  fraction.

In the transect soils, phytoliths were found to be most highly concentrated in the surface horizon with sharp declines in phytolith levels below the surface, indicating little influence of plant-top phytoliths in the lower horizons. Phytolith distribution with depth appeared to be associated with soil texture.

Phytolith quantities at comparable depths were higher in the Typic Argiudoll than in the Typic Argiustoll. However the Udic Argiustoll was lower in phytolith content than both of the above soils rather than

intermediate as would be expected,

Based on percentages occurring in the 5-20 $\mu$ , 20-35 $\mu$ , and 35-50 $\mu$  soils fractions, phytoliths were found to be smaller in the Typic Argiustoll and Udic Argiustoll than in the Typic Argiudoll.

Panicoid and Elongate phytolith shapes were present in residue from the tall grass species studied while Chloridoid and Elongate shapes were present in the short grass studied. Festucoid, Chloridoid, and Elongate forms were found in the Typic Argiustoll, while these same shapes plus Panicoid phytoliths were found in the two remaining transect soils. With the exception of the Elongate class of phytolith shape, characteristic shapes were essentially absent from the 20-35 $\mu$  and 35-50 $\mu$  soil fractions and from horizons below the surface.

Distribution of phytoliths in the bisequum soil was low and relatively uniform with depth. The opaline phytolith data did not support evidence of a buried A horizon in the soil.

### Conclusions

Conclusions drawn from the study are as follows:

1. Opaline phytoliths in all of the Oklahoma soils studied were more common in the 5-20 $\mu$  than in the 20-50 $\mu$  fraction of soils except in Fitzhugh, the Udoll, where the two fractions were approximately equal. This is interpreted to mean that in general plants growing on these soils have relatively small cells with the resultant deposition of small phytolith grains.
2. Opaline phytolith sizes were smaller in the two Ustolls than in the Udoll, suggesting the existence of a phytolith size-annual rainfall relationship. Additional transect points are

required to fully confirm this relationship.

3. Opaline phytolith levels of the sizes studied are not sufficiently high at lower soil depths to be useful in establishing lower pedon boundaries.
4. Phytoliths originating from plant tops appears to be concentrated in the surface horizon of residual soils with phytoliths below the surface originating primarily in roots.
5. In order to fully characterize phytoliths from buried soils, some part of a buried Mollic epipedon should be present.
6. In Oklahoma, phytolith shapes of the Festucoid, Chloridoid, and Panicoid class most commonly occur in the 5-20 $\mu$  fraction of surface horizons.

#### LITERATURE CITED

- Baker, G. 1959a. Fossil opal-phytoliths and phytolith nomenclature. Australian J. Sci. 21:305-306.
- \_\_\_\_\_. 1959b. Opal phytoliths in some Victorian soils and "red rain" residues. Australian J. Bot. 7:64-87.
- \_\_\_\_\_. 1960. Hook-shaped opal phytoliths in the epidermal cells of oats. Australian J. Bot. 8:69-74.
- \_\_\_\_\_ and G. W. Leeper. 1958. Phytoliths in Victorian soils. Australian J. Sci. 21:84.
- Beavers, A. H. and I. Stephen. 1958. Some features of the distribution of plant-opal in Illinois soils. Soil Sci. 86:1-5.
- Brewer, Roy. 1964. Fabric and mineral analysis of soils. John Wiley and Sons, Inc., New York.
- Daubenmire, R. F. 1948. Plants and environment. John Wiley and Sons, Inc., New York.
- Dwyer, Don D. 1961. Activities and grazing preferences of cows with calves in Northern Osage County, Oklahoma. Okla. State Univ. Expt. Sta. Bull. B-588.
- Folger, D. W., L. H. Burckle, B. C. Heezen, 1967. Opal phytoliths in a North Atlantic dust fall. Sci. 155:1243-44.
- Fronde!, Clifford. 1962. The system of mineralogy of James Dana and Edward Dana, 7th ed. Vol. III, Silica minerals. John Wiley and Sons, Inc., New York.
- Giesecking, J. E., H. J. Snider, and C. A. Getz. 1935. Destruction of organic matter in plant material by the use of nitric and perchloric acids. Ind. and Eng. Chem. (Analytical Ed.) 7:185-186.
- Gill, Edmund D. Stability of biogenetic opal. Sci. 158:810.
- Harlan, J. R. 1958. Grasslands of Oklahoma. Okla. State Univ. Processed Teaching Manual, Agronomy Dept.
- Jackson, M. L. 1956. Soil chemical analysis-advanced course. Mimeo. Publ. by author, Dept. of Soil Science, Univ. of Wis., Madison.

- Jones, L. H. P. and K. A. Handreck. 1963. Effects of iron and aluminum oxides on silica in solution in soils. *Nature* 198:852-853.
- 
- \_\_\_\_\_. 1967. Silica in soils, plants, and animals in *Advances in Agronomy* 19:107-149, A. G. Norman, Ed. Academic Press, New York.
- Jones, L. P. H., A. A. Milne, and J. V. Sanders. 1965. Tabashir: an opal of plant origin. *Sci.* 151:464-466.
- Jones, L. P. H., A. A. Milne, and S. M. Wadham. 1963. Studies of silica in the oat plant. II. Distribution of the silica in the plant. *Plant and Soil* 18:358-371.
- Jones, Robert L. and A. H. Beavers. 1963a. Some mineralogical and chemical properties of plant opal. *Soil Sci.* 96:375-379.
- 
- \_\_\_\_\_. 1963b. Sponge spicules in Illinois soils. *Soil Sci. Soc. of Amer. Proc.* 27:438-440.
- 
- \_\_\_\_\_. 1964a. Aspects of catenary and depth distribution of opal phytoliths in Illinois soils. *Soil Sci. Soc. Amer. Proc.* 28:413-416.
- 
- \_\_\_\_\_. 1964b. Variation of opal phytolith content among some great soil groups in Illinois. *Soil Sci. Amer. Proc. Notes* 28:711-712.
- Kanno, Ichiro and Shizuiki Arimura. 1958. Plant opal in Japanese soils. *Soil and Plant Food* 4:62-67.
- Kilmer, V. J. and L. T. Alexander. 1949. Methods of making mechanical analysis of soils. *Soil Sci.* 68:15-24.
- Lanning, F. C., B. W. V. Ponnaiya, and C. E. Crumpton. 1958. The chemical nature of silica in plants. *Plant Physiol.* 33:339-343.
- Maximov, N. A. 1929. *The plant in relation to water.* The MacMillan Company, New York.
- McKeague, J. A. and M. G. Cline. 1963. Silica in soils in *Advances in Agronomy* 15:339-396. A. G. Norman, Ed. Academic Press, New York.
- Parry, D. W. and Smithson, F. 1957. Detection of opaline silica in grass leaves. *Nature* 179:975-976.
- 
- \_\_\_\_\_. 1966. Opaline silica in the inflorescences of some British grasses and cereals. *Ann. Bot.* 30:525-538.
- Pease, D. S. 1967. Opal phytoliths as indicators of paleosols. (Unpub. M. S. Thesis, New Mexico State University).

- Pease, D. S. and J. U. Anderson. 1969. Opal phytoliths in Bouteloua eriopoda torr., roots and soils. Soil Sci. Soc. Amer. Proc. Notes 33:321-322.
- Schoen, Robert and Donald E. Lee. 1964. Successful separation of silt-size minerals in heavy liquids. U. S. Geol. Survey Prof. Paper 501-B:B154-B157.
- Smithson, F. 1956a. Silica particles in some British soils. J. Soil Sci. 7:122-129.
- \_\_\_\_\_. 1956b. Plant opal in soil. Nature 178:107.
- \_\_\_\_\_. 1959. Opal sponge spicules in soils. J. Soil Sci. 10:105-109.
- Soil Survey Staff, SCS, USDA. 1960. Soil classification, a comprehensive system, 7th approximation, as amended June, 1964.
- Tomanek, G. W., Edwin P. Martin and F. W. Albertson. 1958. Grazing preference comparisons of six native grasses in the mixed prairie. J. Range Management 11:191-193.
- Twiss, P. C., Erwin Suess, and R. M. Smith. 1969. Morphological classification of grass phytoliths. Soil Sci. Soc. Amer. Proc. 33:109-114.
- United States Salinity Laboratory Staff. 1964. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60, L. A. Richards, Ed. U. S. Govt. Printing Off., Washington, D. C.
- Wilding, L. P. 1967. Radiocarbon dating of biogenetic opal. Sci. 156:66-67.
- Wilding, L. P., Robert E. Brown, and N. Holowaychuk. 1967. Accessibility and properties of occluded carbon in biogenetic opal. Soil Sci. 103:56-61.
- Witty, John E. and Ellis G. Knox. 1964. Grass opal in some chestnut and forested soils in North Central Oregon. Soil Sci. Soc. Amer. Proc. 28:685-687.
- Yoshida, Shoichi, Yoshiko Onishi and Kauzo Kitagishi. 1959. The chemical nature of silicon in rice plant. Soil and Plant Food 5:23-27.

APPENDIX A  
SOIL PROFILES



## Fitzhugh Profile

The profile was located on the O. S. U. Eastern Oklahoma Pasture Research Station near Muskogee, Oklahoma, in Muskogee County. It was located 1320 feet north and 1120 feet east of the SW $\frac{1}{2}$  Sec 33 T14N R17E. The site was in a cultivated field. It was on a convex 2% slope. All characteristics are for moist conditions. Described by R. Yeck.

Horizon	Depth cm	Description
Ap	0-13	Dark brown (7.5YR 3/2) fine sandy loam; weak fine granular structure; very friable; plentiful roots; abrupt, smooth boundary.
A1	13-33	Dark brown (7.5YR 3/2) fine sandy loam; moderate, fine granular structure; very friable; few, fine, black concretions; common worm casts; few crotovinas filled with yellowish red loam (cylindrical 6 mm diameter); roots plentiful; clear, smooth boundary.
A&B	33-48	Reddish brown (5YR 4/4) and dark brown (7.5YR 3/2) loam; very weak fine subangular blocky structure; very friable; common fine, black concretions; crotovinas as noted above common; roots plentiful, clear smooth boundary.
B21	48-68	Reddish brown (5YR 4/4) heavy loam; weak fine, subangular blocky structure; very friable; discontinuous clay films; common medium (up to 6 mm diameter) black concretions; common worm casts; few fine roots; clear, smooth boundary.
B22	68-88	Yellowish red (5YR 4/8) clay loam; moderate fine subangular blocky structure; friable; few faint yellowish red (5 YR 5/6) mottles; then discontinuous clay films; very common medium black concretions; few roots; clear, smooth boundary.
B3	88-103	Mottled brownish yellow (10YR 6/8) and yellowish red (5YR 4/8) clay loam; moderate to strong fine subangular, blocky structure; friable; discontinuous clay films; many medium black concretions; clear, smooth boundary.
C1	103-140	Yellowish brown (10YR 5/6) light gritty clay; massive; friable; many medium prominent, red (2.5YR 4/6) mottles; concretions increase to approximately 75% of the volume near the lower horizon boundary; few fine roots.

## Meno-Like Bisequum Profile

This soil was sampled on the O. S. U. Peanut Research Station near Fort Cobb, Oklahoma, in Caddo County. It was located 535 feet south and 805 feet east of the northeast corner of the SE $\frac{1}{4}$  Sec 21 T8N R12W. The site was in a cultivated, nearly level field. All characteristics are for the moist condition. Described by R. Yeck and C. Stahnke.

Horizon	Depth cm	Description
Ap	0-13	Brown (7.5YR 4/4) fine sandy loam; weak fine granular structure; very friable; clear, smooth boundary.
A1	13-28	Brown (7.5YR 4/4) fine sandy loam; weak fine granular structure; very friable; few discontinuous sand lenses; clear, smooth boundary.
B2	28-38	Reddish brown (5YR 4/4) heavy fine sandy loam; weak moderate, subangular blocky; friable; abrupt, smooth boundary.
C1	38-48	Brown (7.5YR 4/4) loamy fine sand; massive, very friable; abrupt smooth boundary.
IIB21tb	48-73	Dark brown (7.5YR 3/2) clay loam; moderate, medium to coarse subangular blocky structure; friable; then discontinuous clay films; clear, smooth boundary.
IIB22tb	73-98	Dark grayish brown (10YR 4/2) silty clay loam; moderate medium subangular blocky structure; firm; thin, discontinuous clay films; few, very fine (up to 3 mm diameter) black concretions; clear, smooth boundary.
IIIC1	98-113	Brown (10YR 4/3) clay loam; massive, friable; few faint strong brown (7.5YR 5/6) mottles; common very fine black concretions; clear, smooth boundary.
IVC2	113-150	Brown (10YR 5/3) loamy fine sand; massive; very friable; common, distinct strong brown (7.5YR 5/6) mottles.

## St. Paul Profile

The sample was taken 1 mile South and 1 1/3 miles east of Seiling, Oklahoma, in Dewey County, or 1700 feet east and 600 feet west of the southwest corner of Sec 10 T19N, R16W. It was in an area with a convex slope of 2%. The vegetation consisted of native grasses in an old field. All characteristics are for the moist condition, except where otherwise noted. Described by R. Yeck and C. Stahnke.

Horizon	Depth cm	Description
Ap	0-13	Brown (10YR 4/3) silt loam; weak fine granular structure; very friable; abundant roots, clear smooth boundary.
A1	13-35	Brown (7.5YR 4/2) silt loam; weak fine subangular blocky structure; friable; abundant roots; gradual, smooth boundary.
B1	35-50	Brown (7.5YR 4/2) silt loam; weak medium subangular blocky structure; friable; plentiful roots; clear smooth boundary.
B21t	50-75	Brown (7.5YR to 5YR 4/2) silty clay loam; moderate, medium, subangular blocky structure; friable; thin continuous clay films; plentiful roots; clear smooth boundary.
B22t	75-95	Reddish brown (5YR 4/3) silty clay loam; moderate, coarse, subangular blocky structure; friable; thin continuous clay films; somewhat more developed than previous horizon; few roots; lower inch of horizon weakly calcareous; clear, smooth boundary.
B23t	95-125	Yellowish red (5YR 4/6) silty clay loam; moderate medium prismatic structure breaking to moderate coarse blocky; hard when dry; thin, discontinuous clay films; calcareous; few lime concretions and threads; gradual, smooth boundary.
B3	125-145	Yellowish red (5YR 4/6) clay loam; moderate, medium angular blocky structure; hard when dry; lime concretions up to 12 mm diameter, common threads of limestone; clear, smooth boundary.
C	145-163	Yellowish red (5YR 4/6) fine sandy loam; massive.

## Zaneis Profile

The sample was taken 1 mile north and  $\frac{1}{2}$  mile west of Stillwater, Oklahoma, in Payne County, or 1962 feet west and 105 feet south of the northeast corner of Sec 10 T19N R2E. The vegetation was native grasses, occurring on a 2% convex slope. All characteristics are for the moist condition. Described by R. Yeck and D. Rogers.

Horizon	Depth cm	Description
A11	0-15	Dark reddish brown (5YR 3/2) light loam; weak fine granular structure; very friable; abundant roots; few worm casts; clear, smooth boundary.
A12	15-29	Dark reddish brown (5YR 3/3) loam; weak fine subangular blocky and moderate medium granular structure; very friable; abundant roots; few worm casts; clear, smooth boundary.
B1	29-40	Reddish brown (5YR 4/4) light clay loam; moderate medium subangular blocky structure; friable; common very fine, faint, yellowish red (5YR 4/8) mottles, abundant roots; very few worm casts; clear, smooth boundary.
B2t	40-65	Red (2.5YR 4/6) clay loam; moderate, medium, subangular blocky structure; friable; common very fine, faint red (2.5YR 4/8) mottles; continuous reddish brown clay films; plentiful roots; clear, smooth boundary.
B3t	65-93	Yellowish red (5YR 5/6) sandy clay loam; moderate medium subangular blocky structure; friable; many distinct, medium pale brown (10YR 6/3) and few medium distinct very pale brown (10YR 7/3) mottles; continuous clay films; few black concretions in lower part of horizon; few fine roots; smooth, gradual boundary.
C	93-108	Light brown (7.5YR 6/4) sandy clay loam; structureless, many medium yellowish brown (10YR 5/8) mottles; many, medium black concretions; horizon consists mainly of weathered sandstone including a few sandstone rocks.
R	108	Sandstone

APPENDIX B  
MECHANICAL DATA

TABLE VI  
SOIL FRACTIONATION AND ORGANIC MATTER PERCENTAGES IN THE FITZHUGH SOIL

Horizon	Depth (cm)	% Silt				% Sand		% Organic Matter
		5-20 $\mu$	20-35 $\mu$	35-50 $\mu$	Total	vfs	Total	
Ap	0-13	12.40	18.92	24.30	55.62	17.20	26.41	3.23
A1	13-33	12.20	19.18	21.07	52.45	16.72	28.26	2.12
A&B	33-48	13.80	20.78	21.00	55.58	17.34	28.26	1.68
B21	48-68	12.67	17.58	18.65	48.90	15.12	25.13	
B22	68-88	19.53	16.02	17.82	53.37	13.34	21.52	
B3	88-103	12.64	16.91	17.46	47.01	13.69	21.56	
C	103-140	12.27	15.37	15.32	42.96	12.63	25.90	

TABLE VII

## SOIL FRACTIONATION AND ORGANIC MATTER PERCENTAGES IN THE ST. PAUL SOIL

Horizon	Depth (cm)	% Silt				% Sand		% Organic Matter
		5-20 $\mu$	20-35 $\mu$	35-50 $\mu$	Total	vfs	Total	
Ap	0-13	7.66	16.72	27.79	42.17	26.06	29.50	1.72
A1	13-35	9.10	16.84	23.50	49.50	21.95	24.73	1.71
B1	35-50	10.28	15.80	21.94	48.02	19.36	23.02	1.46
B21t	50-75	10.66	12.74	16.22	39.62	20.28	22.33	
B22t	75-95	9.90	9.98	13.86	33.74	26.23	31.88	
B23t	95-125	8.21	9.27	14.57	32.05	29.64	35.27	
B3	125-145	7.50	8.53	15.36	31.39	34.06	39.94	

TABLE VIII

SOIL FRACTIONATION AND ORGANIC MATTER PERCENTAGES IN THE ZANEIS SOIL

Horizon	Depth (cm)	% Silt				% Sand		% Organic Matter
		5-20 $\mu$	20-35 $\mu$	35-50 $\mu$	Total	vfs	Total	
A11	0-15	5.80	6.43	8.31	20.54	13.42	59.75	2.92
A12	15-29	5.25	5.41	7.10	17.76	13.78	58.55	2.21
B1	29-40	5.06	4.19	5.54	14.79	13.42	54.59	1.81
B2t	40-65	4.87	4.98	3.54	13.39	13.71	55.14	
B3t	65-93	6.05	1.89	2.80	10.74	14.36	57.65	
C	93-108	7.69	2.51	2.64	12.84	11.46	50.01	



APPENDIX C  
PHYTOLITH DATA

TABLE IX  
 FRACTIONATED OPALINE PHYTLITHS IN HORIZONS OF THE FITZHUGH SOIL

Horizon	Depth (cm)	5-20 $\mu$		20-35 $\mu$		35-50 $\mu$		Horizon total mg/g. of soil
		mg/g of soil	% of Horizon total	mg/g of soil	% of Horizon total	mg/g of soil	% of Horizon total	
Ap	0-13	5.27	55.2	2.99	31.3	1.28	13.5	9.54
A1	13-33	2.90	52.0	1.90	34.0	0.78	14.0	5.58
A&B	33-48	1.86	49.9	1.41	37.8	0.46	12.3	3.73
B21	48-68	0.84	50.6	0.60	36.1	0.22	13.3	1.66
B22	68-88	0.44	61.1	0.26	36.1	0.02	2.8	0.72
B3	88-103	0.09						

TABLE X  
FRACTIONATED OPALINE PHYTOLITHS IN HORIZONS OF THE MENO-LIKE SOIL

Horizon	Depth (cm)	5-20 $\mu$		20-35 $\mu$		35-50 $\mu$		Horizon total mg/g of soil
		mg/g of soil	% of Horizon total	mg/g of soil	% of Horizon total	mg/g of soil	% of Horizon total	
Ap	0-13	0.54	56.8	0.28	29.5	0.13	13.7	0.95
A1	13-28	0.57	63.3	0.25	27.8	0.08	8.9	0.90
B2	28-38	0.41	59.4	0.23	33.3	0.05	7.3	0.69
C1	38-48	0.33	44.0	0.29	38.7	0.13	17.3	0.75
IIB21tb	48-73	0.07	15.2	0.30	65.2	0.09	19.6	0.46
IIB22tb	73-98	0.09		0.03		Trace		

TABLE XI

## FRACTIONATED OPALINE PHYTOLITHS IN HORIZONS OF THE ST. PAUL SOIL

Horizon	Depth (cm)	5-20 $\mu$		20-35 $\mu$		35-50 $\mu$		Horizon total mg/g of soil
		mg/g of soil	% of Horizon total	mg/g of soil	% of Horizon total	mg/g of soil	% of Horizon total	
Ap	0-13	3.65	69.2	1.32	25.4	0.28	5.4	5.27
A1	13-35	1.64	64.6	0.81	31.9	0.09	3.5	2.54
B1	35-50	1.27	81.4	0.25	16.0	0.04	2.6	1.56
B21t	50-75	0.18		0.20		Trace		
B22t	75-95	0.46		Trace				

TABLE XII

## FRACTIONATED OPALINE PHYTOLITHS IN HORIZONS OF THE ZANEIS SOIL

Horizon	Depth (cm)	5-20 $\mu$		20-35 $\mu$		35-50 $\mu$		Horizon total mg/g of soil
		mg/g of soil	% of Horizon total	mg/g of soil	% of Horizon total	mg/g of soil	% of Horizon total	
A11	0-15	2.11	67.0	0.75	24.1	0.28	8.9	3.15
A12	15-29	0.76	65.0	0.40	34.2	0.01	0.8	1.17
B1	29-40	0.28	48.3	0.22	37.9	0.08	13.8	0.58
B2t	40-65	0.08		0.06		Trace		

VITA

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