METHODS OF STUDY OF SAND AND SILT FROM SOILS[1]

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ABSTRACT

Methods of cleaning, segregating and identifying minerals found in the coarse silt and sand fractions of soils are outlined. After a heavy liquid separation, identification was carried out mainly by the use of the petrographic microscope for the coarse silt and fine sand, and the binocular microscope for the medium and coarse sand. The Franz iso-dynamic separator, a staining technique for the identification of the potash feldspars, and index of refraction oils were also used. The distinguishing characteristics of about 35 minerals are described.

INTRODUCTION

On commencing a mineralogical analysis of the fine sand of some Ontario soils, it was found that there was no reference outlining a complete procedure. "Sedimentary Petrography" (Milner, 1940) was the most useful reference, but it was necessary to select information from many sources and to alter the methods when special problems were encountered. Most studies of this nature have concentrated on the heavy minerals. However, it is the light minerals such as quartz, feldspar, calcite and dolomite which make up the bulk of the sand and contribute many of the plant nutrients. Even though there is not as great a variety of these minerals, their identification is more tedious and difficult because of similarities. Using the petrographic microscope, the Franz iso-dynamic separator, staining techniques and index of refraction oils in making identification, a set of standard slides was made by mounting grains of each mineral in Canada balsam. This paper outlines the methods found to be most useful in the mineralogical analysis of these soils and gives complete descriptions of all the minerals present in the coarse silt and sand.

PREPARATION OF SOIL SAMPLES FOR ANALYSIS

Cleaning and Dispersion Procedures

A soil sample as it comes from the field often contains organic matter and iron oxide coatings on the grains. These materials must be removed

[1]Based partly on an unpublished M.A. thesis by the author: *A mineralogical analysis of some Pleistocene deposits of southern Ontario—University of Toronto*, 1958.

and the soil dispersed for easy separation of the desired fractions. Several techniques have been published. Drosdoff & Truog (1935) used hydrogen sulphide to change the free ferric oxides into sulphides. Jeffries (1946) describes a method using a buffer mixture of potassium oxalate and oxalic acid on magnesium ribbon to produce nascent hydrogen. Gilbert (1947) discusses the use of oxalic, phosphoric, citric and tartaric acids. The disadvantage of all these procedures is that the carbonates and apatite are destroyed by the acids. The method adopted in this research was that of Jackson, Whittig & Pennington (1949) in which sodium carbonate is the cleaning agent. This process called for the use of hydrochloric acid in destroying carbonates but this was omitted as was also the use of acidified alcohol in washing.

Segregation Procedures

Sand fractions may be separated from the silt and clay by means of wet sieving. The silt is obtained by sedimentation methods. These procedures are fully outlined in Jackson, Whittig & Pennington (1949). It was found possible to make identification of the minerals down to a particle size of 20 microns. Identification below this limit was unreliable.

Analysis of the Coarse Silt and Fine Sand

The first step was the separation of the coarse silt or fine sand into light and heavy minerals using bromoform. There are various types of heavy liquid separators, some of which are described by Krumbein & Pettijohn (1938) and Milner (1940). After the bromoform separation each fraction was weighed accurately. A representative portion of each was then mounted in Canada balsam on petrographic slides (4.5 by 2.5 cm.). This was done simply by sprinkling the grains onto the heated balsam and adding a cover glass.

Grain counts under the petrographic microscope were made by following parallel traverses across the slide and identifying each grain as it came under the cross-hairs. The grains were identified on the basis of their optical properties which are discussed in detail later. Approximately 250 grains were counted in each slide giving a total of 500 grains for the total sand sample. After each count, the whole slide was re-examined to determine whether any mineral types were present which were not included in the 250 grain count. Naturally, they would be very rare minerals occurring as only one or two grains in the whole slide. After the examination with the petrographic microscope some of the unmounted sand was spread out under the binocular microscope. If a mineral not noted before was seen, it was counted as one grain.

When examining the fine sand it is possible to separate fairly accurately

the calcite from the dolomite. However, in the coarse silt these grains cannot be differentiated under the petrographic microscope when mounted in balsam. When carbonates are present in the coarse silt a sample must be immersed in a liquid with index of refraction of 1.67. The proportion of calcite to dolomite may then be determined since the calcite has both indices of refraction lower than the oil whereas the dolomite has one index higher.

A final step involved the differentiation of microcline, orthoclase and plagioclase with an index of refraction less than balsam. Microcline is easily identified in the slides by its cross-hatch twinning. However, it is impossible to differentiate between orthoclase and untwinned low-index plagioclase grains and they were grouped together in the grain counts of the mounted material. A staining method for loose grains was used to differentiate these minerals (Reeder & McAllister, 1957). The sample was treated with hydrofluoric acid, causing all the feldspar grains to turn chalky white. They were then immersed in a concentrated sodium cobaltinitrite solution which turned the potash feldspar grains yellow. The potash feldspar was then determined by counting the grains under the binocular microscope. The number of microcline grains (which had been determined from the grain counts of the mounted material)was then subtracted from the total number of potash feldspars to give the amount of orthoclase. This was subtracted from the total amount of orthoclase and low-index plagioclase to give the number of low-index plagioclase feldspar grains.

Calculation of Percentage by Weight of the Minerals

The percentage by number of each mineral does not correspond to the percentage by weight because the minerals vary in specific gravity, nor does it strictly correspond to the percentage by volume because of the varying shapes of the grains. However, since it is customary to express mineral counts on a weight basis, particles are assumed to have the same volume and factors must be introduced to compensate for the differences in specific gravity. The procedure is illustrated below using imaginary figures.

Total weight of fraction = 1.0550 gms.
Weight of heavy minerals in fraction = 0.2543 gms.

Mineral	*Number counted*		*Specific gravity*	
A	150	×	3.3	= 495.0
B	50	×	3.2	= 160.0
C	30	×	3.5	= 105.0
D	10	×	2.9	= 29.0
E	10	×	3.1	= 31.0
TOTAL	250			820.0

$$\text{Specific gravity of heavies} = \frac{820.0}{250} = 3.28$$

$$\text{Weight of A in heavy fraction} = \frac{150}{250} \times \frac{3.3}{3.28} \times 0.2543 \text{ gms.}$$

$$\text{This may be reduced to } 495 \times \frac{0.2543}{820} \text{ gms.}$$

$$\text{Percentage by weight of A in fraction} = \frac{495 \times \frac{0.2543}{820}}{1.0550} \times 100$$

The same procedure is followed for the other minerals in the heavy and light fractions.

Dryden (1931) discusses the accuracy of percentage presentation of heavy mineral frequencies and states that by counting about 300 grains satisfactory results may be obtained. No more than two significant figures are justified even when 4000 grains are counted.

Analysis of the Medium Sand

The grains of the medium sand were too large to be mounted in balsam and were examined under the binocular microscope. The heavy and light minerals were first separated and the light minerals then treated with hydrofluoric acid and sodium cobaltinitrite. The grains were spread out under the microscope and 250 grains from each fraction counted. By this method the feldspars were divided into only two groups, the potash feldspars and the plagioclases. In the carbonates it is common for both calcite and dolomite to be present in the same grain making differentiation between these two minerals impossible. Cook (1952) gives good descriptions of the heavy minerals found in the medium sand fraction of some southern Ontario tills. The calculations of the percentages is the same as for the coarse silt and fine sand.

Analysis of the Coarse Sand

No heavy mineral separation was carried out on the coarse sand since many of the grains consist of several minerals. The sample was first treated with hydrofluoric acid to distinguish between the quartz and feldspar and the grains were then examined and counted under the binocular microscope. The percentage by weight of each minerals was found by using the expression:

$$\%\text{ by weight of A} = \frac{\text{No. of A counted}}{\text{Total grains counted}} \times \frac{\text{specific gravity of A}}{\text{specific gravity of sand}} \times 100$$

Use of the Franz Iso-dynamic separator

When this work was first started, it was hoped that a rapid separation of the minerals might be possible using the Franz magnetic separator. One sample was selected and the extremely magnetic minerals such as magnetite removed with a hand magnet. By putting the sample through the separator repeatedly and increasing the amount of current each time, the sand was broken down into a large number of fractions. It was found that it was not possible to isolate each mineral completely although certain groups of several minerals could be separated. On the basis of this experimentation six of the samples were split into five parts—the light minerals into two and the heavy minerals into three. After these six samples had been analyzed, the use of the iso-dynamic separator was abandoned for several reasons. After having studied the minerals of the six sands, the various types were familiar and most of the heavy minerals could be identified by sight. The splitting of the light minerals had been of no help in identification. Furthermore, the splitting and subsequent extra weighing and calculations were very tedious.

Descriptions of Minerals

These descriptions apply to the minerals as they appear in the fine sand and coarse silt.

Actinolite: Actinolite occurs in pale green prismatic fragments usually possessing jagged ends. It is differentiated from tremolite by its green colour and from pyroxene by the nearly parallel extinction.

Andalusite: Andalusite occurs in irregular fragments, many showing a distinct pleochroism from almost colourless to salmon-red. Some thick grains of sphene show a similar change of colour which is, however, not as pronounced.

Apatite: Apatite occurs in clear colourless, elongated or oval grains. It is usually very well rounded although the ends of some grains have been fractured. Inclusions are sometimes present. The interference colours range from grey to pale yellow. This mineral may be confused with quartz but the rounded appearance and moderately high relief are characteristic.

Augite: Augite occurs in irregular to well-rounded grains. It varies from colourless to pale green and is usually clouded with greenish yellow alteration products. This mineral is very difficult to distinguish from diopside which has the same form and colour. Diopside, however, is usually free from alteration coatings.

Biotite: Biotite occurs in thin greenish brown translucent flakes or in

thick, black, shiny mica "books". When the flakes are very thin and light in colour, it is impossible to differentiate them from phlogopite.

Calcite: Calcite occurs in rounded grains with a granular appearance. They are actually fragments of limestone and are found almost entirely in the light mineral fraction. In reflected light they range in colour from buff, greenish yellow to dark grey. Under crossed nicols the grains appear as pale greenish brown fragments showing whiter interference colours around the edges. Many grains are crowded with impurities and approach the composition of a calcareous shale. Some fragments also enclose minute crystals of dolomite.

Chlorite: Chlorite occurs in pale green mica-like flakes. It is readily identified by its green colour, platy appearance and very low birefringence for basal plates.

Clinozoisite: Clinozoisite occurs in pale grey or colourless grains with a rather flaky and irregular appearance. It is identified by its anomalous blue and greenish yellow first order interference colours.

Diopside: Diopside occurs in pale green and colourless, rounded to irregular grains. It is difficult to differentiate from augite but augite is usually coated with greenish yellow alteration products whereas diopside is clear.

Dolomite: Dolomite is found in both the heavy and light fractions. It occurs in rounded and irregular grains and in rhombohedral fragments. Most of the grains possess one or more linear edges and may show cleavage and twinning lines. The grains are colourless or grey and show a change of relief as the microscope stage is rotated. Under crossed nicols, the interference colours are high-order white or pearly grey. This mineral is difficult to distinguish from calcite but in general it appears to be better crystallized, has whiter interference colours and is of a lighter colour in reflected light.

Enstatite: Enstatite occurs in colourless or pale grey prismatic grains. It may be confused with tremolite but tremolite usually has a small extinction angle whereas enstatite exhibits parallel extinction.

Epidote: Epidote occurs in partly rounded and irregular grains and occasionally in prismatic crystals. It is easily identified by its distinctive pleochroism from bright greenish yellow to pale yellow or colourless.

Garnet: Garnet is commonly found in very angular fragments with sharp edges and conchoidal fracture. Rounded grains are sometimes present. It varies from colourless to pale purplish pink, red and reddish brown. Usually it is glassy in appearance but it may have orange-yellow alteration products on the surface. Inclusions are abundant in many grains and some fragments exhibit a weak birefringence.

Gypsum: Gypsum occurs in short prismatic grains, many of which show crystal faces. During the sodium carbonate treatment these grains

are coated white with calcium carbonate. Under the petrographic microscope they are readily recognized by their shape, low index of refraction and rather fibrous appearance.

Hematite: Hematite occurs as dark red translucent or opaque grains with a metallic lustre. It also occurs as a coating on some magnetite grains.

Hornblende: Hornblende occurs in irregular, poorly rounded and prismatic fragments. Many thick grains are of such a dark colour that they appear almost opaque, but under crossed nicols a band of interference colours can be seen around the edge. Most grains show a characteristic pleochroism from pale or yellowish green to dark green. Some blue-green fragments are also present.

Hypersthene: Hypersthene occurs in irregular fragments and prismatic grains. Most grains have a distinctive pleochroism from green to red although a few fragments are so faintly coloured that the pleochroism may be barely noticeable, and the grains may be mistaken for tremolite-actinolite or pale green monoclinic pyroxene. Some fragments contain rows of inclusions. Brownish alteration coatings may be present.

Kyanite: Kyanite occurs in colourless bladed fragments showing parting almost perpendicular to the length of the grain. This gives the minerals a step-like pattern of interference colours which is readily recognized.

Limonite: Limonite occurs in opaque grains which appear as powdery-looking yellow or orange aggregates in reflected light.

Magnetite and Ilmenite: It is not possible to differentiate between magnetite and ilmenite and they are therefore grouped together. They occur in dark grey and black metallic grains, many exhibiting crystal faces with rounded edges. Some grains are partially coated with hematite. Loose grains are readily attracted to a hand magnet.

Microcline: Microcline occurs in colourless, rounded and irregular grains showing typical cross-hatch twinning under crossed nicols.

Muscovite: Muscovite occurs in colourless flakes, some with reddish blotches. It is identified by its flaky appearance and low order interference colours.

Orthoclase: Orthoclase occurs in irregular grains and rectangular cleavage fragments. It is often clouded with alteration products. It is difficult to distinguish from untwinned plagioclase with an index of refraction less than that of balsam, and a staining technique must be used to separate the two.

Phlogopite: Phlogopite occurs in yellow to dark brown single flakes and in mica "books". The thin flakes may be confused with biotite but the thick, yellowish brown mica "books", many having a semi-metallic lustre, are characteristic.

Plagioclase: Plagioclase occurs in rounded or irregular, colourless grains

which often show cloudy alteration patches on the surface. Some fragments are bounded by cleavage planes and twinning lines may also be present. Untwinned, glassy grains are differentiated from quartz only by interference figures.

Pyrite and Marcasite: Sulphides in the form of pyrite and marcasite occur in irregular fragments with a characteristic brassy metallic lustre.

Rutile: Rutile occurs in short prisms or occasionally in irregular grains. The colour varies from yellow to reddish brown. The relief is so high the grains often appear edged with a broad black line with a central area of colour.

Shale Fragments: Shale occurs as black, red, grey, etc., fragments. They often tend to be platy. Under the petrographic microscope they may be opaque or translucent and some contain a large percentage of carbonate.

Sphene: Sphene occurs in irregular platy fragments and in rounded grains. Flat subhedral crystals are occasionally present. The colour varies from colourless, pale yellow to reddish brown. The thicker rounded grains sometime show a faint pleochroism. Sphene has a very high dispersion which causes many of the grains to extinguish to an anomalous blue colour instead of black.

Staurolite: Staurolite occurs in partly rounded and irregular grains showing a distinct pleochroism from straw-yellow to colourless. Irregularly arranged inclusions of quartz are usually present.

Tourmaline: Tourmaline occurs in irregular fragments or occasionally in well-rounded oval grains. Many grains show a distinct pleochroism from yellow to orange-brown or pale brown to black. A few grains with pleochroism from blue to black have been seen.

Tremolite: Tremolite occurs in colourless prismatic grains. It is similar to actinolite except for the lack of colour. Fragments show a small extinction angle and may therefore be distinguished from enstatite.

Zircon: Zircon occurs in a variety of forms, grains showing gradations from euhedral prisms with sharp terminations to oval or rounded fragments. It is usually colourless but pale brown and yellow crystals have been noted. Some grains are crowded with inclusions and contain orange patches. This mineral is easily identified by its high birefringence, very high relief and parallel extinction.

Summary

This paper has outlined the methods of cleaning, segregating and identifying minerals found in the coarse silt and sand fractions of soils. In describing the minerals as seen under the petrographic microscope, particular attention has been paid to the methods of differentiating similar-appearing minerals. It is hoped that this information will aid others engaged in similar mineralogical analyses.

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