



STATENS GEOTEKNISKA INSTITUT

SWEDISH GEOTECHNICAL INSTITUTE

No. 24

SÄRTRYCK OCH PRELIMINÄRA RAPPORTER

REPRINTS AND PRELIMINARY REPORTS

Supplement to the "Proceedings" and "Meddelanden" of the Institute

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Clay Microstructure**

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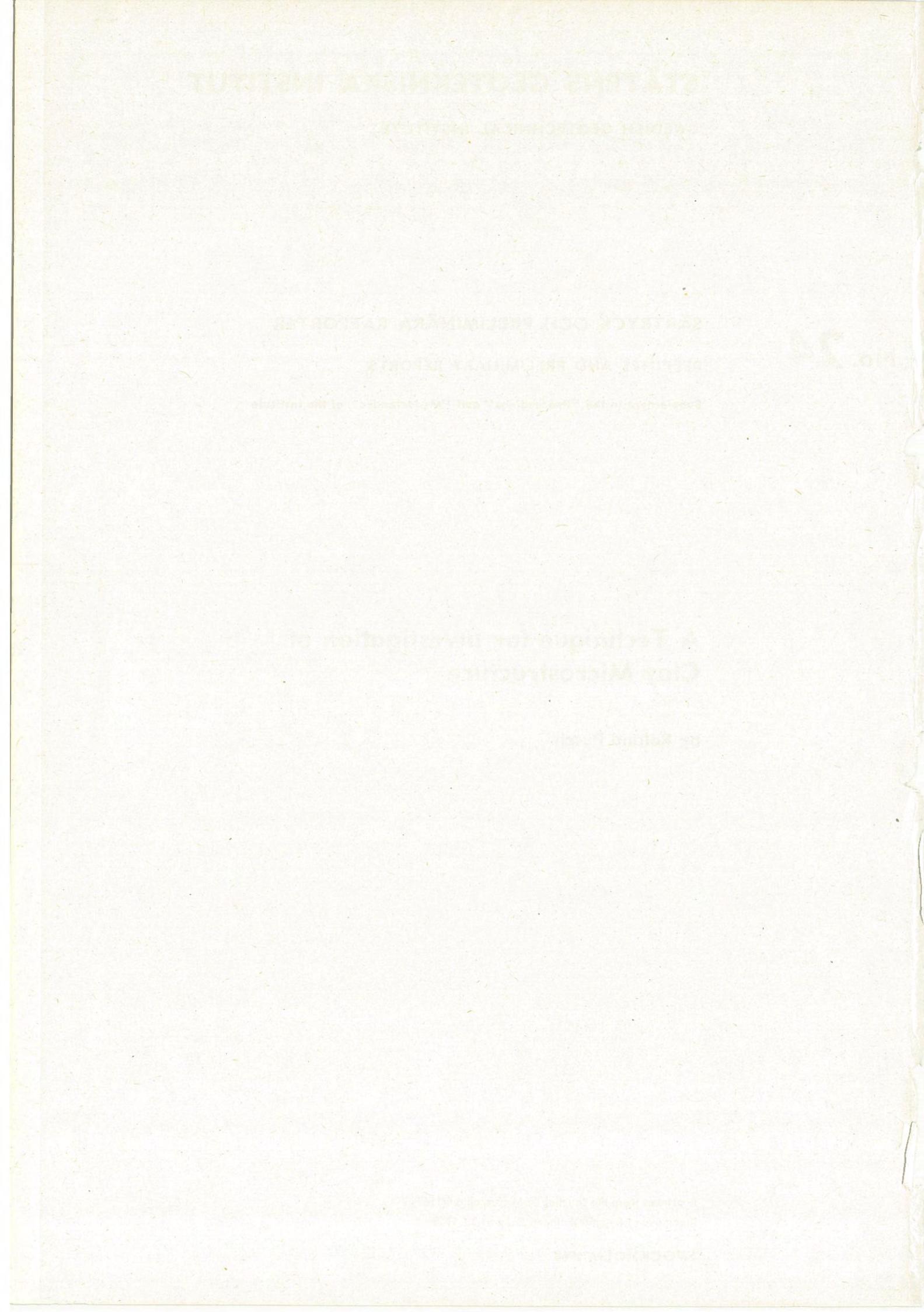
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A TECHNIQUE FOR INVESTIGATION OF CLAY MICROSTRUCTURE

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INTRODUCTION

An important aim in modern pedology and soil mechanics is to find a suitable method for observing detailed clay microstructure. Only electron microscopy offers a possibility of studying the arrangement of clay size particles, and special procedures have been developed for this type of investigation. Thus, several methods for the preparation and the study of replicas of fractured freeze-dried clay samples have been suggested, forming, in certain respects, the basis of the current conception of clay microstructure (cf. Rosenqvist, 1958).

In recent years, the present author has been engaged on investigating microtome-cut clay sections in an electron microscope. Although there are certain difficulties in the preparation of ultra-thin ($\sim 500 \text{ \AA}$) sections of clay by using a microtome, this method offers considerable advantages compared to working with replicas since the very thin sections represent two-dimensional cross sections through the clay which give a characteristic picture of the micro-pore system and of other features. The replica only gives a rough impression of the microstructure depending on the "topographic" variation of the fracture surface being investigated.

The greatest advantage of the ultra-thin section with an almost uniform thickness is that it offers a possibility of statistical presentation of certain microstructural features, such as particle orientation and size, shape and area of the sectioned micro-pores. When research work on clay microstructure was initiated at the Swedish Geotechnical Institute

in 1963, the development of a suitable ultra-thin sectioning technique was considered to be of primary importance.

A preparation method, which has been used in the study of organic tissues, was suggested by Professor Gösta Glimstedt, Department of Histology, University of Lund. It was applied to clay material and has been used experimentally since 1963 in the preparation of several types of Swedish clays. It is the main purpose of this article to give a report on the method and on its accuracy and reliability with respect to the preservation of the original microstructure.

THE ULTRA-THIN SECTION METHOD

SPECIMEN PREPARATION

Small samples of undisturbed clay are treated in such a way that the pore water is replaced by acrylate plastic by means of a diffusion process. The following preparation is used :

1. A prismatic specimen with a base area of approximately $1/8 - 1/2 \text{ cm}^2$ and a length of approximately 1 cm is cut from a clay sample with a thin steel wire or, in the case of stiff clay, with a sharp knife. The clay sample is cut in such a way that the orientation of the thin sections can be related to the clay layers *in situ*.
2. The sample is placed in 50 percent ethyl alcohol for 30 min, transferred to 70 percent alcohol for 5 min, and thereafter put into 90 percent alcohol and finally into 99.5 percent alcohol for 5 min each.
3. The sample is placed in a monomer mixture consisting of 85 percent butyl methacrylate and 15 percent methyl methacrylate for 45 min. This process is repeated once.
4. The sample is placed for 90 min in a solution consisting of 98 percent monomer and 2 percent 2,4-dichlorbenzoylperoxide (EWM) catalyst. Thereafter the sample is placed in a gelatine capsule (Parke, Davis & Co., No. 00) which is filled with monomer and catalyst.
5. After polymerization by incubating the sample for 15 hours in an oven at 60°C , the gelatine capsule is removed by washing with water. The sample can thereafter be trimmed and cut. The trimming is done in such a way that the original orientation of the thin sections obtained in the microtome can be defined. This may be effected by giving the sections a rectangular shape.

TRIMMING AND ELECTRON MICROSCOPY

The shape of the trimmed specimens is illustrated by Figures 1 and 2. In the standard procedure, thin sections taken parallel to the vertical plane *in situ* were obtained with a LKB Ultrotome 4801 A/4802 A. The

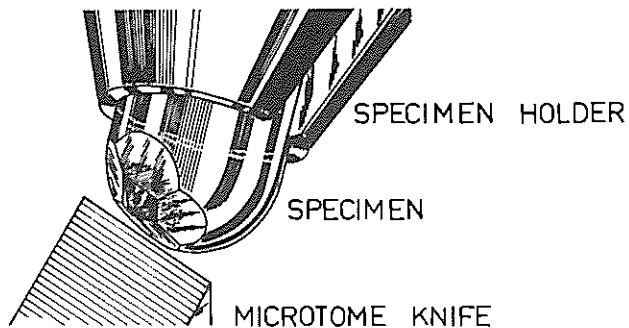


FIGURE 1. Embedded clay specimen trimmed for sectioning.

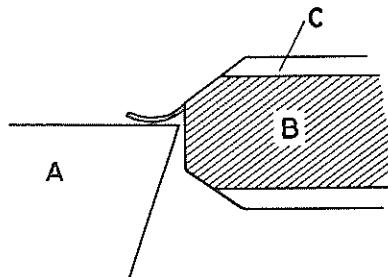


FIGURE 2. Close-up view of knife and specimen. A is the knife, B the plastic-treated clay specimen and C external plastic.

microtome was equipped with a diamond knife and the liquid in the collecting trough consisted of 10 percent acetone solution. The sections, which had a thickness of 400-700 Å, were placed on carbon-coated 150 mesh grids.

The author's investigations were made with a Siemens Elmiskop I which belongs to the Department of Histology, University of Lund. The microscope, which was equipped with a platinum-iridium aperture of 50 μ in the objective lens, was operated at 80 kV.

Ocular inspection took place at a magnification of 400 000 × at maximum and the thin sections were photographed, the micrographs being taken at random. By identification of the rectangular shape of the sections, their orientation in relation to the horizontal plane *in situ* was determined.

The electronic magnification during exposure was 5 000 - 10 000 \times depending on the size of the object. The plates used were Ilford "Special lantern".

With this procedure almost all the thin sections obtained from pure clay can be used. When the diamond edge meets coarser particles being harder than the embedding plastic substance, these particles are sometimes pushed up in front of the edge or are cut loose. This may cause a change in the clay structure in the vicinity of the coarse particles, but by observing the cutting process through the eye-piece of the microtome, it is possible to select only those sections which appear to be intact.

The acrylate method was tested for the first time on soft, illitic clays from Skå-Edeby in 1963. The results of this investigation showed that valuable information concerning microstructural features could be obtained from micrographs of ultra-thin sections of acrylate-treated clay if the original structure is preserved.

TEST PROGRAMME

GENERAL

The introductory studies of the Skå-Edeby clays were followed by an investigation concerning the influence of the preparation method on the microstructure of clays. This investigation comprised the following parts :

1. Determination of the change of specimen dimensions due to the acrylate preparation. In this investigation clay samples from Måltorp (Västervik), Kollahed (Kungsbacka) and Morjärv were used. They represent clay types with largely different properties.
2. Comparative study of electron micrographs of the Skå-Edeby clay using several plastic substances (acrylate, Epon and Durcupan). The ultra-thin section technique was employed.
3. High-voltage electron microscopy.
 - a. Investigation of Burgsvik (Gotland) clay with its natural water content.
 - b. Investigation of polyethylene glycol (Carbowax)-treated samples of Burgsvik clay.
 - c. Investigation of polyethylene glycol-treated samples of Skå-Edeby clay.
4. Analysis of micrographs with reference to artifacts caused by acrylate plastic treatment and microtome operation.

MÉMOIRES ORIGINAUX

CLAY TYPES

Table 1 contains some characteristic geotechnical data of the investigated clays. A full report on the relationship between the microstructure and the geotechnical properties of these clays, which have a negligible content of expanding clay minerals, will be given in a separate publication.

The soft, normally consolidated sediments in the Skå-Edeby area, about 25 km west of Stockholm, were formed during Quaternary glacial and postglacial times in stagnant water with low content of dissolved oxygen. The 10-12 m thick illitic clay layer sequence, which was deposited in fresh or brackish water, contains varves of iron sulphide throughout the whole profile. The lower 5 m thick stratum of glacial clay shows yearly deposited varves.

The samples of Burgsvik clay were taken from layers of very stiff blue-grey plastic clay in a sandstone bed of the Upper Silurian Burgsvik Formation at Kättelviken. These illitic clay layers, which were originally deposited in shallow salt water, are heavily overconsolidated.

The Måltorp clay is similar to the glacial Skå-Edeby clay, while the Morjärv clay bears resemblance to the postglacial Skå-Edeby clay. The Kollahed clay represents the soft marine clay deposits which are common in western Sweden.

TABLE 1
Clays investigated

Clay	Depth m	Unit weight g/cm ³	Shear strength (cone test) kg/cm ²	S _c	w %	w _L %	w _P %	Clay content %	Description
Skå-Edeby	2.0	1.43	0.15	10	107	123	40	58	Green-grey muddy clay
Skå-Edeby	5.0	1.48	0.09	20	105	98	29	80	Grey clay
Skå-Edeby	8.0	1.60	0.11	18	73	57	23	59	Brown-grey clay
Burgsvik	0.5	2.20	~ 50 (unconf. compr. test)	-	12	43	22	18	Grey silty clay
Måltorp	6.0	1.59	0.19	11	75	76	-	-	Brown-grey clay
Kollahed	13.0	1.78	0.17	90	45	34	-	-	Grey (sulphide) clay
Morjärv	4.0	1.25	0.13	26	198	157	79	19	Black clayey sulphide mud

STATISTICAL DESCRIPTION AND EVALUATION OF CLAY MICROSTRUCTURE

Direct comparison between micrographs illustrating clay microstructure necessitates a description based on statistics. A simple method, which involves determination and description of the size distribution of the pores and of the total pore area (P) in percent of the total area (T) of the studied thin section, is generally suitable (cf. Pusch, 1967). The dark details in the micrographs represent the solid phase while the bright fields represent the pore system. The blackness of the dark parts is of special importance in a detailed interpretation of the micrographs. Thus, the angle of inclination between the plane of sectioning and the crystallographic ab-plane of flat phyllosilicate particles can be estimated by observing their blackness, which is a function of the absorption of the electron radiation. For instance, very dark, needle-shaped images of the particles indicate that they are oriented perpendicular to the plane of sectioning.

The dimensions of the pores are defined in accordance with the author's concepts used for clay particles (Pusch, 1962) but in the present investigation only the longest intercept (a_p) was measured (Figure 3). Since the pore image represents a cross section without reference to the real extension and orientation of the pore, the dimension a_p only describes the sectioned part of the pore. Because of the shallowness of

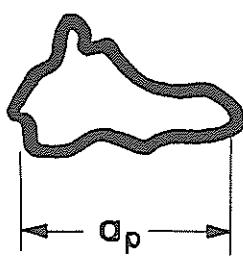


FIGURE 3. Definition of pore size.

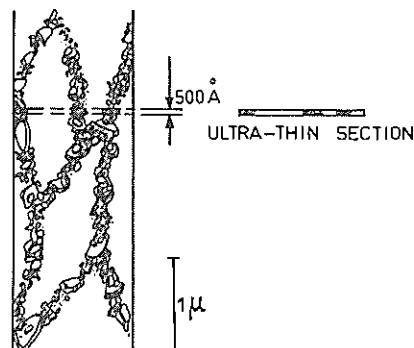


FIGURE 4. Schematic picture of an ultra-thin section of a clay particle network.

the clay section, the microstructural image almost corresponds to a two-dimensional section through the natural clay mass (Figure 4). The determination of the pore area is carried out by means of a planimeter.

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All the measurements are based on drawn images of the micrographs in which no discrete particles are depicted. Pores extending outside the micrographs' edges are measured as if they have this truncated shape.

A histogram of a_p is illustrated in Figure 5. It represents the micrograph and the corresponding drawn image of the acrylate specimen partly shown in Pl. II. The class intervals agree in the main with the ones used by the author in the graphical representation of clay particle size and shape.

Some typical micrographs of acrylate-treated Skå-Edeby clay are shown in Pl. I. Dense flocks or large particles connected by groups or chains of small particles (Pl. I, fig. c) is a characteristic feature of this clay although it is much more pronounced in marine clays.

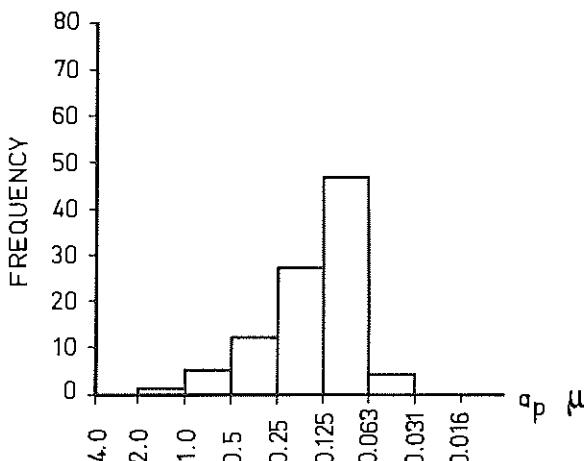


FIGURE 5. Histogram of a_p , corresponding to a micrograph of acrylate-treated clay, which is partly shown in Plate II.

TEST DESCRIPTIONS AND RESULTS

1. CHANGE OF SPECIMEN DIMENSIONS DUE TO ACRYLATE PREPARATION

Volume change due to acrylate treatment of clay specimens is a measure of the structural alteration caused by the treatment. Three different clays were used in an investigation concerning the change of specimen dimensions, namely 1) a fresh- or brackish-water (deposited) clay from south-eastern Sweden (Måltorp), 2) a quick, salt-water clay from the Swedish westcoast (Kollahed) and 3) an organic brackish-water clay (svartmocka) from north-eastern Sweden (Morjärv).

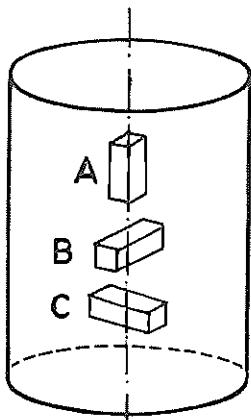


FIGURE 6. Extraction of differently oriented specimens for precision measurement of dimension changes during acrylate treatment. The cylindrical body is a vertically taken Ø 50 mm Swedish standard soil sample.

Specimens of about the same dimensions as in the standard preparation technique were cut in the manner shown in Figure 6.

By using a micrometer-screw the long dimensions of the specimens A, B and C of each clay type were measured before, during and after the acrylate treatment. Air-dried specimens were measured as well. The results of the investigation are given in Table 2. It can be concluded that the acrylate-plastic treatment caused a very small change in the dimensions of the specimens, compared with the effect of drying. The tendency of the specimens to swell during the alcohol treatment and in the polymerization process can be assumed to have had a negligible influence on the microstructure.

From the measurements of the air-dried specimens, it can be seen that the shrinkage was almost the same in the vertical and horizontal directions. This indicates an isotropic type of microstructure in these clays.

A separate investigation of a specimen of the Burgsvik clay was made by means of X-ray diffraction technique. An undisturbed specimen was investigated after water saturation and after ethyl alcohol treatment according to the standard procedure. The pronounced particle alignment in this clay gave very clear reflection patterns. The investigation gave identical peak distances and reflection intensities in the range of the investigated 2θ -values, $5-25^\circ$, indicating no measurable influence on the natural microstructure by the alcohol treatment.

2. COMPARISON WITH OTHER EMBEDDING SUBSTANCES

Plastic embedding techniques using Epon epoxy resin (Epon 812, Shell Chemical Company) and Durcupan (Fluka AG, Chemische Fabrik)

TABLE 2
Change of specimen dimensions due to acrylate preparation

Sample	Alcohol treatment		Acrylate treatment (monomer)		Acrylate treatment (polymerized)		No treatment		Remarks
	Fresh cut	Treated	Alcohol satur.	Treated	Fresh cut	Hardened	Fresh cut	Air dry	
	Change in percent of fresh cut specimen	mm	Change in percent of fresh cut specimen	mm	mm	mm	mm	mm	
184	A	14.96	+ 1.2	15.14	+ 0.3	9.87	+ 2.2	8.79	- 15.8
	A	15.07	+ 0.5	15.15	+ 0.3	9.44	+ 7.6	10.02	- 15.1
	B	15.85	+ 0.7	15.96	- 0.1	10.50	- 0.5	10.41	- 14.3
	B	-	-	-	-	9.64	+ 5.7	10.16	- 14.2
	C	17.46	+ 0.9	17.61	+ 0.9	10.45	+ 8.9	9.10	"
	C	-	-	-	-	8.28	+ 4.7	9.13	- 11.7
Average		+ 0.8	Average	+ 0.4	Average	+ 3.0	Average	+ 3.0	Average - 13.4
5076	A	17.88	+ 0.7	18.00	+ 0.3	9.74	- 2.9	10.06	- 8.8
	A	16.69	+ 1.0	16.85	0	9.21	- 1.2	8.85	- 8.0
	B	17.64	+ 1.5	17.90	+ 1.0	10.07	+ 6.6	10.57	- 8.6
	B	-	-	-	-	10.68	+ 3.3	10.39	- 7.2
	C	17.75	+ 0.3	17.80	- 0.2	6.41	+ 4.1	9.68	- 10.7
	C	-	-	-	-	8.27	- 4.8	9.54	- 7.5
Average		+ 0.9	Average	+ 0.3	Average	+ 0.9	Average	+ 0.9	Average - 8.5
2089	A	11.71	+ 1.0	11.83	+ 0.2	-	-	9.20	- 25.5
	A	11.28	+ 2.5	11.56	+ 1.8 *	9.31	+ 3.7	10.41	- 25.8
	B	9.66	+ 2.8	9.93	+ 1.9	8.40	- 1.2	7.65	- 23.0
	B	-	-	-	-	-	-	8.02	- 20.4
	C	9.67	+ 3.5	10.01	+ 2.5	8.92	- 6.4	8.62	- 20.9
	C	-	-	-	-	7.46	- 10.8	7.68	- 22.8
Average		+ 2.5	Average	+ 1.6	Average	- 3.2	Average	- 3.2	Average - 23.1
Average of all tests		+ 1.4	Average of all tests	+ 0.7	Average of all tests	+ 0.6	Average of all tests	+ 0.6	Average of all tests - 15.0

* indicate swelling — indicate shrinkage.

have been used on Skå-Edeby clay samples to provide a check on the acrylate method.

The Epon treatment is as follows :

1. Replacement of water by ethyl alcohol by the same procedure as in the acrylate method.
2. The specimen is kept in propylene oxide for 15 minutes. This treatment is repeated once.
3. The specimen is stored for 1 hour in a mixture of 50 percent propylene oxide and 50 percent (Epon + DDSA) / (Epon + NMA). The Epon + DDSA fraction consists of 62 ml Epon and 100 ml DDSA (dodecetyl succinic anhydride) and the Epon + NMA fraction consists of 100 ml Epon and 89 ml NMA (nadic methyl anhydride). The ratio between the Epon + DDSA and the Epon + NMA fractions is 3 : 1.
4. The specimen is stored in 100 percent (Epon + DDSA) / (Epon + NMA) for 5 hours. An aromatic tertiary amine accelerator, DMP - 30 (2, 4, 6 - tri [dimethylaminomethyl] phenol) is added to 2 percent by volume.
5. Embedding of the specimen in the mixture mentioned in (4). The specimen, which is kept in a gelatine capsule, is incubated for 12 hours at 35 °C, for 12 hours at 45 °C and for 6 days at 60 °C.

The Durcupan treatment, which was applied to specimens not subjected to alcohol, is as follows :

1. The freshly cut specimen is placed in a solution of 30 percent distilled water and 70 percent Durcupan, component A, for 30 minutes.
2. The procedure in (1) is repeated with 10 percent distilled water and 90 percent Durcupan, component A.
3. The specimen is placed in 100 percent Durcupan, component A, for 45 minutes. This procedure is repeated once.
4. Embedding of the specimen in Durcupan consisting of the components A, B, C and D in ratio 1 : 2.34 : 0.65 : 0.24. The specimen, which is kept in a gelatine capsule, is incubated for 12 hours at 4 °C and for 48 hours at 40 °C.

Thin sections of the same thickness and quality as in the acrylate method were cut from the Epon-treated specimens. The hardness of these specimens was comparable with that of the acrylate-treated specimens, whereas the Durcupan-treated specimens were considerably softer. Consequently, the majority of the Durcupan sections were distorted by the microtome operation.

MÉMOIRES ORIGINAUX

In all cases, the trimming and cutting procedures as well as the mounting operations were observed in oculars. Thus, it was possible to select those sections that could be used for microscopic investigation.

By using the Siemens Elmiskop I microscope, a series of micrographs were taken of the Skå-Edeby clays treated with the different plastic substances (Pl. II). The statistical treatment of the images drawn from the micrographs gave the values in Table 3. Each value was calculated on the basis of 3 micrographs. A detailed description of the micrographs, which represent sections taken parallel to the vertical plane *in situ*, will be given in a separate report.

TABLE 3
*Statistical parameters of the pore dimension a_p in μ and $\frac{P}{T}$ in percent,
determined from ultra-thin sections of Skå-Edeby clay*

Sample, depth	Acrylate			$\frac{P}{T}$ %	Epon			$\frac{P}{T}$ %	Durecupan			$\frac{P}{T}$ %			
	a_p in μ				a_p in μ				a_p in μ						
	M	UQ	LQ		M	UQ	LQ		M	UQ	LQ				
2570 2 m	0.21	0.34	0.14	17.4	-	-	-	-	-	-	-	-			
2853 5 m	0.11	0.20	0.08	9.4	0.22	0.37	0.13	28.5	0.19	0.32	0.14	17.5			
3217 7 m	0.13	0.19	0.10	13.8	0.22	0.35	0.17	19.2	-	-	-	-			
3700 8 m	0.24	0.40	0.18	21.5	0.15	0.25	0.11	24.9	-	-	-	-			
3917 9 m	0.20	0.31	0.14	24.9	0.13	0.19	0.10	14.4	-	-	-	-			
4046 10 m	0.19	0.32	0.11	11.3	0.12	0.20	0.10	15.4	-	-	-	-			
Averages ¹⁾	0.18	0.29	0.13	16.4	0.17	0.27	0.12	20.5							

M = median value.

UQ = upper quartile.

LQ = lower quartile.

1) Arithmetic mean of the median and quartile values respectively.

The median values of the pore size for the acrylate-treated specimens indicate that the clay matrix of the soil profile at Skå-Edeby is fairly uniform as regards the microstructure, although both organic post-glacial clay and varved glacial clay are represented. Although this matter is very interesting, a general discussion would be beyond the scope of this work. The discussion will therefore be confined to the variation of a_p and $\frac{P}{T}$.

According to the results from the analysis of the acrylate-treated clay, the median values (0.18μ as an average) are found in the range

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of $0.11 - 0.24 \mu$. The Epon-treated clay gave median values (0.17μ as an average) within the range of $0.12 - 0.22 \mu$.

Acrylate treatment gave a minimum of the median value in the specimens 2853 and 3217, whereas Epon treatment gave a maximum of the median value in these specimens. This discrepancy is probably accidental. In fact, the median value of a_p varies within the range of about $0.10 - 0.25 \mu$ in the profile.

Also, the quartile values are of the same order in the differently treated specimens.

As regards the $\frac{P}{T}$ -values, the variation is similar to that of the a_p -values. It can be assumed that $\frac{P}{T}$ varies within the range of about 9-30 percent. Experience shows that this rather narrow range is typical for this fresh- or brackish-water clay and quite different from those valid for highly organic or marine clays.

It can be concluded that the acrylate- and Epon-treated clays show similar microstructural patterns. This is also the case with the Durcupan-treated clay. However, the Durcupan treatment was not successful in most cases, only one specimen (2853) being hard enough to permit sectioning with the microtome.

3. HIGH-VOLTAGE ELECTRON MICROSCOPY

By courtesy of Professor Gaston Dupouy, Toulouse, Head of the Laboratoire d'Optique Electronique du Centre National de la Recherche Scientifique, Toulouse, France, the author had the opportunity of studying sections of clay in a high-voltage electron microscope. In this microscope, which can operate at a potential up to 1.5 MV, the penetrability of the electron radiation is much greater than in ordinary electron microscopes. This property offers a possibility of studying comparatively thick specimens as well as specimens enclosed in a cell. Using the cell, in which an air pressure of 1 atmosphere can be kept, $1-10 \mu$ thick sections of clay at the natural water content can be investigated in the microscope. Unfortunately, such thin clay specimens cannot generally be cut without severe disturbance. Soft clay, for instance, cannot be used for this reason but thin slices peeled from a laminated stiff clay can be studied successfully.

Moist specimens of the stiff Burgsvik clay were chosen for microscopic investigation. Since acrylate-treated specimens of this clay had been studied previously in an ordinary electron microscope, a direct comparison could be made.

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The great penetrability of the electron radiation also made possible an investigation of specimens of soft clay from Skå-Edeby, treated with polyethylene glycol (Carbowax 6000) according to Mitchell's (1956) method. Carbowax has been used frequently in X-ray analyses of clay microtexture and it has been that the treatment does not affect the microstructure (Martin, 1965). Also, Carbowax-treated specimens of the Burgsvik clay were studied.

Due to the softness of the embedding substance, microtome-cut sections of specimens saturated with Carbowax cannot generally be thinner than $1 - 5 \mu$ without being distorted. A section thickness of this magnitude means that ordinary electron microscopes cannot be used.

The electron microscope has been described in a series of publications (Dupouy and Perrier, 1960; 1963). For the author's investigations, the microscope was equipped with an aperture of 30μ in the objective lens. The residual air pressure in the microscope column was 10^{-5} torr and the voltage 1 MV. The plates used were Ilford "Special lantern contrasty". In all the tests, except the ones where the closed cell was used, the thin sections were placed on 200 mesh copper grids covered with a zapon/carbon film with a thickness of about 500 Å. Data concerning the preparation and investigation in the high-voltage microscope are given in Tables 4, 5 and 7.

Ocular inspection at magnifications of $10\,000 - 150\,000 \times$ was made and the sections were photographed, the micrographs being taken at random. The electronic magnification was $6\,000 \times$ in the study of Carbowax-treated clays. Since the absorption of the electron radiation was high in the penetration of the closed cell, the upper limit of magnification was $3\,500 \times$ in the study of the moist clay.

A Porter-Blum microtome equipped with a glass knife was used in the author's investigations. The sections were made as thin as possible in each case in order to facilitate a study of microstructural details.

a. MOIST BURGSVIK CLAY

The preparation of the untreated, moist Burgsvik clay commenced with a trimming of truncated pyramids. This operation, as well as the subsequent cutting, was performed rapidly in order to prevent water loss. The cutting procedure did not involve any real shearing in this case, it was merely a question of peeling off thin laminae. The slices obtained were transferred directly to the small cell ("microchambre") which, after having been closed, was placed in the microscope for investigation (Figure 7). The 0.1μ thick supporting glass film on which the slices rested in the cell, was cooled to about 15°C during the mounting procedure in order to reduce water evaporation. The basin of the cell con-

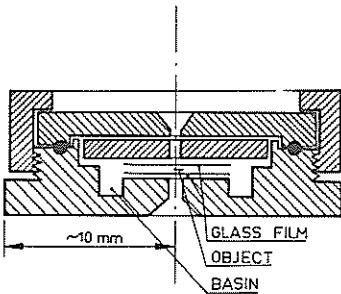


FIGURE 7. Schematic cross section of the cell for investigation of moist clay. The basin of the cell is partly filled with water.

tained distilled water and it was assumed that, owing to the humid conditions in the enclosed space, no great change of the moisture content of the clay occurred during the microscopic investigation. Thus, it was assumed that the moist clay was studied in its natural state since water content and air pressure were the same as in this state. All the sections of the moist Burgsvik clay had to be taken parallel to the known direction of the particle orientation, e.g. horizontally *in situ*. The thickness, which was the same for all the sections, was estimated at 1-10 μ . Table 4 contains basic preparation data of the micrographs used in a statistical investigation concerning the size of the micropores. For this purpose, images of the micrographs were prepared as in the investigation mentioned previously. Some representative micrographs are shown in Pl. III, IV, V and VI.

TABLE 4
Pore size parameters of the moist Burgsvik clay

Micrograph	a_p in μ			$\frac{P}{T}$ %	Electronic magnif. (times)	Microtome operation	Cell number
	M	UQ	LQ				
12165	0.42	0.70	0.35	8.9	3500	Thin sections picked from the microtome knife.	59
12167	0.60	1.00	0.50	8.9	3500		59
12175	0.42	0.67	0.35	9.9	2500	Thin sections taken from the liquid in the microtome trough.	61
12177	0.35	0.63	0.25	10.2	2500		61
Averages	0.45	0.75	0.36	9.5			

b. CARBOWAX-TREATED BURGSVIK CLAY

The Carbowax treatment was made according to Mitchell's procedure. The preparation of the Carbowax-treated specimens commenced with a trimming of truncated pyramids that were embedded in paraffin. The paraffin was trimmed to conform with the internal pyramidal shape.

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All the sections were taken parallel to the vertical plane *in situ*, this being the standard plane of sectioning in all sedimentary clays. The thickness, which was the same for all the sections, was estimated at somewhat more than 1 μ .

Due to the paraffin coating, the thin sections of the Carbowax-treated clay were surrounded by paraffin frames, the stiffening effect of which made possible the sectioning perpendicular to the plane of stratification (Figure 8).

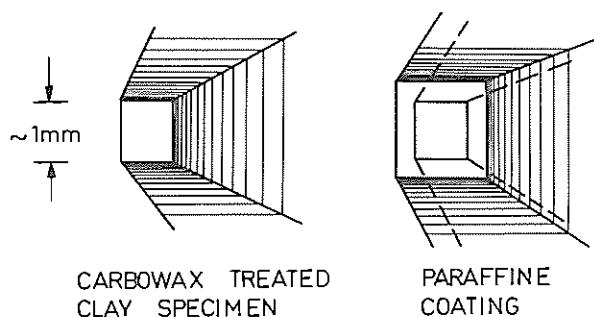


FIGURE 8. Schematic picture of trimmed Carbowax-treated clay specimen. Right picture shows the specimen after trimming of the paraffin coating. Sectioning in the microtome gives slices with paraffin frames.

Table 5 shows some basic preparation data and the statistical parameters of some representative micrographs. Two of these are shown in Pl. VII and VIII.

TABLE 5
Pore size parameters of the Carbowax-treated Burgsvik clay

Micrograph	a_p in μ			P T %	Electronic magnif. (times)	Preparation	Microtome operation
	M	UQ	LQ				
12142	0.34	0.48	0.25	12.4	6000	Paraffin treatment by repeated dipping of the trimmed clay pyramids in liquid paraffin.	Thin sections taken from the liquid in the mierotome trough.
12148	0.55	0.95	0.32	10.8	6000		
12149	0.24	0.45	0.20	14.7	6000		
12150	0.24	0.37	0.17	7.6	6000	Trimming of the paraffin envelope after solidification.	
Averages	0.34	0.56	0.24	11.4			

For comparison, micrographs of previously studied ultra-thin sections of acrylate-treated Burgsvik clay were also analysed. The sections had been cut with the LKB microtome and investigated in the Siemens microscope, following the standard procedure for acrylate-treated specimens. All the sections were taken parallel to the vertical plane *in situ*. The thickness was of the order of 700 Å.

Table 6 contains the statistical parameters of the representative micrographs shown in Pl. IX, X and XI.

TABLE 6
Pore size parameters of the acrylate-treated Burgsvik clay

Micrograph	a_p in μ			P T %
	M	UQ	LQ	
15749	0.15	0.24	0.11	39.5
15751	0.24	0.40	0.19	37.5
15755	0.21	0.34	0.11	34.6
Averages	0.20	0.33	0.14	37.2

c. CARBOWAX-TREATED SKÅ-EDEBY CLAY

Samples from 2, 5 and 8 m depth were investigated. As in the previous case, the preparation of the Carbowax-treated specimens included paraffin embedding. However, in the cutting operation the paraffin frame was lost in most cases and a slight curling of the sections could not be avoided. The curling was partly eliminated by spraying distilled water on the zapon/carbon film before application of the sections. By the action of tension of the water, the thin slices were then stretched. The thickness of the sections, which were taken parallel to the vertical plane *in situ*, was estimated at $0.5 - 1 \mu$. Table 7 contains basic preparation data and statistical parameters of a number of representative micrographs. Such micrographs are illustrated in Pl. XII and XIII.

CONCLUSIVE COMMENTS ON a, b AND c

The investigation of the Burgsvik clay shows that the median and quartile values of the pore size are somewhat higher (0.45μ as an average of the median values) for the moist specimens than for the Carbowax-treated specimens (0.34μ). This discrepancy is explained by the somewhat greater thickness of the moist sections in which a larger number of small pores were concealed and, to a lesser extent, by the different orientations of the cutting planes in this layered type of structure. Also, these factors explain the somewhat higher average value of $\frac{P}{T}$ of the Carbowax-treated specimens.

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TABLE 7
Pore size parameters of the Carbowax-treated Skå-Edeby clay

Sample	Micrograph	a _p in μ			$\frac{P}{T}$ %	Electronic magnif. (times)	Preparation	Microtome operation
		M	UQ	LQ				
4201 (2m depth)	12207	0.23	0.33	0.20	4.0	6000	Paraffin treatment according to Table 5. The paraffin envelope was generally lost in the cutting process.	Thin sections picked from the microtome knife. In certain cases a slight curling of the sections occurred. On placing these sections on the grid films, drops of water were first applied, permitting the sections to be stretched.
	12208	0.28	0.40	0.20	1.0			
	12211	0.24	0.35	0.13	13.7			
	12212	0.29	0.42	0.20	12.4			
	12219	0.32	0.41	0.25	5.8			
	12222	0.20	0.33	0.16	5.7			
	Averages	0.26	0.37	0.19	7.1			
2086 (5m)	12152	0.27	0.44	0.20	15.8			
	12156	0.22	0.35	0.18	7.7			
	12158	0.22	0.29	0.18	4.2			
2575 (8m)	Averages	0.24	0.36	0.19	9.2			
	12182	0.20	0.39	0.11	5.7			
	12184	0.16	0.23	0.12	2.5			
	12186	0.15	0.24	0.11	3.3			
	12190	0.24	0.68	0.16	7.1			
	12192	0.23	0.46	0.15	4.9			
	12193	0.23	0.45	0.13	5.5			
	12196	0.32	0.55	0.22	5.4			
	Averages	0.22	0.43	0.14	4.9			

The ultra-thin sections of acrylate-treated Burgsvik clay show even smaller values of the pore size, which is explained by the large number of revealed pores with very small dimensions. The value of $\frac{P}{T}$ is large in comparison to the moist and Carbowax-treated specimens. This is caused by the increase of the sectioned pore area due to the increased number of small pores revealed in the ultra-thin section.

The most interesting conclusion is that the same types of characteristic microstructural features are recognized in all three cases. Thus, the typical feather-like or, in some cases, domain-like arrangement which is common in the moist sections and which can also be identified in the Carbowax-treated clay, is very clearly seen in the acrylate-treated specimens.

Of equal importance is the typical feature of groups or chains of small particles forming links between dense flocks or between larger particles in all the micrographs (Pl. XIV and XV). The true existence of this microstructural arrangement in the natural state, which may be of primary importance as regards the physical properties of clay in bulk, is thus proved.

The median and quartile values of the pore size of the Carbowax-

treated Skå-Edeby clay are somewhat higher (0.24μ as an average of the median values) than those of the acrylate-treated clay (0.19μ for 2, 5 and 8 m depth, Table 3). This discrepancy is explained by the greater thickness of the Carbowax-treated sections. The higher average value of $\frac{P}{T}$ of the acrylate-treated sections is also explained by the different section thicknesses.

As in the case of the acrylate-treated specimens, the median values of the pore size indicate that the clay matrix is fairly uniform in the Skå-Edeby soil profile.

The investigations of the Burgsvik clay in the high-voltage microscope support the assumption that Carbowax treatment does not have any extensive influence on the original clay microstructure. Despite the discrepancy between the median values of the pore size in the acrylate- and Carbowax-treated Skå-Edeby sections, it can be concluded that the clay microstructure is similar in the two cases, which accordingly indicates that the acrylate treatment does not have any extensive influence on soft clay microstructure. In the micrographs of the acrylate-treated clays the particle orientation in the Skå-Edeby sediments is almost random, while large particles tend to be oriented parallel to the plane of sectioning in the Carbowax-treated clays. This is explained by the fact that the soft Carbowax was easily distorted when the plane of sectioning crossed large particles. The microtome operation was successful only in those cases where this plane coincided with the plane of orientation of almost parallel large particles. The same observation was also made in the study of the moist Burgsvik clay, in which case the plane of sectioning coincided with the known direction of particle orientation, e.g. the sedimentation plane.

Some of the micrographs of the moist and Carbowax-treated clays give the impression that the pore space contains a greater amount of small, dispersed particles than in the acrylate-treated clays, which might be due to a phase separation in the latter case. However, in comparison with acrylate-treated soft marine clays, which are characterized by a very distinct phase separation (Pl. XVI, fig. a), the acrylate-treated Skå-Edeby clay shows a much higher degree of dispersion which, in fact, corresponds to the low salinity in the sedimentation basin during the formation of this clay. Thus, acrylate treatment does not bring about a standard microstructure pattern, which indicates that the original distribution of small particles in the open parts of the particle network is probably not seriously affected. The deficiency of such particles in the acrylate-treated clays, in comparison to moist or Carbowax-treated clays, is only apparent and it can be explained by the much shallower depth of the acrylate sections.

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A more detailed analysis of the pore parameters in relation to the section thickness is of no value, owing to the uncertainty concerning the exact thickness of the sections and of the depicted structure within the sections in the direction of observation.

The observed resolution at the high-voltage microscopy was within the 10 - 20 Å range. Since the focal distance was 7.2 mm, the depth of field was greater than about 12 000 Å implying that all the details in the sections were in focus at once except in the thicker sections of the moist Burgsvik clay.

As can be seen from Tables 4, 5 and 7, several methods of transferring the sections from the microtome were used. The standard method of picking up the sections from the liquid in the microtome trough could not always be used because of the tendency of the sections to disintegrate in the liquid, which consisted of a water/acetone mixture or of plain water. In the preparation of the moist Burgsvik clay and of most of the other specimens, it was necessary to pick up the sections directly from the glass knife with a thin needle.

No diamond knife was available during the investigations in Toulouse.

No changes of the observed specimens occurred during the investigation in the high-voltage microscope. The objects were perfectly stable but the paraffin frames surrounding certain clay sections melted as soon as the electron radiation was applied.

4. ARTIFACTS CAUSED BY ACRYLATE PLASTIC TREATMENT AND MICROTOME OPERATION

Generally, the uniform white-greyish areas, which represent the plastic substance in the micrographs, occupy the complete space between particles in aggregates and in pores. However, certain micrographs show that the plastic has not filled the space completely. This is the case in Pl. XVI, fig. b where the incomplete replacement may be due to the existence of entrapped air or gas, as can be inferred from the smooth contour.

The diffusion processes in the preparation technique may have an influence on particles which are not rigidly linked in the particle network. Thus, the introduction of organic molecules creates new bonds by which particle rotation or displacement may be developed. In particular, the location of free or very loosely connected particles may be changed. The mutual particle arrangement at the periphery of the micropores may therefore be different from the arrangement in the natural state. Probably the pore size is influenced to a smaller degree.

A well-known disadvantage of the ultra-microtome technique is the deformation of the thin section caused by the cutting operation. Thus, according to Hirsch et al. (1965, pp. 39-42), a hard material deforms elastically whereas a soft material deforms plastically and may undergo slip on a fine scale. When soft embedding substances are used, such as the polyethylene glycol in Mitchell's method, such deformation often causes curling when the section is thinner than a few μ . However, the present investigations show that acrylate-treated clay sections with a thickness down to about 300 Å can be produced without any signs of curling or stepwise deformation if a diamond knife without defects is used.

Distortion of the thin section may be caused when the diamond edge meets coarse particles. In the case of particles or crystals with a layer structure having a plane of easy cleavage, the layers will generally be parted without distortion of the surrounding material if the cutting and cleavage planes coincide. This is regardless of the particle size. If the particles are not oriented in this manner they may be pushed up ahead of the edge and the thin section may be distorted. When the particles are smaller than about 0.2 μ such influence is generally very small. In many cases, even large clay mineral particles seem to have been parted by the knife although the cutting has not followed any cleavage plane. This is illustrated by Pl. XVI, fig. c and d.

Pushed or turned larger particles are easily detected in the micrographs. This is illustrated by Pl. XVI, fig. e in which some particles turned by the knife are seen. The bright part (hole) with distinct contours in the greyish acrylate material adjacent to the particles indicates their original positions.

The experience from the work which has been carried out so far, shows that artifacts caused by the microtome operation are easily identified.

CONCLUSIONS

The precision measurement of the specimen dimensions in the chemical preparation indicates that the change of the dimensions is so minute that it cannot probably be connected with any large microstructural change. The comparison between the acrylate, Epon and Durcupan embedding plastic substances, which have been used for several years in medicine for preparation of organic tissues, shows that the acrylate treatment does not have any special effect on clay microstructure. The most important conclusion from the high-voltage microscope investigations

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concerns the frequency of identical structural features in moist, and in Carbowax- and acrylate-treated specimens of the same clay. This similarity, which is also indicated by the statistical analysis of the pore size, shows that characteristic parts of the natural microstructural pattern are preserved in acrylate-treated clay specimens. Thus, although there is no definite proof that acrylate treatment does not influence illitic clay microstructure, all the tests support the assumption that the influence of such treatment cannot be large.

The mechanical preparation of ultra-thin sections produces artifacts. However, by using perfect diamond knives, a large number of good sections of specimens rich in clay can generally be obtained. The unavoidable distortions of the sections are easily recognized. The importance of first-class equipment and of extremely careful handling cannot be over-emphasized.

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SUMMARY

A technique for investigation of clay microstructure based on acrylate embedding and microtome sectioning is described. Small specimens of undisturbed clay were treated with ethyl alcohol and butyl/methyl methacrylate and subsequently polymerized, by which the water was replaced by the plastic substance through diffusion. The treated specimens were hard enough to permit sectioning by means of a microtome in slices about 500 Å thick.

In order to investigate the influence of the preparation technique on natural clay microstructure, a series of tests was carried out which comprised 1. precision measurement of the specimen dimensions during the chemical treatment, 2. comparative study of the effect of several plastic substances and 3. high-voltage electron microscopy. The last-mentioned test was carried out by using the 1.5 MV electron microscope at the Laboratoire d'Optique Electronique, Toulouse, France. The great penetrability of the electron radiation of this microscope permitted a study of

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Carbowax-treated specimens and certain moist specimens. A direct comparison of micrographs of moist and Carbowax-treated specimens and of Carbowax- and acrylate-treated specimens, respectively, indicated that the influence of acrylate treatment on natural clay microstructure is probably not large. This conclusion is also supported by the first-mentioned tests. As regards artifacts caused by the microtome operation, an analysis of the micrographs indicated that such effects are easily recognized.

RÉSUMÉ

L'auteur décrit une technique d'étude de la microstructure des argiles, basée sur l'imprégnation des échantillons, suivie d'une coupe au microtome. De petits spécimens d'argile brute sont traités par l'alcool éthylique, puis par du métacrylate de butyl-méthyl que l'on polymérise par la suite. L'eau contenue dans l'échantillon original est donc remplacée par la nature polymérisable, suivant un mécanisme de diffusion. Les échantillons ainsi traités sont assez durs pour fournir des coupes d'environ 500 Å d'épaisseur.

Pour déterminer l'influence de la technique de préparation sur la texture de l'argile, on a mesuré de façon précise les dimensions de l'échantillon pendant le traitement chimique; on a comparé l'effet de plusieurs substances utilisées pour l'imprégnation; on a employé la microscopie électronique à très haute tension. Ce dernier test a été rendu possible par l'emploi du microscope électronique de 1,5 MV du Laboratoire d'Optique Electronique de Toulouse (France). Le grand pouvoir pénétrant des électrons a permis l'étude des échantillons imprégnés au «Carbowax» et l'étude de certains échantillons en présence d'eau. Une comparaison des clichés obtenus dans ces deux derniers cas indique qu'il n'y a sans doute pas beaucoup d'effet du traitement acrylique sur la texture de l'argile (conclusion à laquelle les autres tests ont également permis de parvenir). En ce qui concerne les effets dûs au microtome, il est facile de les reconnaître par l'examen des micrographies.

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Plate I. — Micrographs of acrylate treated Skå-Edeby clay.

Fig. a. — Sample 2570 (2 m depth), Micrograph 15037; $\times 30\,000$.

Fig. b. — Sample 4046 (10 m depth), Micrograph 15051; $\times 30\,000$.

Fig. c. — Network of flocks and large particles connected by groups or chains of small particles (Micrograph 15037); $\times 30\,000$.

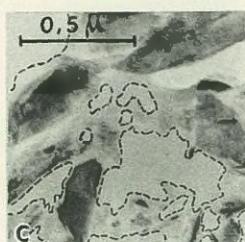


Plate II

Micrographs and drawn pore images of Skå-Edeby clay treated with different plastic substances. Sample 2853 (5 m depth).

Fig. a. — Acrylate-treated specimen. Micrograph 15,040; $\times 24,000$.

Fig. b. — Epon-treated specimen; $\times 25,000$.

Fig. c. — Durcupan-treated specimen; $\times 25,500$.

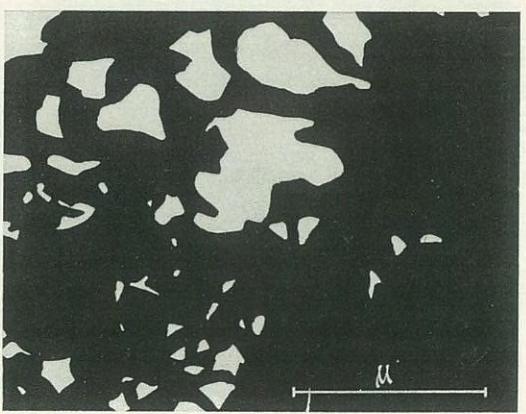
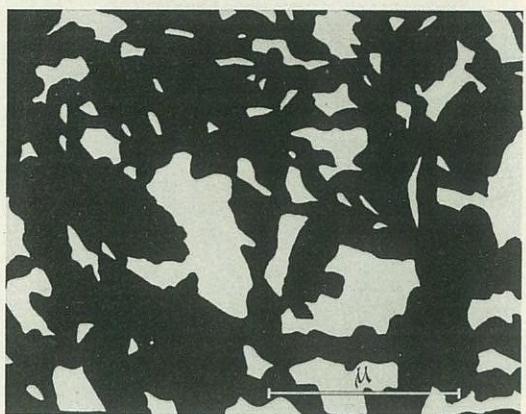
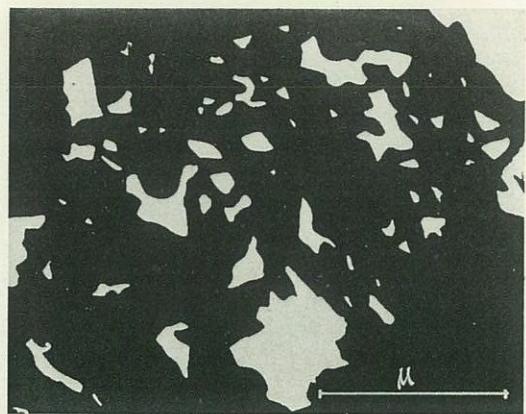


Plate III

Micrograph of moist Burgsvik clay. Micrograph 12,165; \times 8,500.



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Plate IV

Micrograph of moist Burgsvik clay. Micrograph 12,167; $\times 8,500$



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Plate V

Micrograph of moist Burgsvik clay. Micrograph 12,175; $\times 8,500$.



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Plate VI

Micrograph of moist Burgsvik clay. Micrograph 12,177; $\times 8,500$.



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Plate VII

Micrograph of Carbowax-treated Burgsvik clay. Micrograph 12,142; $\times 22,000$.



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Plate VIII

Micrograph of Carbowax-treated Burgsvik clay. Micrograph 12,150; \times 19,000.

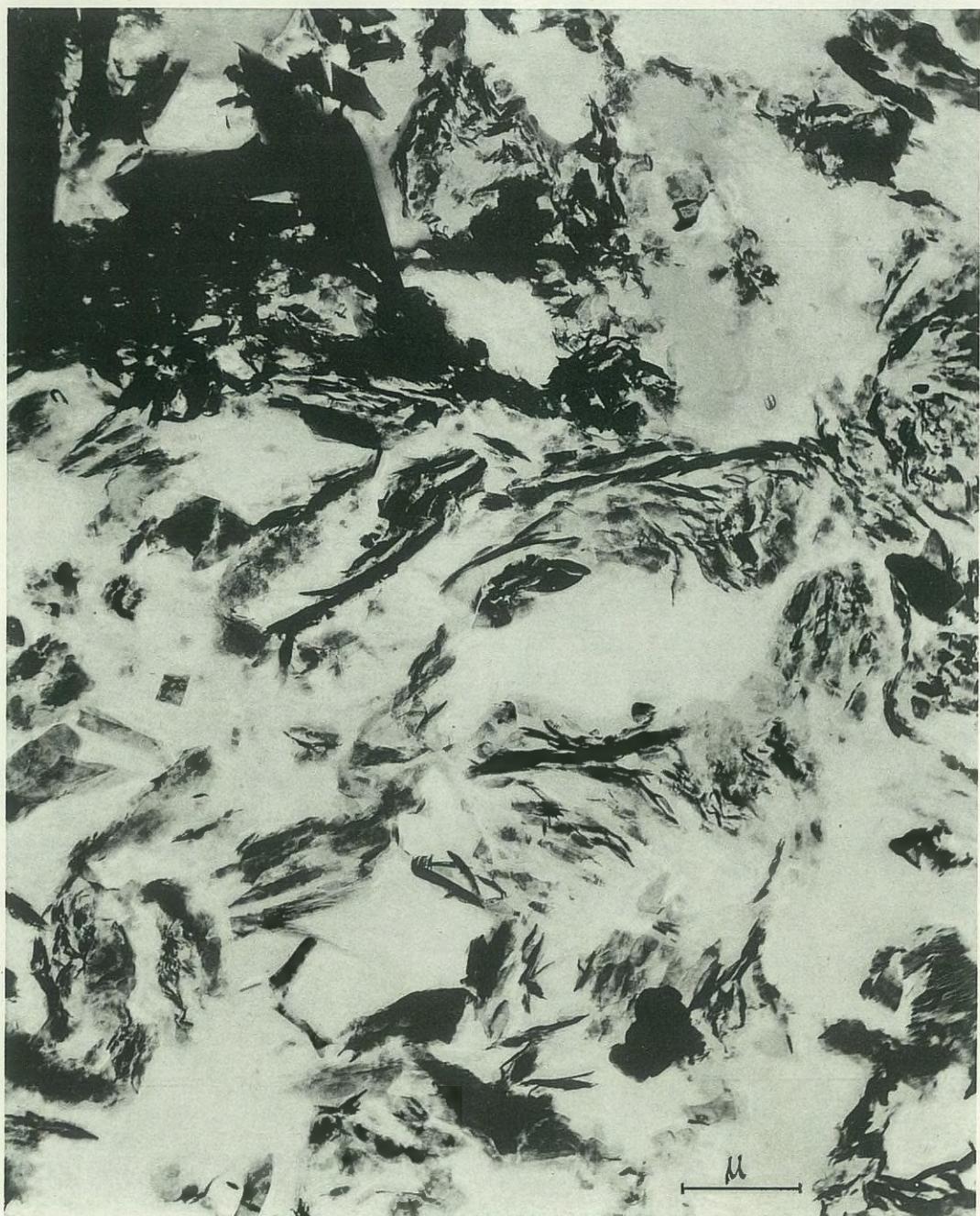


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Plate IX

Micrograph of acrylate-treated Burgsvik clay. Micrograph 15,749; \times 12,000.

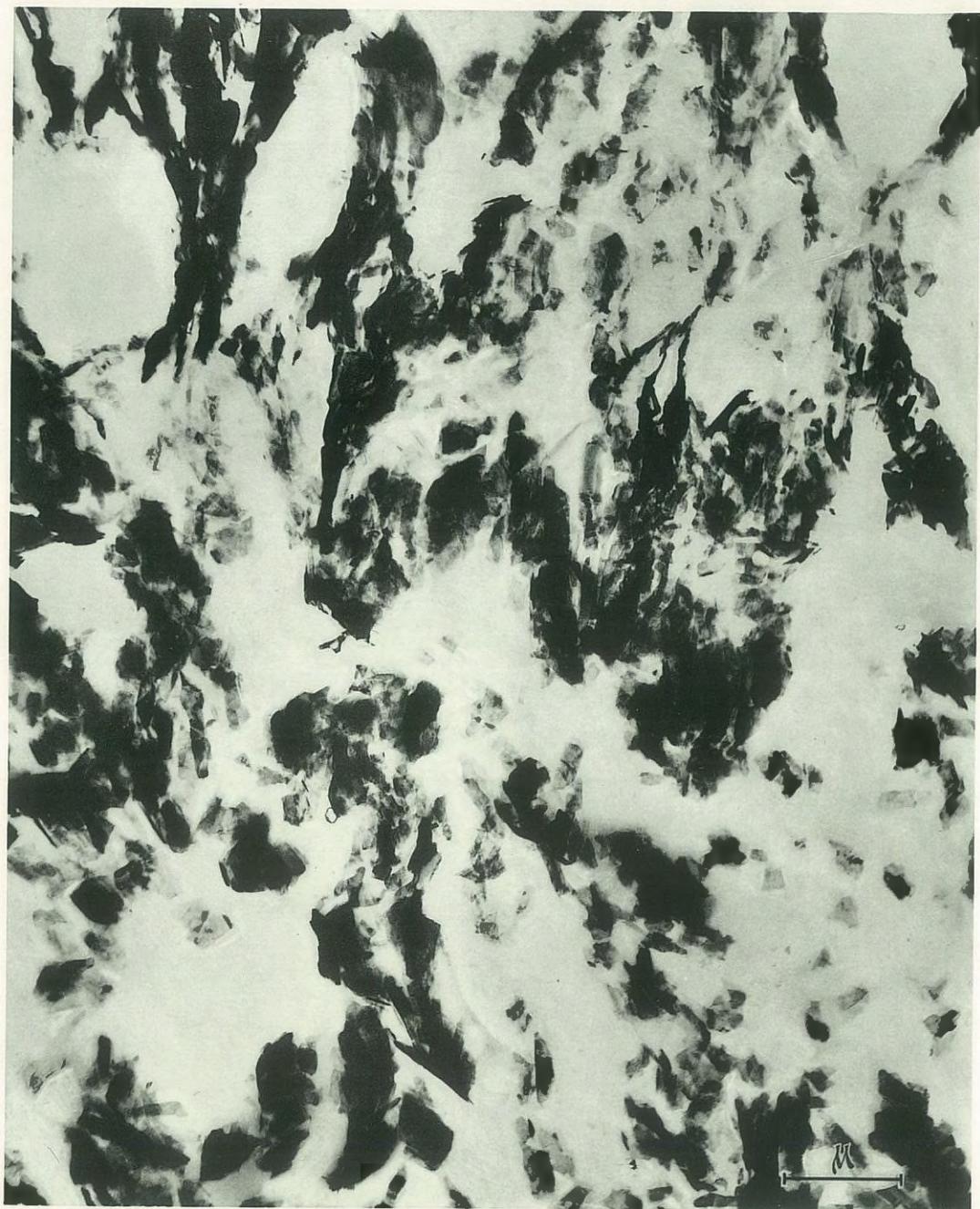


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Plate X

Micrograph of acrylate-treated Burgsvik clay. Micrograph 15,751; $\times 12,000$.

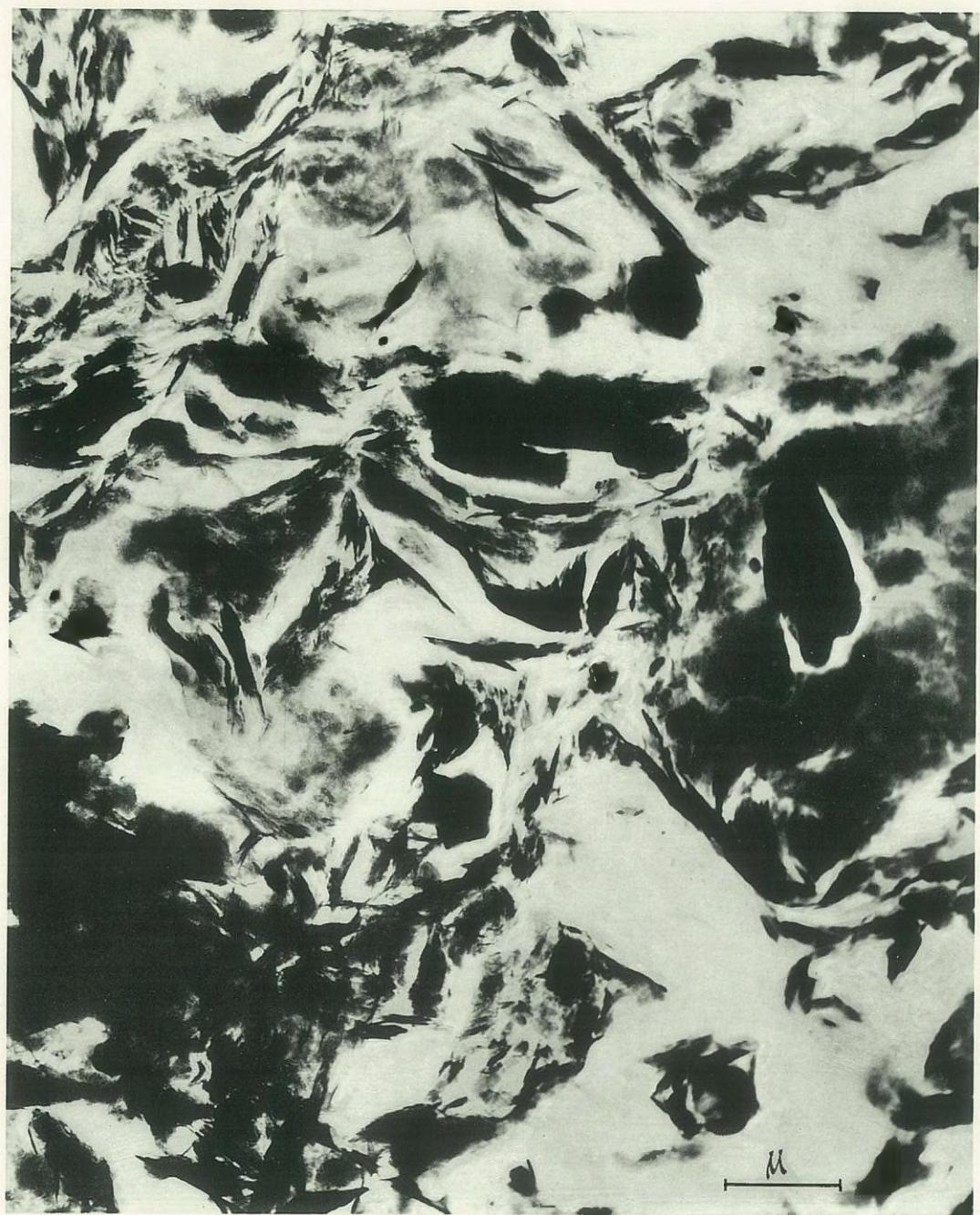


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Plate XI

Micrograph of acrylate-treated Burgsvik clay. Micrograph 15,755; $\times 11,500$.



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Plate XII

Micrograph of Carbowax-treated Skå-Edeby clay. Micrograph 12,207; \times 34,000.



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Plate XIII

Micrograph of Carbowax-treated Skå-Edeby clay. Micrograph 12,156; \times 34,000.



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Plate XIV

Micrographs showing large particles or aggregates linked by groups or chains of small particles. This is a common feature of clay microstructure.

Micrographs of Carbowax-treated Skå-Edeby clay :

Fig. a. — 12,207; \times 16,000.

Fig. b. — 12,158; \times 18,000.

Fig. c. — 12,186; \times 17,000.

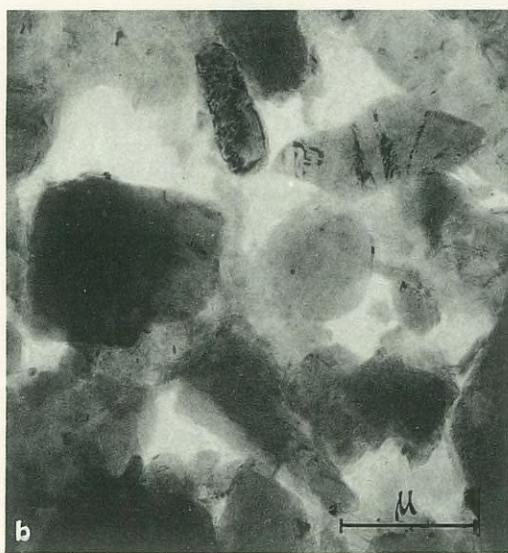
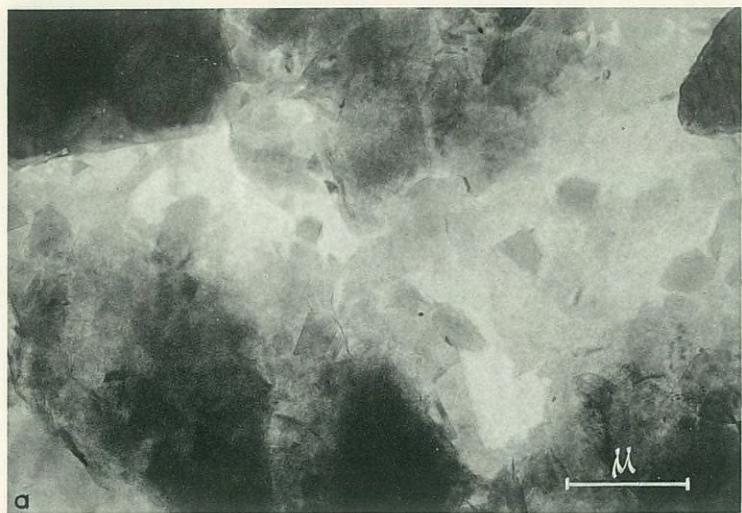


Plate XV

Fig. a. — Micrograph of Carbowax-treated Burgsvik clay. Micrograph 12,142; $\times 31,500$.
Fig. b. — Micrograph of moist Burgsvik clay. Micrograph 12,165; $\times 8,000$.



Plate XVI

Fig. a. — Typical micrograph of an acrylate-treated specimen of a marine clay; $\times 32,500$.

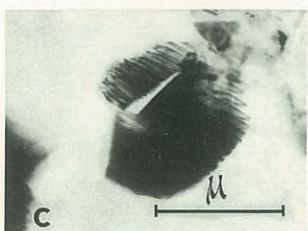
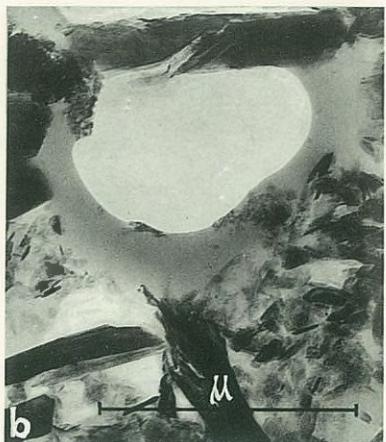
Fig. b. — Artifact caused by incomplete replacement of pore water with acrylate plastic. Micrograph 15,037; $\times 32,500$.

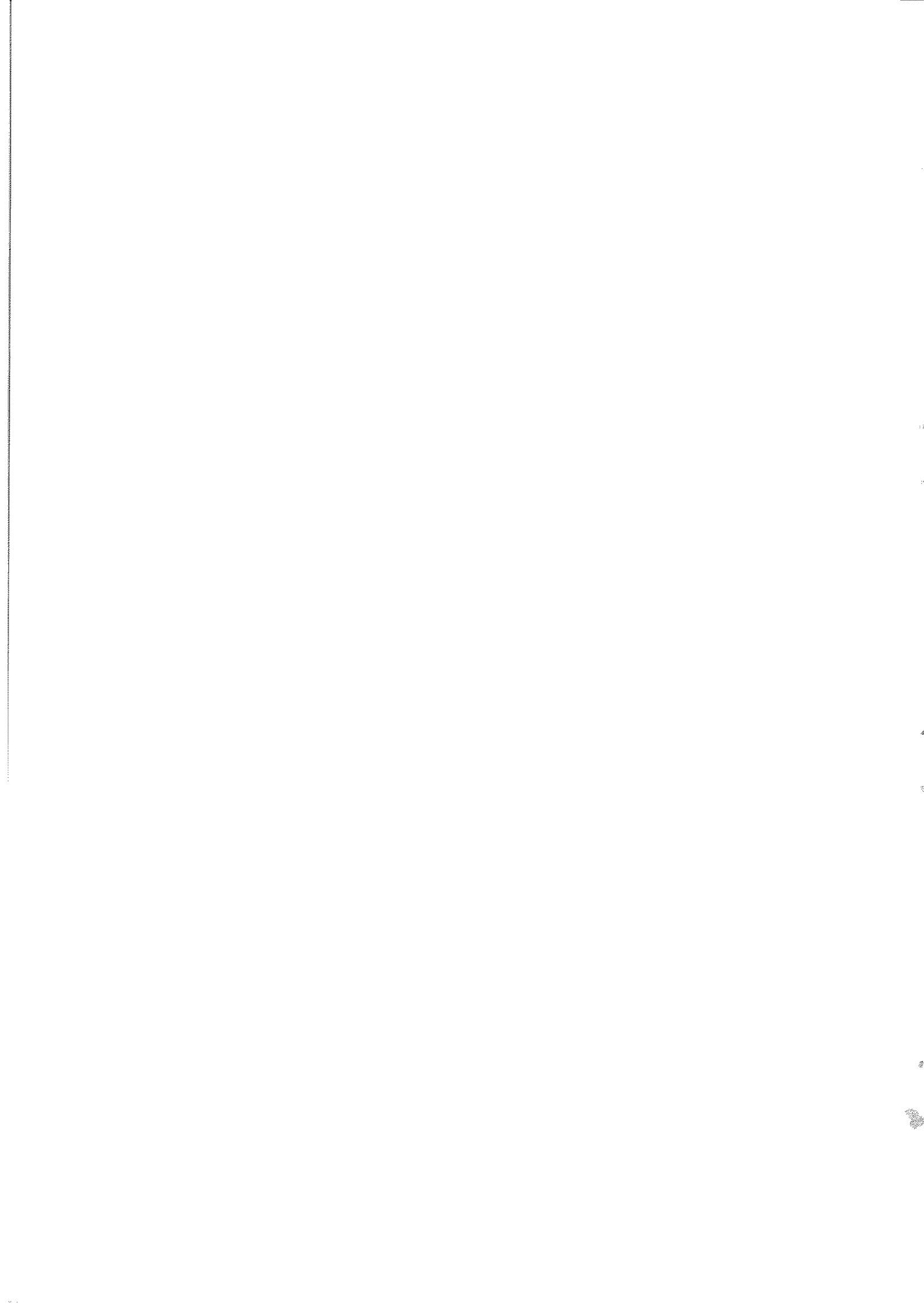
Fig. c and d. — Sectioned clay particles without distortion of the embedding substance.

Fig. c. — Acrylate-treated Skå-Edeby clay specimen from 8 m depth. Micrograph 15,032; $\times 16,500$.

Fig. d. — Acrylate-treated marine clay specimen; $\times 33,000$.

Fig. e. — Typical artifact caused by the microtome knife in the cutting operation. Acrylate-treated Skå-Edeby clay specimen from 5 m depth. Micrograph 15,040; $\times 16,500$.





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