

## Micromorphological Analysis of Selected Indiana Soils<sup>1</sup>

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### *Abstract*

Soil micromorphology is a technique using the principles of optical mineralogy for the detailed study of soils. This method is particularly useful in studies of soil genesis to demonstrate the occurrence or absence of certain processes during soil formation. Preparation of thin sections of unconsolidated soil materials presents special problems. A review of the methods used by the authors to prepare thin sections is presented. Principles of micromorphological analysis are demonstrated using selected Indiana soils.

This paper presents the methods used by the authors to prepare soil thin sections for micromorphological analysis, illustrates some of the features of the soil material, and discusses their genetic significance. The terminology used for describing micromorphological features is that suggested by Brewer (2).

### **Materials**

The available equipment determines to a large extent the specific techniques used for sample preparation. In some cases it might be more convenient to use commercially prepared thin sections, but to examine a particular feature one can control better the area to be examined if he makes his own. The impregnation equipment used here consisted of a vacuum desiccator and vacuum pump with accessories (Fig. 1). After the samples were thoroughly embedded in plastic, a diamond saw (Highland Park Model EZ) and a polisher-grinder (Beuhler Ltd. Cat. No. 53-1502) were used to complete the thin sections.

The valve assembly at the top of the desiccator was removed and a rubber stopper inserted. The rubber stopper contained two glass tubes; one was connected to a vacuum pump and the other led from the impregnation mixture reservoir to the samples. The line leading to the vacuum pump contained a valve to release the vacuum, flask traps to prevent pump damage, and a manometer to measure the vacuum in the desiccator. The soil samples were put in lead cups or other suitable containers and placed in a circle beneath the delivery tube on a brass rotating platform. By placing the cups on the outside edge of the rotating platform many samples could be treated without having to break the vacuum. The platform was rotated by means of two magnets, one attached to the platform and the other on the outside.

The following chemicals were used in making the thin sections, Vestopal-H (obtained from Henley and Co., Inc., 202 E. 44th St., New York, N.Y. 10017), monostyrene, 60% methyl ethyl ketone peroxide, 1% cobalt-naphthenate, diamond saw lubricant and white gasoline.

<sup>1</sup> Journal Paper No. 5323, Purdue University Agricultural Experiment Station.

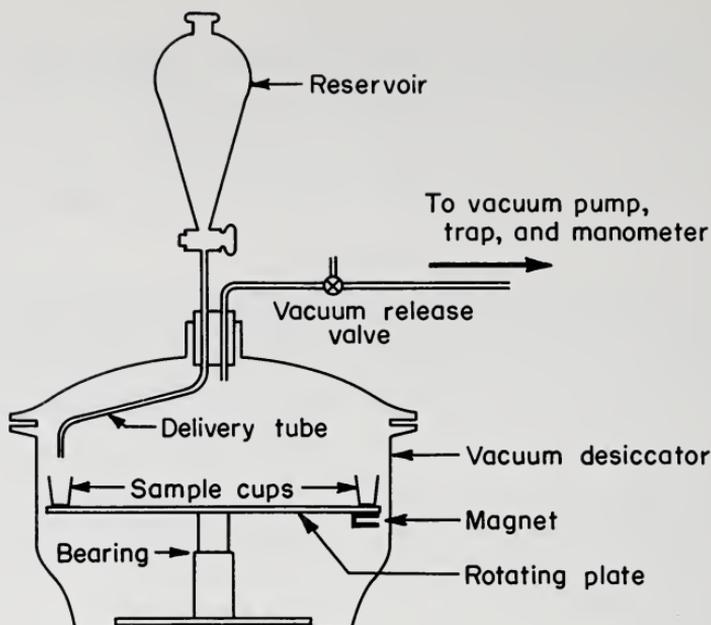


FIGURE 1. Vacuum apparatus for plastic impregnation of samples, after Brewer (2).

### Methods

In this study two procedures for impregnation are discussed; one using a rapid cure technique and the other a slow cure. These methods are modifications of procedures suggested earlier by Altemüller (1), Jongerius and Heintzberger (3), Brewer (2), and Kubiena (pers. comm.).

A discussion of the theoretical aspects of the polymerization of the impregnation mixture is given by Jongerius and Heintzberger (3). It suffices to say here that Vestopal-H is a polyester resin that is viscous in the usual form. To lower the viscosity it is dissolved in monostyrene. This better enables the mixture to penetrate the sample, resulting in a more thorough impregnation. The catalyst, which might be considered an initiator, is a peroxide, in this case methyl ethyl ketone peroxide. The splitting of its oxygen bridges provides free radicals to activate the double carbon bonds in the resin and the styrene causing them to co-polymerize and form a three dimensional network structure. An accelerator, Co-naphthenate, is added to insure that the peroxide molecules will split, by reduction, at room temperature. Excess monostyrene is evaporated off during the course of the polymerization.

These are the steps followed in making thin sections:

- 1) Select samples which are small enough to be 1.5 cm below the top of the lead sample cups. Samples were usually 2 to 3 cm by 3 to 4 cm.
- 2) Dry samples overnight in a 105°C oven and cool in a desiccator. It is important that the samples be free of water for the plastic completely to impregnate the sample.
- 3) Prepare impregnation mixtures. The slow cure mixture suggested by Jongerius and Heintzberger (3) has this composition ratio:

Vestopal-H .....	1000 ml
Monostyrene .....	667 ml
1% Cobalt-octoate, accelerator .....	1.3 ml
50% Cyclohexanone peroxide, catalyst .....	2.7 ml

In this mixture we substituted 1% cobalt naphthenate for cobalt octoate, and 60% methyl ethyl ketone peroxide for 50% cyclohexanone peroxide.

The rapid cure mixture of Kubiena had this composition ratio:

Vestopal-H .....	1000 ml
Monostyrene .....	250 ml
Co-naphthenate .....	3.1 ml
Methyl ethyl ketone peroxide .....	6.2 ml

Special care must be taken when preparing this mixture to avoid bringing the Co-naphthenate and methyl ketone peroxide in contact with each other as this will result in an explosion. To avoid this problem the Co-naphthenate and methyl ethyl ketone peroxide were added separately to the Vestopal-H and monostyrene mixture. The Co-naphthenate is a deep purple color which can be seen readily until it is thoroughly mixed with the other components. When the entire mixture is clear it is safe to add the methyl ethyl ketone peroxide. At temperatures higher than 22°C only one-half of the Co-naphthenate and methyl ethyl ketone peroxide are used.

4) Place the impregnation mixture under moderate vacuum to remove air bubbles that have been caused by stirring.

5) Place the samples in the lead cups on the outside edge of the rotating circular platform in the vacuum desiccator and apply a vacuum of 63 cm of mercury as recorded on the manometer. Leave the soil samples under vacuum for 20 min to remove entrapped air sufficiently.

6) Pour plastic into the funnel and open the valve to allow the plastic to flow out the spout and down the side of the cup. Be careful not to put the plastic on the sample itself. This avoids the problem of plastic entering the soil sample from more than one direction, causing incomplete impregnation. Initially, the sample cup is filled to about one-third full and allowed to stand until plastic has moved to the top of the sample by capillary action. At this point, the sample is allowed to set for ½ hour and then the cup is filled to the top. An attempt is made to keep the plastic 1.5 cm above the surface of the sample. This is to allow for the evaporation of the excess monostyrene without exposing the surface of the sample.

7) Leave the samples in the desiccator under vacuum for 12 hours. At the end of this time, place the samples under a ventilated hood to harden. The expected time for completion of the rapid cure samples is 4 to 7 days. The slow cure samples are expected to take 4 to 10 weeks. Our experience has shown that the rapid cure method should be used only on coarse-textured samples, because incomplete impregnation is common in other soils. The slow cure method can be used for either fine or coarse textured samples.

Monostyrene lost by evaporation is replaced by the following mixture:

Vestopal-H .....	1000 ml
Co-naphthenate .....	2.5 ml
Methyl ethyl ketone peroxide .....	2.5 ml

No monostyrene is added at this stage because little penetration is needed since the sample is already saturated with partially polymerized mixture.

8) After curing, the samples prepared according to the two different methods are handled in the same manner. Remove the sample containers. Saw off an approximately ¼-inch thick section and trim with the diamond saw.

9) At this point, check the section for thoroughness of impregnation with a binocular microscope. The section should be surface reimpregnated with the refilling mixture if needed.

10) Obtaining a smooth surface on the section to affix to the glass slide is a critical part of the procedure because this is the surface that will be seen in the finished thin section. This polishing is done manually on a piece of 1-inch thick plexiglass using Carborundum papers of 240, 320, 400 and 600 grit moistened with diamond saw lubricant. Water cannot be used because even successfully impregnated samples will have clay and organic matter that react with the water and cause pits at the surface. Successively finer

abrasive papers are used to remove imperfections left by the coarser papers. White gasoline is used to clean the diamond saw lubricant and loose particles from the surface of the section.

11) Cement the smooth side of the section to the glass slide by the same mixture used to refill the slow cure samples. Place the plastic mixture on the polished surface of the section and then place the glass slide on the polished surface and remove air bubbles. Clamp the slide and affixed sections together and allow to harden.

12) After the plastic has cured, reduce the size of the section to about 1/16 inch by cutting with the diamond saw. A special holder was made for the diamond saw to hold the glass slides.

13) The final grinding is similar to that described for preparation of the section for mounting on the glass slide. Use the grinding wheel with 120 and 240, 320 or 400 grit abrasive papers to reduce the section to approximately 60 $\mu$  as determined with a petrographic microscope and interference color chart. Complete the final grinding with 600 grit paper. The final section should have a thickness of 30 $\mu$  which is first order gray to yellow for quartz.

14) Complete the thin section by affixing a cover glass with the same plastic as used for mounting. The procedure used is to place the thin section on a table and apply a thin layer of plastic on it. Attach the cover glass from one edge in a hinge-like motion. Any air bubbles induced should be removed.

### Results and Discussion

Figures 2 and 3 are photographs of various micromorphological features found in some Indiana soils.

Figure 2, Photograph 1 is a photomicrograph of a thin section made from a Miami B2t horizon. The Miami soil is classified as a Hapludalf in Soil Taxonomy (4). The feature labeled CH is a longitudinal section of a root channel which continues across the slide but is masked by an argillan, a concentration of oriented clay particles, labeled A. It is likely that this argillan covered the entire void but parts of it were removed when the section was polished. The remainder of the material is considered to be the s-matrix, the soil matrix material within peds, labeled SM with the larger grains being quartz. This example shows the importance of channels and planes in the movement of water through soils. Clay is carried in aqueous suspension and as the water moves into the s-matrix, clay particles are deposited on the surface as argillans. These small regions that are extremely high in clay result in the overall fine texture of the entire horizon.

Photograph 2 in Figure 2 is from a fragipan in the Bx horizon of a Clermont soil (Ochraqulf). The features labeled P and CH are cross sections of root channels or former root channels, and the s-matrix is labeled SM. Again these channels are lined by argillans and the clay is highly oriented perpendicular to the plane of the photograph. Unlike the channel shown previously, the channel, P, has at least four stages of development. Starting with an initial channel, an argillan, A, was deposited, followed by deposition of an argillan high in manganese and iron, B. The void then was filled by a cutan, C. Its birefringence and its appearance in the photograph indicate that the cutan is primarily silt with some clay. The last filling is primarily manganese and iron oxides and constitutes a pedotubule (P). These different stages have resulted from different soil conditions; one possibility is a change in oxidation state of the surrounding soil material.

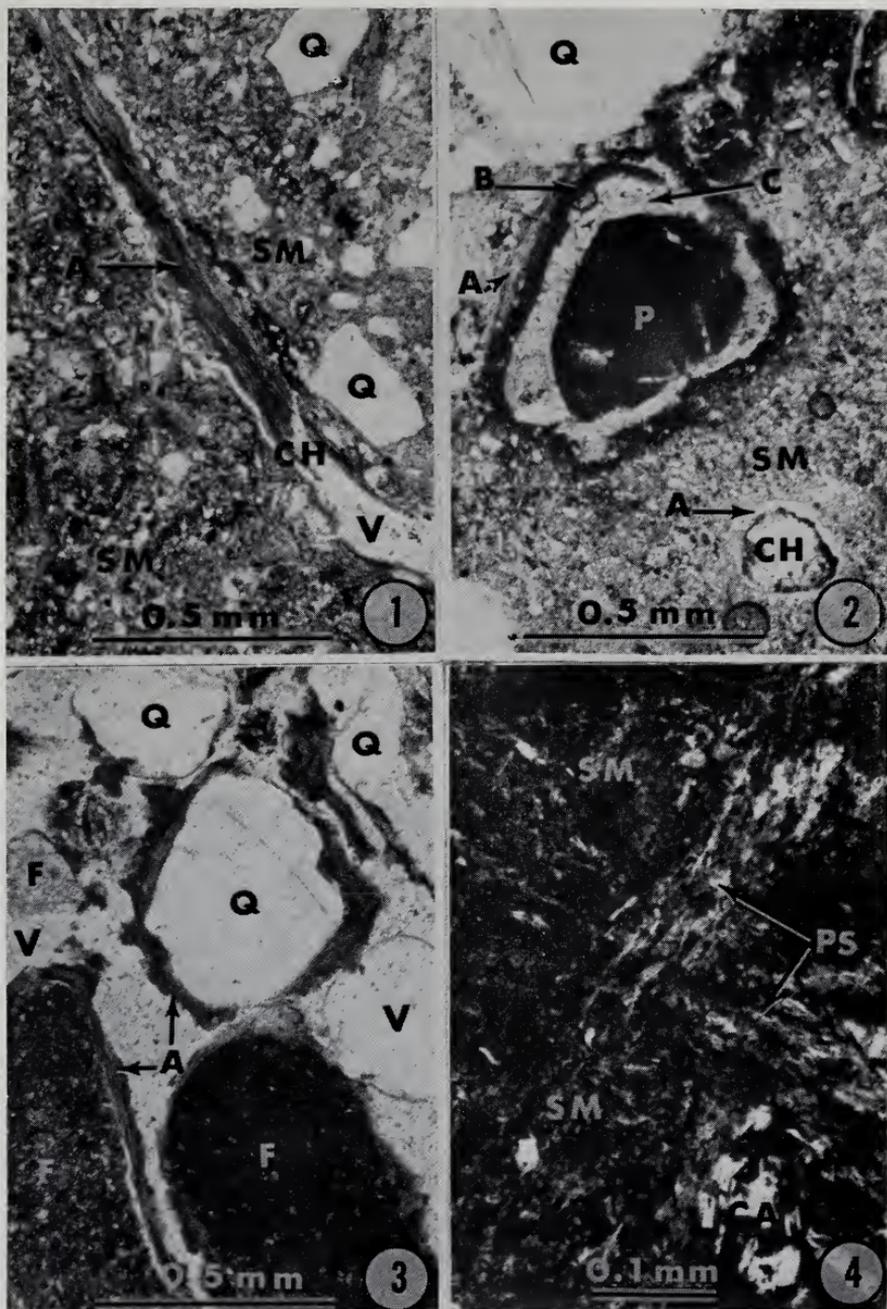


FIGURE 2. Photomicrographs in plane polarized light of: 1) Miami B2t, 2) Clermont Bx, 3) Ockley 11B2t; and under crossed polarizers, 4) palcosol in Illinoian till. Features illustrated: A (argillan), B (argillan high in iron and manganese), C (cutan), CA (clay aggregate), CH (channel), F (feldspar), P (pedotubule), PS (plasma separation), Q (quartz), SM (s-matrix), and V (void). All thin sections were prepared using slow cure methods.

Photograph 3 in Figure 2 is of a thin section from an Ockley IIB2t horizon (Hapludalf). This soil has coarser soil particles than the soils in the pictures above. The slide shows primarily skeleton grains and argillans. The skeleton grains are quartz, labeled Q, and weathered feldspar, F. The dark features surrounding each of the skeleton grains, A, are argillans. These grain argillans coat some sand grains and bind neighboring grains together. Practically all of the clay in the horizon is oriented, suggesting that it has been transported and re-deposited.

Photograph 4 of Figure 2 is a thin section of the B horizon of a paleosol developed in Illinoian till. The picture shows the s-matrix, labeled SM; plasma separations (PS), rearrangements of soil constituents; and an aggregate of clay particles in similar orientation (CA). The plasma separations are arranged in two linear patterns at right angles to each other. They probably are the result of external stresses on the soil.

Photographs 5 and 6 of Figure 3 are from a Miami B2t horizon. Picture 5 was taken in plane polarized light and Picture 6 was taken under crossed polarizers. These pictures again demonstrate the significance of voids in the movement of clay and water in the soil profile. The features labeled CH are root channels lined by argillans, A. These are surrounded by the s-matrix, labeled SM. The argillan is not highly developed. This would indicate that this particular void may have had less water moving through it than moved through the other voids

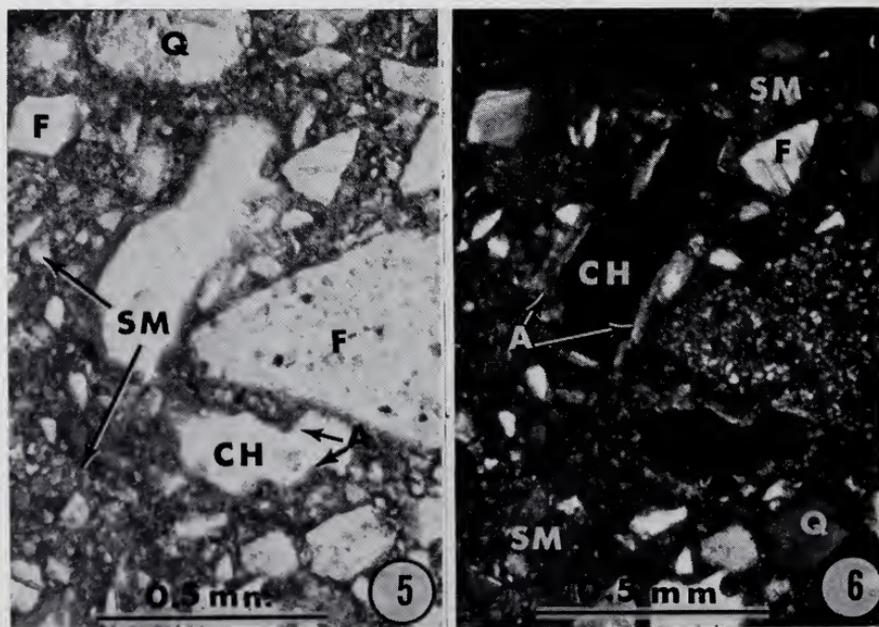


FIGURE 3. Photomicrographs of Miami B2t, 5) plane polarized light and 6) crossed polarizers. Features illustrated: A (argillan), CH (channel void), F (feldspar), Q (quartz), and SM (s-matrix).

pictured. There is a considerable difference between the features seen under plane polarized light and under crossed polarizers. The weathered feldspar, F, which shows clearly under crossed polarizers is very similar to quartz in plane polarized light. Similarly the argillan which shows its oriented nature by birefringence under crossed polarizers is not clear at all in plane polarized light. The unusual shape of the root channel is probably due to an oblique cut.

These results indicate that much information can be obtained about soil forming processes by micromorphological analysis. The new soil classification system, Soil Taxonomy (4), with its emphasis on natural properties shows the need for micromorphological analysis. However, it is also clear that more basic research needs to be done to correlate the soil forming processes with specific micromorphological features.

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