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# The Use of Heavy Minerals in Studies of the Origin and Development of Soils

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# The Use of Heavy Minerals in Studies of the Origin and Development of Soils\*

J. F. HASEMAN AND C. E. MARSHALL

## INTRODUCTION

The soil science of the present day seeks to express, as far as possible, in quantitative form, the fact that soils are derived from geological materials, which were later subjected to a small group of definable factors leading to the development of soil profiles as we now observe them. It is evident, therefore, that the recognition of parent materials and the measurement of the changes which have taken place within them are experimental problems of the highest importance. The present communication deals with methods, largely adapted from those used by sedimentary petrographers, which lend themselves directly to the solution of these problems. Several previous researches, some of them begun here and completed elsewhere (27, 41, 42, 46), had already provided a considerable measure of experience in the attainment of this general aim. This was especially the case with regard to the recognition of parent materials, since here the techniques were already known. Thus the soils worker had merely to select the particular combination of methods which would best serve his own purpose. Part II of the present study is devoted to this subject.

The measurement of the changes which have occurred during soil formation has not previously been accomplished in a rigorously quantitative way. A considerable amount of experimentation with different methods and with different soils as described in Part III preceded the successful attainment of this end. Finally in Part IV a quantitative study of a Grundy soil profile, derived from loess, is described. The mineral zircon is here used for the first time as the immobile indicator of gains or losses. It is believed that this provides the most satisfactory basis at present attainable in the calculation of changes undergone in soil development.

In spite of the difficulties, these efforts are probably well spent even if only one profile out of five selected for study should prove sufficiently homogeneous in origin to make a strict application of the new technique possible. One need only reflect how greatly soil science would be advanced if ten completely successful studies, well distributed amongst the major soil groups, were available.

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\*The experimental material here presented is taken from the Ph.D. Thesis entitled "An investigation of the use of heavy minerals in determining the origin and course of profile development in soils," by J. F. Haseman in the University of Missouri, June, 1944.

## PART I.—REVIEW OF LITERATURE

Minerals are frequently classified into two groups on a basis of their specific gravity. The so-called light minerals are generally taken to include those with a density less than about 2.85, whereas the heavy minerals have a density greater than this. The light fraction includes quartz, calcite, feldspar, clay minerals and the micas. These minerals generally make up about 98% of a sediment. The heavy minerals, though they constitute only about 2% of the sample, include a much greater variety of mineral species and are, therefore, generally more useful than the light fraction in characterizing a particular deposit. Most of the accessory minerals are heavy minerals.

Minerals differ widely in their resistance to chemical and physical destruction. This resistance depends upon numerous factors such as hardness, cleavage, coefficient of expansion, original cracks in the crystal, solubility under a given environment, etc. Minerals which are stable under one set of conditions may be quite unstable under another.

Numerous attempts have been made to arrange minerals in the order of their resistance to weathering. Pettijohn<sup>50\*</sup> has calculated an order of persistency by comparing the minerals in sedimentary rocks of increasing geologic age with those of recent sediments. His series is as follows: —3 anatase, —2 muscovite, —1 rutile, 1 zircon, 2 tourmaline, 3 monazite, 4 quartz, 5 garnet, 6 biotite, 7 apatite, 8 microcline, 9 ilmenite, 10 magnetite, 11 staurolite, 12 kyanite, 13 epidote, 14 hornblende, 15 andalusite, 16 topaz, 17 titanite, 18 zoisite, 19 augite, 20 sillimanite, 21 hypersthene, 22 diopside, 23 actinolite, and 24 olivine. The negative sign means that anatase, muscovite, and rutile are more abundant in ancient sediments than they are in recent sediments. This is due to the fact that they are frequently secondary as well as primary minerals.

Other investigators list their order of resistance somewhat differently. Smithson<sup>58</sup>, for example, places zircon, rutile, tourmaline, and apatite in the stable group, monazite in the somewhat stable group, garnet, kyanite, and staurolite in the unstable group, and the ferro-magnesium minerals in the very unstable group. Goldrich<sup>15</sup>, after studying the degree of weathering of minerals in granite gneiss, gives the following stability series: biotite, potash feldspar, muscovite, and quartz > hornblende and plagioclase feldspar > augite > olivine.

We generally consider zircon and tourmaline as stable under any weathering conditions. They are very hard and are not noticeably attacked by any acids or bases. However, work by J. H. Druif<sup>10</sup> on

\*Superscript numerals refer to references in the bibliography.

the tropical soils of Sumatra indicates otherwise. Druif concludes that all minerals dissolve under the chemical conditions in the soil in the tropics. Therefore, he suggests that the minor nutrient elements, such as boron and zinc, are probably furnished to many soils from the weathering of such very resistant minerals as tourmaline and green spinel. If this does occur, then the importance of the mineral reserve in the soil is even greater than has been generally thought possible.

Observations by other authors also indicate the possibility that zircon is attacked chemically. Butterfield<sup>3</sup> and Smithson<sup>56</sup> have recorded cases of secondary zircon. Butterfield's study of the Millstone Grit of the Pennine Region discloses a number of instances where secondary outgrowths of zircon occur in optical continuity with the parent zircon. Many zircons in the deposit show marked etching.

Goldrich<sup>15</sup>, from a study of the weathering of granite gneiss in Minnesota, concluded that zircon is sometimes chemically attacked. In such cases it displays a dusky, granular appearance.

The possibility that tourmaline may sometimes be authigenic is indicated by some oil well core samples in West Virginia, described by Martens<sup>48</sup>. Authigenic tourmaline has also been described in the Oriskany Sandstone by Stow<sup>59</sup>. Such cases show that under certain conditions the constituents of tourmaline exist in solution.

Russell<sup>53, 54</sup> has contributed some valuable information to our knowledge concerning the resistance of mineral to weathering and abrasion. He examined hundreds of samples of sand from the Mississippi river between Cairo, Illinois, and the Gulf of Mexico, and was forced to conclude that there was no apparent destruction or sorting of any minerals by the river. We must, therefore, conclude that the disappearance of minerals, such as hornblende and augite, which are being deposited in the oceans, but which are uncommon in consolidated sedimentary deposits of older geologic age, have resulted from a chemical attack on these minerals after deposition. Possibly this change is brought about by sea water. Smithson's<sup>58</sup> work on the Estuarine Series of Yorkshire gives further support to the idea of mineral destruction by intrastratal solutions.

There have been a number of investigations of the rich assemblage of heavy minerals occurring in glacial till and loess deposits. Lamar and Grim<sup>37</sup> have studied sands and gravels of the glacial period and compared them with similar ones of Cretaceous age. They found a distinct difference in the heavy mineral suites. The glacial material suggested an igneous rock source and the Cretaceous sediments a metamorphic rock source.

Studies of the weathered rock in, on top of, and underlying glacial drift in New Hampshire have been made by Goldthwait<sup>16</sup>. He included a soil profile in his heavy mineral examination. However, the work is mostly qualitative and gives us no basis for computing

soil formation processes. The extent of weathering indicates a much longer time in the interglacial or pre-glacial period than in the post-glacial period.

A red clay-like deposit near Kansas City, Kansas, was examined by Hoover<sup>25</sup> who concluded from a mineralogical study that it was highly weathered glacial till. It might be difficult in some cases to distinguish such deposits from loess.

Griffiths<sup>17</sup> has traced the limits of glacial drift sheets in South Wales by means of the minerals of a particular grade size. Petrographically distinct drifts were produced by two local ice caps combined with a continental lobe of distant origin.

The difficulty of differentiating some drift sheets by the heavy mineral suite alone was pointed out by Kruger<sup>33</sup>. A study of several drift sheets of Minnesota revealed that their mineralogy is complex due to admixtures and reworking, and that few mineral species are diagnostic of any one drift sheet. If the amount of soluble carbonate and the heavy mineral assemblage are taken together, however, a fairly certain identification of the drift can be made. Furthermore, Kruger found that the distribution of heavy minerals in all the drifts is strikingly similar to their distribution in freshly crushed granite.

Two theoretical concepts were used with success by Krynine<sup>35</sup> in the solution of some complex problems dealing with glacial sediments reworked by streams. One is that heavy minerals decrease in size with increasing distance from the source, due to abrasion and sorting. This being true, one would expect local contaminations from tributaries of streams to produce an increase in heavy minerals in the coarse fractions. The other concept is that certain original heavy mineral ratios tend to persist even though the deposit becomes highly contaminated.

The number of papers written on petrographic studies of loess deposits in this country is rather small. Oefelern<sup>49</sup> has made an important study of loess deposits near St. Charles, Missouri. Heavy minerals studies were made on samples of grey loess and of buff loess which overlies the grey loess, in order to see if the two horizons represent different periods of deposition. Some important points were brought out in this study. In the first place, the per cent of heavy minerals varied considerably between samples taken from different places within both the buff and grey loess. The list of heavy minerals and their relative abundance corresponds remarkably well with those obtained in practically all of the many samples of loess examined in our present study from various loess covered areas of the Missouri and Mississippi valleys. Oefelern concluded that the two colored loesses were of the same age, and that the color distinctions were due to different amounts of the clay mineral beidellite which was deposited with the loess.



An interesting insight into the conditions of loess deposition is revealed by Smith<sup>55</sup>. He took loess samples at increasing distances from the river valley, and chiefly from the mechanical analysis and carbonate content of the samples, reached the following conclusions: 1. Differences between loess profiles are not closely associated with differences in texture, but rather with differences in carbonate content and time of exposure to weathering. 2. The climate of the loess deposition was not far different from that of today. 3. The rate of loess deposition decreases rapidly with increasing distance from its source. This results in greater weathering of the loess and increased losses of calcium carbonate as distance from the river increases. This weathering and carbonate loss is believed to have occurred mostly at the time the loess was being deposited.

Differentiation of loess and glacial till, which consisted of a large proportion of reworked loess has been described by Krynine<sup>35</sup>. For criteria he used evidence from comparisons of (1) the mechanical analysis, (2) minerals of the medium sand fractions, and (3) heavy minerals from the medium and fine sand fractions.

Different methods are used by different sedimentary petrologists to obtain quantitative data on the heavy minerals. Cogen<sup>8</sup> suggests it is best to separate the sediment into several grade sizes and make determinations of each fraction. He points out, however, that data so obtained cannot be compared with those of unseparated samples. He also mentions that mineral ratios often vary considerably between size groups of the same sediment. He further suggests the counting of all the heavy minerals in a given small weight of material separated with a micro-splitter.

Dorothy Carroll<sup>7</sup> suggests that studies be made on the fractions 0.2-0.02 mm. after treatment with 1:1 HCl to remove particle coatings of iron and aluminum. The use of Clerici solution in addition to bromoform was sometimes found to be a valuable aid in identification. She suggests counting only enough grains to give an accuracy of 5 per cent, since greater accuracy is in general not practical. The use of numbers to express percentage ranges is also suggested, and the results can be expressed in triangular diagrams which are very useful in reconnaissance work.

Goldman and DallaValle<sup>14</sup> compute the weight percentage of a given mineral by counting several hundred grains and then recording the diameter of each particle measured along a given straight line. From the median value of the diameter and the geometric standard deviation of the diameters, the diameter of a particle having the average volume is calculated. Knowing this average diameter, the number of particles of the species, the density of the species, and a shape factor, the weight of the component is computed. The method of calculation of the shape factor is also given.

Smithson<sup>57</sup> has written a very stimulating series of papers dealing

with methods of heavy mineral analysis. He believes it is better in making counts to use microscopic fields rather than lines, because lines tend to exaggerate the number of large-grained constituents. His method consisted in measuring the diameter of the minerals in two directions at right angles, and counting the grains at the same time. All grains were used which touched the micrometer scale during the traverse of the slide. Since the heavy minerals were not fractionated, it was necessary to make a correction for the exaggeration of large grains. Using the geometric mean of the length and width measurements, the counts, and assumed specific gravities, the percentage of the species was calculated. Smithson found that within the same deposit, the ratio of the average size of a given heavy mineral to that of another remains constant. Another interesting observation is that authigenic anatase is generally found associated with zircon larger than itself, whereas detrital anatase is associated with zircon smaller than itself. The same observation was made here in samples of the Tilsit silt loam soil profile.

The advantages and disadvantages of various techniques in sedimentary petrology have been discussed by Grout.<sup>18</sup> He finds that a preliminary magnetic separation often aids in identification and saves time in counting. Centrifuging with heavy liquids did not appreciably affect the quantity of heavy minerals recovered, neither did evacuation of the centrifuged sample, nor did use of several types of separation glassware. It is probable that Grout did not use the very fine sand and silt fractions in these studies. Grout further suggests that when the ratio between two mineral species only is desired, it saves time and is more accurate to count these two species only and to lump all other grains together. Counts made on an artificial mixture of garnet and mica showed that large errors may be introduced in counts which embrace grains of extreme tabular shape.

A very important paper by Rubey<sup>52</sup> points out the possibility of erroneous conclusions when one compares either (1) different size fractions of sediments, or (2) the same size fraction of sediments of different grain size. From a study of different grain-size samples of the same origin, Rubey found that epidote, kyanite, andalusite, rutile, and hypersthene are increasingly abundant in increasingly coarse grained samples, whereas magnetite, ilmenite, zircon, muscovite, and biotite increase in the successively finer grained samples. By considering the deposition of sediments according to Stokes' law, he concludes that the effect of shape on settling velocity is small except in extreme cases, such as those of the micas. Density, however, is an important factor resulting in deposition of small dense minerals under the same conditions as larger less dense ones. The

study of unfractionated samples is objectionable since effects of sorting cannot be corrected for. The best way to compare samples would be to use heavies from all possible fractions of the sediments, but this is too time consuming. The author thus suggests the examination of two fractions only. The same actual grain-size fraction was used for one, since it eliminated errors due to abrasion and irregular size distribution in the parent rock. The same grain-size relative to the size distribution curve was used for the other fraction since it eliminated errors due to sorting.

A paper by the Dutch investigator, Doeglas<sup>9</sup> gives us a somewhat different method of approach. He believes that errors due to sorting are small, and that therefore it is necessary to examine only one comparable fraction of heavy minerals from each sample. This avoids errors in mineral percentages. He also favors line counting over counting of fields since the first method eliminates personal differences and exaggerates the larger grains more in accordance with their true percentage by volume or weight. The non-opaque grains, insoluble in strong acids, since they are more diagnostic and are easily separated and identified from all types of sediments, are used for comparisons. The opaque grains are lumped together and their percentage determined. The author considers the counting of only 100 non-opaque grains sufficiently accurate for stratigraphic correlations. He favors a less detailed examination of many samples as the best procedure in tracing out petrographic provinces. Variations of percentage of a mineral species in different samples within an area or stratigraphic section are generally due to the presence or mixing of detritus of different origin, and not due to sorting differences, according to this author's belief.

Curves for the determination of the probable error, due to counting of minerals of varying abundance for any given number of grains counted, have been given by Dryden<sup>11</sup>, and extended to include smaller percentages by Rittenhouse<sup>51</sup>. Such curves are very useful in showing approximately how many grains must be counted to produce a desired degree of accuracy. As has been pointed out by Krumbein and Pettijohn<sup>34</sup>, however, it is impossible to calculate the true probable error from experimentally determined frequencies. Other errors enter into a heavy mineral determination and must be included to obtain the true error. Such errors are those due to non-random sampling during the separation procedure, and those due to incorrect identification of mineral grains.

Among the German workers, Tröger<sup>60</sup> has published a very important paper concerning the usefulness of the elutriator in quantitative heavy mineral determinations of sediments. Heavy minerals separated from sieved samples and from elutriated samples of three grain-size sandstones were compared. It was concluded that the

percentage of a mineral calculated from elutriated samples is, in many cases, more accurate than that calculated from sieved samples. Five elutriator fractions with narrow size limits were separated, and the zircon and tourmaline were isolated quantitatively according to weight from each fraction. From these determinations, Tröger constructed curves showing the percentage, by weight and by number of grains, of tourmaline and zircon. Zircon/tourmaline ratios were then calculated for the various size fractions of the sandstone samples separated by elutriator and sieve methods. These ratios were compared with the ideal ratios obtained by making the corrections necessary so as to have equal-volume grains. The zircon/tourmaline ratios by elutriation were much too low in the coarse fractions but approached the ideal in the 0.1-0.05 mm. fractions. The sieved fractions were shown to have ratios much too high in most cases, but the ratios came fairly near to the ideal in the 0.1-0.05 mm. fractions.

The difference between the ideal zircon/tourmaline ratio and that obtained from the sieved or elutriated sample, Tröger called the "distortion factor". He points out that many results in the literature have no scientific value since this factor is not considered. The "distortion factor" was shown to be quite similar for the three different grain-size sandstones when comparing the same size fractions. Thus, one is fairly safe in comparing similar fractions. It is necessary, however, in quantitative work to determine which technique, the sieving or the elutriation process, will result in smaller distortion of the mineral ratios from the ideal for any particular sample. Tröger believes the "distortion factor" can be used throughout any geologic formation which has been derived from the same source area.

Only a few important heavy mineral studies have been made on soils. Dorothy Carroll<sup>18</sup>, from a mineralogical study of the soils of Western Australia, concluded that: (1) The character of the heavy residue of a soil often gives a clear indication of the nature of the parent rock. (2) The diagnostic value is enhanced by the total amount of heavy minerals, by distinctive features of a mineral species, and by the amount of magnetic material. (3) A study of the soil heavy minerals can be very useful in geologic mapping.

The process of soil development under lateritic conditions has been investigated by several workers. Hardy and Rodrigues<sup>19, 20</sup> have studied lateritic soils from the British West Indies and from our southern states. They examined profiles chemically, physically, and mineralogically and found rather uniform compositions throughout the profiles and in the different size fractions. The chief contribution of their studies is evidence that laterization is a primary process, occurring within the inch or two of weathered crust of the



parent rock. The succeeding process of soil development is apparently a podsollic process, even under tropical conditions, as is indicated by an increase in quartz at the surface.

Harrison<sup>22</sup> has written an extensive treatise on the chemical, microscopical, and petrological aspects of the katamorphism of igneous rocks under the humid tropical conditions of British Guiana. He concluded that primary laterization is due to the action of carbonated ground water resulting in the liberation of silica, calcium, magnesium, sodium, and potassium while leaving most of the aluminum as gibbsite and most of the iron as limonite. This primary process is the same under tropical conditions at both high and low altitudes, but the succeeding processes differ materially. At low altitudes and with igneous rocks low in quartz, a portion of the silica set free crystallizes as quartz. At high altitudes the same rock appears to lose quartz. The primary laterite may later have most of the silica leached out resulting in surficial ironstones and bauxites. Acid igneous rocks under humid tropical conditions katamorphose differently and do not produce primary laterite. They produce pipe- or pot-clays, or more or less quartziferous, impure kaolin. Basic igneous rocks under humid temperate conditions katamorphose to produce more or less complex hydrated silicates.

Several British workers have recognized the value of a mineralogical study of soils as an aid to soil classification. Hendrick and Newlands,<sup>23, 24</sup> and later Hart<sup>21</sup>, made studies of many glacial soils of England and Scotland. They separated the minerals from the fine sand fractions by means of heavy liquids into the orthoclase group, the quartz group, and the heavy minerals group. Microscopic studies were found to be useful as an indication of the nature of the parent rock and of the reserve plant nutrients existing in the form of silicates. Local differences in silicate content of the soils reflected local differences in the parent rock from which the preponderance of the till is derived.

One of the first mineralogical studies of soils in the U. S. A. was carried on by McCaughey and Fry<sup>45</sup>. They outlined in detail the methods used by the geologist in mineral studies of consolidated sediments, and showed how these methods can be applied to soil studies. The sand and silt fractions of many soil types from all parts of the United States were studied by qualitative mineralogical methods. The authors found that epidote and hornblende are common in practically all soils. The alkali feldspars were found to be among the most abundant soil forming minerals. Microcline and the micas were shown to be very persistent minerals displaying little alterations. The authors conclude, from their rather exhaustive study, that any mineral commonly occurring in rocks may be expected in soils, no matter what the origin of the particular soil may be. Their data and conclusions suggest the possibility of the

contamination of surface soils over most parts of the United States by windblown materials.

Fry<sup>13</sup> further discussed the application of petrographic methods to soils laboratories in a more recent paper. The progress of such investigations toward quantitative methods is suggested by the outlining of a simple procedure for making such determinations from carefully sized fractions.

Jeffries and White<sup>28, 29, 30, 31</sup> are among the first in this country to study soil profiles in detail from a mineralogical point of view. Their series of important papers deals with soils derived from various limestones, dolomites, and shales of our eastern states. The very fine sand fractions were separated quantitatively into the heavy group, the quartz group, and the feldspar group by means of heavy liquids. Microscopic studies and counts were made on the heavy fractions. The authors found all soils examined quite similar in their qualitative minerals content, differing only in the proportion and total quantities of various minerals. They, therefore, believe that in comparing soils it is best to choose only the more outstanding heavy minerals and to correlate on relative percentages. The total quantity is also thought to be useful in correlation. From a comparison of the minerals of the very fine sand fraction of soils with those of the parent rock, the authors found that the mineralogical characteristics of the parent rock are reflected in the soil. All soils derived from limestone and dolomite were found to have a high feldspar content which is thought to account partially for their outstanding agricultural properties. The authors were especially interested in studying the weathering of these feldspar minerals.

Thin section mineral studies have been applied to soils for the interpretation of their genesis and morphology by Allen<sup>1</sup>. The soil profile was sampled in a columnar unit, and oriented thin sections were made at the horizon desired without destroying the soil structure. The materials were impregnated with kolloid and then ground to the desired thickness. Microscopic examination of a gumbo till profile showed definite weathering of feldspar, hornblende, and epidote to produce beidellite. It was further strongly indicated that under good drainage conditions the clay produced from weathering in the upper horizons is translocated and deposited in lower horizons.

Lutz<sup>38, 39</sup> showed the value of heavy mineral studies in investigating disturbances of the normal profile caused by the uprooting of trees.

Cady<sup>4</sup> has compared the heavy minerals of the A and C horizons of true podsoils with those of brown podsolitic forest soils. Podsolization was found to cause significant reduction in the hornblende, whilst epidote, garnet and magnetite were little affected.

Buckhannan and Ham<sup>2</sup> have used heavy mineral studies to decide questions of provenance affecting the classification of certain Oklahoma soils.

Nikiforoff and Drosdoff<sup>48</sup> have investigated claypan formation in the Dayton silt loam with particular emphasis on origin of the claypan. Mechanical analysis and chemical analysis were made on the profile and soil development was calculated on the assumption that silica is the most stable constituent and does not migrate appreciably. This assumption is probably erroneous, since clay contains a high percentage of silica and most soils workers believe that it migrates downward in the profile. From their study, the authors conclude that most of the clay in the B horizon of the Dayton silt loam was formed in place by decomposition of silt and that clay migration was unimportant. They also believe that the highly leached A<sub>2</sub> horizon has resulted from degradation of material similar to that in the B horizon, which means that large amounts of clay were decomposed with the release of silica. Clay is generally thought to be very resistant to weathering and its decomposition is unlikely under these conditions. The authors have undertaken the problem of soil development with highly dubious assumptions and consequently their work is of doubtful quantitative value.

Michelson<sup>46</sup> has continued in Ohio work begun in Missouri on three soils derived from glacial deposits. These were soils of the Miami, Bethel, and Brookston series formed from highly calcareous drift of late Wisconsin age. The methods used were essentially similar to those here presented, the ultimate aim being a quantitative measure of soil-forming processes. Much valuable information was obtained as to rates of weathering of particular minerals. Apatite was found to persist under neutral or slightly alkaline conditions in the Brookston soil whereas it had disappeared from the acid Miami and Bethel soils. The plagioclase feldspars, pyroxenes, amphiboles and pyrophyllite, were also found to have weathered much more extensively from the Miami and Bethel soils than in the Brookston. The presence of large amounts of limestone in the parent materials of these soils made the interpretation of quantitative results much more difficult than has been the case in our work with non-calcareous soils. Although not apparent at the time, it would now seem that the profiles used by Mickelson were not taken to a sufficient depth for reliable calculation of gains and losses based on constant zircon.

Marshall<sup>41</sup> has outlined a petrographic method for use in studies of soil formation processes which is based on the assumption that certain resistant minerals remain constant during soil development. It was this method of approach that has been used throughout the profile studies described below. This method will be discussed elsewhere.

## PART II.—SEMI-QUANTITATIVE HEAVY MINERAL STUDIES LEADING TO THE IDENTIFICATION OF PARENT MATERIALS

### Method of Investigation

Heavy minerals can be used to determine the origin of a soil without highly precise work. Qualitative and approximately quantitative methods are generally all that is required. In making such a study it is necessary first to become familiar with the various heavy mineral assemblages to be expected in the soils of the area. This can be done by examining several samples of each of the various parent rocks and rock materials which weather to form the soils. The sedimentary rock samples should be crushed and treated with acid, if necessary, in order to separate them into free, mineral particles. These methods have been well outlined by Milner<sup>47</sup>. When dealing with igneous and metamorphic rocks it may also be advisable to make thin section examination such as was done by Humbert<sup>26, 27</sup>. Unless it is desired to make weathering studies, however, one can use samples of the highly weathered rock as parent material, which saves time and should be perfectly satisfactory if care is used to obtain uncontaminated samples.

All samples were thoroughly cleaned by boiling with strong HCl in order to remove coatings from heavy mineral grains and to give a cleaner heavy minerals separation. After the acid was washed out, the silt and very fine sand fractions were isolated. Quantitative heavy mineral separations could then be easily made by means of a heavy liquid and the centrifuge. Tetrabromethane of about 2.9 specific gravity is satisfactory in most cases. The density of the liquid should be the same for all samples to be compared.

The heavy mineral residue was next examined under the microscope and all possible species were identified and listed according to their relative abundance on a rough scale. Counting is not always necessary with a practised observer who is thoroughly familiar with the mineral assemblage. The writers found it convenient to list both identifiable, and coated and opaque minerals as very abundant, abundant, very common, common, fairly common, uncommon, rather rare, rare, and very rare. These terms were assigned numbers from 1 to 9 according to decreasing abundance. Some may question the advisability of using such a broad scheme of classification, but quite uniform results were found to be obtainable with practice.

After examining the heavy minerals from the typical parent material or parent rocks of the area, and becoming familiar with differences to be expected both within the same material and between different materials, one can proceed to studies of comparable fractions from soils. Either the very fine sand or the coarse silt fraction can be used for heavy mineral comparisons. One fraction is usually enough to establish a correlation and this saves consider-



able time when many samples are to be examined. The heavy minerals in the soil are listed according to their abundance as was done with the parent materials. Block diagrams plotting the frequency of the most characteristic and abundant minerals are very useful in making correlations.

Studies of this nature were made upon about seventy-five different samples of soil and parent material taken mostly from Missouri. The majority of the soils were thought to be of loessial origin, but some samples were also included which were thought to be of glacial till and sedimentary rock origin.

TABLE I. — NAME AND LOCATION OF SAMPLES

Name of sample	Location
Unweathered loess*	Four separate samples taken close to the Missouri river bluff at Sioux City, Iowa, Oregon, McBaine and Cape Girardeau, Missouri.
Glacial drift (Kansan or Nebraskan)	Five miles east of McBaine, Mo. in a river bluff underlying loess.
LaMotte weathered sandstone	Road cut 4 miles west of Fredericktown, Mo.
Grundy Silt loam profile	1 1/2 miles west of Midway, Mo. on Missouri horticulture farm.
Marshall silt loam	In river bend upland west of Marshall, Mo.
Putnam silt loam profile	Tucker's pasture south of Moberly, Mo.
Marion silt loam	Road junction 3 miles north of McBaine, Mo.
Mississippi loess soil*	Central Mississippi.
Missouri river sand	River edge at Booneville Mo.
Dust of June 1938	Dust collector, McBaine, Mo.
Tilsit silt loam	Road cut 4 miles west of Fredericktown, Mo.
Oswego silty clay loam	South of Clinton, Mo. overlying shale.
Boone fine sandy loam	North of Clinton, Mo. overlying sandstone.
Bates silt loam	Near Deepwater, Mo. overlying sandstone and shale.

\* Samples furnished by H. B. Vanderford

### Results.

Tables I and II list some of the most interesting samples together with information concerning location, depth of sampling, particle size studied, and the per cent heavy minerals in the fraction examined. The heavy minerals analysis of these samples is summarized in Table IV where the original numbers used to express frequency have been transferred into percentages. These percentages were obtained by counting at least 1,000 particles in the 0.05-0.02 mm. fractions of the Tilsit, Lamotte sandstone, and Marion

samples. Percentages were then calculated from the counts. Number estimates were made on these same samples and the numbers

TABLE II. — DATA ON SAMPLES USED FOR HEAVY MINERALS STUDY

Sample number	Name	Depth	Fraction size in mm.	% heavies in fraction
1 to 4	Loess	below profile	0.1--0.02	1.5
5	Glacial drift	10 feet	0.1--0.02	1.5
6	LaMotte	44 inches	0.1--0.02	1.0
7	Grundy	58-62 in.	0.125--0.046	3.27
8	Grundy	58-62 in.	0.046--0.025	2.0
9	Grundy	58-62 in.	0.025--0.011	1.6
10	Grundy	12--16 in.	0.046--0.025	1.5
11	Grundy	108--114 in.	0.046--0.025	1.3
12	Marshall	10 inches	0.1--0.02	1.5
13	Putnam	18-28 in.	0.05--0.02	1.0
14	Putnam	48--60 in.	0.05--0.02	1.0
15	Putnam	7--8 ft.	0.05--0.02	1.0
16	Putnam	12--13 ft.	0.05--0.02	1.0
17	Marion	15--19 in.	0.05--0.02	1.5
18	Miss. loess soil	B. horizon	0.05--0.01	2.0
19	River sand	---	0.1--0.02	1.5
20	Dust	---	0.05--0.02	1.5
21	Tilsit	0--8 in.	0.1--0.02	1.4
22	Oswego	10 in.	0.05--0.01	low
23	Boone	10 in.	0.1--0.02	1.5
24	Bates	10 in.	0.1--0.02	1.0

were transferred into a percentage range using Table III. The value from the counts in all cases fell within the percentage range covered by the number estimate. The count percentages showed that uniformity was obtained in the number estimates. In tabulating the results of the number estimates, a percentage figure was assigned to each number which was approximately equal to the average of the range covered by that number. These are the percentages given in Table IV.

TABLE III. — CONVERSION OF NUMBER ESTIMATES TO PERCENTAGE

Descriptive term	Frequency number	Per cent range covered	Per cent plotted
Very abundant	1	> 35	40*
Abundant	2	20 - 35	30
Very common	3	10 - 20	15
Common	4	5 - 10	7
Fairly common	5	3 - 5	4
Uncommon	6	2 - 4	3
Rather rare	7	1 - 3	2
Rare	8	1 - 2	1
Very rare	9	0 - 1	1

\* or over, according to rough estimate or actual count.

In all samples where counts were made, the count percentage has been used in the data rather than the number estimate percentage because of its higher accuracy. The error of the number estimate percentages is indicated by the range covered by this

number as is shown in Table III. Errors greater than this range are possible but are not likely in view of the agreement with the counts.

TABLE IV. — PERCENTAGES OF MINERAL SPECIES IN THE HEAVY MINERALS FRACTION.

Sample	Zircon (Zr)	Tourmaline (Tn)	Epidote (Ep)	Hornblende (H)	Basaltic hornblende (BH)	Tremolite (Tr)	Colorless garnet (CG)	Browngarnet (BG)	Pink garnet (PG)	Anatase (An)	Rutile (Ru)
1	3	2	30	40	2	4	7	1		1	1
2	7	4	30	30	3	3	7	1		1	1
3	7	4	30	30	4	3	7	1		2	2
4	7	7	30	30	7	7	3	1		4	3
5	2	7	30	30	1	4	4		1	2	1
6	20	5	tr.					tr.		40	2
7	9	4	15	30	2	2	7	2		2	7
8	7	4	30	25	1	3	7	1		4	15
9	4	8	40	3	1	3	3	1		4	30
10	7	7	30	15	2	4	4	1		7	15
11	7	15	30	7	2	4		1		4	15
12	10	6	30	20	4	7	4	1		4	7
13	7	7	30	20	4	7	2	1		7	15
14	7	4	30	15	4	4	3	1		2	7
15	15	4	30	4	3	4	1			3	4
16	30	15	4	1		3				3	4
17	7	4	30	15	2	5	2	1		5	5
18	10	4	30	7	1	7	4	1		7	4
19	10	10	15	15	4	4	7	1		2	3
20	4	15	15	7	7	4				1	4
21	21	7	10	1	tr.	1				24	2
22	7	7	40	15	4	4	2			2	15
23	15	15	15	15	4	7	1	1		2	10
24	30	7	15	1	1	4	3	3		7	15

In most samples opaque and coated grains made up 20% or more of the heavies. They did not appear useful in correlation because of difficulty in identification and have therefore not been listed in the analysis. There are also a few grains of other minerals which have not been included.

### Discussion.

Figures 1 to 4 show the variations to be expected in heavy minerals content of loess samples taken between Sioux City, Iowa, and Cape Girardeau, Missouri. These figures show the remarkable similarity of loess over large distances. Most of the differences indicated are within the experimental error.

Figures 1 to 6, 19 and 20 indicate the striking difference in mineral assemblages and in mineral frequencies that can be expected in soils derived from sandstone or shale, as compared with those from loess or glacial till. It should be noted that Missouri river

Per Cent of the Indicated Mineral in the Heavy Mineral Fraction

Fig. 1 -- Sioux City Loess,  
0.1--0.02 mm. Fraction

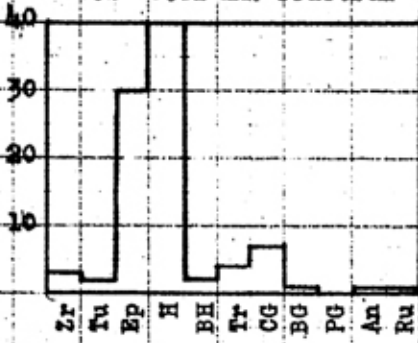


Fig. 2 -- Oregon Loess,  
0.1--0.02 mm. Fraction

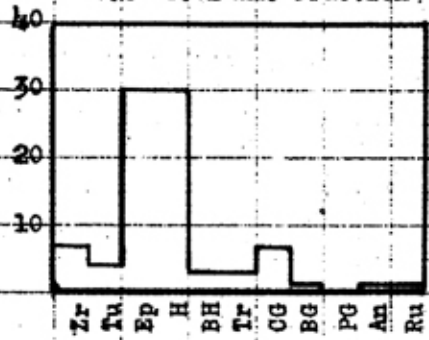


Fig. 3 -- McBain Loess,  
0.1--0.02 mm. Fraction

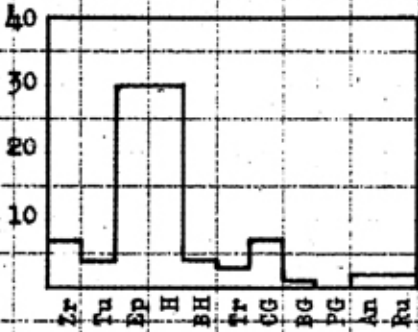


Fig. 4 -- Cape Girardeau  
Loess,  
0.1--0.02 mm. Fraction

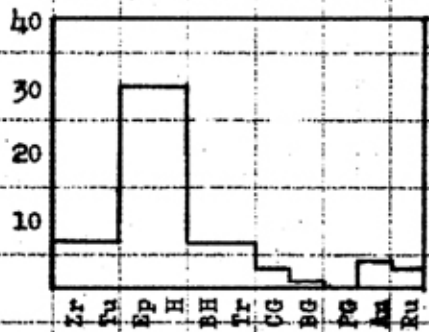


Fig. 5 -- Glacial Drift,  
0.1--0.02 mm. Fraction

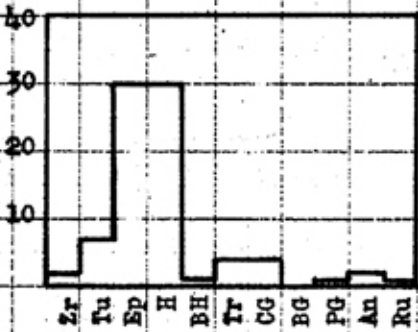


Fig. 6 -- LaMotte Weathered  
Sandstone,  
0.1--0.02 mm. Fraction

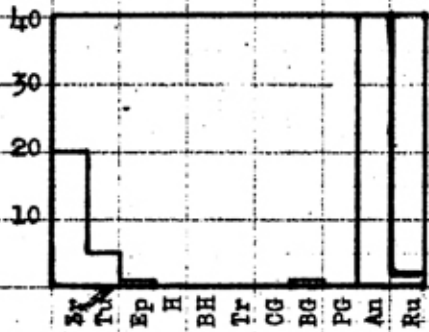




Fig. 7 -- Grundy Silt Loam,  
58--62 Inch Horizon,  
0.125--0.046 mm. Fraction

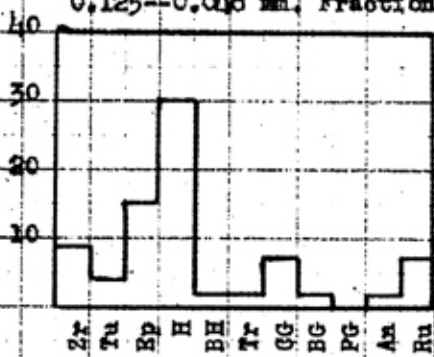


Fig. 8 -- Grundy Silt Loam,  
58--62 Inch Horizon,  
0.046--0.025 mm. Fraction

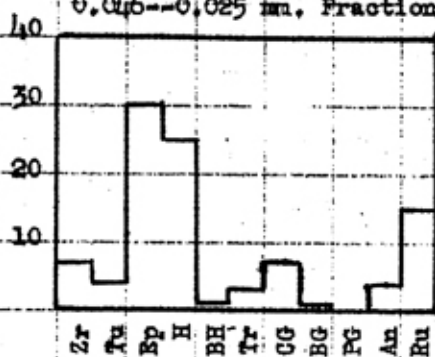


Fig. 9 -- Grundy Silt Loam,  
58--62 Inch Horizon,  
0.025--0.011 mm. Fraction

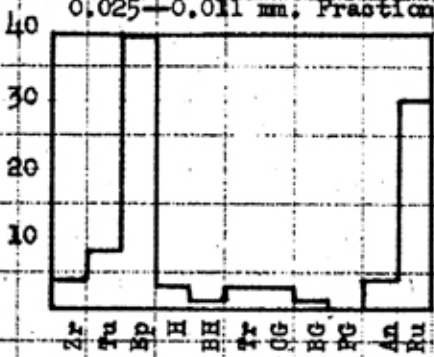


Fig. 10 -- Grundy Silt Loam,  
12--16 Inch Horizon,  
0.046--0.025 mm. Fraction

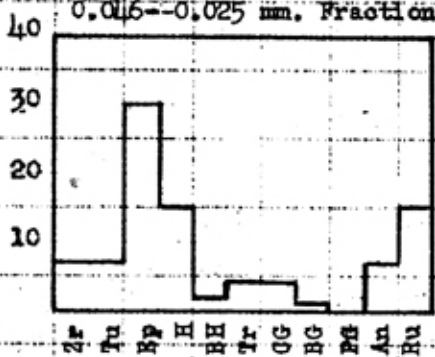


Fig. 11 -- Grundy Silt Loam,  
108--114 Inch Horizon,  
0.046--0.025 mm. Fraction

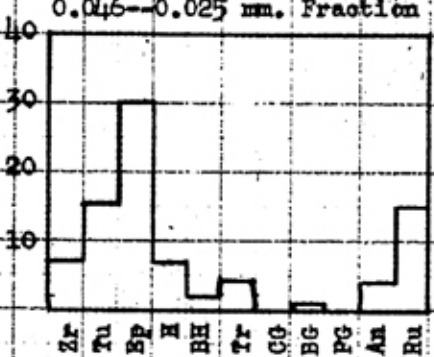
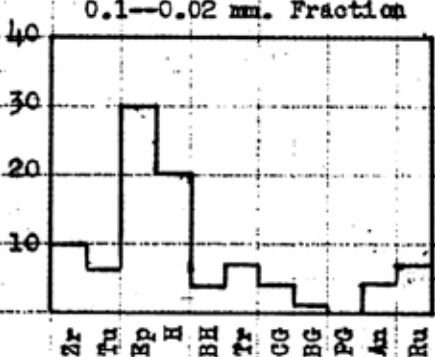


Fig. 12 -- Marshall Silt Loam,  
0.1--0.02 mm. Fraction



Per Cent of the Indicated Mineral in the Heavy Mineral Fraction

Per. Cent of the Indicated Mineral in the Heavy Mineral Fraction

Fig. 13 -- Putnam Silt Loam,  
18--28 Inch Horizon  
0.05--0.02 mm. Fraction

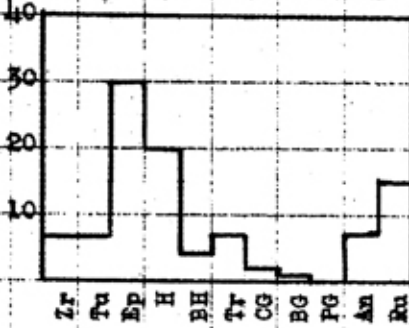


Fig. 14 -- Putnam Silt Loam,  
48--60 Inch Horizon,  
0.05--0.02 mm. Fraction

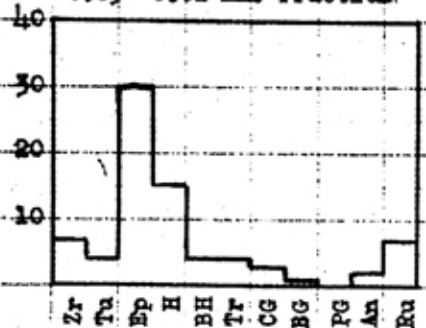


Fig. 15 -- Putnam Silt Loam,  
7--8 Foot Horizon,  
0.05--0.02 mm. Fraction

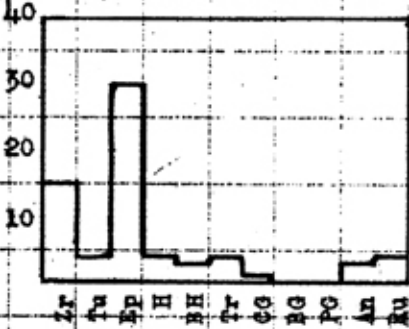


Fig. 16 -- Putnam Silt Loam,  
12--13 Foot Horizon,  
0.05--0.02 mm. Fraction

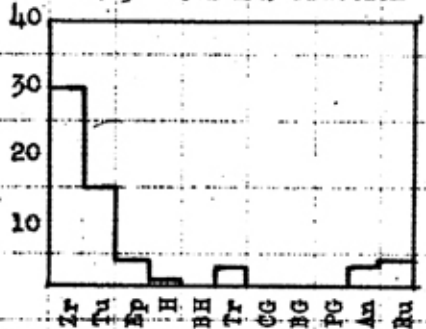


Fig. 17 -- Marion Silt Loam,  
0.05--0.02 mm. Fraction

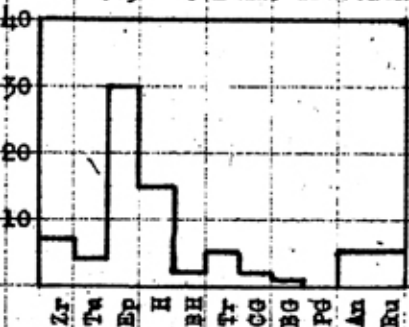
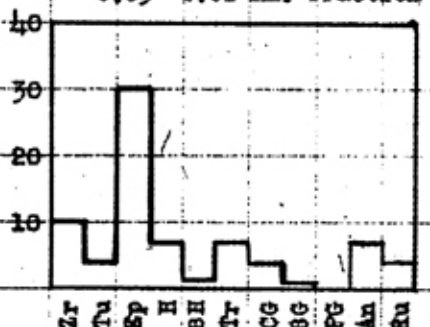


Fig. 18 -- Mississippi Loess,  
Soil,  
0.05--0.01 mm. Fraction



Per Cent of the Indicated Mineral in the Heavy Mineral Fraction

Fig. 19 -- Missouri River Sand, 0.1--0.02 mm. Fraction

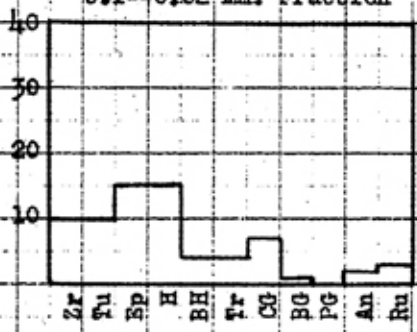


Fig. 20 -- Dust of June 1938, 0.05--0.02 mm. Fraction

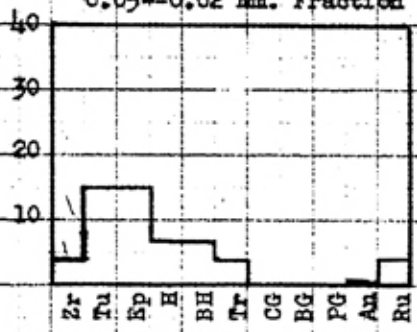


Fig. 21 -- Tilsit Silt Loam, 0.1--0.02 mm. Fraction

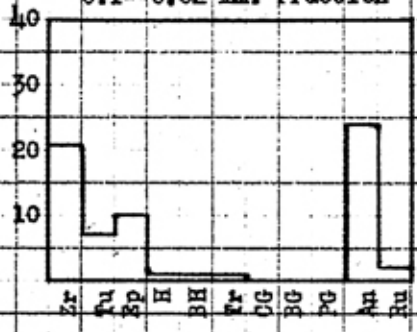


Fig. 22 -- Oswego Silty Clay Loam, 0.05--0.01 mm. Fraction

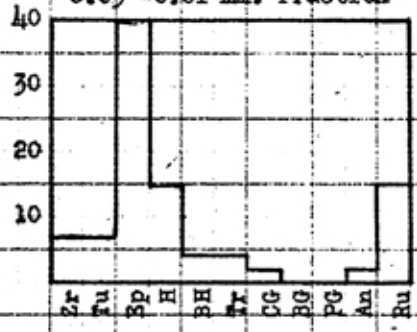


Fig. 23 -- Boone Fine Sandy Loam, 0.1--0.02 mm. Fraction

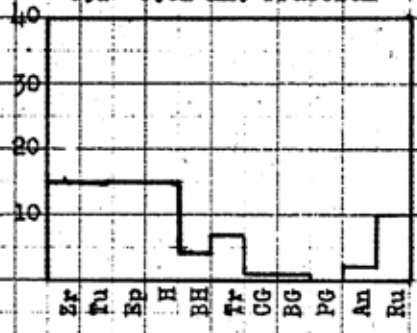
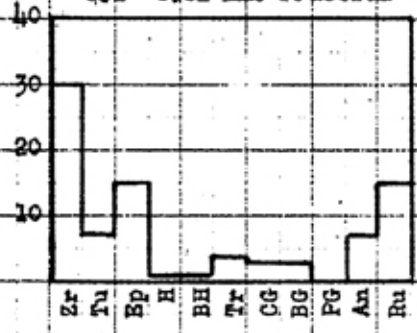


Fig. 24 -- Bates Silt Loam, 0.1--0.02 mm. Fraction



sand and recent dust both contain the heavy mineral components characteristic of loess or glacial till.

The differences to be expected in the heavy minerals of different size fractions of a soil are shown by comparing the fractions 0.125-0.046, 0.046-0.025, and 0.025-0.011 mm. of the Grundy silt loam (Figures 7, 8, and 9). There is no difference in the kinds of minerals present, but the increase in the percentages of epidote and rutile with a decrease in particle size is quite apparent. Likewise there is a sharp decrease in the percentage of hornblende with a decrease in particle size. These differences emphasize the importance of comparing the same size fractions of soils.

The most striking point brought out by the studies is the remarkable similarity in heavy mineral assemblage shown by most of the surface soils. The assemblage is characteristic of either loess or glacial till and strongly indicates that the soils contain appreciable quantities of this material.

Of the soils examined, those thought to have a possible loessial origin were the Marshall, Marion, Grundy, and Putnam. They are shown by heavy mineral analysis to be similar to fresh loess in both kinds and ratios of resistant heavy minerals. Slight differences in ratios of heavy minerals could be due either to weathering or to the differences in deposition which would be expected with increasing distance from the source of the loess. In surface soils of mixed origin, the nearer the soil to the source of the loess, the more it is like the loess as regards heavy mineral ratios.

The Tilsit, Oswego, Boone, and Bates soils all occur south of the glaciated area in Missouri and they were thought to have been derived from the local sedimentary rocks which lie underneath. The heavy minerals studies (see Figures 21, 22, 23, and 24) indicate that the upper 10 inches of these soils contain material added as loess. The evidence of loess contamination is strongest for the Tilsit soil, since a more detailed analysis is available than for the other soils. In this case the evidence is based on the fact that the minerals epidote, colorless garnet, hornblende, and basaltic hornblende either do not occur at all in the parent material or else are very rare here, whereas in the soil they are much more abundant. Furthermore, it is very unlikely that any of these minerals could have formed from the weathering of minerals present in the parent material, which is essentially quartz and microcline. In addition, the ratios of these "foreign" heavy minerals to one another is about the same as the ratios of these minerals in the 0.025-0.011 mm. fraction of loessial soil (see Figure 9). The mechanical analysis furnishes additional evidence of loess contamination in the Tilsit. From a detailed study of the Tilsit, loess contamination was calculated to be about 16% in the upper horizons. (See Part III, B.)

In the case of the Oswego, Boone, and Bates soils loess contamination can only be inferred but not proved, since samples of parent materials were not examined.

The A horizon of most Missouri soils has a very high silt content, regardless of the character of the underlying material. The heavy minerals studies suggest that loess can be held responsible for part of this silty texture. This conclusion is not unreasonable when one recalls that there are great thicknesses of loess within two hundred miles or less of any part of the state. A single dust storm of recent years deposited a perceptible layer of silt over most of the middle west and dust was observed as far east as the Atlantic coast.

Further interesting data are found by comparing the heavy minerals of a soil profile. Figures 13 to 16 show various horizons of the Putnam silt loam. The material appears to be quite uniform to a depth of 5 feet. The 7-8 foot layer shows some significant differences which are represented by an increase in zircon and a decrease in hornblende. The 12-13 foot zone is very distinctly different, as is shown by a big increase in zircon and tourmaline, and a marked decrease in epidote and hornblende. The 12-13 foot zone of this Putnam profile has apparently been derived mostly from the local bed rock of shale, sandstone, and possibly limestone. The 7-8 foot zone is undoubtedly glacial drift. It contains a heavy mineral assemblage suggestive of a mixture of igneous, metamorphic, and sedimentary rocks, and it is definitely different from the overlying material which is thought to be loess.

The Grundy silt loam profile (Figures 8, 10, and 11) is very similar to the Putnam, but the bedrock zone was not reached because samples were not taken deep enough. Down to a depth of 5½ feet the profile is uniform and is very similar to the typical loess. Below this depth, significant changes in heavy mineral ratios are observed. At a depth of 9 feet the proportion of tourmaline has increased markedly and the hornblende percentage has significantly decreased. The Grundy from about 5½ feet to 9½ feet is thought to be formed from glacial drift.

The Putnam and Grundy profiles are indicative of the deposit that might be expected to occur near the front of a stagnant continental glacier. A possible explanation of this condition is as follows. During the advancing ice stage, local bedrock is incorporated in the base of the ice mass. This debris becomes slowly mixed through the glacier as a result of differential movements in the ice. This slow mixing would cause the higher levels of the ice sheet to contain more debris of a foreign origin than do the lower layers. As the ice becomes stagnant and melts, this debris would be deposited in the same order. Thus the surface drift would contain the highest proportion of foreign material and the change downward to bedrock



would be gradual. As the ice sheet retreated farther north, a layer of outwash material may have been deposited on top of the till. The presence of outwash is indicated by the stratification of some glacial deposits of this region. As the ice retreated still farther north, loess was deposited on top of the glacial drift or outwash, as the case may be. The mineral content of the loess, the glacial outwash, and the uppermost glacial drift, therefore, might be almost identical.

It is interesting to compare the B horizon of the loess soil from Mississippi (Figure 18) with loess from Missouri (Figures 1 to 4). The heavy minerals assemblage is the same and the mineral ratios are, for the most part, very similar. The Mississippi soil, however, shows a much lower percentage of hornblende. This is readily explained when one considers that the Mississippi loess soil was subjected to the severe weathering of a moist southern climate. Hornblende is not generally considered to be a very resistant mineral and would tend to disappear under these conditions. Pronounced weathering and loss of hornblende were observed also in the B horizons of the Marion and Grundy. This is shown by comparing the percentages of hornblende in Figures 10 and 17 with those of the fresh loess shown in Figures 1 to 4. It appears therefore, that hornblende might be a very useful mineral in indicating the severity of weathering which occurs in various horizons of a soil, and under various climatic conditions.

### Conclusions.

The following conclusions seem justifiable from the data of the heavy minerals studies.

1. The origin of a soil is reflected in the kinds and amounts of heavy minerals present. A qualitative and roughly quantitative determination of the heavy minerals is usually sufficient to show what this origin is, after one has determined the kind and relative abundance of heavy minerals in the possible parent materials.

2. Loess is very uniform in heavy minerals composition over large areas.

3. In making heavy minerals comparisons between soils and parent materials one should use the same size fractions.

4. The Marshall, Marion, Grundy, and Putnam profiles examined are apparently formed from loess. The deeper horizons of the Grundy and Putnam profiles are probably of glacial drift origin. Below 12 feet, the Putnam profile seems to be formed mostly from local sedimentary rock.

5. All the Missouri soils examined contained a significant amount of loess in the A horizon.

6. Loess is responsible in part for the characteristic high silt content in the A horizon of many Missouri soils.

7. Differences in the origin of the parent material at different depths are readily shown by a heavy minerals analysis.

8. Profiles like the Putnam and Grundy can be explained by considering them to have been formed in loess underlain by debris deposited from a stagnant continental glacier near the margin of its advance.

9. Hornblende, because of its wide distribution and moderate resistance to weathering forces, is a good mineral to use in comparing the severity of weathering of different horizons of a soil and of soils formed under different climatic conditions.

### PART III.—THE TECHNIQUE OF QUANTITATIVE HEAVY MINERAL STUDIES

#### A. General Considerations.

The procedure outlined in Part II is adequate for the purpose of recognizing geological differences in soil materials. It may also furnish information of a preliminary and qualitative character on depositional variation and on the intensity of the weathering processes to which the surface layers have been subjected. A much more strictly quantitative method is needed, however, for the detailed study of soil formation processes. It should enable us to detect even small depositional variations, and in case these are absent, to provide a firm basis for the calculation of gains or losses through the precise determination of immobile constituents.

The results discussed in Part III are therefore illustrative of the difficulties arising in the quantitative extension of the heavy mineral technique. The two soils here considered were, as it turned out, not suitable for the quantitative measure of soil-forming processes. Each, however, presented problems of origin and of technique the solution of which greatly facilitated later work upon the Grundy silt loam profile described in Part IV.

When these investigations were begun no previous work had been done with the object of using resistant heavy minerals as a means of assessing changes in the soil. Mickelson's studies<sup>46</sup> proceeding contemporaneously with ours, have also served to illustrate the difficulties inherent in this approach.

From what follows it will be seen that two major difficulties are found in the use of quantitative microscopic methods. One arises through the presence of grains with highly resistant coatings which make accurate identification impossible. The second involves the assumptions and errors inherent in the translation of the numerical count of grains into a weight percentage of the soil. These difficulties are here considered in some detail.

The statistical errors involved in the counting of grains under the microscope are very easily evaluated. The conditions encountered fit those dealt with in the Poisson series, which gives the mean error as the square root of the number of grains of a particular species counted. Thus if in a series of counts, 100 grains of tourmaline were seen, the mean error would be  $\pm 10$  grains which is  $\pm 10$  per cent of the total tourmaline. This mean error corresponds to the standard deviation of the mean as discussed by R. A. Fisher and the probability table given by him can be used to determine by what factor this must be multiplied to give any specific probability. We find for 20 or more grains of a single species counted that the mean error multiplied approximately by 2 would bring us to the 5 per cent point, that is the odds are 19 to 1



that an error of twice the mean error would not be exceeded. Thus in the above example the odds are 19 to 1 that the tourmaline should lie between 80 and 120 grains.

## B. The Tilsit Silt Loam Profile.

### (1) Field and Sampling Data

*Location and Description of Profile.* The profile used for this study is exposed in a road cut about 4 miles west of Fredericktown, Missouri. The soil is supposedly derived from the LaMotte Sandstone of Upper Cambrian age which outcrops in the same road cut immediately underlying the profile. A brief description of the profile is as follows:

- 0- 8 in. Pale yellowish brown silt loam. Volume weight 1.30.
- 8-17 in. Light yellow silt loam. Volume weight 1.36.
- 17-28½ in. Highly mottled, yellow and brown silty clay. Volume weight 1.49.
- 28½-35 in. Yellowish brown, highly weathered sand containing sandstone fragments toward the base.

The underlying sandstone was found to be very poorly sorted and arkosic in nature. Different layers of the sandstone varied considerably in texture and in resistance to weathering.

*Method of Sampling.* A trench was dug several feet into the bank of the road cut in order to obtain undisturbed horizons. The profile was sampled in volume weight cans to a depth of 28½ inches. Below this depth it was impossible to obtain volume weight samples because of the presence of sandstone fragments. A can holding about 110 cc. was carefully driven into the sides of the trench at depths of 2, 4, 6, 10, 12, 14½, 18½, 22, and 26 inches. After the volume weights were determined in the laboratory, the three samples representing a given horizon were thoroughly mixed.

Representative samples of the horizon 28½-35 inches and of the weathered sandstone were taken in sacks. A fresh sandstone sample was taken in the road cut about 50 feet away from the profile proper. It represented a layer which could be traced at a depth of about 60 inches in the profile. Except for the fresh sandstone, all samples were taken in the same vertical profile. Sampling was done in October 1940 under favorable moisture conditions.

*Pretreatment of Samples.* The dry samples were disaggregated by crushing carefully in a mortar. The sandstone samples were poorly consolidated, but required a steel mortar before they could be easily disintegrated. By using some care it was fairly easy to break the aggregates apart without crushing the sand grains, and very little powdered material resulted.

The disintegrated samples were passed through a 2 mm. sieve

in order to get rid of any large undesirable pieces of gravel. Only a very small amount of gravel was found and it was discarded.

## (2) Mechanical Analysis.

A standard mechanical analysis was run on all samples in duplicate according to the combined sieve and pipet method outlined in the appendix. The average results are shown in Table V.

The silt fractions 0.05-0.02, 0.02-0.01, and 0.01-0.005 mm. equivalent diameter were then separated by stirring the well-dispersed sample in a beaker, allowing to stand the required period of time, and siphoning off the supernatant liquid.

TABLE V - MECHANICAL ANALYSIS OF THE TILSIT SILT LOAM PROFILE

Diameter in millimeters	Per cent of various sizes in horizons of the profile					
	0-8 in.	8-17 in.	17-28 1/2 in.	28 1/2-35 in.	Weathered sandstone	Fresh sand- stone
2--1	1.81	0.16	1.00	13.83	6.82	5.99
1--.6	3.87	0.61	2.95	14.07	12.94	9.15
.6--.2	3.96	2.86	3.21	11.90	10.48	32.95
.2--.1	3.87	3.14	3.22	10.21	11.58	14.48
.1--.06	5.92	4.32	4.98	12.72	26.63	27.39
.06--.02	28.25	30.02	23.89	16.52	21.44	4.64
.02--.01	19.82	22.90	18.48	6.33	3.31	1.18
.01--.005	8.72	8.83	11.64	3.56	1.90	0.56
.005--.002	5.60	10.46	5.14	2.22	1.58	0.66
<.002	14.10	19.49	25.96	8.81	3.91	2.60
Total	95.92	102.79	100.47	100.18	100.59	99.60

An examination of Table V immediately suggests that considerable differences in parent material must have been present from the outset. From the surface down to 28½ inches, however, the mechanical analyses show fair uniformity as regards the seven coarsest fractions from 2 mm. down to .01 mm. The 28½-35 inch layer, the weathered sandstone and the fresh sandstone differ very markedly from the layers above in their higher content of coarse material. At the same time they differ very significantly from each other in this respect. We may conclude therefore that geological or de-

positional differences may have existed between the parent materials of the different soil horizons.

### (3) Quantitative Separation of Heavy Minerals.

Heavy mineral separations were made on the fractions 0.2-0.1, 0.1-0.05, 0.05-0.02, 0.02-0.01, and 0.01-0.005 mm. to see which contained the major portion of the heavy minerals. Separations were made by means of a tetrabromoethane-nitrobenzene mixture having a specific gravity of  $2.85 \pm 0.01$ . A centrifuge tube of special design (see appendix) was used. By taking one- or two-gram samples and centrifuging at 1000 r. p. m. for 5-10 minutes, it was possible to obtain good quantitative separations on all the fractions used. In order to obtain a complete recovery of heavy grains it was necessary to shake and recentrifuge each sample four times. Practically all the heavy minerals were found in the fractions 0.1-0.05, 0.05-0.02, and 0.02-0.01 mm. and these were, therefore, selected for heavy mineral study.

TABLE VI. - HEAVY MINERAL DISTRIBUTION IN FRACTIONS OF THE TILSIT PROFILE.

Horizon depth in inches	Fraction .1-.05 mm.		Fraction .05-.02 mm.		Fraction .02-.01 mm.		Total % heavies in layer
	% H. M. in		% H. M. in		% H. M. in		
	Fraction soil		Fraction Soil		Fraction soil		
0-8	0.35	0.021	1.41	0.398	1.07	0.212	0.631
8-17	0.28	0.012	1.04	0.312	0.83	0.189	0.513
17-28 1/2	0.26	0.013	1.07	0.256	0.68	0.146	0.415
28 1/2-35	0.25	0.032	1.01	0.167	0.57	0.036	0.235
Weathered sandstone	0.19	0.051	0.98	0.210	0.35	0.012	0.273
Fresh sandstone	0.25	0.069	3.49	0.162	8.24	0.097	0.328

Microscopic examination of the heavy minerals showed a very poor quantitative separation. Many compound quartz-anatase and iron-coated quartz grains were found in the heavy fraction. There was also a large number of heavy mineral grains so completely coated that identification was impossible. Boiling with concentrated oxalic acid or strong hydrochloric acid, helped to eliminate this difficulty. It was then decided to treat all samples that were to be used for heavy mineral separations with 15% HCl and to heat them on the steam plate for one hour. Preliminary microscopic examination failed to reveal any identifiable heavy minerals which were dissolved by this treatment.

In order to have enough heavy residue for mineral counts and for chemical analysis, it was necessary to make separations on from 10 to 30 grams of each fraction. This meant that from 4 to 15 separations were made. From this number of separations, a very good

average was obtained. Most determinations checked within one tenth of one per cent. The average results of the heavy minerals separations are given in Table VI.

TABLE VII - HEAVY MINERAL COUNTS ON THE 0.1-0.05 MM. FRACTIONS OF THE TILSIT SILT LOAM PROFILE

Mineral	Number of Particles in various horizons					
	0-8 in.	8-17 in.	17-28 1/2 in.	28 1/2-35 in.	Weathered sandstone	Fresh sand- stone
Anatase and rutile *	258	273	316	1,019	252	330
Zircon	405	329	378	530	262	138
Tourmaline	150	208	212	232	162	135
Epidote	43	32	14	4	4	5
Amphiboles	16	9	6	3	0	0
Muscovite	0	0	0	0	2	3
Quartz **	0	0	0	0	136	222
Opaque	940	983	674	87	10	9
Coated unknowns	270	272	320	469	373	279
Totals	2,082	2,106	1,920	2,344	1,201	1,121

\* 98% anatase

\*\*compound quartz-anatase grains

#### (4) Microscopic Study of Fractions.

The heavy minerals from each fraction of several horizons were studied until complete familiarity with all possible identifiable grains was attained. The heavy minerals indicated that the profile was fairly uniform geologically. However, an increase in hornblende and epidote and the occurrence of some garnet in the upper horizons suggested the probability of some contamination here with wind-blown material. The heavy minerals in the order of their abundance were anatase, zircon, tourmaline, epidote, opaques (probably mostly ilmenite), and amphiboles. There were also very minor amounts of rutile and garnet. In spite of the acid treatment there was still a considerable number of coated unknown minerals as well as compound quartz-anatase grains, especially in the deeper horizons.

The anatase was practically all secondary in origin, as was in-

licated by the perfect rectangular platy crystals occurring in all samples, especially in the fresh sandstone. Weathering of anatase in the upper horizons was not great although some rounding, pitting,

TABLE VIII - HEAVY MINERAL COUNTS ON THE 0.05-0.02 MM. FRACTIONS OF THE TILSIT SILT LOAM PROFILE

Mineral	Number of Particles in Various Horizons				Weathered sandstone	Fresh sandstone
	0-8 in.	8-17 in.	17-28 1/2 in.	28 1/2-35 in.		
Anatase and rutile *	267	405	427	1,347	977	2,014
Zircon	172	151	158	392	291	191
Tourmaline	88	103	81	176	88	37
Epidote	97	119	56	23	18	15
Amphibole	9	5	13	19	0	1
Garnet	1	4	2	2	0	0
Muscovite	3	0	3	5	6	2
Biotite	2	0	0	0	0	0
Quartz **	10	6	16	131	146	79
Opaque	321	91	94	23	9	6
Coated unknowns	143	208	213	327	683	113
Totals	1,113	1,092	1,063	2,445	2,218	2,458

\*98% anatase

\*\*Mostly compound quartz-anatase below 28 1/2 inches.

and coating of grains was evident. Zircon and tourmaline in all samples were more or less rounded, especially the larger grains. This was probably due to abrasion during formation of the sandstone, although these grains could have occurred in this form in the original igneous rock. Zircon and tourmaline did not appear to undergo weathering in the profile. Many of the zircon crystals were coated, but this occurred in the fresh sandstone as well as in the soil. Coatings do not necessarily mean weathering of the mineral itself but may be deposits of silica, iron, or aluminum from percolating solutions.

Counts of from 500 to 2,000 grains were made on each fraction of every horizon in order to have results of statistical significance. The mineral grains were mounted in a medium of refractive index 1.70. The particles were scattered so as to give no more than 25 grains

per field. All grains which appeared completely within the field of vision were counted. The slide was moved from field to field by using cross-hatched marks on the microscope stage to prevent counting of portions of the same field more than once. The results of the counts are given in Tables VII, VIII, and IX.

TABLE IX - HEAVY MINERAL COUNTS ON THE 0.02-0.01 MM. FRACTIONS OF THE TILSIT SILT LOAM PROFILE

Mineral	Number of Particles in Various Horizons				Weathered Sandstone	Fresh sandstone
	0-8 in.	8-17 in.	17-28 1/2 in.	28 1/2-35 in.		
Anatase and Rutile *	309	302	300	376	425	398
Zircon	23	24	15	39	20	3
Tourmaline	50	20	36	9	12	0
Epidote	69	71	31	17	11	7
Amphibole	10	3	6	5	2	0
Muscovite	11	5	16	0	6	1
Quartz	19	14	15	18	43	11
Coated unknowns	85	120	103	55	69	23
Totals	576	559	522	519	589	443

\*98% anatase

A cursory examination of these tables immediately suggests that the epidote, garnet and amphiboles present in the 0-28½ inch layers could not possibly have come entirely from parent material similar to that of the lower layers, since the zircon and tourmaline, which are even more resistant chemically, do not show the same high relative abundance in the upper as compared with the lower layers. Some outside source would seem to have provided all the garnet and the bulk of the epidote and amphiboles. It will be seen also that the increase in epidote in the surface layers is especially marked in the finest fraction (.02-.01 mm.), suggesting that loessial material may have been responsible. The relative amounts of epidote, amphiboles (hornblende) and garnets are also such as would be found in surface soils weathered from loess. (See Figs. 10, 12, 13, 17.)

*Calculations From Counts.* The percentage by weight of each mineral was calculated from counts after assuming that the average sizes were the same. Each mineral was given an assumed specific



gravity which was obtained from the average of the specific gravity range found in Dana's Textbook on Mineralogy<sup>12</sup>. Those used were: anatase and rutile 3.9, zircon 4.7, tourmaline 3.1, epidote 3.3, amphibole 3.2, garnet, 3.7, muscovite 2.8, biotite 2.8, and quartz 2.66. All opaque

TABLE X. — AVERAGE SPECIFIC GRAVITY OF HEAVY MINERAL FRACTIONS CALCULATED FROM COUNTS AND DETERMINED WITH THE PYCNOMETER BOTTLE.

Horizon depth in inches	S. G. of Various Fractions			
	.1--.05 mm. from counts	.05--.02 mm.		.02--.01 mm. from counts
		From counts	With pyc. bottle	
0--8	4.41	3.88	3.75	3.69
8--17	4.37	3.84	3.76	3.76
17--28 1/2	4.32	3.90	3.71	3.75
28 1/2 - 35	4.09	3.86	3.74	3.88
Weathered sandstone	4.11	3.49	---	3.78
Fresh sandstone	4.09	3.87	3.77	3.86

and coated unknown grains were assumed to have a specific gravity which was equal to that of the average of the known grains. The error introduced by this assumption is small provided that their proportion is not too large, and would tend to be eliminated when the various samples are compared with one another.

The accuracy of the average specific gravity calculation for the heavy grains of the 0.05-0.02 mm. fractions was checked by determining their specific gravity with a pycnometer bottle. The procedure for this determination is outlined in appendix D. It was found that the

TABLE XI. — DEPOSITIONAL VARIATION AS SHOWN BY THE RELATIVE PROPORTIONS OF HIGHLY RESISTANT MINERALS (TOTALS FOR FRACTIONS 0.1 - 0.01 MM.)

Depth inches	Percentages by weight of soil				$\frac{Z}{T}$	$\frac{Z}{A+R}$
	Total H. M.	Zircon	Tourmaline	Anatase + Rutile		
0-8	0.631	0.090	0.042	0.219	2.1	.41
8-17	0.515	0.065	0.030	0.224	2.2	.29
17-28 1/2	0.415	0.054	0.025	0.192	2.2	.28
28 1/2 - 35	0.235	0.048	0.017	0.122	2.8	.39
Weathered sandstone	0.273	0.055	0.017	0.099	3.2	.56
Fresh sandstone	0.328	0.036	0.011	0.232	3.3	.16

calculated average gravity was consistently a little too high (see Table X). This is probably due to the fact that some of the assumed specific gravities are too high. It was very difficult to obtain good results with the pycnometer on small samples of such fine grained material because

of surface tension effects. This method was therefore abandoned in favor of the calculation from counts which gave consistent, though high, results.

TABLE XII. — DEPOSITIONAL VARIATION AS SHOWN BY THE VARIATION IN PARTICLE SIZE DISTRIBUTION OF ZIRCON, TOURMALINE AND ANATASE + RUTILE.

Depth inches	Mineral	Percentages of 0.1-0.01 mm. fraction		
		0.1-0.05 mm.	0.05-0.02 mm.	0.02-0.01 mm.
0-8	Zircon	5	83	12
8-17	"	3	81	16
17-28 1/2	"	5	85	10
28 1/2-35	"	17	76	7
Weathered sandstone	"	25	74	1
Fresh sandstone	"	31	67	2
0-8	Tourmaline	3	60	37
8-17	"	3	79	18
17-28 1/2	"	4	63	33
28 1/2-35	"	14	84	2
Weathered sandstone	"	33	63	4
Fresh sandstone	"	68	32	0
0-8	Anatase + Rutile	1	44	55
8-17	"	1	52	47
17-28 1/2	"	1	53	46
28 1/2-35	"	11	67	22
Weathered sandstone	"	11	79	10
Fresh sandstone	"	10	52	38

*Conversion of Counts to Weight Percentages.* Utilization of the microscopic counts and the specific gravities of species identified is likely to lead to serious error in calculated weight percentages in certain cases. Apart from the difficulty of dealing with unidentified grains there is also a possibility that the average volumes of the particles of different species in a given fraction may not be the same. This in itself would not vitiate the quantitative comparison of soil horizons unless the volume factor varied from one layer to another. This, however, is only probable when there are geological or depositional differences. A good illustration of this effect showed itself in the Tilsit profile, where grains of the .05-.02 mm. fraction were measured in two directions at right angles in addition to being counted. Sixty to eighty grains of each species were measured. For the 0-8 inch, 8-17 inch, and 17-28½ inch layers the ratios of the diameters of anatase, zircon and tourmaline grains to those of all the gains, varied little from 1.00, but in the lower layer values ranging from .76 to 1.73 occurred. The broader the limits of particle size in a given fraction the more likely will be such observations.

However, measurements in two dimensions do not enable one to compute with accuracy the volumes of the particles measured, so that where the size ratios vary appreciably from 1.00 no reliable weight percentage can be calculated. If, however, the ratio remains constant down the profile, the proportionate errors will be the same for all layers and calculations on weathering can still be made.



In Tables XI and XII, therefore no great accuracy can be claimed for percentages of individual minerals in the layers of soil below 28½ inches. Nevertheless, these tables illustrate clearly that depositional variations are reflected both in the weight ratios of different resistant minerals to one another and in the distribution of each mineral amongst the various size groups.

The two types of criteria for depositional variation are seen to show marked differences in parent materials where the mechanical analyses also indicated differences. It might perhaps be thought that the mechanical analysis alone would be adequate to decide these questions. In many cases, especially those where weathering has left a large fraction of the original soil skeleton intact, there is no doubt that mechanical analyses would clearly indicate variations in parent materials. But where more extensive weathering takes place, giving coarser fractions which are themselves secondary in origin, only these two types of heavy mineral criteria can be used. As to which will prove the better, insufficient experience is yet available for any decision, and several mineral ratios or distributions should be examined.

#### **(5) Chemical Determination of Zircon in Soil Fractions.**

Because of the large number of coated grains which were found in most of the fractions in the profile, it was decided to check the zircon determined by counts with a chemical method for estimating zircon. Since most of the zircon occurred in the 0.05-0.02 mm. fractions, this fraction was used for the determination.

A method for the determination of zirconium in the mineral zircon which could be used in the presence of all other elements found in the heavy fractions was outlined (Appendix D.) This procedure was then applied to some pure zircon crystals and to an artificial mixture of zircon and apatite. It was found possible to obtain quantitative results for zircon in the presence of 25% apatite according to the procedure. Because of the high percentage of zircon in the Tilsit samples and since a large amount of heavy minerals had been separated, it was not necessary to use the 5-place weighing technique described in the appendix. In this case 0.1 gram samples were used and the precipitates were weighed to the fourth decimal place only. Duplicate or triplicate determinations were made and results checked within one per cent.

We see from Table XIII that the zircon determined chemically is somewhat higher than that determined from the counts. This would suggest either that the zircon is not of the theoretical composition, that some of the assumed densities of the various heavy minerals are incorrect, that some of the coated unknown grains are in reality zircon, or that the shapes and sizes of the grains of different species

may change relatively especially in the lower layers. The first three results suggest that where there is good uniformity of parent material the zircon determined chemically is strictly proportional to that given by counts.

TABLE XIII. - ZIRCON IN 0.05-0.02 MM. HEAVY FRACTIONS OF THE TILSIT PROFILE DETERMINED FROM COUNTS AND BY CHEMICAL ANALYSIS

Horizon depth in inches	Zircon in per cent of Heavy Fraction	
	From counts	From chemical analysis
0-8	18.72	21.08
8-17	16.91	19.43
17-28 1/2	17.92	19.75
28 1/2 - 35	19.37	24.90
Weathered sandstone	15.67	25.26
Fresh sandstone	9.24	20.18

#### (6) Conclusions and Summary.

The results of the heavy minerals study on the Tilsit profile indicate that the upper part of this soil was developed in a mixed material of LaMotte sandstone and loess. The sharp increase in epidote and amphiboles, and the presence of small amounts of basaltic hornblende and garnet in the upper horizons of the profile, is strong evidence of loessial contamination. These minerals are all absent from, or are very rare, in the sandstone parent material, and it is very unlikely that they could have been formed from the decomposition of other minerals present in the sandstone. The amounts of these "foreign" minerals seem relatively small, and one might think that loessial contamination is not very great. However, if we assume that these "foreign" minerals have been added as loess, and that their relative abundance in the loess was the same as that in the Missouri loess samples previously examined, it is estimated that the contamination above 17 inches is about 16 per cent. The method used to calculate loess contamination was based on epidote and is as follows:

- 0.0591 gm. epidote of 0.10-0.01 mm. size in 100 gm. soil, average for 0-17 inch horizons.
- 0.0021 gm. epidote of 0.10-0.01 mm. size in 100 gm. weathered sandstone.
- 0.0570 gm. epidote of 0.10-0.01 mm. size *added as loess* to 0-17 inch horizon (difference).
- 0.3596 gm. epidote of 0.125-0.011 mm. size in 100 gm. of average loess.

$$\frac{0.0570}{0.3596} \times 100 = 15.9 \text{ per cent loess contamination.}$$

Amphiboles can be used in the same way to make this calculation, but they are more susceptible to weathering than epidote and would give low results.

Addition of loess in the general size range of 0.02-0.01 mm. is strongly indicated. This is shown by the fact that in the Tilsit profile, the biggest differences are in the 0.02-0.01 mm. fractions. Large increases in the percentage of this size occur in the upper horizons. Then, too, the largest percentage of the "foreign" heavy minerals occur in the 0.02-0.01 mm. fractions. And most important of all, the ratio of amphibole to epidote in the Tilsit is about the same as the ratio of these two minerals in the 0.025-0.011 mm. fraction of loess. The addition of loess of the average particle size indicated above would be reasonable to expect at this distance from the nearest source of loess.

The possibility that the increase in the 0.02-0.01 mm. size material in the surface horizons is due to the mechanical breakdown of coarser material is not in line with the evidence given above. Mechanical breakdown of sand grains in the profile is thought to be small.

It is also possible that the sandstone parent material of the upper horizons contained more of the 0.02-0.01 mm. size material than does the underlying sandstone. This is supported by the evidence of the heavy minerals and zircon distribution among the fractions, and it is probably true to a certain extent. However, the evidence from the "foreign" heavy minerals shows that a considerable portion of this 0.02-0.01 mm. size material must have come from loess.

From the data of the mechanical analysis, and from the distribution of total heavy minerals and of zircon among the various fractions of the profile, we have indisputable evidence of depositional variations within the profile. The mechanical analysis shows significant differences in particle size distribution between almost all the horizons of the profile. This is particularly evident in the coarser fractions. The distribution of total heavy minerals among the various fractions shows significant differences, especially between the deeper horizons of the profile. Weathering of the less resistant heavy minerals would cause some differences in particle size distribution, so the criterion of totals is not as useful as that offered by one very resistant mineral.

Because of its high resistance to weathering, zircon is especially good for showing differences in deposition between layers of the profile. Probably the criteria offered by zircon distribution among the fractions is the most sensitive measure we have of depositional variations. The distribution of zircon among different particle sizes in the profile shows distinct depositional differences. We must remember, however, that small differences in zircon distribution, as well as in total heavy minerals distribution, may be due to errors arising in the fractionation of the samples.

As final evidence of depositional or geological differences between layers in the profile, we have the ratio of zircon to tourmaline using the combined fractions. These ratios indicate uniformity down to a depth of 28½ inches. Below 28½ inches, the ratios are significantly different. By combining the data from the three size fractions in calculating these mineral ratios, errors arising in the separation of fractions are avoided. However, from the results it appears that the mineral ratios of the combined fractions are not as sensitive to small depositional differences as are the mechanical analysis and the zircon distribution.

When one considers the nature of the LaMotte sandstone, it is not surprising that depositional differences occur. Outcrops of the sandstone show thin interbedded layers of relatively coarse, poorly-sorted, arkosic sandstone and well-sorted finer textured sandstone. There are also thin beds of shale and siltstone in some places.

Apparently this Tilsit profile has been developed in a fairly uniform admixture of loessial material and sandstone down to a depth of about 17 inches. From 17 to 35 inches loess contamination decreases rapidly, but it is still apparent. Below 35 inches, there is no evidence of loess. Below 28½ inches the profile is formed from alternate beds of coarse and fine sandstone which differ markedly from the parent material that gave rise to the profile above it.

The way in which the loess became mixed with the other material in the profile is not clear. Possibly the mixing took place during the period of loess deposition. It does not seem likely that the loess could have been translocated downward by leaching alone to any appreciable extent, although some of the finest silt size particles may have been moved. Perhaps plant roots and insects in the soil are largely responsible for this mixing.

*Summary.* From the studies on the Tilsit silt loam profile, the following conclusions seem justifiable.

1. The profile shows both geological and depositional differences between the upper and the lower horizons.
2. The profile has been developed in mixed material of LaMotte sandstone and loess.
3. Loess makes up roughly 16 per cent of the total material above 17 inches depth and about 10 per cent of the material between 17 and 28½ inches. Traces of loess extend to a depth of somewhere between 28½ and 35 inches.
4. Down to a depth of about 28½ inches the profile is relatively uniform geologically and depositionally.
5. Below 28½ inches the layers differ markedly from those above both geologically and depositionally.
6. The layers below 28½ inches differ from each other depositionally and are apparently formed from thin beds of coarse and fine sandstone.

7. The way in which loess was mixed with the upper horizons of the profile is not known, but it could have occurred as a result of the action of plant roots or of worms and insects in the soil.

### C. The Marion Silt Loam Profile.

#### (1) Field and Sampling Data.

*Location and Description of Profile.* The profile samples were taken in a field about 100 yards from a road junction three miles north of McBaine, Missouri. The location was under blue grass pasture cover and had not been in cultivation for several years. The topography was gently undulating and the soil was typical upland prairie type. The profile is thought to have been developed from loess parent material as is indicated by its nearness to the Missouri river and by the occurrence of loess outcrops only a short distance away. A brief description of the profile is as follows:

- 0-10 in. Grey-brown silt loam containing numerous iron concretions. Becomes greyer toward base.
- 10-20 in. Grey silt loam containing iron concretions. Becomes somewhat heavier in texture towards base.
- 20-38 in. Grey and brown mottled heavy clay. Contains numerous iron concretions. Chert fragment found at 22 inches.

TABLE XIV. - AVERAGE VOLUME WEIGHT AND MOISTURE CONTENT OF HORIZONS OF THE MARION PROFILE.

Depth in inches	Horizon	Volume weight	Per cent moisture
0-10	A <sub>1</sub>	1.37	28.0
10-15	A <sub>2</sub>	1.48	22.6
15-20	A <sub>2</sub>	1.48	20.4
20-28	B <sub>1</sub>	1.36	31.0
28-34	B <sub>1</sub>	1.44	28.1
34-38	B <sub>2</sub>	1.48	27.0

*Method of Sampling.* A pit was dug about two feet wide and five feet long to a depth of 38 inches. Because of the presence of a chert fragment indicating possible non-loessial material, it was decided not to sample deeper. Volume weight samples were taken in duplicate or triplicate at the desired horizons using cans holding about 110 cubic centimeters. The cans were driven into the sides of the pit at carefully measured depths centered at 6, 12½, 17, 24, 31, and 37 inches. The samples were taken on March 13, 1941, and the soil was a little too wet for ideal sampling. Volume weights were determined in the laboratory a few hours after sampling, and the results are given in Table XIV.



## (2) Mechanical Analysis.

The samples were treated, in general, the same as those of the Tilsit profile. A mechanical analysis was run in duplicate, both with and without strong acid treatment, and the average results are given in Table XV. The fractions 0.13-0.06, 0.06-0.02, and 0.02-0.01 mm. equivalent diameter of the acid treated samples were separated by the beaker method, as was done in the Tilsit profile. It is interesting to note from the tables that the strong acid treatment had little effect on any parts of the soil except the iron concretions and the clay fraction.

TABLE XV. — MECHANICAL ANALYSIS OF THE MARION SILT LOAM PROFILE WITH AND WITHOUT STRONG ACID TREATMENT.

Particle diameter in mm.*	Standard mechanical analysis of various horizons expressed in per cent of the total soil					
	4-8 in.	10-14in.	15-19 in.	22-26in.	29-33in.	35-39 in.
Plus 1.0	1.59	1.84	1.03	0.05	0.04	0.25
1.0-0.6	1.71	2.14	1.56	0.30	0.32	0.46
0.6-0.2	1.47	1.47	1.20	0.38	0.36	0.55
0.2-0.1	0.62	0.69	0.71	0.32	0.29	0.33
0.1-0.06	2.50	2.14	2.89	1.25	1.57	2.01
0.06-0.02	44.29	42.03	38.87	23.24	25.06	28.13
0.02-0.01	22.78	22.66	22.80	14.93	16.77	17.74
0.01-0.005	8.37	9.48	9.06	8.34	7.88	9.16
0.005-0.002	5.52	6.23	7.10	5.11	6.84	6.07
Minus 0.002	13.98	13.93	17.61	43.79	41.59	35.72
Total	102.83	102.61	102.83	97.71	100.72	100.42
Standard mechanical analysis after 15% HCl treatment						
Plus 1.0	none		none			none
1.0-0.6	0.04		none			none
0.6-0.2	0.05		none			none
0.2-0.1	0.08		0.04			0.01
0.1-0.06	1.97		1.59			0.83
0.06-0.02	43.83		39.25			28.12
0.02-0.01	20.73		23.04			12.65
0.01-0.005	8.19		9.72			6.93
0.005-0.002	4.98		6.98			5.14
Minus 0.002	13.93		14.21			23.07
Total	93.80		94.83			76.75

\*Most of fractions coarser than 0.1 mm. are concretions.

The mechanical analysis would seem to indicate a reasonable degree of uniformity of parent material in this profile. The apparent break at 22 inches is due largely to the great increase in the clay fraction at this point. However, the figures for the coarser fractions, after removal of concretions by acid, show that the upper layers of the loess contain significantly higher proportions of particles in the range 1-0.02 mm. than do the lower. It would seem, therefore, that minor depositional variations are present.

### (3) Quantitative Separation of Heavy Minerals.

The heavy minerals separations were carried out in the special centrifuge tubes. The heavy liquid in this case, however, was adjusted to a specific gravity of 2.91 plus or minus 0.01. The commercial tetrabromoethane was found to have about this specific gravity and it was easy to adjust the specific gravity of the recovered liquid to this figure. Another advantage of this higher specific gravity was the complete elimination of the micas, a few grains of which were found to settle out with the heavy grains when separations were made at specific gravity of 2.85. To save time, only the 4-8, 15-19, and 35-39 inch horizons were used at first for the heavy mineral studies. It was feared that the profile might not be of uniform origin.

Difficulty was encountered in making heavy minerals separations on the fractions less than 0.02 mm. diameter, and recentrifuging of the light fraction was found to be necessary in order to make a complete separation. Sometimes only 50% of the total heavies were recovered in the first separation. Incomplete heavy mineral separations in the finer fractions may be explained in several ways. It may be due to (1) too small a difference in density between some of the minerals and the heavy liquid used, (2) air bubbles entrapped on the particles which prevent them from settling out, (3) the entrapping of heavy particles by lighter ones which carry them upward, (4) or electrical attraction of the particles for one another, resulting in coagulation of the light and heavy minerals together.

The density of the heavy liquid used was 2.91 and all of the heavy minerals have densities differing from this by at least 0.1 unit. This should be large enough for quantitative separations. At any rate, by extending the time and increasing the speed of centrifuging, all such grains should eventually come down. However, samples centrifuged for 30 minutes at 1800 r.p.m. failed to give any increase in heavy minerals.

If the cause were due to entrapped air, then evacuation of the heavy liquid mineral mixture should correct the difficulty. To see if this could be the trouble, several samples were run after thorough evacuation with a water suction pump. No measurable increase was found in the heavy minerals recovered, so this explanation was abandoned.

If the cause is the entrapment of heavy particles with lighter ones, then a better separation would be expected if the centrifuge were run at a higher speed, or if the samples were shaken and recentrifuged repeatedly, or if smaller samples were used. All of these possibilities were tried but with only very slight increases in heavy minerals recovered. Thus this explanation appears to apply only to a limited extent.

One must then fall back on the fourth possibility, that of electrical

attraction. This is supported by the observation that in all cases where poor recovery of heavy minerals occur, coagulation of the particles in the heavy liquid takes place within a few seconds after shaking. Usually this coagulation becomes more marked when shak-

TABLE XVI. — DISTRIBUTION OF HEAVY MINERALS IN THE FRACTIONS OF THE MARION SILT LOAM PROFILE SEPARATED BY BEAKER METHOD.

Horizon depth in inches	Per cent heavies of various equivalent diameters						Total per cent heavies in soil
	0.1-0.05 mm.		0.05-0.02 mm.		0.02-0.01 mm.		
	% in frac-tion	% in soil	% in frac-tion	% in soil	% in frac-tion	% in soil	
4-8	0.44	.0087	1.57	.688	0.97	.200	0.897
15-19	0.42	.0069	1.30	.510	0.51	.117	0.634
35-39	0.54	.0045	1.45	.395	0.44	.055	0.455

ing the tube the second time after the first centrifuging. When this coagulation occurs, it is impossible to obtain more than a small fraction of the total heavy minerals. This coagulation does not always take place. Using a small enough sample it should be prevented. However, in such cases the amount would become too small for accurate weighings, and it would take hundreds of separations to obtain enough heavy residue for chemical and microscopic studies.

The most practical method was to make a double separation of the heavy minerals. The light fraction from the first separation was saved and run again. A third separation was also tried on several samples but only a trace of heavy grains was found. Microscopic examination of heavy minerals from the first and second separations failed to reveal any differences in size, kind, or relative abundance of minerals present.

The results of the heavy mineral separations on the Marion profile are shown in Table XVI.

#### (4) Microscopic Study of Fractions.

Microscopic examination of the various heavy fractions indicated a large variety of minerals which from qualitative studies appeared to be uniform throughout the profile. The most abundant minerals identified in the order of their frequency were epidote, green and brown hornblende, zircon, tourmaline, rutile, anatase, and tremolite. There were lesser amounts of basaltic hornblende and colorless garnet. A few grains of brown garnet, staurolite, hematite, titanite, and diopside were also identified in all of the samples. At least 50% of the grains in all the fractions were opaque or coated; it was desirable therefore to reduce this unidentifiable portion as much as possible.

*Magnetic Separations.* A magnetic separation was made on the heavy fraction in order to see what portion of the opaque minerals was magnetite. An electromagnet and a bar magnet were both tried

and it was found that the latter worked just as well as the more powerful electromagnet. Only 1.5-2.5 per cent of the heavy fraction was found to be magnetite, and in addition, it was very difficult to obtain a clean separation on such fine-grained material. The magnetic separation was therefore abandoned.

*Heavy Mineral Separation Using Thallium Formate.* It was next decided to make a separation within the heavy minerals fraction using a thallium formate solution. A solution saturated at 25 degrees centigrade was used, and it was found to have a specific gravity slightly greater than 3.5. This corresponds very well with that found by E. Clerici (outlined in Johannsen's Manual of Petrographic Methods<sup>32</sup>). Samples of heavy minerals weighing about 0.1 gram were used with the thallium formate in small size centrifuge tubes. The heavier fraction from this separation was transferred with a few drops of the heavy liquid to a small crucible. The bulk of the thallium formate was then drained out of the tube into a beaker using care to prevent loss of the lighter minerals floating on top. The lighter minerals, together with the last fraction of a cubic centimeter of the heavy liquid, were transferred to a second crucible. Both fractions were washed with distilled water several times. The washings were decanted off and saved for recovery of the thallium formate by evaporation.

Because of the very small size of the heavy mineral grains, the surface tension effects of the water used for washing made quantitative recovery of the grains very difficult. In fact, it was impossible to obtain quantitative results on the fraction 0.02-0.01 mm. Fairly good quantitative separations were obtained on the 0.06-0.02 mm. fractions. There was too small an amount of the coarser 0.13-0.06 mm. heavy fractions for separations by this method. The results on the 0.05-0.02 mm. fractions are shown in Table XVII.

TABLE XVII.—HEAVY MINERALS SEPARATIONS ON THE 0.05-0.02 MM. FRACTIONS OF THE MARION PROFILE USING TETRABROMOETHANE AND THALLIUM FORMATE.

Specific gravity of heavy fraction	Per cent heavies in the soil at various depths		
	4-8 inches	15-19 inches	35-39 inches
Greater than 3.5	0.3212	0.2334	0.1896
2.9-3.5	0.3670	0.2769	0.2057

Microscopic examination of the two heavy fractions from the three horizons showed that most of the opaque and coated grains occurred in the fraction greater than 3.5 specific gravity. There was still a moderate amount of these grains in the lighter fraction, however. It

was disappointing to discover that epidote occurred in both heavy separates. Most of the epidote, however, was floated off in the fraction 2.91-3.5 specific gravity.

TABLE XVIII.--HEAVY MINERAL COUNTS ON THE 0.05-0.02 MM. FRACTIONS OF THE MARION PROFILE

Mineral	Number of grains counted in various samples					
	Sp. gr. over 3.5			Sp. gr. 2.9-3.5		
	4-8 in.	15-19 in.	35-39 in.	4-8 in.	15-19 in.	35-39 in.
Grey and brown hornblende	9	5	4	246	202	192
Basaltic hornblende	0	0	0	25	13	22
Tremolite	0	0	0	47	95	49
Epidote	64	28	48	364	351	356
Tourmaline	10	1	2	109	136	105
Zircon	250	198	177	10	6	13
Rutile	85	105	65	10	10	17
Anatase	96	71	70	0	0	0
Colorless garnet	31	11	36	2	1	5
Brown garnet	5	4	9	0	0	0
Titanite	7	0	8	2	1	3
Diopside	0	0	0	2	5	4
Hematite	5	7	8	0	0	0
Staurolite	4	1	0	0	0	0
Biotite	0	0	0	5	5	7
Muscovite	0	0	0	5	9	9
Quartz	5	3	9	95	26	17
Coated unknowns	269	237	454	176	137	220
Opagues	185	394	150	28	15	27
Totals	1025	1065	1040	1126	1012	1046

*Heavy Mineral Counts.* Although it now seemed probable that this secondary heavy minerals separation could not be used very successfully quantitatively on fine textured materials, it was decided to make counts on the 0.06-0.02 mm. fractions already separated. Counts of approximately 1,000 grains were made on these heavy fractions for the 4-8, 15-19, and 35-39 inch horizons. The data are given in Table XVIII.

Mineral ratios were calculated from the counts in this one fraction in the same way as was done in the Tilsit profile. All grains appeared to be of approximately the same average diameter. The calculated percentages of the most abundant heavy minerals are given in Table XIX, and Table XX shows the mineral ratios.

Small depositional differences are suggested by these mineral ratios, but it is quite possible that greater uniformity would be indicated if counts had been made upon the other heavy fractions and the results



had been combined. It should be pointed out that the 0.06-0.02 mm. fraction which was used for these counts is the one in which the upper size limits are set by sieves and the lower limits by sedimentation.

TABLE XIX. - PER CENT OF IDENTIFIED HEAVY MINERALS IN THE MARION PROFILE CALCULATED FROM COUNTS ON THE 0.05-0.02 MM. FRACTIONS

Mineral	Assumed Specific gravity	Per cent in soil of 0.05-0.02 mm. size		
		4-8 in.	15-19 in.	35-39 in.
Total amphiboles	3.3	0.1067	0.0845	0.0523
Epidote	3.4	0.1388	0.1039	0.0779
Rutile and anatase	4.1	0.0598	0.0389	0.0291
Zircon	4.7	0.0899	0.0477	0.0389
Tourmaline	3.14	0.0362	0.0355	0.0194
Colorless garnet	3.53	0.0079	0.0019	0.0054
Opagues	4.7	0.0631	0.0869	0.0297
Unknowns	3.9	0.1552	0.0948	0.1316

Therefore, the heavy mineral grains would cover a much greater range of size than is indicated by these limits. Furthermore, the great abundance of opaque and coated grains, which together make up 50-75 per cent of the heavy fraction, leave the counting technique open to considerable error due to improper identification. This could easily explain the differences in mineral ratios shown by the counts.

*Fusion of Heavy Minerals with Potassium Acid Sulfate.* Fusion with potassium acid sulfate has been suggested<sup>40</sup> as a method of removing such minerals as magnetite, ilmenite, pyrite, and anatase from the remainder. Experiments were carried on by fusing the heavy minerals with this reagent at various temperatures for various periods of time. It was found that fusion for 1 hour at 500 degrees centigrade removed all the opaques, anatase, and rutile. Zircon and garnet were not affected except that some coated zircon grains

TABLE XX. - MINERAL RATIOS IN HORIZONS OF THE MARION BASED ON COUNTS IN THE 0.05-0.02 MM. HEAVY MINERAL FRACTIONS

Sample depth in inches	Ratio <u>zircon</u> / <u>tourmaline</u>	Ratio <u>epidote</u> / <u>zircon</u>	Ratio total <u>amphiboles</u> / <u>zircon</u>	Ratio <u>anatase</u> / <u>zircon</u>
4-8	2.49	1.54	1.19	0.66
15-19	1.34	2.18	1.77	0.82
35-39	2.00	2.00	1.35	0.75

were partially cleaned. Tourmaline was very slightly rounded and the pleochrism of the brown variety was somewhat reduced. Inclusions in some tourmaline grains were attacked. The hornblendes were only very slightly rounded. All varieties of hornblende, and especially the basaltic hornblende, appeared to be quite resistant

to this treatment. Epidote was moderately attacked, resulting in a dusty grey appearance on the margins which was very characteristic of all grains. It seems probable that this vigorous treatment might have considerable merit in improving the accuracy of counts on sand fractions where the opaque minerals and the titanium minerals are very abundant, and where it is desirable to remove them. However, in a quantitative procedure on small samples of fine-grained material, it is another possible source of error because of the difficulty of handling small weights of heavy residues in a quantitative way. The biggest trouble was in recovery of the unfused portion of the heavy residue. The procedure followed in this case was to dissolve the fused cake in a crucible with a small amount of water. The water was decanted off or sucked off through a small capillary tube, and 5 or 6 washings were necessary to remove all the soluble material. It was difficult to prevent loss of the very small heavy mineral grains during the decanting process. Surface tension forces caused some of these grains to float on the surface of the water and it was practically impossible to prevent some loss.

TABLE XXI. — ELUTRIATOR MECHANICAL ANALYSIS OF THE MARION SILT LOAM PROFILE

Sample depth in inches	Per cent of various equivalent diameters			Total per cent
	0.13-0.046 mm.	0.046-0.025 mm.	0.025-0.011 mm.	
4-8	5.05	29.37	31.51	65.93
10-14	4.85	28.29	32.06	65.20
15-19	6.54	25.64	29.58	61.76
22-26	3.47	16.41	21.49	41.37
29-33	3.93	16.49	22.56	42.98
35-39	2.85	19.37	22.53	44.75

#### (5) Elutriator Fractions for Heavy Minerals Studies.

After a study and comparison of the methods available for obtaining fractions to be used for heavy mineral analysis, it was finally decided that elutriation should work best when one is dealing with such fine-grained material as occurs in the Marion profile. There are several advantages in the elutriation procedure in such cases. Sieves are practically useless with such fine textured profiles. Only a very small percentage of the sample is retained even on the finest mesh sieve that can be used. By using only one separation procedure for all fractions of the sample, the errors arising in the change from sieve to sedimentation methods are avoided. Such errors can be large, as was shown in the studies of the Tilsit profile, and they are difficult to correct for.

Because of the narrow size range within which most of a loess sample falls, it is desirable for the separation procedure to give narrow limits to the size of each fraction studied in order to provide

data on two or more fractions. Furthermore, by having a small size range within a fraction, the accuracy of the assumed average size of any given mineral species is increased.

TABLE XXII. — DISTRIBUTION OF TOTAL HEAVY MINERALS AMONG ELUTRIATOR FRACTIONS OF THE MARION PROFILE.

Sample depth in inches	Heavies of various equivalent diameters								Total % heavies in soil .125 - .011 mm.
	.125-.046 mm.		.046-.025 mm.		.025-.011 mm.		.011-.005 mm.		
	% of size	% in soil	% of size	% in soil	% of size	% in soil	% of size	% in soil	
4-8	2.62	.132	1.58	.464	1.71	.539	0.20	.017	1.135
10-14	5.98	.290	1.53	.432	1.77	.567	0.05	.005	1.290
15-19	3.00	.196	1.49	.382	1.67	.494	0.10	.010	1.072
22-26	2.31	.080	1.53	.251	1.41	.303	trace	---	0.634
29-33	2.42	.095	1.63	.269	1.47	.332	0.03	.003	0.695
35-39	2.62	.075	1.75	.339	1.60	.360	0.28	.026	0.774

There are several disadvantages in the use of an elutriator. Only one small sample can be run at a time and it requires several hours to make a determination. Thus the total time necessary for making the separations is quite long. Then, too, it is impossible to make absolutely clean separations at the theoretical size boundaries. Imperfect separation may arise because of convection currents, sticking of particles to the sides of the vessels, interference of particles with one another, slight differences in viscosity of the water due to temperature changes, and the tendency for the small particles to remain as aggregates unless they are dispersed immediately preceding elutriation.

By using care and a standard procedure, it was found possible to avoid most of these errors and very uniform results were obtained. The procedure adopted for the elutriation of samples is found in the appendix.

The fractions 0.125-0.046, 0.046-0.025, and 0.025-0.011 mm. equivalent diameter were separated by elutriation from the horizons of the Marion profile. All horizons were run in quintuplicate and the average results are given in Table XXI. Quantitative heavy mineral separations were made on all three fractions according to the methods outlined in the appendix. The results of the heavy mineral separations are shown in Table XXII.

Table XXI, like Table XV, suggests some dispositional variation between the layers of soil above 22 inches and those below. Table XXII gives, in addition to the three standard elutriator fractions between 0.125 mm and 0.011 mm, the results of heavy mineral separations on the 0.011-0.005 mm fraction. This was necessary with the Marion profile since a large proportion of the heavy minerals were found in the 0.025-0.011 mm fraction, which was not the case in the Tilsit. However, only minor amounts of heavy minerals were found in the 0.011-0.005 mm size range.

**(6) Chemical Determination of Zircon in Soil Fractions.**

Because of the large amount of unidentifiable material in the heavy minerals of the Marion, and in order to save time and increase accuracy, it was decided to determine zircon chemically rather than by counts on the elutriator fractions. This, of course, prevented one from obtaining mineral ratios. It seemed probable, however, that accurate data on this one mineral species from the three fractions of the profile would give the data needed to decide as to uniformity.

The chemical analysis was carried on according to the procedure outlined in the appendix, and the results are shown in Table XXIII.

The zircon distribution shows some very considerable variations from layer to layer, although it must be remembered that these figures are probably highly sensitive to small variations in the operation of the elutriator. Taking this evidence along with that of the mineral ratios derived from counts and with the mechanical analyses it would seem that minor but significant variations in deposition are present. It would certainly not be safe to use this profile for the calculation of losses and gains based on constant zircon. This illustrates the very great care which must be exercised in the selection of profiles for quantitative study.

TABLE XXIII. — DISTRIBUTION OF ZIRCON AMONG ELUTRIATOR FRACTIONS OF THE MARION SILT LOAM PROFILE.

Sample depth in inches	Distribution of zircon of various equivalent diameters						Total zircon in soil
	.125-.046 mm		.046-.025 mm.		.025-.011 mm.		
	% in fraction	% in soil	% in fraction	% in soil	% in fraction	% in soil	
4-8	10.61	.0140	8.72	.0405	4.18	.0225	.0770
10-14	11.31	.0328	6.78	.0293	3.20	.0182	.0803
15-19	11.07	.0217	8.37	.0320	5.73	.0283	.0820
22-26	9.80	.0079	6.11	.0153	4.12	.0125	.0357
29-33	10.31	.0098	6.66	.0179	3.95	.0131	.0408
35-39	10.54	.0079	6.50	.0220	4.46	.0161	.0460
	Distribution of zircon as percentage of total zircon.						
4-8	18.2		52.7		29.1		
10-14	40.9		36.5		22.6		
15-19	28.5		39.0		34.5		
22-26	22.1		42.8		35.1		
29-33	24.0		43.9		32.1		
35-39	17.2		47.8		35.0		

**(7) Conclusions and Summary.**

The heavy minerals studies on the Marion profile were of an exploratory nature with the purpose of finding out the best procedure to follow when dealing with loessial soils. The results showed that

fractions separated by elutriation were superior to those separated by a combination of sieving and decantation. The theoretical advantages of elutriation were discussed. The most important advantages shown for loessial samples were that several closely-sized fractions were obtainable in the finer sieve and sub-sieve sizes. These fractions were all comparable since their separation was based on sedimentation alone. In addition, the elutriator fractions, because of their narrow size limits, would bring to light small depositional differences between the various horizons of the profile.

Microscopic studies and counts showed that the heavy mineral fraction of this loess contained a large variety of minerals, many of which were easily identified. However, the large amount of opaque and coated unidentifiable grains made it difficult to calculate accurate percentages from counts. The opaque and unidentifiable grains could be removed, along with the anatase and rutile, by a controlled fusion with potassium acid sulfate. Other heavy minerals were not seriously attacked by this treatment. However, it was difficult to work quantitatively on small samples of fine grained particles, so this fusion treatment was not very satisfactory for the present studies.

The further subdivision of the heavy mineral fraction by thallium formate at specific gravity 3.5 was not found advantageous in this case, although it might be of use in other instances.

The mineral counts clearly indicated the geological uniformity of the parent material and showed also that depositional variation was minor. The same conclusions were drawn from the mechanical analyses. As regards ratios of heavy minerals as a test of uniformity, the variability found is perhaps exaggerated by the fact that so many unidentifiable grains were present. The test of zircon distribution among the elutriator fractions is not open to this objection, but it may be somewhat too sensitive to slight variation in the conditions of operation of the elutriator. Of course the same limitation would apply no less to fractions obtained by decantation. As was pointed out earlier, it is important to accumulate further evidence on the sensitivity of these tests in different cases.

In this Marion profile it seems clear that slightly coarser loess overlies finer loess and therefore the heavy clay layer of the B horizon cannot without objection be ascribed wholly to soil-forming factors.

*Summary.* Conclusions may be summarized as follows:

1. The Marion silt loam profile investigated showed by the mechanical analysis figures that some small variations in parent material were present.

2. Heavy mineral separations followed by identification and counting of the grains showed clearly that the soil was uniform geologically and that some depositional variations were present.



3. Magnetic separations and specific gravity separations with thallium formate did not facilitate appreciably the detailed study of the heavy minerals.

4. Controlled fusion with potassium acid sulfate removed opaque grains and made identification of the unattacked grains easier. It was notable that hornblende and basaltic hornblende were relatively resistant to this treatment.

5. The separation of fractions by the elutriator was found to be very advantageous in the case of this loessial soil, because of the close fractionation achieved.

6. Chemical determination of the zirconium in the heavy minerals from the elutriator fractions provided the basis for comparisons of the distribution of zircon according to particle size in the different layers. This is a very sensitive test for depositional variation.

## PART IV. THE QUANTITATIVE STUDY OF SOIL FORMATION AND DEVELOPMENT: THE GRUNDY SILT LOAM PROFILE.

### A. Data On Which Calculations Are Based.

#### (1) Field and Sampling Data.

*Location and Description of Profile.* Samples were taken under blue grass sod on the horticulture farm of the University of Missouri, located about 1½ miles west of Midway, Missouri, on U. S. highway number 40. The topography is gently undulating, resulting in fair surface drainage of the soil. From all indications, the soil had not been cultivated for at least 10 years, but it was not a virgin prairie soil. The site is about 4 miles from the nearest portion of the Missouri river flood plain, which is located to the southwest.

From the texture of the soil and its location, one is led to believe that it is of loessial origin. This is further indicated by the occurrence of loess deposits of 5-8 feet thickness in road cuts only a few hundred yards away. A brief description of the profile follows. Profile changes below 22 inches are gradational. Therefore, distinction of horizons below this depth is only approximate.

- |                |            |  |
|----------------|------------|--|
| A <sub>1</sub> | 0- 12 in.  | Greyish-brown silt loam.   |
| A <sub>2</sub> | 12- 17 in. | Light grey silt loam. A weak grey layer.   |
| B <sub>1</sub> | 17- 22 in. | Greyish-brown silty clay loam. Mottled with light brown silty clay toward the base.  |
|                | 22- 50 in. | Light brown and grey mottled, heavy silty clay, becoming slightly lighter in texture toward base. Numerous iron concretions.                 |
|                | 50- 63 in. | Light grey silty clay mottled with light brown silty clay (more grey than brown). Numerous iron concretions. Not as heavy textured as above. |
|                | 63- 96 in. | Same as above. Lighter texture and greyer.   |
|                | 96-114 in. | Light grey, light textured silty clay loam. Not as heavy in texture as above.  |

*Method of Sampling.* An L-shaped pit was dug to a depth of 6 feet. The arms of the L were each 5½ feet in length. At the center bend of the L, the main profile samples were taken in volume weight cans each holding about 110 cc. Three cans placed closely together were driven into the side of the pit at each horizon of the profile at which samples were desired. Samples were taken at depths of 4-8, 12-16, 18-22, 25-29, 34-38, 46-50, 58-62, 64-68, 70-74, 84-96, and 108-114 inches. The samples of the lower two depths were taken with a hand auger by boring into the bottom of the pit. Care was used to select uncontaminated cuttings representative of the desired horizon.

In order to study horizontal variations to be expected, profile samples were taken in volume weight cans at either end of the L

at depths corresponding to 12-16, 34-38, and 64-68 inches of the main profile. A few other comparable samples were also taken.

This triple method of sampling is of considerable importance from a statistical standpoint. The assumption that the parent material was originally uniform is, after all, an approximation about whose limits of tolerance little can be said as regards the particular profile site selected for detailed study. Some variation will always be found experimentally. The question as to whether the small differences found have any significant relation to depth can only be settled by a parallel investigation of nearby profiles. In this way we get an idea of the normal horizontal variation in parent material in the locality and provided the differences found in the profile selected for study do not exceed the normal horizontal variation, it seems safe to proceed. The practice of taking one main sample plus two subsidiary samples oriented along two lines at right angles has therefore been adopted. An L-shaped trench facilitates this kind of sampling.

TABLE XXIV. — AVERAGE VOLUME WEIGHTS OF THE THREE GRUNDY PROFILES

Horizon	Depth in inches	Volume weights in the profile		
		Grundy 1	Grundy 1E	Grundy 1N
A <sub>1</sub>	0-12	1.39	1.31	1.35
A <sub>2</sub>	12-17	1.30	1.37	1.36
B <sub>1</sub>	17-22	1.35	1.32	---
B <sub>2</sub>	22-32	1.30	1.32	---
	32-42	1.47	1.46	1.48
	42-55	1.45	1.44	---
	55-63	1.42	---	---
	63-69	1.40	1.42	1.40
	69-75	1.44	---	---

Sampling was done on January 24, 1942, a rather undesirable time of year. The soil was quite moist at the time and the upper one inch was frozen. Since the surface samples were taken below the 4 inch depth, it is doubtful if frost action had any effect on the volume weights. The results of the volume weight determinations are given in Table XXIV.

## (2) Mechanical Analysis.

The mechanical analysis was carried on according to the procedure outlined in the appendix, except that the sieves were not used, since only a trace of material coarser than 0.1 mm., other than iron concretions, was found in any of the samples. Pipet samples were taken for the 0.011-0.005, 0.005-0.002, and less than 0.002 mm. fractions. The coarser fractions were determined on separate samples in the elutriator.

**Elutriator Mechanical Analysis.** The procedure followed in the elutriator mechanical analysis is outlined in the appendix. The

fractions 0.125-0.046, 0.046-0.025, and 0.025-0.011 mm. equivalent diameter were separated by this means, and they were used for heavy minerals analysis. The results of the elutriator mechanical analysis cannot rightly be combined with those of the standard mechanical

TABLE XXV. — MECHANICAL ANALYSIS OF THE GRUNDY NO. 1 PROFILE

Depth in inches	Method used for determining fractions							Total %
	Sieve	Elutriator			Pipet			
	% Over .125 mm.	% .125 to .046 mm.	% .046 to .025 mm.	% .025 to .011 mm.	% .011 to .005 mm.	% .005 to .002 mm.	% <.002 mm.	
0-12	.05	6.38	30.32	30.13	9.64	5.32	15.58	97.42
12-17	.04	7.37	25.93	28.52	11.63	5.93	21.78	101.10
17-22	.02	4.07	19.23	25.21	8.78	5.62	38.78	101.71
22-32	.03	3.62	18.43	28.68	10.64	6.96	41.03	109.39
32-42	.02	3.91	20.85	28.51	10.04	6.51	36.07	105.91
42-55	.03	3.45	20.98	31.60	14.03	4.90	34.21	109.20
55-63	.03	4.29	21.56	29.75	13.05	7.09	28.33	104.13
63-69	.03	4.67	22.77	29.27	12.24	6.50	27.23	102.71
69-75	.12	4.47	22.34	29.66	13.80	8.93	26.25	105.47
108-114	1.18	10.77	22.69	22.37	5.00	6.28	34.39	102.68

analysis, since separate samples given radically different treatments were used in the two procedures. It will be recalled, however, that pipet mechanical analyses were run on Marion samples, with and without strong acid treatment, and they gave almost identical results in the silt fractions. It is interesting, therefore, to see what the combined pipet and elutriator mechanical analysis shows. From Table XXV we see that, in most cases, the results total over 100 per cent. Ordinarily we might expect the results to be below 100, due to losses in the elutriated samples resulting from the strong acid

TABLE XXVI. — MECHANICAL ANALYSIS OF COMPARABLE HORIZONS OF THE GRUNDY PROFILES

Fraction size in mm.	Profile number and depth in inches								
	1			1E			1N		
	12-17	12-17	12-17	32-42	32-42	32-42	63-69	63-69	63-69
>.125	.04	.12	.05	.02	.02	.02	.03	.07	.03
.125-.046	7.37	6.40	5.09	3.91	3.79	4.27	4.67	5.27	4.85
.046-.025	25.93	25.33	25.23	20.85	17.76	19.67	22.77	23.27	20.87
.025-.011	28.52	28.38	29.35	28.51	27.27	28.48	29.27	28.79	29.67
.011-.005	11.63	13.17	13.29	10.04	11.98	10.39	12.24	12.94	13.65
.005-.002	5.93	5.86	6.67	6.51	6.88	6.92	6.50	5.98	6.75
<.002	21.78	21.45	20.61	36.07	34.89	35.31	27.23	27.82	28.12
Total	101.10	100.74	100.29	105.91	102.59	105.06	102.71	104.14	103.94

treatment. Apparently, there was little loss in the silt fractions from this treatment, as has already been shown for the Marion profile.

The fact that the combined results of the two mechanical analyses total more than 100 per cent suggests that the elutriator does not make the theoretical separations calculated for it. This is probably

TABLE XXVII. — TOTAL HEAVY MINERALS DISTRIBUTION AMONG FRACTIONS OF THE GRUNDY NO. 1 PROFILE.

Depth in inches	Per cent heavies of various sizes						Total % heavies in soil
	.125-.046 mm.		.046-.025 mm.		.025-.011 mm.		
	in fraction	in soil	in fraction	in soil	in fraction	in soil	
0-12	3.72	.237	1.67	.506	1.77	.534	1.277
12-17	2.55	.188	1.53	.397	1.25	.357	.942
17-22	3.20	.130	1.42	.273	1.23	.310	.713
22-32	3.05	.110	1.37	.253	1.00	.287	.650
32-42	2.92	.110	1.48	.309	1.14	.325	.744
42-55	3.20	.110	1.59	.334	1.29	.408	.852
55-63	3.27	.140	1.95	.421	1.65	.490	1.051
63-69	2.74	.128	2.07	.471	1.70	.498	1.097
69-75	3.37	.151	2.13	.476	1.84	.546	1.173
108-114	2.27	.245	1.32	.300	1.16	.260	.805

due mostly to the difficulty in obtaining complete dispersion after the strong HCl treatment. However, since all elutriated samples were treated similarly and good checks were obtained, we are justified in comparing one elutriated sample with another.

The mechanical analysis of comparable horizons of the three profiles is given in Table XXVI. The results show remarkable horizontal uniformity over these short distances.

The results given in Table XXV clearly show a depositional difference between the 108-114 inch layer and those above. The 69-75 inch layer may be very slightly contaminated with this glacial till, as the figures for the  $>.125$  mm. fraction suggest. However, from 69 inches upwards there is no sudden break, and the evidence of the mechanical analyses strongly favors a high degree of uniformity in the parent material.

TABLE XXVIII.—TOTAL HEAVY MINERALS IN COMPARABLE HORIZONS OF GRUNDY PROFILES.

Depth in inches	Total heavy minerals in various Grundy profiles per cent of total soil		
	No. 1	No. 1E	No. 1N
12-17	0.941	0.893	0.933
32-42	0.744	0.715	0.675
63-69	1.097	0.768	0.723

### (3) Quantitative Separation of Heavy Minerals.

The heavy minerals were separated in centrifuge tubes especially designed for the purpose (see appendix). The results of the separa-



tions are given in Table XXVII. The procedure used in making the separations was the same as that previously discussed for the Marion profile. Heavy minerals separations on comparable horizons of the three profiles are given in Table XXVIII, and they show uniformity in all but the 63-69 inch horizon, where some difference is indicated.

#### (4) Microscopic Observations.

Temporary slides were made of heavy minerals from the 0.125-0.046 mm. fractions of the various horizons in the profile. A brief description of these follows:

Black opaque minerals made up about 50 per cent of all heavy fractions. These opaque minerals were mostly leucosine and ilmenite with a small per cent of magnetite.

The amphibole group of minerals was very abundant. Several varieties of hornblende were recognized. There was a green variety showing green to greenish brown pleochroic colors, a brown variety showing brown to pale brown pleochroic colors, and a pale grey variety showing very weak pleochroism. The grey variety was perhaps the most common. The colorless amphibole, tremolite, was also common. Basaltic hornblende was another very interesting amphibole which occurred in small amounts. It was recognized by the characteristic amphibole cleavage, high index, dark brown to pale yellow pleochrism, and parallel to about 5 degree extinction angle. All varieties of hornblende showed evidence of weathering by slight rounding and cloudy alteration products on their margins. In general, the grains were only slightly altered in the horizons below 50 inches. Above 50 inches weathering was much more pronounced.

The epidote group was represented by two distinct varieties. The normal green epidote with moderately high birefringence and moderate pleochroism was rather uncommon. Much more abundant was a colorless variety with low birefringence and high index. These grains looked like colorless garnet, but unlike garnet they were anisotropic. Epidote frequently had a gray coating on the surface and an etched appearance which indicated that it had been slightly weathered.

Zircons were common, occurring in perfect euhedral forms and in grains with rounded terminations which were often more or less zoned. Rounded forms were often coated with grey dusty material which made identification difficult and indicated possible weathering. These rounded zircons were probably derived from a sedimentary rock, whereas the fresh euhedral crystals probably came directly from igneous rocks.

Tourmalines were fairly common and included a brown highly pleochroic variety, a pale brown moderately pleochroic variety often with dusty inclusions, and a bluish-grey moderately to highly pleochroic variety. Some tourmalines showed differences in pleochroic color in

different parts of the same crystal. Both euhedral and irregular crystals were present in about the same proportions. A few of the less pleochroic tourmalines showed etched edges indicating some weathering. These tourmalines, like the rounded zircons, were probably derived from a sedimentary rock which would explain their "aged" appearance.

The titanium minerals, anatase, rutile, and titanite were observed. Anatase was present in small amounts. It showed its characteristic square outline and pale yellow color and was more or less clouded on the surface by coatings. Rutile was fairly common and was mostly of deep honey yellow color. Pale yellow colored varieties were also present and semi-opaque, dark reddish brown grains were fairly common. Rutile grains were irregular in shape with a tendency toward rod-like forms. A few knee-shaped twins were observed. Coatings were characteristic of many rutile and anatase grains making identification difficult. A few grains of titanite were seen. They were colorless to very pale yellow, showed incomplete extinction, high dispersion, and had the characteristic incomplete interference figure. Titanite grains were more or less rounded to slightly diamond shaped and resembled zircon in many respects.

Garnet was present in both colorless and coffee brown varieties. The brown variety was rare. The colorless variety was common and it resembled chips of broken glass.

Qualitative microscopic studies failed to reveal any differences in heavy mineral varieties throughout the profile. Weathering of the less resistant grains was quite noticeable above the 50 inch level and increased in intensity toward the surface. Below 50 inches, the minerals displayed a remarkably fresh appearance. The deepest sample,

TABLE XXIX. — ZIRCON IN FRACTIONS OF THE GRUNDY NO. 1 PROFILE DETERMINED BY CHEMICAL ANALYSIS.

Depth in inches	Per cent zircon of various sizes						Total zircon % of soil
	.125 -- .046 mm.		.046 -- .025 mm.		.025 -- .011 mm.		
	in fraction	in soil	in fraction	in soil	in fraction	in soil	
0-12	10.56	.0251	5.30	.0268	4.63	.0247	.0766
12-17	11.12	.0209	5.55	.0220	4.85	.0173	.0602
17-22	11.11	.0145	6.07	.0166	4.28	.0133	.0444
22-32	8.63	.0095	5.00	.0126	5.11	.0147	.0368
32-42	8.43	.0093	4.86	.0150	4.59	.0149	.0392
42-55	8.21	.0091	4.67	.0156	4.07	.0166	.0413
55-63	8.34	.0117	5.79	.0243	3.43	.0168	.0528
63-69	8.60	.0110	4.97	.0234	3.80	.0189	.0533
69-75	8.97	.0135	4.85	.0231	3.62	.0198	.0564
108-114	12.58	.0308	8.42	.0252	4.76	.0124	.0684

108-114 inches, showed a marked difference in relative abundance of various heavy minerals, indicating a difference in sorting or a difference in origin. The 58-62 inch horizon showed a slight increase in

the average size of the heavy minerals in the 0.125-0.046 mm. fraction. This may be due to a small sorting difference. The petrographic studies indicated a similar geologic origin for all horizons down to a depth of 75 inches.

TABLE XXX. — PERCENTAGE DISTRIBUTION OF ZIRCON AMONG FRACTIONS OF THE GRUNDY NO. 1 PROFILE DETERMINED BY CHEMICAL ANALYSIS

Depth in inches	Total Zircon % of soil	Zircon distribution, % of total		
		.125-.046 mm.	.046-.025 mm.	.025-.011 mm.
0-12	.0766	33	35	32
12-17	.0602	35	36	29
17-22	.0444	33	37	30
22-32	.0368	26	34	40
32-42	.0392	24	38	38
42-55	.0413	22	38	40
55-63	.0528	22	46	32
63-69	.0533	21	44	36
69-75	.0564	24	41	35
108-114	.0684	45	37	18

#### (5) Chemical Determination of Zircon.

After ascertaining that the profile was made up of a material of the same geologic origin, it was necessary to investigate the possibility of sorting, or depositional, differences. Depositional differences would be expected to show up in the mechanical analysis, in the distribution of total heavy minerals among the various fractions, and in the distribution of one or more mineral species among the various fractions.

From the data of the mechanical analysis and of the total heavy minerals (see Tables XXV and XXVII), a remarkable uniformity in deposition is indicated down to the 75 inch depth. The small variations that occur are within the experimental error.

It is desirable, however, to have the further evidence of uniformity indicated by the distribution of one or more mineral species among the fractions. The tedious and less accurate counting procedure was abandoned in this case in favor of the more accurate chemical methods. A semi-microchemical technique for determination of zircon was adopted which was applicable to small samples of heavy minerals. This method is outlined in the appendix. From 2 to 4 samples were run for each heavy fraction and good checks were generally obtained. Table XXIX shows the average zircon determined by chemical analysis. Table XXX shows the zircon distribution between the different fractions expressed as a percentage of the total zircon. The differences in percentage down to a depth of 75 inches are small and give confirmation to the conclusion that the profile is uniform, both geologically and depositionally. The small variations in zircon distribution above 75 inches can easily be explained by slight differences in the operation of the elutriator. Below 75 inches, the percentages

are significantly different from the horizons above indicating difference in origin.

### B. Calculations of Soil Formation and Development.

*Outline of Method.* Now that we have shown that the profile is uniform both geologically and depositionally, we can proceed to calculations of soil formation and development. These calculations are made most easily by considering a soil column of 1 square centimeter cross section. The thicknesses of the layers sampled are also expressed in centimeters. Since the volume weights are determined on a 110 degree Centigrade basis in grams per cc. then

$$\text{Volume weight} \times \text{thickness} = \text{total weight of layer.}$$

Let the experimentally determined resistant mineral indicators be expressed as fractions, or percentages, by weight  $R_a, R_b, \dots$   $R_p$ , of the present-day layers, the total weights of which are  $W_a, W_b$ , etc. Then the weights of the original layers  $W'_a, W'_b$ , etc., which

$$W'_a = W_a \frac{R_a}{R_p}, \quad W'_b = W_b \frac{R_b}{R_p}, \text{ etc.}$$

Thus we reconstruct an original profile whose total weight is  $W'_a + W'_b$ , etc. We are, therefore, in a position to assess the net gain or loss of the profile as a whole as well as that of each separate layer. If we care to make the further assumption that the volume weight was originally uniform and equal to that now found for the assumed parent material, we can calculate the original thickness of the profile and of its layers according to the equation:

$$\text{Original thickness of A} = \frac{\text{present thickness A} \times \text{Vol. Wt. A} \times \% \text{ zircon in A}}{\% \text{ zircon in parent material} \times \text{vol. wt. of parent material}}$$

The foregoing calculations apply to the whole soil dried at 110 degrees centigrade or calculated on any other suitable basis. The same procedure can be applied to individual parts of the soil, to fractions obtained in mechanical analysis, and to elements determined chemically.

*Applications to the Grundy No. 1 Profile.* In making our calculations we must first decide on the parent material. The choice of parent material falls naturally on the 63-69 inch depth, since the 69-75 inch depth may be slightly contaminated and the mechanical analyses at the 55-63 and the 63-69 inch levels are almost identical.

The most reliable index is probably that offered by the total zircon, since this figure is not appreciably affected by slight variations in the operation of the elutriator. The total losses and gains calculated on

this basis for the different layers of the profile are shown in Table XXXI. The gain of 13.50 grams, or 6.4 per cent, in the profile as a whole is important, although from the nature of the calculation, the

TABLE XXXI. — TOTAL LOSSES AND GAINS OF ONE SQUARE CENTIMETER CROSS SECTION OF THE GRUNDY NO. 1 PROFILE BASED ON CONSTANT TOTAL ZIRCON AND ON CONSTANT FINE SAND.

Depth in inches	Wt. now in grams	Changes based on zircon			Changes based on .125-.046 mm. sand.		
		Original wt. in grams	change in grams	% change in wt.	Original wt. in grams	change in grams	% change in wt.
0-12	42.37	60.89	-18.52	-30.4	58.05	-15.68	-27.0
12-17	16.51	18.66	- 2.15	-11.5	26.09	- 9.58	-36.7
17-22	17.15	14.25	+ 2.90	+20.4	14.95	+ 2.20	+14.7
22-32	33.02	22.78	+10.24	+44.9	25.59	+ 7.43	+29.0
32-42	37.34	27.44	+ 9.90	+36.1	31.25	+ 6.09	+19.5
42-55	47.88	37.01	+10.87	+29.4	35.38	+12.50	+35.3
55-63	28.85	28.59	+ 0.26	+ 0.9	26.51	+ 2.34	+ 8.9
63-69	21.34	21.34	--	---	21.34	---	---
0-63	223.12	209.62	+13.50	+ 6.4	217.82	+ 5.30	+ 2.43

experimental error may be considerable. This figure is greater than the organic matter which probably does not exceed 4 grams. It must, therefore, largely be ascribed to hydration and oxidation of mineral matter. If this is so, there should be an even greater increase in the total secondary minerals. These minerals are to be found in the clay fraction and perhaps also in the finest silt fraction. It will also be noted that losses from the A horizon and the gains in the B horizon are well marked, the latter extending well into the 40-56 inch layer. The interpretation of these results can best be made by considering the losses and gains for the mechanical analysis fractions. These are shown in Tables XXXII and XXXIII.

TABLE XXXII. — LOSSES AND GAINS IN GRAMS OF THE FINE SILT AND CLAY FRACTIONS OF THE GRUNDY NO. 1 PROFILE BASED ON TOTAL ZIRCON.

Depth in inches	Fraction .001-.005 mm.			Fraction .005-.002 mm.			Fraction <.002 mm.		
	Wt. now	Original wt.	Change	Wt. now	Original wt.	Change	Wt. now	Original wt.	Change
6									
0-12	4.08	7.45	-3.37	2.25	3.96	-1.71	6.60	16.58	-9.98
12-17	1.92	2.28	-0.36	0.98	1.21	-0.23	3.61	5.08	-1.47
17-22	1.51	1.74	-0.23	0.96	0.93	+0.03	6.65	3.88	+2.77
22-32	3.51	2.79	+0.72	2.30	1.48	+0.82	13.55	6.20	+7.35
32-42	3.75	3.36	+0.39	2.43	1.78	+0.65	13.47	7.47	+6.00
42-55	6.72	4.53	+2.19	2.35	2.41	-0.06	16.38	10.08	+6.30
55-63	3.76	3.50	+0.26	2.05	1.86	+0.19	8.17	7.79	+0.38
63-69	2.61	--	--	1.39	--	--	5.81	--	--
0-63	25.25	25.65	-0.40	13.32	13.63	-0.31	68.43	57.08	+11.35

As anticipated above, there is an appreciable gain in the clay fraction. The increased hydration thereby conferred would probably



not exceed 3 grams for the entire profile. All fractions coarser than 0.002 mm. show losses or gains too small to be significant. However, it should be noted that the fractions 0.046-0.002 mm., while showing no total change, appear subject to translocation within the profile. The fraction 0.125-0.046 mm. shows no evidence of either loss or translocation.

TABLE XXXIII. — LOSSES AND GAINS IN GRAMS OF THE FINE SAND AND COARSE SILT FRACTIONS OF THE GRUNDY NO. 1 PROFILE BASED ON TOTAL ZIRCON\*

Depth in inches	Fraction .125-.046 mm.			Fraction .046-.025 mm.			Fraction .025-.011 mm.		
	Wt. now	Original wt.	Change	Wt. now	Original wt.	Change	Wt. now	Original wt.	Change
0-12	2.70	2.84	-0.14	12.86	13.49	-0.63	12.77	17.34	-4.57
12-17	1.22	0.87	+0.35	4.23	4.13	+0.10	4.66	5.31	-.65
17-22	0.70	0.67	+0.03	3.24	3.16	+0.08	4.25	4.06	+0.19
22-32	1.20	1.06	+0.14	5.51	5.05	+0.46	8.59	6.49	+2.10
32-42	1.46	1.28	+0.18	7.33	6.08	+1.25	10.02	7.81	+2.21
42-55	1.65	1.73	-0.08	9.12	8.20	+0.92	13.74	10.54	+3.20
55-63	1.24	1.34	-0.10	5.96	6.33	-.37	8.23	8.14	+0.09
63-69	1.00	----	-----	4.73	---	-----	6.08	----	-----
0-63	10.17	9.79	+0.38	48.25	46.44	+1.81	62.26	59.69	+2.57

\*Fractions corrected so as to add up to 100 per cent in the mechanical analysis by dividing the error equally between the 0.046-0.025 and 0.025-0.011 mm. elutriator fractions.

From the fact that the silt fractions from 0.002 mm. to 0.046 mm. appear to be almost constant in total amount, we may argue as follows. The fine sand fraction, 0.125-0.046 mm. shows no evidence of change in amount nor of translocation. Therefore it cannot have lost appreciable amounts by weathering in any horizon. The silt fraction immediately smaller, 0.046-0.025 mm., thus cannot have gained from it. It is very unlikely that the 0.046-0.025 mm. fraction could have gained from the finer silt or clay fractions. Hence, the changes in this fraction within the profile might be ascribed purely to translocation. One cannot, however, extend this argument down throughout all the silt fractions, since it would lead to the conclusion that no silt whatever had been weathered. But if no silt had been weathered, where did the extra clay come from? In dealing with the finer silt fractions it is dangerous to assume that there has been no gain by synthesis. It would appear in this example that the gains approximately balanced the losses. Unfortunately the errors in the operation of the elutriator preclude any closer examination of this matter.

Since there has been no appreciable loss or translocation in the 0.125-0.046 mm. fraction, it could be used like zircon as an index to profile changes. Calculations of the total losses and gains in layers of the profile based on this fraction are shown in Table XXXI. The total profile gain in this instance is 5.3 grams as compared with 13.5 grams using zircon.

Since the total heavy minerals were determined accurately, their weathering can be followed in the same way. Results of this calculation are shown in Table XXXIV. It is found that appreciable losses

TABLE XXXIV - LOSSES AND GAINS OF TOTAL HEAVY MINERALS IN THE GRUNDY NO.1 PROFILE BASED ON CONSTANT ZIRCON.

Depth in inches	Wt. H. M. now in grams	Wt. H. M. originally in grams	Change in grams	Change in per cent
0-12	0.5311	0.6680	-0.1269	-19.00
12-17	0.1553	0.2047	-0.0494	-24.13
17-22	0.1223	0.1563	-0.0340	-21.75
22-32	0.2148	0.2356	-0.0208	- 8.83
32-42	0.2778	0.3010	-0.0232	- 7.71
42-55	0.4079	0.4060	+0.0019	+ 0.47
55-63	0.3032	0.3136	-0.0104	- 3.32
63-69	0.2341	---	---	---
0-63	2.0224	2.2852	-0.2628	-11.50

occur down to 42 inches, but not below this depth. The breakdown is vigorous in the 0-22 inch horizons where it amounts to about 20 per cent. It then changes abruptly to about 8 per cent for the horizons 22-42 inches. Below 42 inches, weathering of heavy minerals is negligible.

The changes in thickness or volume of various horizons of the profile are shown in Table XXXV. Since the calculations are based on the assumption that the original volume weight of all horizons was the same as that of the present-day parent material, the results may not be as accurate as those based on weight changes. Nevertheless, these changes are large enough to be significant.

TABLE XXXV. - CHANGES IN DEPTH OF ONE SQUARE CENTIMETER. CROSS SECTION OF THE GRUNDY NO. 1 PROFILE.

Depth in inches	Thickness now in cm.	Thickness originally in cm.	Change in cm.	Per cent change
0-12	30.48	43.43	-12.95	-29.8
12-17	12.70	13.31	- 0.61	- 4.6
17-22	12.70	10.17	+ 2.53	+24.9
22-32	25.40	16.28	+ 9.12	+56.0
32-42	25.40	18.60	+ 6.80	+36.7
42-55	33.02	26.42	+ 6.60	+24.9
55-63	20.32	20.42	- 0.10	- 0.5
63-69	15.24	15.24	----	----
0-63	160.02	148.63	+11.39	+ 7.7

Down to a depth of 17 inches, the profile has shrunk 13.5 centimeters or about 25 per cent. Below 17 inches a large increase in volume occurs reaching a maximum of 56 per cent in the 22-32 inch horizon and tapering off at greater depths. The profile shows a net increase in thickness of 11.39 centimeters or 7.7 per cent.

### C. Conclusions and Summary.

The weathering processes in this soil probably center chiefly around the decomposition of the feldspars, since ferromagnesium minerals are present only in very small amounts. The equations normally used to express such decompositions indicate the formation of kaolinite and the removal in the drainage water of the bases and of silicic acid. Under the conditions here present from the start, namely a relatively high content of reactive colloidal clay in the parent material, such a result would seem highly improbable. The feldspars present are a mixed group and there is no reason why they should not, after decomposition, be directly resynthesized into clays of the beidellite-montmorillonite group. The silica would thus be completely utilized while sodium, calcium, and potassium would enter into the exchange complex of the original clay present, as well as into that of the synthesized clay. Losses in the drainage, therefore, would be small and the profile as a whole would show an increase in weight. It will be necessary, however, to proceed to detailed chemical analyses before the exact status of gains and losses for each element can be assessed.

A point of practical interest in soil survey work comes out very strongly in this example. Profile development, even where, as in this case, it is not extreme in character, extends to a greater depth than that which would normally be taken as the base of the B horizon. It can readily be shown, using the figures given above, that if the assumption had been made that the sample at 32-42 inches depth represented parent material, a completely erroneous picture would have been obtained. Instead of a slight gain in total weight, the profile to 40 inches would have shown a 20 per cent loss.

This error in the total weight is only one of several, the others being even more serious as regards quantitative interpretation. The choice of the 32-42 inch layer as the basis for calculation automatically makes the assumed parent material too high in clay content. Hence the apparent losses from the A horizon become greatly exaggerated as can be seen by the comparison given in Table XXXVI.

Conclusions may be drawn regarding the process of formation of the heavy B horizon. Here the increase in volume and in weight has been much greater than could be accounted for by a simple deposition of clay in cracks and interstices. In other words, the B horizon is grossly swollen. The accumulation of clay is, therefore, accompanied by microplastic movements which lead to a considerable increase in volume. Thus the present-day B horizon is much thicker than the layer of parent material from which it started.\*

The losses of clay and silt from the A horizons naturally cause

\*In a previous brief account of this work (42) some of the depths of the layers were incorrectly copied; hence some slight differences between the two sets of calculations. The general conclusions are in no way affected.

a decrease in total volume. Compared with the parent material this layer has lost 66 per cent of its clay, but this represents only the minimum possible movement since clay has certainly been formed

TABLE XXXVI -ERRORS ARISING IN INCORRECT CHOICE OF ASSUMED PARENT MATERIAL IN CALCULATING TOTAL GAINS AND LOSSES\*

Depth in inches	Wt. now in grams	Weight originally in grams	
		Incorrect 32-42 inch layer as the basis	Correct 63-69 inch layer as the basis
0-12	42.37	82.62	60.89
12-17	16.51	25.42	18.66
17-22	17.15	19.38	14.25
22-32	33.02	31.03	22.78
32-42	37.34	37.34	27.44
0-42	146.39	195.79	144.02

\* Calculations based on total zircon as the immobile indicator.

concurrently. In order to separate clay formation from clay movement, it would be necessary at present to make assumptions regarding the rates of clay formation in different horizons. We shall, therefore, not pursue this matter further, except to point out that it can be treated experimentally, namely, by a complete quantitative study of the feldspars in the profile.

The noncalcareous character of this profile implies that the conditions were similar to those found by Smith<sup>55</sup> in Illinois loess. The absence of carbonates is explained by assuming that aerial deposition was of long duration and that leaching was sufficient to remove the carbonates as fast as they were deposited. The calculations which have been made, therefore, give us a picture only of the later stages of soil formation and development.

### Summary.

1. Data from mechanical analysis and from heavy mineral analysis show that the Grundy profile is uniform in origin both geologically and depositionally down to a depth of 69 inches. Above 69 inches the profile is of loessial origin; below this depth an increasing amount of glacial drift contamination is indicated.

2. Horizontal variations in profile development and mineral composition were small when taken over short distances.

3. A simple method was outlined for calculating the changes taking place during profile development, based on zircon, or some other resistant mineral, as an immobile indicator.

4. Zircon was chosen as the best indicator and it was shown that the present-day profile was heavier than the original parent material. This weight increase was due partly to organic matter and

partly to oxidation and hydration of minerals leading to clay formation.

5. A quantitative measure of the clay formed was obtained together with a partial picture of its movement.

6. The silt fractions from 0.002 mm. up to 0.046 mm. remained almost constant in total amount and showed some evidence of translocation.

7. The coarsest sand present in quantity (0.125-0.046 mm.) was apparently quite immobile and unaffected by weathering. It could, therefore, be used instead of zircon as an indicator of losses and gains.

8. A study of total heavy minerals revealed that mineral breakdown was vigorous in the 0-22 inch layers, that it fell off with increasing depth down to 42 inches, and that it was negligible at greater depths.

9. The profile was shown to have a pronounced volume increase in the B horizon and a net volume increase of about 8 per cent. The swelling necessary in the B horizon must have been accompanied by microplastic movements.

10. It is emphasized that profiles examined be taken to a sufficient depth, otherwise completely erroneous conclusions may be drawn.



## APPENDIX.

## A. Method Used For Standard Mechanical Analysis.

Twenty-gram samples in duplicate were placed in 300 cc. beakers and treated with 6% hydrogen peroxide on the steam plate to remove organic matter. The samples were then removed, cooled, and HCl added until a .1 normal solution was obtained. The soil was stirred thoroughly and allowed to stand for about 2 hours. The clear supernatant liquid was then drawn off slowly by means of a suction pump, using care to prevent loss of any sediment. The beakers were then filled with distilled water, stirred, and allowed to stand until a clear supernatant liquid was again obtained. This liquid was also drawn off. The process was repeated three or four times or until the clay started to disperse and would not settle out in 12 hours. Then the liquid was sucked off by means of a Berkefeld filter. Reversed pressure from the distilled water faucet was applied to the filter at the end of each filtration in order to wash any clay particles from the pores back into the sample. Each sample was then washed through a nest of standard, calibrated sieves with a jet of water from a wash bottle. A rubber policeman was used at times to hasten the sieving process. The sieves used represented the sizes 1, .6, .22, .13, and .06 mm. The portion of the sample remaining on each sieve was dried and weighed. The suspension passing through the sieves was reduced to a small volume by sucking off most of the liquid with the Berkefeld filter. Back pressure from the distilled water faucet was again used to wash the fine particles from the pores of the filter. The samples were next treated with 10 cc. of 1 normal NaOH and diluted to 500 cc. Dispersion was brought about by agitation with a high speed electric stirrer for twenty minutes. The stirrer had rubber stoppers attached to a steel rod to act as agitators. In this way a high rate of shear was obtained giving a very thorough dispersion without fracturing the sand or silt grains. It was found that twenty minutes stirring with this apparatus gave as complete dispersion as several hours in a shaking machine.

After being dispersed, the samples were transferred to large glass cylinders and made up to 1,000 cc. The mechanical analysis was then carried on at 30 degrees Centigrade in a constant temperature chamber according to the pipet method. Samples were taken to represent the fractions .02-.01, .01-.005, .005-.002, and less than .002 mm. equivalent diameter. The fraction .06-.02 mm. was recovered by transferring the sediment remaining in the bottom of the cylinder to a tall beaker. The beaker was filled, stirred, and allowed to stand for 4 minutes. The liquid was then siphoned off to a depth of 10 cm. This process was repeated until a clear solution occurred to a depth of 10 cm. after standing 4 minutes. The remaining sediment was then dried and weighed.

When it was desirable to save the .01-.005 and .02-.01 fractions for heavy minerals separations, this was done as follows. The sediment from the cylinder of the pipet analysis was transferred to a tall beaker. All particles which did not settle through 10 cm. in 66 minutes were siphoned off and discarded by repeated treatment with distilled water. This was the  $< .005$  mm. fraction. The .01-.005 mm. fraction was then obtained by saving the material remaining in suspension in the beaker after standing  $16\frac{1}{2}$  minutes. Finally the .02-.01 mm. fraction was recovered by saving the portion that remained suspended after 4 minutes.

### B. Description of Special Centrifuge Tube Used For Heavy Minerals Separations.

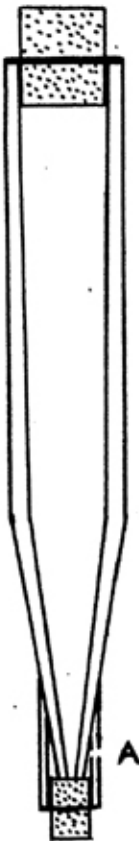


Fig. 25.—Special centrifuge tube. A, aperture for admittance of air.

The tube (originator not known) has an outside diameter of about 2 cm. for 8 cm. of its length, and then tapers gradually to an opening at the tip of about 1 mm. The total length of the tube is 13 cm. The walls of the tapered portion are thickened considerably in order to give it more strength. Commencing 1 cm. from the tapered end, a glass tubing of 1 cm. diameter and 1.5 cm. length is welded over the tip so that it extends a short distance below this end. In this way a cork can be fitted into the base of the welded portion and be pushed in until it seals off the end of the tapered tube. When centrifuging, the weight of the tube and its contents rests on the cork stopper which helps still further to seal the tube and prevent leakage of the heavy liquid. The glass tube fits inside the metal jacket of the centrifuge. It was found best to protect and support the lower end of the glass tube by allowing it, together with its cork stopper, to rest inside a hole bored part way through a large cork which rests in the bottom of the metal tube. The upper portion of the tube is also supported by using a #7 rubber stopper with a hole punched through its center large enough for the glass tube to fit snugly. This rubber stopper fits into the top of the metal centrifuge cylinder.

### C. Method Used For Heavy Mineral Separations.

The quantitative separation of heavy minerals was carried on with tetrabromoethane of specific gravity either 2.85 or 2.91, depending on the sample separated. Two-gram samples were weighed out

and transferred to each of four centrifuge tubes which were filled to within 3 centimeters of the top with heavy liquid. The tubes were balanced by adding a few drops of the heavy liquid to the proper tubes. They were then stoppered, vigorously shaken, and immediately centrifuged at 1,000-1,200 r. p. m. for five to ten minutes or until the liquid was perfectly clear. At this speed it is not necessary to use a liquid around the glass tube in order to protect it from breakage. All the heavy minerals did not come down with the first centrifuging, so each tube was shaken again and recentrifuged. This was repeated until each tube had been centrifuged 4 times. It was found by experiment that there was no increase in heavy minerals with more than 4 centrifugings and that only a slight increase occurred between 3 and 4 centrifugings.

After centrifuging, the tubes were removed and the heavy mineral grains transferred quantitatively to a small crucible by pulling the cork from the bottom of the tube and forcing them out together with a few drops of the heavy liquid, by means of the air pressure developed in pushing the upper cork tightly into the tube. This method of transfer worked very well and only in a few cases were any heavy minerals lost. The grains were washed free of the heavy liquid by treating them several times with acetone. The acetone was carefully decanted off and the heavy minerals were dried and weighed. The clear heavy liquid in the tubes was drained off into a beaker, filtered, and used again. The light fraction, together with a small amount of the heavy liquid, was transferred to another beaker and washed free of the heavy liquid with acetone. It was dried and saved.

The heavy liquid was later recovered by distilling off the acetone in a hot water bath. The recovered liquid was then brought up to its former density by heating to about 90 degrees Centigrade and cooling several times. After recovering the heavy liquid over and over again, it became darkened. A useful way to lighten the color, if it is not extremely dark, was discovered by accident in the course of the heavy minerals separations. It was found that the finer silt fractions had a pronounced adsorption for the color. In the course of several separations of heavy minerals from these fractions practically all of the dark color was removed from the liquid. Thus by repeated mixing of a darkened liquid with the finer silt fraction of a loess soil, and then filtering, it is possible to obtain a clear liquid again. When the liquid had become extremely dark, this treatment did not work. Such a darkened liquid can be recovered by distillation under a partial vacuum in an oil bath at 160 degrees Centigrade.

To separate enough heavy residue for chemical and optical studies, from 4 to 16 one- or two-gram samples were required for each fraction. Duplicate samples checked very closely in most cases. However, with the finest fraction, .025-.011 mm., poor results were

frequently obtained, and it was found necessary to make a second separation on the light fraction in order to get an accurate quantitative result. A second separation of the light fraction of some of the .046-.025 mm. fractions was also made, but in practically all cases only a trace of heavy minerals was found in the second run. Contamination of the heavy fraction with light minerals amounted to only about 1%. Before chemical analysis or mineral counts were made, however, the whole accumulated heavy minerals fraction was recentrifuged with the heavy liquid.

#### D. Method Used For the Chemical Determination of Zircon.

Samples of heavy residue weighing .015-.025 grams were placed in small transparent quartz crucibles. Weights were accurate to the fourth decimal place. The samples were fused with about 3 grams of potassium pyrosulfate. During fusion, the crucibles were covered and heat was applied slowly and with increasing intensity using care to prevent spattering. Two hours total fusion time was found to be necessary to completely dissolve all zircon grains. After cooling for a few minutes, the crucible with its contents and cover were placed in a 50 cc. beaker, and 10 cc. of concentrated sulfuric acid was added. The crucible cover was removed as soon as any spattered spots of the melt had dissolved. The fused cake was then dissolved by heating several minutes on a Bunsen burner to the temperature at which acid fumes started to escape. When the cake had completely dissolved, the temperature was increased and the sulfuric acid was permitted to boil slowly for a few minutes under a hood. This digestion caused coagulation of the silica and made the subsequent filtering operation more rapid.

When the acid solution had partially cooled, it was filtered through a sintered glass filtering crucible using suction. The silica residue was washed four or five times with 10 cc. portions of cold water acidified with a few drops of sulfuric acid. The filtrate was transferred to a 150 cc. beaker marked at the 100 cc. level. About 5 cc. of 30%  $H_2O_2$  were added to transform titanium to its higher valence form. Then .5 gram of  $(NH_4)_2HPO_4$  was added from a freshly prepared and filtered 10% water solution. The beaker was filled to the 100 cc. mark with distilled water, covered, and allowed to stand in an oven at 60 degrees Centigrade for several hours or until the zirconium phosphate had completely precipitated.

After cooling, the precipitate was recovered by filtering through a 9 cm. #42 filter paper. Each beaker was carefully scrubbed twice with a rubber policeman, and all precipitate washed on to the filter paper. Each precipitate was washed with 150 cc. of 5%  $NH_4NO_3$  in 15 cc. portions. The filter paper and precipitate were transferred to weighed crucibles and ignited carefully until the residue became white. The crucibles were cooled in a desiccator over calcium



chloride and weighed to the fifth decimal place after standing in the balance case 10 minutes to reach equilibrium. The weight of precipitate times the factor .69094 gives the weight of zircon present in the sample, using the formula  $ZrSiO_4$ .

By being careful to keep dust out of the precipitate, and by using a standard weighing technique, it was possible to obtain good checks in most cases. The weight of filter paper ash was determined by igniting several papers and taking their average weight. This small correction was applied to all samples. From two to four samples were run for each heavy minerals fraction.

#### **E. Method For Determining the Average Specific Gravity of Heavy Minerals Fractions With a Pycnometer Bottle.**

Small 5 cc. pycnometer bottles were used and their volume was determined with recently boiled distilled water in a constant temperature bath held slightly above room temperature. All subsequent measurements were made at this same temperature in order to avoid corrections for errors due to expansion of the glass. The pycnometer bottle was weighed; empty, filled with distilled water, and containing .5000 grams of heavy minerals plus distilled water. From this data the volume of the heavy minerals sample was found and the specific gravity calculated. To obtain checking results, it was necessary to remove absorbed air from the mineral particles in the pycnometer bottle. This was done by placing the bottle, plus minerals, plus a small amount of water, under an aspirator for about fifteen minutes. A cap, consisting of the lower part of a small test tube, was placed over the top of the pycnometer bottle when it was removed from the constant temperature bath in order to prevent evaporation before weighing.

#### **F. Method Used For Elutriator Separation of Fractions For Heavy Minerals Analysis.**

In order to get rid of iron concretions and to remove coatings as much as possible from the mineral grains to be examined, it was found best to treat all samples with strong acid. Thus the following procedure was adopted.

Twenty-gram samples were put in a 400 cc. beaker and treated on the steam plate for one hour with hydrogen peroxide to remove organic matter. Then concentrated HCl was added to produce a final acid concentration of about 6 normal. The samples were stirred and allowed to remain on the steam plate for another hour. After cooling, the supernatant liquid was carefully drawn off by suction and discarded. The beakers were then filled with distilled water and thoroughly stirred. After standing one hour, the supernatant liquid was again drawn off. The process was repeated until the acid was removed as was indicated by dispersion of the clay. No



attempt was made to save any of the sample that did not settle in the beaker in one hour since this fraction would be removed by elutriation anyway. However, care was used in drawing off the supernatant liquid to avoid any loss of sediment in the bottom of the beaker. The samples were then rubbed through a .125 mm. sieve with a rubber policeman and the very small amount of residue on the sieve was dried and weighed. The samples were then dispersed by treating with 10 cc. of 1 normal NaOH, diluting to 500 cc., and agitating for 20 minutes in the electric stirrer used for the standard mechanical analysis. The dispersed samples were transferred to one-liter beakers and permitted to stand for at least one hour. The supernatant liquid containing most of the clay was then carefully drawn off and discarded. The residue was transferred to the small tube of the elutriator with a jet of water from a wash bottle. The tube was filled nearly full with distilled water and then stoppered and vigorously shaken for several seconds to break up any lumps and soft aggregates which might have formed as a result of the time that the sediment stood in the beaker. Fine sand, approximately two grams of which had been previously added to all the tubes in order to reduce convection currents during elutriation, aided in producing this final dispersion. The small tube was then connected to the other tubes of the elutriator train, and, after standing for about five minutes, the current of water running through the apparatus was turned on and adjusted to a very slow rate. It was allowed to flow at this rate until the clay and finer silt had passed into the second and third tubes. Then the current was gradually increased until the desired velocity was attained after about one hour had passed. The elutriation was continued at the maximum rate until the water at the top of each tube was practically clear. This required a total time of about three to four hours and meant the passage of about 15 liters of water through the apparatus. Finally each fraction was washed through a .125 mm. sieve to recover the sand used to reduce convection currents, and the remaining fraction was dried and weighed.

The temperature of the distilled water used was controlled as accurately as was possible without using a constant temperature chamber. The water was allowed to reach room temperature before use. This temperature was not permitted to vary from 27 degrees Centigrade by more than two degrees, and in most cases it varied less than one degree.

A constant head of water was maintained in the elutriator supply bottle by means of an automatic siphoning arrangement connecting it with a larger storage bottle. The rate of flow remained constant and was adjusted by means of screw clamps and a gauge. The gauge was calibrated just prior to its use, together with the outlet used throughout these experiments.

The elutriator itself consisted of the usual two cylindrical tubes and a large pear-shaped tube, all with openings at both top and bottom. They were connected in series according to increasing diameter so that the liquid passing from the top of one tube flowed into the bottom of the succeeding tube. The effective diameter of each tube was measured as accurately as possible, and the size for quartz particles retained in each tube was calculated for various rates of flow according to Stokes law. It was decided to use a rate of flow separating the samples into the following fractions: .125-.046, .046-.025, and .025-.011 mm. These fractions in general included about 80 per cent of that portion of the samples greater than .002 mm. equivalent diameter. They also contained about 98 per cent of the heavy minerals which it was possible to extract.

In order to obtain sufficient material for heavy minerals studies, it was necessary to use 100 grams of material. Thus five samples were run through the elutriator for each horizon. From the five samples, a very good average was obtained.

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