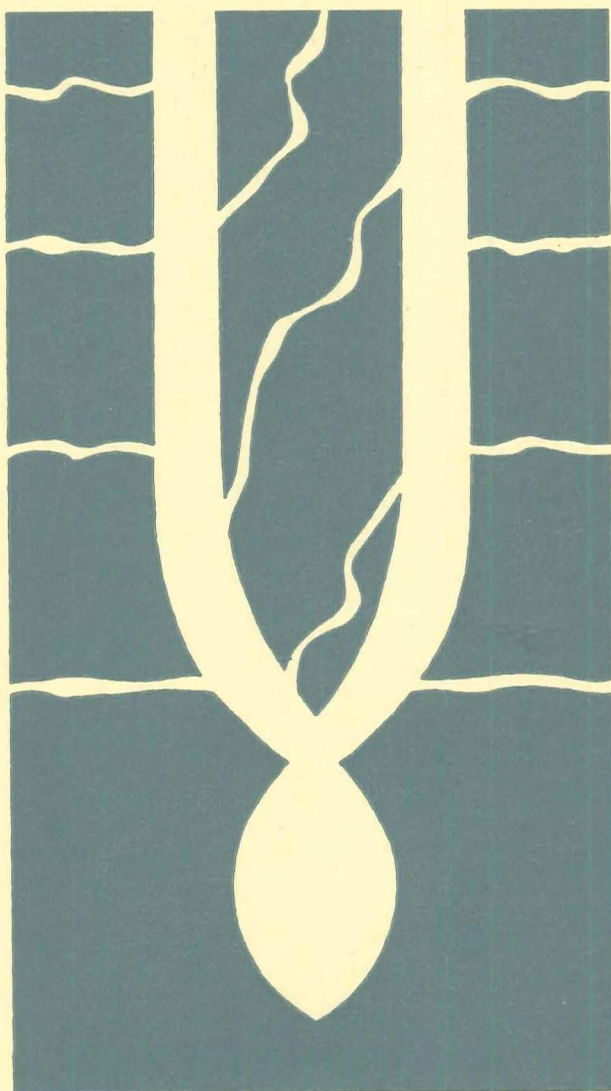


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# Soil Survey Papers, No. 10

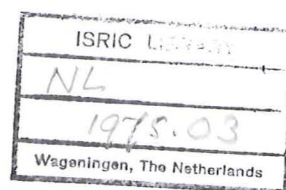
METHODS IN SOIL  
MICROMORPHOLOGY.  
A TECHNIQUE FOR THE  
PREPARATION OF LARGE  
THIN SECTIONS



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G. Heintzberger

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## PREFACE

Soil Survey Papers No. 1 ('The preparation of mammoth-sized thin sections') which was published in 1963, has been out of print for several years. There is, however, still a great deal of interest for this publication.

It was decided, therefore, to reprint this issue. The considerable changes in the methodology, however, which have taken place over the last few years, e.g. the application of freeze-drying and high-pressure impregnation and the introduction of new sawing and grinding equipment, compelled the authors to rewrite large parts of the text and to add a new chapter (concerning drying).

In order to emphasize these considerable changes, this monograph is published under a new title as a new volume in the Soil Survey Papers series.

The Director of the  
Netherlands Soil Survey Institute,

R. P. H. P. van der Schans



## 1. INTRODUCTION

The micropedologist's main aid in studying the soil is the thin section, viz. a very thin cross-section of the undisturbed soil obtained by grinding. A preparation of this kind must be of sufficient quality to allow accurate observations to be made at high microscopic magnifications.

Soil, being a soft and often friable material, needs to be artificially hardened to allow it to be ground. The technical basis for this hardening process was laid by Ross (1924). Many methods have since been developed. Most of them involve the principle of impregnating (usually in vacuo) an air-dried sample of undisturbed soil. The material used for this process must be on the one hand highly fluid during impregnation, while on the other it should afterwards become so hard at room temperature that the sample can be ground.

Over the years many materials or combinations of materials have been used as impregnants, amongst others: bakelite lacquer diluted with a mixture of 1 part ether and 1 part methyl alcohol (ROSS, 1924), Canada balsam (KUROTORI and MATSUMOTO, 1958), Canada balsam and shellac (VAN STRAATEN, 1951), plasto resin no. 15 and resinol no. 3 (VOLK and HARPER, 1939), resinol (ROTTER, 1941), lakeside 70 (DALRYMPLE, 1957), vernicolor (FREI, 1947; JONGERIUS, 1957) and kolloolith (KUBIENA, 1938; REDLICH, 1940; JONGERIUS, 1957).

Although fairly good results were obtained with these and similar hardeners, each of them has one or more properties limiting their suitability or adversely affecting the quality of the thin sections.

For example, disturbances may occur in the structure even during impregnation. Several hardeners are sufficiently fluid only at fairly high temperatures (of up to about 150° C). As a result the soil sample is 'cooked' in the impregnant. This is often accompanied by violent foaming, which can easily disrupt the microstructure of less coherent materials. A high temperature by itself, however, has never been found to affect the microstructure or morphology of the organic matter. At the same time intense heating causes the hardener to become gradually more brittle and it leads to a dark discoloration.

In addition to brittleness, impregnants may suffer from various other shortcomings, such as insufficient hardness, lack of impregnating power, lack of cementing power, a low melting point. These shortcomings may lead to various difficulties during grinding, such as the grinding away of sand grains, the contamination of thin sections by grinding powders, smearing, etc. In fact, it is practically impossible to make flawless slides which are thinner than 25 to 30  $\mu\text{m}$ , which is already too thick for many investigations. Another result of these deficiencies is that it is usually not possible to obtain thin sections larger than 3 cm square. Finally, a number of the conventional hardeners have a refractive index which differs considerably from that of Canada balsam, so that the thin sections prepared from them are less suitable for mineralogical research.

This outline of the problems, incurred with the different hardeners, should serve to indicate that the percentage of inferior thin sections and of failures was usually high, while the dimensions of the preparations were bound to limits which were practically impossible to exceed. There are especially great problems with sandy soils (disruption of the microstructure, removal of sand grains, etc.) and with very dense soils (insufficient impregnation).

Since the middle of the fifties, however, great improvements have been obtained with

cold polymerizing plastics, such as unsaturated polyester resins and some epoxy resins. Although BOURBEAU and BERGER (1947), HAGN (1953) and ALEXANDER and JACKSON (1954) amongst others had already used these materials, it was ALTEMÜLLER (1956, 1962) in particular who made a very important contribution towards the solution of many of the difficulties referred to above by introducing the unsaturated polyester resin Vestopal-H. However, the complete impregnation of dense samples requires a very elaborate treatment with his method, involving repeated exposures to impregnants. BORCHERT (1961, 1962) and KURON and HOMRIGHAUSEN (1959) solved this problem by using Plexigum 7466. Their sections however, are very thick and not suitable for detailed microscopic examination. Furthermore, Plexigum 7466 does not adhere to glass. The hardened samples must therefore be glued to thick (4 mm) plexiglass and left exposed after grinding. An important advantage of the Plexigum 7466 is that it permits the preparation of large sized sections. Initially, this appeared impossible with Vestopal-H; Altemüller's method was originally developed for sections 28 x 48 mm large. Using his method, large sections (so called Grosschliffe, 6 x 9 cm) can also be made, but these have the disadvantage of being quite thick (approx. 30  $\mu$ m). Here again, dense samples require repeated impregnation (ALTEMÜLLER, 1962).

Building on the work of Altemüller and also using Vestopal-H, JONGERIUS and HEINTZBERGER (1963) have developed a method for preparing thin sections as thin as 15  $\mu$ m (or less if required) and as large as 8 x 15 cm. These sections can be made at room temperature and from any type of soil, including the heaviest. On the one hand they provide an immediate picture of the macrostructure of a horizon, while on the other hand they allow a very detailed examination at the highest microscopic magnifications. The only defect of such mammoth-sized thin sections is that the hardened Vestopal-H has a refractive index somewhat higher than that of Canada balsam.

Over recent years, however, the authors changed their method in several respects. For instance, another plastic is used nowadays (Synolite), a freeze-drying procedure has been developed and the impregnation technique was totally changed. Therefore, we decided to rewrite the first publication (1963) entirely.

For a better understanding of the method, the paper will first turn to the theory of the hardening process.

## 2. THEORY OF THE HARDENING PROCESS

Hard setting polyester resins consist of a solution of an unsaturated reactive polyester in a vinyl or allyl monomer, usually monostyrene. Polyesters of this type usually have a linear structure and they are prepared from dibasic acids and glycols (ref. amongst others BERNDTSSON and TURUNEN, 1954). The nature of these materials determines the properties of the resins. Thus the unsaturated character of these polyesters depends on the incorporation of maleic or fumaric acid and their reactivity increases as the content of these acids increases (MALTHA, 1957).

In many hard setting resins the transition from the liquid to the solid phase depends on condensation reactions. The synthetic resins of the type described, however, pass into the solid state, via a gel phase, as the result of copolymerisation of polyester and styrene, i.e. the building of styrene bridges between the polyester chains and the formation of a spatial net structure (cross-linking).

This copolymerisation is not a spontaneous reaction, but one that requires an activation of the monomers. Although these are unsaturated as a result of the presence of one or more double carbon bonds, they can only be made reactive by activation of these double bonds.

This may be achieved by supplying energy in the form of heat or ultraviolet light, or by means of catalysts; for our purpose only the latter method is important. The catalysts used for the copolymerisation of unsaturated polyester resins are organic peroxides such as benzoyl peroxide, methyl ethyl ketone peroxide and cyclohexanone peroxide. The splitting of their oxygen bridge ( $O = O$ ) releases free radicals capable of activating the double carbon bonds. Simultaneously with the formation of a new free radical, addition of the catalyst radical then occurs. The new radical may combine in turn with another double carbon bond, and so on. The monomers combine in this way to form an increasingly large, three-dimensionally constructed polymer.

Since the free radicals formed from the organic peroxides are built into the growing polymer molecules, the term 'catalyst' is not really correct in this case. It would be better to speak of an initiator (CONIX, 1950; CYWINSKI, 1960). In spite of this, the term 'catalyst' will be used in the remainder of the paper as it accords with general usage.

The oxygen bridge of the catalyst can be broken only with the application of energy (heat or ultra-violet light), or the addition of an accelerator (MALTHA, 1957). The accelerators enable the peroxides to be split at room temperature, even peroxides having a critical temperature of from 60° to 80° C. Accelerators are materials that split the peroxides by reduction. Various materials can serve as accelerators. Thus, tertiary amines (especially dimethyl aniline) are used to split benzoyl peroxide, and metal soaps, particularly soluble Co-soaps such as Co-napthenate and Co-octoate are used for hydroperoxides (DAMEN and KEVER, 1961).

The foregoing can be summed up by stating that copolymerisation of unsaturated polyester resin dissolved in monostyrene at room temperature is possible only in the presence of both a catalyst and an accelerator. The process takes place in a number of phases which can be clearly distinguished (ANONYMOUS 1, no date) because of the exothermic nature of the polymerisation. By following the temperature changes in the polymerising resin (which needs to be insulated in order to make measurements at room temperature) and plotting temperature against time, the so-called polymerisation curve is obtained (BERNDTSSON and TURUNEN, 1954; MALTHA, 1957; CYWINSKI, 1960). At first the temperature in the plastic does not rise, as can be seen in Figure 1. During



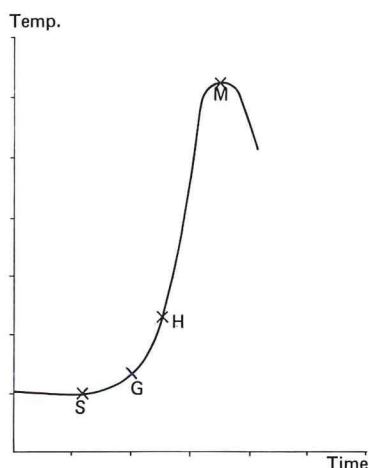


Fig. 1. Theoretical representation of a polymerisation curve at 20°C. S = starting point of the polymerisation reaction; G = gelling point; H = point after which the plastic has hardened; M = point of maximum heat evolution ('peak exotherm').

this early period (termed the pot life), the free radicals formed from the catalyst are used up by the inhibitor (see below). It is only on completion of this phase (Fig. 1, S) that polymerisation begins. This is revealed by, amongst other things, a rise in temperature, followed by gelling of the material (gelling point: Fig. 1, G). This rapidly raises the reaction rate and therefore the temperature also, and in particular the free monostyrene still present is almost entirely polymerised out (FUNKE, 1959). The rise in temperature during the gelling may be so great that the viscosity of the plastic may be reduced for a short time (BEYER, 1955; MALTHA, 1957). Only after the plastic has hardened (Fig. 1, H), does the maximum heat evolution occur (Fig. 1, M). This indicates that the polymerisation also continues in the hard product.

The hardened end-product may be regarded as a three-dimensional combination of the following polymerisates (BERNDTSSON and TURUNEN, 1954):

- a. Polyester chains bound via carbon bonds by bridges of one or more styrene molecules.
- b. Branches of non-bridge-forming polystyrene chains from the double carbon bonds of the polyester chains.
- c. Independently developed styrene chains.

The rate of the polymerisation process plays a very important aspect in the preparation of thin sections of soil. It is determined by several factors.

*The type of plastic.* The unsaturated polyester resins may differ fairly considerably in their chemical compositions. Consequently the number of carbon double bonds can vary widely, which means that the reactivity will also vary considerably. One example of the differences in the polymerisation rate of plastics can be found in CYWINSKI (1960: Table 13, p. 43).

*The concentration and type of catalyst.* According to KÜCHLER (1951), the concentration (c) is proportional to the polymerisation rate (h) according to the formula  $h = k.c^n$  in which  $n = 1/2$  and k is a constant. BERNDTSSON and TURUNEN (1964) found a similar relationship for the gelling time, with the proviso that the exponent n may have entirely different values for different catalysts. This means, therefore, that different catalysts may differ substantially in activity, a fact which many other scientists (FARKAS and PASSAGLIA, 1950; CYWINSKI, 1960; DAMEN and KEVER, 1961) have also established.

*The concentration and type of accelerator.* In general it can be said that at least

when the accelerator concentration is fairly low, an increase in the amount of accelerator produces an increase in the rate of polymerisation, and sometimes a very substantial increase. There is, however, no simple functional relationship as in the case of the catalysts. Increasing concentrations of dimethyl aniline (in combination with benzoyl peroxide) raises the polymerisation rate. It is noticeable, however, that at higher concentrations of dimethyl aniline the polyester resin is not hardened by increasing the concentration (MALTHA, 1957). BERNDTSSON and TURUNEN (1954) found that the plastic Soredur 9155 B gelled more rapidly at room temperature with cyclohexanone peroxide as they increased the additions of Co-naphthenate. But at a certain concentration of Co-naphthenate, the rate of gelling peaks, and above a given concentration of accelerator (about 2 % of 6 % Co-naphthenate) polymerisation actually proceeds more slowly than at lower concentrations. MALTHA (1957) also found that an unspecified polyester resin had a much shorter gelling time with 2 % of 50 % methyl ethyl ketone peroxide as catalyst when increasing amounts of 1 % Co-naphthenate were added. However, at higher concentrations (of up to 12 % of accelerator) the gelling time remained constant when amounts were increased. Co-soaps are much more reactive (i.e. they have a higher polymerisation rate) than other metal soaps both with cyclohexanone peroxide and methyl ethyl ketone peroxide (MALTHA, 1957; DAMEN and KEVER, 1961).

*The operating temperature.* This has a very large influence on the polymerisation rate. A rise of only 3° to 5° C during the hardening of Vestopal resins with cyclohexanone peroxide and Co-naphthenate at room temperature reduces the gelling time by half (ANONYMOUS 2, no date). On the other hand, the unsaturated polyester resins do not usually harden properly at temperatures below about 15° C (ANONYMOUS 3, 1955).

*The amount of polyester resin and the shape of the cast.* A large amount of polymerising polyester resin produces more heat than a small amount. In the first case, the amount of heat that can be rapidly dissipated is relatively smaller than in the second case, in which the temperature rises more strongly and the reaction proceeds more rapidly. In this respect, the shape of the cast plays an important part, in addition to the concentrations of catalyst and accelerator. There will be little rise in temperature in a thin sheet of a given volume, while the opposite will apply to the same amount of plastic in the shape of a cube.

*Fillers.* Various kinds of fillers such as chalk, talc, mica, quartz, asbestos and kaolin are used in the plastic-processing industry for both economical and technical reasons. The addition of such a filler to the liquid plastic usually results in a lengthening of the gelling time. The various fillers differ considerably as to the effect they have: thus the addition of quartz only slows down polymerisation to a slight degree, whereas carbon black slows it down considerably (MALTHA, 1957). In addition to the kind of filler, the concentration (vol. %) of the filler is also very important: the higher the concentration, the slower the polymerisation proceeds. The delaying effect of fillers may be due to many reasons. In general, it can be said that the addition of a filler to liquid polyester resin means a dilution of reactive double bonds, which results in a slower polymerisation reaction (BERNDTSSON and TURUNEN, 1954). At the same time, electrolytes or traces of different metals in the fillers may slow down the reaction considerably and, finally, the catalysts and/or accelerators may be adsorbed on the surface of various fillers (MALTHA, 1957).

*The moisture content of the filler.* Even small traces of moisture will reduce the rate of polymerisation (ANONYMOUS 3, 1955). In impregnating a soil, which was not quite air-dry, with Vestopal-H the authors found that, in so far as it had penetrated into the soil, the plastic did not harden at all, or at least not for several months.

*Inhibitors.* These are materials, usually of a phenolic type, such as hydroquinone,

tertiary butyl catechol, etc., which block the free radicals and thus considerably delay the initial reaction. They are added in small amounts to the unsaturated polyester resins and to monostyrene in order to increase the keeping quality of these materials.

*The diluent content.* The liquid unsaturated polyester resins are usually fairly viscous. Their viscosity can be substantially reduced by adding monostyrene or acetone (FITZPATRICK, 1970) to the resin. This, however, affects the polymerisation rate, and the effect may be either positive or negative. Thus BERNDTSSON and TURUNEN (1954) found that at 20° C the gelling time of the plastic Soredur 9155 B with cyclohexanone peroxide and Co-naphthenate was reduced with an increasing styrene content, whereas when benzoyl peroxide and dimethyl aniline were used with Soredur 9155 B it rose with an increasing styrene content.

Several of the factors discussed above do not only influence the polymerisation but they also have a great effect on the properties of the polymerisate. This will be discussed as far as necessary in Chapter 4.



### 3. SOIL MOISTURE: A PROBLEM IN THIN SECTION PREPARATION

#### 3.1 Air drying

To stabilize a soil sample with a plastic, water should be thoroughly removed beforehand (Cf. Chapter 2). To achieve this, one usually lets the water evaporate from the sample at room temperature. However, this procedure is very time-consuming, particularly if the sample is clayey. According to A. H. BOUMA (1969), air drying of bulky clayey samples may even require six months to two years. Of course the drying can be sped up considerably if it is conducted at higher temperatures, but this has several disadvantages, such as a high shrinkage, oxidation of organic matter and conversions in certain crystalline materials. Nevertheless, in practice we did not find essential differences between samples dried at room temperature and at 40° C respectively. Consequently in the authors' department soil samples are dried in an oven at the latter temperature. The progress of the drying is controlled by means of a hygrometer, the procedure being completed when the humidity in the oven remains constant.

Though for pedogenetic micromorphological investigations drying at room temperature or at 40° C maximum gives satisfactory results in many cases, the method has a number of limitations and shortcomings:

1. Samples of unripe or partly ripened clayey sediments and of peats are subject to severe shrinkage and (eventually) crack formation. Accumulations of mineral, and in particular, of organic gels will shrink to such an extent that usually they are not recognizable as such any more.
2. Soluble salts may diffuse to the outer surfaces of the sample (i.e. the zones of highest evaporation), giving rise to the formation of artificial efflorescences.
3. According to STEPHAN (1969) argillans may form during drying, if clay particles occur in suspension.
4. In a slowly drying sample burrowing soil animals (earthworms) withdraw to its interior in an attempt to survive. By doing this, they usually cause considerable changes in the original structure of the sample.
5. No soil animals are found in the dried samples, a part of them having left the samples during drying and the remaining having died and decomposed.
6. During drying in unripe or partly ripened clayey materials, very pronounced plasmic fabrics of an artefactual nature may form.

To anticipate the above-mentioned defects several methods have been developed for the removal or neutralization of water present in soil samples. These can be grouped into replacement, critical point drying, 'wet' impregnation, and freeze-drying methods.

#### 3.2 Replacement of the soil moisture with organic liquids

Replacement is based on the ability of water to mix with other liquids, such as acetone and ethanol. It is a common method of drying in biology (ethanol) and in electron microscopy (acetone; BURCK, 1966). To avoid shrinkage as much as possible the replacement should be carried out slowly, i.e. stepwise with increasing concentrations of the replacing liquid(s).

LUND and BEALS (1965) employed this principle to prepare thin sections of soil. First, living roots are fixed by a repeated FAA treatment (FAA is a 1 : 1 : 10 mixture of

acetic acid, formaldehyde and ethanol). After this the sample is dehydrated with 70 %, 95 % and pure ethanol successively. The duration of these treatments and the required number of repetitions depends strongly on the clay and moisture content of the sample. A rather dry sandy soil, for instance, requires only one treatment each with FAA, ethanol 70 % and ethanol 95 % respectively, and two treatments with pure ethanol. However, to obtain a satisfactory drying of a rather wet clay sample, all treatments should be repeated three times and each should be carried on much longer than in the former case.

To impregnate wet or humid samples of clay soils with the epoxy resin Araldite without pre-drying, SINGH (1969) replaces the soil moisture with acetone and monostyrene successively. The monostyrene-saturated sample is impregnated with the Araldite. For 1.5 cm thick samples of the clay tested a complete replacement of a liquid is reached in one week, if (1) the replacing liquid and the sample have a 20 : 1 volume ratio, (2) evaporation of the liquid is prevented, and (3) the system is agitated for some seconds each day. Thicker samples can be treated successfully in a similar manner, but it is advised to use a smaller volume ratio and to replenish the replacing liquid several times during the treatment.

MIEDEMA, PAPE and VAN DE WAAL (1974) use a one-step diffusion process: soil moisture is replaced with acetone and the acetone-saturated sample is impregnated with the unsaturated polyester resin Synolite (Cf. Chapter 4). In this method as well the replacing liquid is entirely replenished several times.

Replacement techniques are preferable to air drying for certain clayey samples, but the mentioned methods have a strong limitation. They are not suited to treating samples which contain humic substances, which would be partly dissolved by the used organic liquids. Moreover, replacement treatments do not preclude shrinkage entirely. SINGH (1969) reports the occurrence of some cracking and a shrinkage of up to 3 per cent, and for pure montmorillonite even of about 5 per cent. MIEDEMA, PAPE and VAN DE WAAL (1974) did not find any artificial cracking, but they noted a shrinkage of about 5 per cent (which they attributed mainly to the shrinkage of the setting polyester resin).

### 3.3 Critical point drying

This method is based on the principle that, above a certain critical limit of temperature and pressure, the physical properties of a liquid and its vapour become the same. So, applying a temperature and a pressure above the critical point of water, this can be removed of a soil sample without damage caused by capillary forces. However, the temperature and pressure required for such a direct drying are very high. To overcome this drawback, the soil water can be replaced by a miscible liquid which has a lower critical point.

The authors having no experience with this method, the reader is referred to GILLOT (1974).

### 3.4 'Wet' impregnation

'Wet' impregnation refers to a direct consolidation of the sample at field humidity with an agent that removes the soil moisture. Such agents are:

### *Carbowax 6000*

This material is a polyethylene glycol with an average molecular weight of 6000 to 7500. At room temperature it is a crystalline solid with a tallowy consistency, and it melts at about 55° C. Carbowax 6000 is soluble in water and in many organic liquids. For a detailed review of its properties refer to GREENE-KELLY and CHAPMAN (1970).

It was first used to impregnate moist samples of shale (MITCHELL, 1956) later it was used also for hardening of clays and clayey soils (TOURTELOT, 1961; SINGH, 1969; GREENE-KELLY and CHAPMAN, 1970), and organic soil materials (MACKENZIE and DAWSON, 1961).

Most authors soak the wet or moist samples in molten Carbowax 6000 at 60° C for 3 days. However, this may cause such a rapid dehydration that artificial cracks occur. To avoid this, GREENE-KELLY and CHAPMAN (1970) impregnate wet or moist soil materials at room temperature with aqueous Carbowax 6000 before the final consolidation with the molten agent takes place. The concentration of the wax solution with which one should start, depends on the moisture content and the cohesion of the soil material. For a cohesive wet clay ( $pF < 2$ ), for instance, the initial concentration is but 10 %, whereas a dryer ( $pF 2-3$ ) or less cohesive soil sample can be treated directly with a 50 % solution. In the former case the 10 % treatment should be followed by a 25 % and a 50 % treatment successively. Relatively dry samples ( $pF > 3$ ) are impregnated directly with molten Carbowax at 60°-70° C. The duration of the treatments increases from 5 minutes for the 10 % immersion to 5 days for that with molten wax.

For the sake of completeness it should be mentioned that WILLOUGHBY and WALSH (1972) impregnate air-dried samples with molten Carbowax 4000 at 65° C for 5-7 days to produce thin sections of 200-300 sq.cm.

Though useful in certain cases, the Carbowax method has several disadvantages (e.g. TOURTELOT, 1961, GREENE-KELLY and CHAPMAN, 1970), such as:

1. Carbowax is soft and has a low melting point. Hence, surface smearing and cementation of grinding powders during processing occur easily.
2. The sections are rather thick (30  $\mu m$  or more).
3. During impregnation a rather high shrinkage may occur (e.g. QUIGLEY and THOMPSON, 1966), and thus, also, the formation of artificial cracks (e.g. A. H. BOUMA, 1969, Fig. 2.8, p. 119).
4. The wax is optically anisotropic. Dominant interference colours are usually yellow and red of the second order.
5. Carbowax 6000 crystallizes in spherulites. This may hinder the microscopic investigation of small particles, but the study of grains larger than 50  $\mu m$ , plasmic fabrics and the orientations in argillans presents no problems.
6. Possibly, the wax may form complexes with expanding clay minerals (QUIGLEY and THOMPSON, 1966).

### *Arigal C*

This melanine-formaldehyde compound is soluble in water as well. It has been used by MÜLLER-BECK und HAAS (1961) for the preservation of wood, and later (1963) by REINECK for the consolidation of non-dry clays and sandy clayey samples. For a duration of 7 days, the samples are impregnated with Arigal C at room temperature subsequently they are placed for 2 days in an oven at 65° C to allow fixation of the Arigal, and finally they are dried for another 2 days at room temperature. Next, because the Arigal C is not sufficiently hard to permit further processing of the samples



(i.e. sawing and grinding), the samples are re-impregnated with Araldite F. For sandy clays a treatment lasting 24 to 36 hours at 120° C is needed, and clays are impregnated for 16 days at 40° C followed by 2 days at 120° C.

According to WERNER (1966) it is nearly impossible to avoid the formation of artifi-

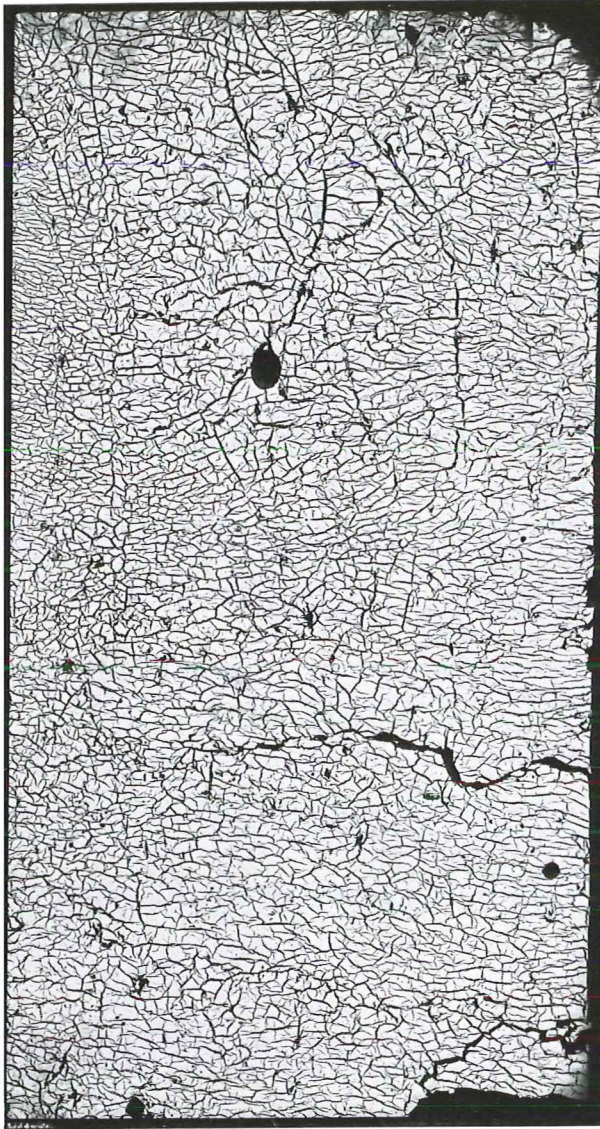


Fig. 2. Destruction of the natural soil structure, caused by ice crystal formation. Thin section of a sample (15 × 8 cm) of a clay soil, frozen in air at -70°C. Negative full-size photo; the cracks are black.

cial cracks when using Arigal. In pictures shown by A. H. BOUMA (1969, Fig. 2.9, p. 121) such cracks are also clearly visible. Another disadvantage of the Arigal method is that only thick sections can be made in this way.

#### *Other impregnating materials*

Some other compounds used for 'wet' impregnation are (BREWER, 1964) latex sols and Durcupan. Latex sols are suited for stabilizing wet soil materials, but a reimpregnation with a plastic is necessary. Durcupan can be used to impregnate slightly moist porous soil materials, but due to its polarity the fabric of swelling soils can be disturbed.

Considering the aforesaid it is understandable that several investigators (a.o. BURGESS and NICHOLAS, 1961; WERNER, 1966; STEPHAN, 1969) tried to solve the problem of the removal of soil moisture in an entirely different way, i.e. by means of freeze-drying.

### **3.5 Freeze-drying**

The procedure consists of two steps, i.e.:

1. the freezing of the samples,
2. the drying of the frozen samples via evaporation of the ice and sublimation of the ice vapour on a cool aggregate (ice condensator).

Compared with the methods described above, the big advantage of this method is that the capillary forces which may cause shrinkage are entirely eliminated.

#### *The freezing*

When freezing soil moisture, the water crystallizes first. The result is that an increasingly concentrated solution of salts and other compounds is left. Usually that solution will also crystallize when a critical eutectic temperature zone is reached. It is also possible, however, that due to the presence of certain compounds the salt solution will turn — via viscous phases — into a hard undifferentiated mass, i.e. a kind of 'salt glass' (REY, 1959). This glassy material may cause complications during the drying procedure, as it practically always softens and may even melt locally when the samples are heated. It should be stressed that we did not experience any problems of this kind. However, salts may also damage the samples due to hygroscopicity and crystal growth. To avoid this it is advised (WERNER, 1966) to desalt samples from marine sediments — by means of repeated oversaturation with distilled water and subsequent decantation — before the freeze-drying procedure takes place.

The freezing should be carried out in such a manner that the samples do not shrink or show any damage. The degree to which such a result is attained depends mainly on the freezing temperature and the freezing speed.

*The freezing temperature.* At rather high freezing temperatures recrystallizations lead to the growth of ice crystals. This causes the formation of artificial cracks and a complete destruction of the natural soil structure (Fig. 2; Cf. also a.o. J. BOUMA, 1969). According to REY (1959) such recrystallizations do not occur at temperatures lower than  $-120^{\circ}\text{C}$  (Fig. 3).

The freezing temperature also determines the kind of ice that is formed and its density. D'ANS-LAX (1967) discerns three kinds of ice (Table 1).



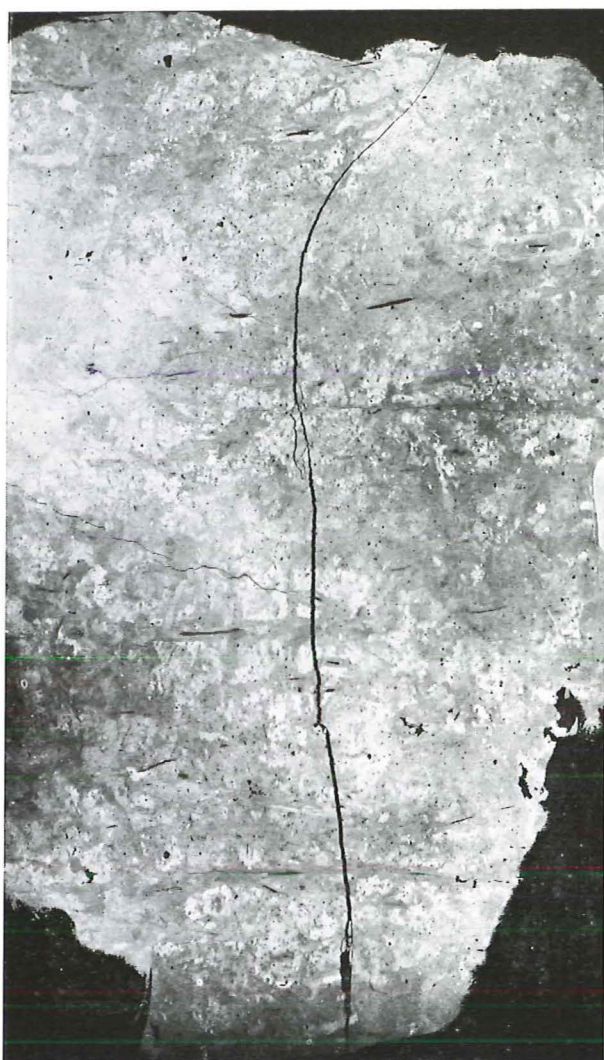


Fig. 3. Thin section of a clay soil sample ( $15 \times 8$  cm), frozen at about  $-165^{\circ}\text{C}$  (in Freon). Negative full-size photo.

Table 1. Kinds of ice formed at different freezing temperatures (after D'ANS-LAX, 1967).

	Freezing temperature	Crystal form	Density ( $\text{g}/\text{cm}^3$ )
Ice I	$-0 - -115^{\circ}\text{C}$	hexagonal	0.918
Ice II	$-115 - -155^{\circ}\text{C}$	rhombic	1.15
Ice III	lower than $-155^{\circ}\text{C}$	tetrahedral	—



From the data of table 1 it follows that the freezing temperature should be lower than  $-155^{\circ}\text{C}$ , for it is only at such very low temperatures that no crystal growth or formation of ice II (shrinkage) will take place.

However, freezing water at temperatures lower than  $-155^{\circ}\text{C}$  causes some extension, though less than when it is frozen at higher temperatures. This is shown in Fig. 4, which presents a graph based on data of EISENBERG and KAUFMANN (1969). According to this graph, which is partly in contradiction with the data of D'Ans-Lax, at  $-180^{\circ}\text{C}$  (i.e. approximately the maximum freezing temperature reached with liquid nitrogen) the volumetric extension is about 7 %. In practice, however, the linear extension of a soil sample frozen at such a low temperature often appears to be less than 1 %. This is probably due mainly to the fact that most samples are not entirely saturated with moisture, so that the air present within the samples may act as a buffer.

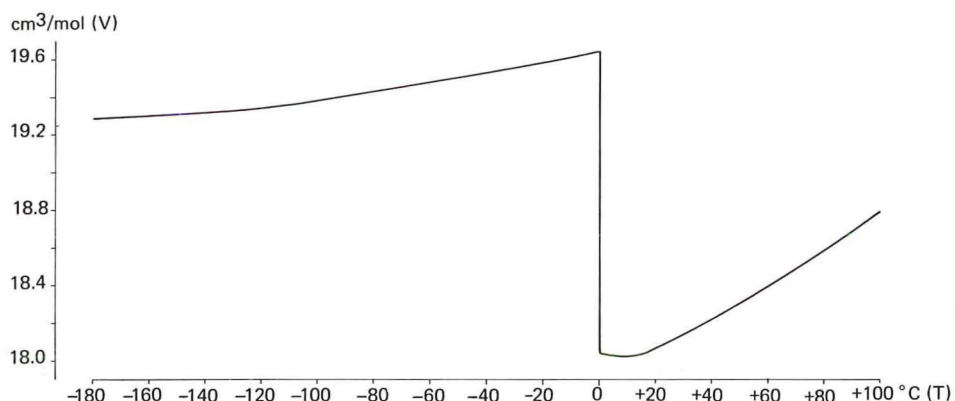


Fig. 4. The relation between temperature and volumetric extension of water and ice. The graph is based on data of EISENBERG and KAUFMANN (1969).

*The freezing speed.* Freezing at  $-170^{\circ}\text{C}$  to  $-180^{\circ}\text{C}$  in itself is no guarantee against the recrystallization of ice crystals. For that, it is also necessary for the cooling down of the sample to take place very quickly. In fact, the freezing speed must be so high that more heat constantly is withdrawn from the sample than is liberated by the ice formation (REIMER, 1959). To achieve this the sample must be rather thin (2 cm or less) and the freezing liquid must be in close contact with the soil. The latter condition cannot be fulfilled using liquid nitrogen, because it forms a gas mantle around the soil sample. This problem can be overcome, however, if the sample is frozen in Freon 12 ( $\text{CCl}_2\text{F}_2$ ), which in its turn is cooled in liquid nitrogen. Using Freon, the freezing takes place at  $-158^{\circ}\text{C}$ .

#### *The drying (dry freezing)*

The drying is based on evaporation of the ice from the frozen sample and subsequent sublimation of the ice vapor on a cold surface, the so-called ice condenser. To ensure a smooth course in the drying, a vacuum is needed and there must be a rather high pressure gradient between the frozen sample and the ice condenser.

Therefore, it would seem that for an optimal drying the vacuum level should be very high and the temperature of the ice condenser very low.

In practise, however, this is not the best combination (ANONYMOUS 6, 1969). In order to evaporate, the ice molecules have to overcome very strong forces. Consequently, the evaporation causes a loss of energy in the frozen sample which results in a decrease of its temperature. This, in turn, leads to a slowing down of the rate of evaporation. To avoid this, the lost energy must be supplemented by heating the samples. Usually this is done by heating the plates on which the samples are standing during the drying procedure. The problem is that generally the contact between samples and plates is not at a maximum: at some points the contact is direct, at other points the heat must be transferred by radiation and convection. As a consequence, the heat transfer is unequal (at least at very high vacuum), and therefore the total amount of transferrable heat will be less than could be expected at any particular temperature. Of course, the amount of transferrable heat can be raised by increasing the temperature of the plates, but this may cause damage at the points of direct contact (melting of the ice). However, the heat transfer can be increased by applying a 'worse' vacuum, i.e. a less high vacuum, because then air molecules occur between plates and samples. The flow of the ice vapour is not essentially inhibited by the air molecules, at least if the partial pressure of the air is not more than 1/10 of the average pressure occurring in the drying-room.

Concerning the temperature of the ice condenser it should be stressed that its optimum temperature is determined to a high degree by the temperature of the frozen sample, as is illustrated in Table 2.

Table 2. The relation between the temperature of the frozen sample and of the ice condenser respectively, and the pressure gradient (From: ANONYMOUS 6, 1969).

	Sample	Ice condenser	Pressure gradient
1. temperature	-30	-60	
pressure	0.2859	0.00808	0.27782
2. temperature	-40	-70	
pressure	0.0965	0.00194	0.09466
3. temperature	-20	-40	
pressure	0.776	0.0966	0.6794
4. temperature	-21	-50	
pressure	0.705	0.02955	0.67545

As is visible from table 2, in cases 1 and 2 the difference in temperature between sample and ice condenser are the same, but the pressure gradient in case 1 is three times as high as in case 2. In case 4 the temperature of the sample is but 1° C lower than in case 3. However, to obtain the same pressure gradient in both cases, the temperature of the ice condenser must be decreased by 10° C in case 4.

### 3.6 The drying procedure

Which drying procedure is followed depends on the nature of the soil material and the purpose of investigation. Samples which are non-coherent (like sandy materials) are dried at 40° C, as are most samples which are taken for micromorphogenetic investi-

gation. On the other hand, those samples which are susceptible to shrinkage and intended for practical purposes such as micromorphometric porosity investigations, are freeze-dried.

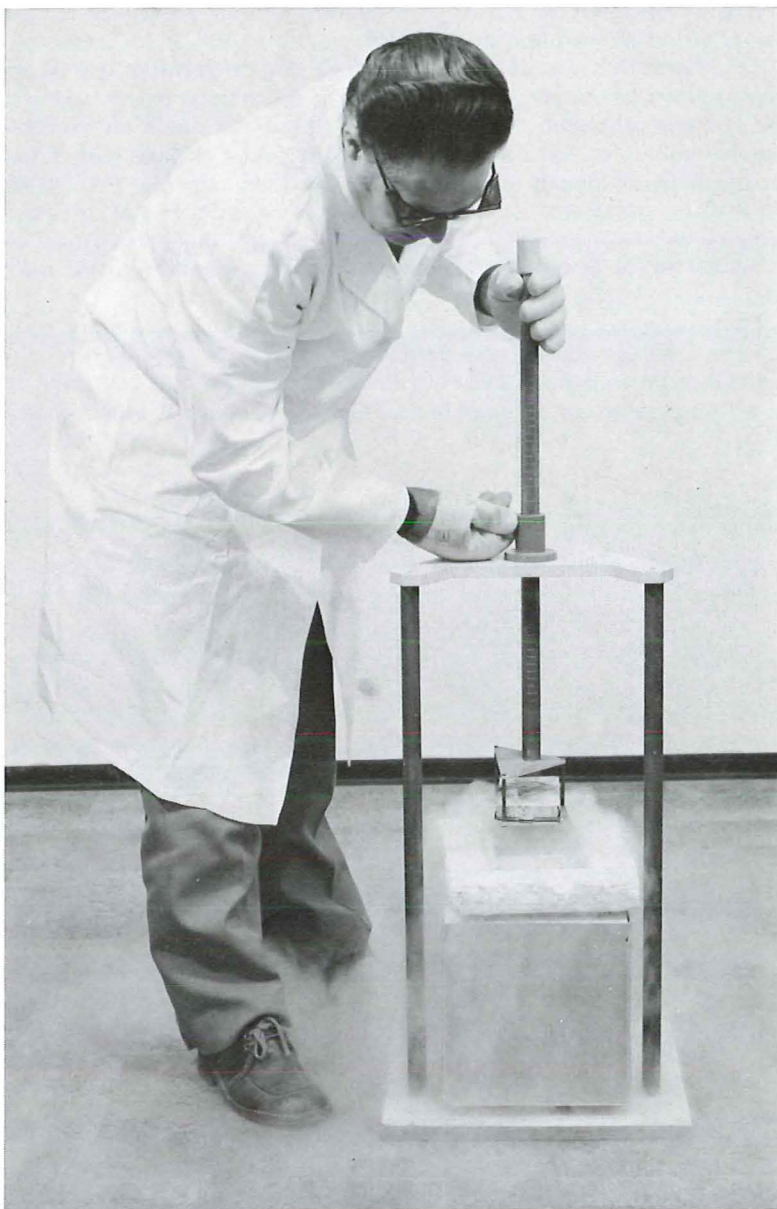


Fig. 5. The freezing of a soil sample in Freon 12, which is cooled in liquid nitrogen. The sample, which is placed on a perforated holder, is moved down into the Freon by means of a vertical bar which is clamped in a support.



Samples are taken in brass tins, which are 15 x 8 x 5 cm or 15 x 8 x 2 cm large, depending on whether drying will be at 40° C or whether the freeze-drying process will be applied.

In the former case the samples are placed in an oven at 40° C. The progress of the drying is controlled by means of a hygrometer; the procedure is complete when the humidity in the oven remains constant.

Samples to be freeze-dried are precooled in a refrigerator at 6° C immediately after arrival in the laboratory. This has proven to be essential to the satisfactory operation of the freezing procedure. Samples kept in closed tins which are wrapped in plastic foil can be stored for some weeks under refrigeration without any shrinkage occurring. Before they are frozen, the samples are taken out the tins and placed on perforated brass plates which are 15.5 x 8.5 cm large. This is done to avoid any distortion of the samples due to extension during freezing, and to simultaneously obtain a maximum contact with the Freon. The samples are quickly re-placed in the refrigerator, even if they will be frozen very soon.

Each sample is frozen separately. It is taken out the refrigerator and placed horizontally on a holder which is also perforated. The sample is plunged for three minutes into a small metal container filled with Freon 12 (Fig. 5). This container, in its turn, hangs in a Dewar vessel, or a double-walled metal box, which is filled beforehand with liquid

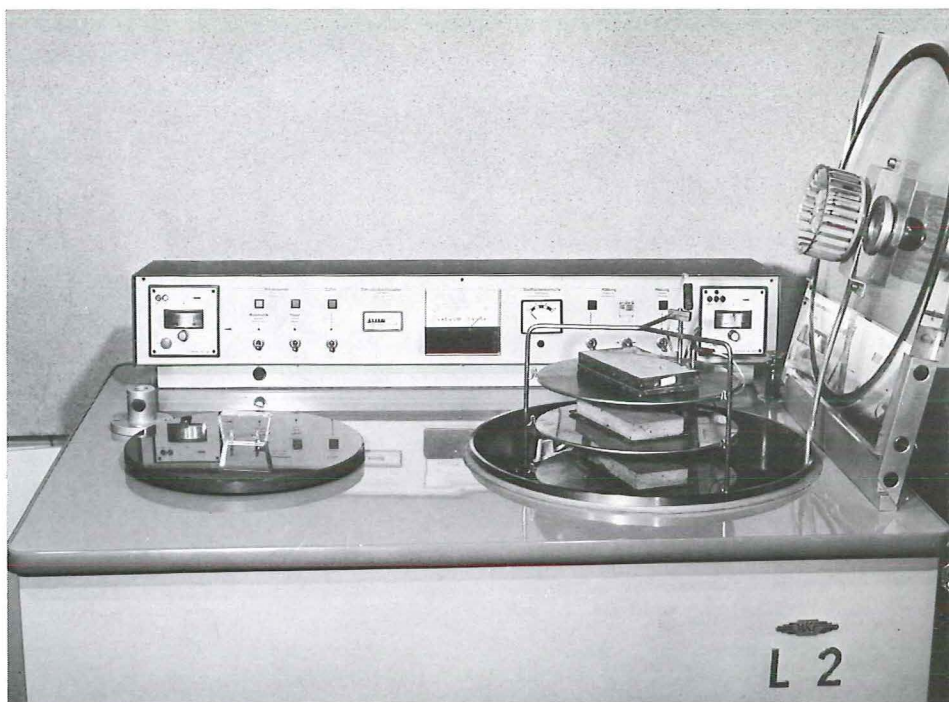


Fig. 6. A W.K.F. L2/70 freeze-drying equipment. The samples are dried in the chamber which is situated at the right hand side of the apparatus. In the photograph the set of heating-plates is shown in a somewhat upheaved position.

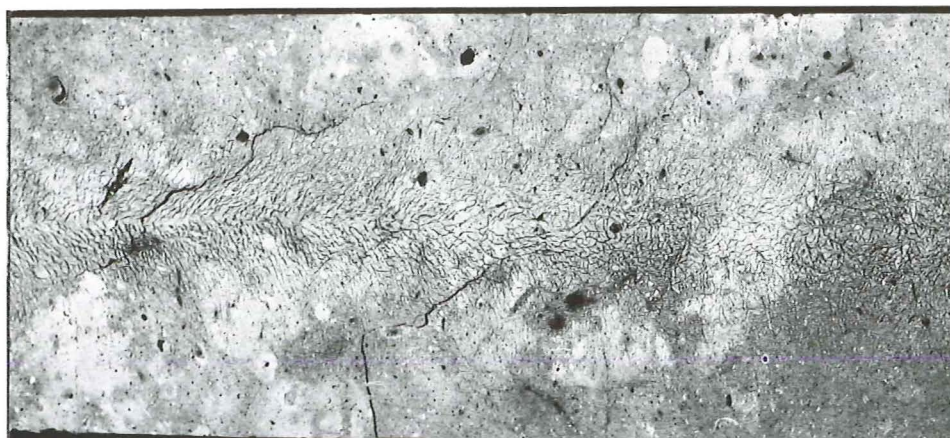


Fig. 7. Cross section of a freeze-dried clay sample of 2 cm thickness. Its rims are unchanged but the central zone shows a crack pattern which is due to ice crystal formation during freezing.

nitrogen. A metal box is preferable because, as we experienced, the glass mantle of a Dewar vessel bursts very easily when the liquid nitrogen is poured in.

Immediately after the samples have been frozen, they are placed in a WKF freeze-drying equipment type L2/70 (manufactured by Weinkauf, Brandau, Germany) (Fig. 6) on a set of plates which can be heated. The set, consisting of six plates, is connected with the machine by means of two thermocouples, one of which is attached to one of the plates to regulate the temperature of the set, whereas the other one is frozen into a test sample. Because two samples can be placed on a plate, eleven frozen samples (+ the test sample) can be dried simultaneously.

After the apparatus is closed, a ventilator which is situated at the inner side of the cover is set into action. The air-circulation obtained in this way maintains an equal temperature throughout the desiccator. Gradually the temperature in the system rises, so that after about two hours the samples are at  $-70^{\circ}\text{C}$ . At this point a vacuum pump is switched on, and at the same time the ventilator stops automatically; this is necessary to prevent the motor of the ventilator from burning. After about ten minutes the partial vacuum is approx. 2 mm Hg, and the heating of the plates starts with a plate temperature of  $-50^{\circ}\text{C}$ . The vacuum continues to rise till about 0,2 mm Hg, a pressure which is kept by means of a micro aeration valve. One waits until the temperature of the samples has risen to  $-50^{\circ}\text{C}$  and then the plates are heated till  $-40^{\circ}\text{C}$ . This procedure is repeated till the samples are entirely desiccated, which is the case at about  $+20^{\circ}\text{C}$ . The entire drying procedure lasts approx. 48 hours.

Samples treated in this way may show very narrow cracks in the centre, but their rims are unchanged all around over a width of approx. 4 mm at the least (Fig. 7). Thus two large-size thin sections can be made from one frozen sample.



## 4. IMPREGNATION AND HARDENING

### 4.1 The pre-hardening

Before impregnation with an unsaturated polyester resin can taken place, less coherent samples — sandy materials — should be pre-hardened by soaking them with a 1 : 1 mixture of cellulose varnish and acetone (JONGERUS, 1957).

### 4.2 The resin

The resin used is Synolite 544, manufactured by Synres Chemical Industries, Hook of Holland, The Netherlands (Synres products are also manufactured in France, Spain, Portugal, Brazil and Mexico). It is an almost colourless, slightly viscous product (viscosity about 6-8 poise) capable of unlimited dilution with monostyrene and acetone. It can be stored at room temperature, in the dark, for about six months. In the polymerised state the resin is very hard, practically colourless, vitreous and resistant to numerous chemicals. Synolite 544 shows a volume shrinkage of 6.5 % during hardening. In the presence of fillers, however, the shrinkage is reduced, probably in proportion to the amount of these fillers (ANONYMOUS 2, no date). Consequently, practically no shrinkage of the cured resin can be observed in hardened soil samples. For detailed characteristics of Synolite 544, see ANONYMOUS 3, 1965.

### 4.3 The catalyst and accelerator

The most commonly used catalyst-accelerator combinations for curing at room temperature are benzoyl peroxide + dimethyl aniline and cyclohexanone peroxide or methyl ethyl ketone peroxide + Co-soap. The latter combinations are preferred as there is no danger of insufficient hardening ('Untervernetzung') of the polymerisate (DAMEN and KEVER, 1961). Since the preparation of thin sections requires a long gelling time, as will be shown below, the less reactive, slower cyclohexanone peroxide is preferred to the rapid methyl ethyl ketone peroxide. Co-octoate is used as an accelerator, instead of Co-naphthenate, to which frequent reference is made in the literature. Co-octoate can be accurately formulated, while Co-naphthenate may also contain other Co-compounds (DAMEN and KEVER, 1961). For catalyst and accelerator the authors use: Cyclonox LNC: a solution containing approx. 40 % of pure cyclohexanone peroxide <sup>1)</sup>. Accelerator NL-49: a solution of Co-octoate with 1 % Co <sup>1)</sup>.

Other more highly concentrated Co-octoate solutions are also available but they are less suitable, as the very small amounts used (see 4.4) are much more difficult to measure at higher concentrations.

### 4.4 The impregnation

For the complete impregnation and proper hardening of soil samples as large as

<sup>1)</sup> Manufactured by AKZO Chemie Nederland, Deventer, The Netherlands.



15 x 8 x 5 cm with Synolite 544, several conditions must be fulfilled (see also ALTEMÜLLER, 1962):

1. The impregnation should take place in a fairly high vacuum. If the soil material is very compact, next a pressure of about 40 atm. (see below) should be applied to ensure an optimum impregnation.
2. The viscosity of the plastic has to be considerably reduced. This can be done by diluting with monostyrene (ALTEMÜLLER, 1956, 1962; JONGERIUS and HEINTZBERGER, 1963), or acetone (FITZPATRICK, 1970). The application of the last mentioned diluent gives better results, but it can only be used in soil materials which are poor in humic substances, these being partly dissolved in the acetone.
3. The excess monostyrene or acetone has to be removed from the samples in good time, since a high styrene or acetone content reduces the polymerisation rate of the resin and adversely affects various properties of the polymerisate, such as its resistance to various chemicals, modules of rupture, and shrinkage (a.o. BERNDTSSON and TURUNEN, 1954). This may lead to difficulties in the grinding of the sections, while the shrinkage may cause birefringence in the hardened polyester. This birefringence reveals itself under crossed nicols as light margins in cavities and around peds in the thin sections. This makes such slides unsuitable for microphotography (ALTEMÜLLER, 1956).
4. In order to allow the excess monostyrene or acetone to evaporate and to make sure that the samples have been completely impregnated by the Synolite 544, a long gelling time is required. This is achieved by adding only low concentrations of catalyst and accelerator. This brings with the advantage that the exothermic heat peak (see Fig. 1) is low. This peak decreases with decreasing catalyst and accelerator concentrations (ANONYMOUS 2, no date) and the amount of heat evolved can be more readily dissipated. This considerably reduces the risk of internal stresses leading to birefringence or even fracture of the polymerisate. The chances of the polymerized resin being colourless are also best, as a high Co concentration causes a green to reddish-violet discoloration. The latter is particularly induced at higher temperatures (DAMEN and KEVER, 1961). Finally, research by Damen and Kever has shown that very low concentrations of Co-accelerator give a better hardening than higher concentrations. For these reasons, the same low concentrations of accelerator are also used on sandy samples, where their greater porosity would seem to warrant a shorter gelling time than for dense samples. Experience has shown a gelling time of anything from 4 to 6 weeks to give the best results with all types of soil.

The impregnation procedure is as follows: The dried samples are impregnated in series of four. Each series is placed in a 10 cm high steel box, which is 34 x 16 cm at the bottom and 37 x 19 cm at the (open) top side. This tapered shape serves to facilitate the removal of the hardened blocks. To avoid sticking of the plastic to the steel, the inside of the box should first be treated with a special release-varnish (type F86, manufactured by Frencken, Weert, The Netherlands). The samples are laid alongside each other, and their laboratory numbers are indicated on the outside of the box.

This method of working has two advantages:

1. The bottom of the box is evenly filled with soil, which is very important for a successful hardening. If the samples did not fill the box uniformly it would contain large empty spaces which would obviously become full of plastic during the impregnation. Such large plastic nuclei polymerise more rapidly than the plastic in the soil samples (see Chapter 2: the amount of polyester resin and the shape of the cast), resulting in so much heat being liberated that the polymerisation in the soil samples is also accelerated, thereby causing the disadvantages mentioned above.

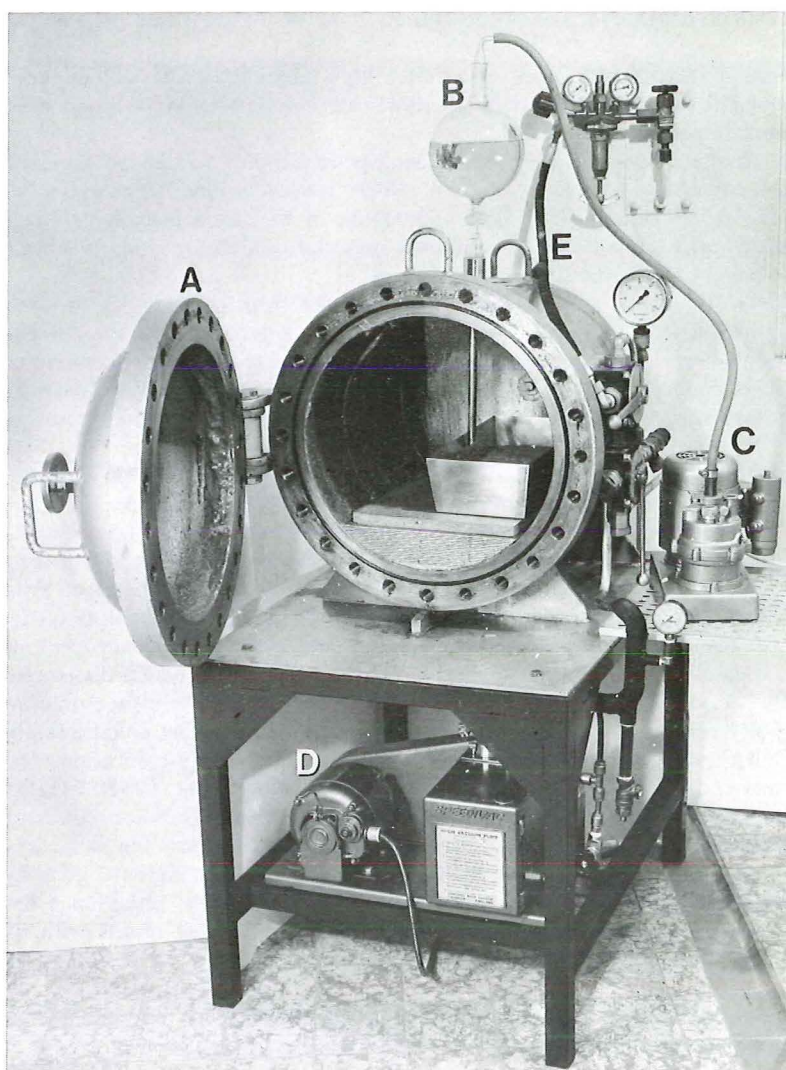


Fig. 8. High pressure device for the impregnation of soil samples. In the steel cylinder, which can be closed by means of a convex lid (A), four boxes each containing four samples of  $15 \times 8 \times 5$  cm or  $15 \times 8 \times 2$  cm can be placed. On top of the cylinder is a 3 l glass sphere (B), which can be replaced by a heavy steel bolt when high pressure is applied. A small Vacuubrand pump (C) serves for the evacuation of the air occurring in the glass sphere filled with impregnation liquid, whereas the cylinder is connected with an Edwards high vacuum Speedivac Ed 100 pump (D). The nitrogen cylinders used to apply high pressure, are connected with the impregnation pan via line E. They are not visible on the photograph because for safety reasons they are situated in an adjoining room.



2. In practice four large samples can conveniently be treated together. With a view to continuous production of thin sections, simultaneous impregnation of a number of samples is required. This can be done by treating the samples separately in series, but this involves the regular cleaning of a lot of glassware (taps, tubes, etc.). This applies much less to the procedure mentioned above. At the same time the impregnation of more than four or five large samples in a single block is not recommended. This would demand not only very large impregnation equipment, but would also make the hardened blocks too large and heavy to handle.

It must be emphasized that steel boxes are only necessary, if the plastic is diluted with acetone. Samples which are going to be impregnated with a mixture of plastic and monostyrene, can be placed in cardboard boxes lined with plastic foil (JONGERUS and HEINTZBERGER, 1963).

The impregnation occurs in a specially constructed device (manufactured by the Technical and Physical Engineering Research Service, Wageningen), in which vacuum ( $10^{-2}$  mm Hg maximum) as well as high pressure (50 kg/cm<sup>2</sup> maximum) can be applied at a maximum temperature of 40° C (Fig. 8).

The cylindrical impregnation pan, which is horizontally assembled, has a total length of 770 mm, and an external diameter of 470 mm. The pan is made of 12.5 mm thick steel, which is specially tested for this purpose. The front end of the cylinder is a convex lid, which is attached to it by a hinge. The lid can be closed by means of 24 steel bolts. Cylinder and lid are furnished with flanges, and an O-ring, which is placed between them, guarantees a hermetic sealing.

On top of the cylinder is a small round aperture, which is closed with a heavy steel bolt, when high pressure is applied. In order to add the impregnation liquid, which takes place in a vacuum, the bolt is replaced by a raised edge which is screwed into the aperture. A 3 litre glass vacuum sphere which is provided with a dosage tap, is placed into the edge.

The impregnation pan is connected to an electric vacuum pump (an Edwards high vacuum Speedivac Ed 100, manufactured by Edwards High Vacuum, Crawley, U.K.), and, via a reducing valve, to nitrogen cylinders. For this reason the equipment is provided with two three-way stop cocks, which must be operated one after the other, at reduced pressure as well as at excess pressure. A special locking-mechanism on both three-way stop cocks serves to avoid a possible simultaneous connection of the cylinder to vacuum and pressure.

The boxes filled with the soil samples are placed one by one in the impregnation cylinder, on top of which the glass vacuum sphere is fitted. The sphere is filled with a mixture of Synolite, the diluent, the catalyst and the accelerator. The composition of the mixture depends on the nature of the materials which are to be impregnated.

To impregnate four normally dried samples (15 x 8 x 5 cm) of sandy soil material, or material with a high organic matter content, the following mixture is used:

2900 cc Synolite 544

2100 cc Monostyrene

4 cc Cyclonox LNC

2 cc Cobaltoctoate 1 %.

For clayey samples and strongly ferruginous soil materials the composition of the mixture is as follows:

2500 cc Synolite 544

2500 cc Acetone

5 cc Cyclonox LNC

3 cc Cobaltoctoate 1 %.

As freeze-dried samples are less voluminous (18 x 8 x 2 cm), only three fifths of the above-mentioned volume, i.e. 3 litre of mixture, is necessary for their impregnation (in sets of four).

The amounts of catalyst and accelerator only hold for operating temperatures of about 22° C maximum. If the temperature is higher (as for instance in summer) only half the amount of both catalyst and accelerator should be used (Cf. Chapter 2: the operating temperature).

As soon as possible before impregnation, the liquid is prepared in a beaker. First of all the plastic is poured in, followed by the monostyrene or the acetone. Both are thoroughly mixed with a stirring rod. The accelerator measured off by graduated pipette is then added, and stirred in until the Co-octoate is uniformly distributed throughout the liquid. The intense violet hue of the accelerator makes this easy to check. Finally, the cyclonox is added to the liquid, also from a measuring pipette, and thoroughly mixed into it. Catalyst and accelerator should never be mixed at the same time with the plastic as this may produce a violent explosion. It should be noted here that a somewhat modified procedure is also possible (ALTEMÜLLER, 1962; ANONYMOUS 2, no date). In this case a stock solution is made of plastic, monostyrene (or acetone) and accelerator. This stock solution can be stored for a fairly long time at room temperature. For impregnation the required amount of solution is taken out and the corresponding volume of catalyst added.

The sphere is then filled with the mixture, and before impregnation starts (with the connecting tap to the cylinder closed), the sphere is connected through a ground glass joint to an electric vacuum pump (a Vacuubrand RE-2N, manufactured by Rudolf Brand, Wertheim a.M., West-Germany). The evacuation, which lasts only a few minutes, serves to remove as much air as possible from the impregnation liquid (the large amount of stirring can introduce a great many microscopic air bubbles). In the mean time the cylinder is also evacuated (by means of the Edwards pump) to the required vacuum, which is 20 mm Hg if the impregnation takes place with a plastic-monostyrene mixture, and 100 mm Hg if a plastic-acetone mixture is used. This difference is due to the fact, that acetone is more volatile, i.e. 'boils' at a higher reduced pressure, than monostyrene.

The above-mentioned vacuum being reached, the connecting tap between sphere and cylinder is opened sufficiently for the liquid to stream very gradually into the box of samples. The box should be placed so, that the plastic does not drop onto but between the samples. This prevents any air still present in larger voids from being occluded by the plastic. At the end of the impregnation procedure, which lasts for about an hour at a constant vacuum of 20 mm Hg and 100 mm Hg resp., the plastic mixture entirely covers the samples. The box of impregnated samples is then removed from the vacuum cylinder, and three other boxes with samples are treated successively in the same way.

Immediately after the completion of the above procedure, the four boxes are placed together in the cylinder, and subjected for one hour to a pressure of 40 atm. nitrogen for one hour to ensure an optimum impregnation. As a result, some nitrogen gas is absorbed in the impregnation liquid. This nitrogen must be removed immediately, as it causes a milky turbidness of the plastic. The boxes are therefore evacuated for another hour.

The boxes with the impregnated soil samples are then placed for a few days in a fume cupboard. During this time the air, which can still be trapped in the very fine voids in the centre of the samples, will be replaced by the impregnation liquid. To prevent a quick evaporation of the acetone, boxes which are filled with plastic-acetone mixtures are covered by a glass plate during this phase of the procedure.





Fig. 9. Fume cupboard in which the temperature is kept constant at  $20^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ . The boxes with impregnated samples are placed on grates in a continuous air stream. The two large boxes contain large impregnated samples which will be processed to mastodont-sized sections. The diluting agent in the left-hand box (steel!) is acetone, that in the right-hand one is monostyrene (a cardboard box lined with plastic foil).

Again the boxes are evacuated for one hour, after which time 2 litres of impregnation liquid is poured into these boxes which contain larger (normally dried) samples.

The next stage in the work is the expulsion of the large excess of monostyrene or acetone from the plastic. To this end the boxes with impregnated samples are placed in a fume cupboard in which the air is drawn off by a large ventilator operating for 24 hours per day (Fig. 9). The temperature is kept constant at  $20^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  in the cupboard, to ensure the long gelling time which is required for a proper consolidation of the samples (see below).

The evaporating monostyrene or acetone causes the liquid level to fall fairly rapidly. This fall usually continues for about two weeks. The impregnating liquid still covers the samples to a depth of about 1.5 cm. Thus in contrast to the procedure which has been used in the past (JONGERIUS and HEINTZBERGER, 1963) no replenishing with concentrated unsaturated polyester resin is needed during the period of evaporation.

From the above, one could get the impression that in order to obtain a maximum impregnation of the samples with the plastic, a very high dilution of the resin with monostyrene or acetone, combined with the application of high pressure, would suffice, and that the length of the gelling time is less important (though, of course, it should be longer than the time required for the evaporation of the diluting medium). However, a low viscosity by itself does not guarantee a complete impregnation with enough plastic. ALTEMÜLLER (1962), for instance, using a high dilution and a rather short gelling time (the polymerisation took 4-6 days) had to re-impregnate his (small) samples in several cases. JONGERIUS and HEINTZBERGER (1963), on the other hand, reported that a high dilution of the plastic (Vestopal-H) and a gelling time of 4-5 weeks generally resulted in an excellent impregnation, even if the samples had a volume of about 40 times those of

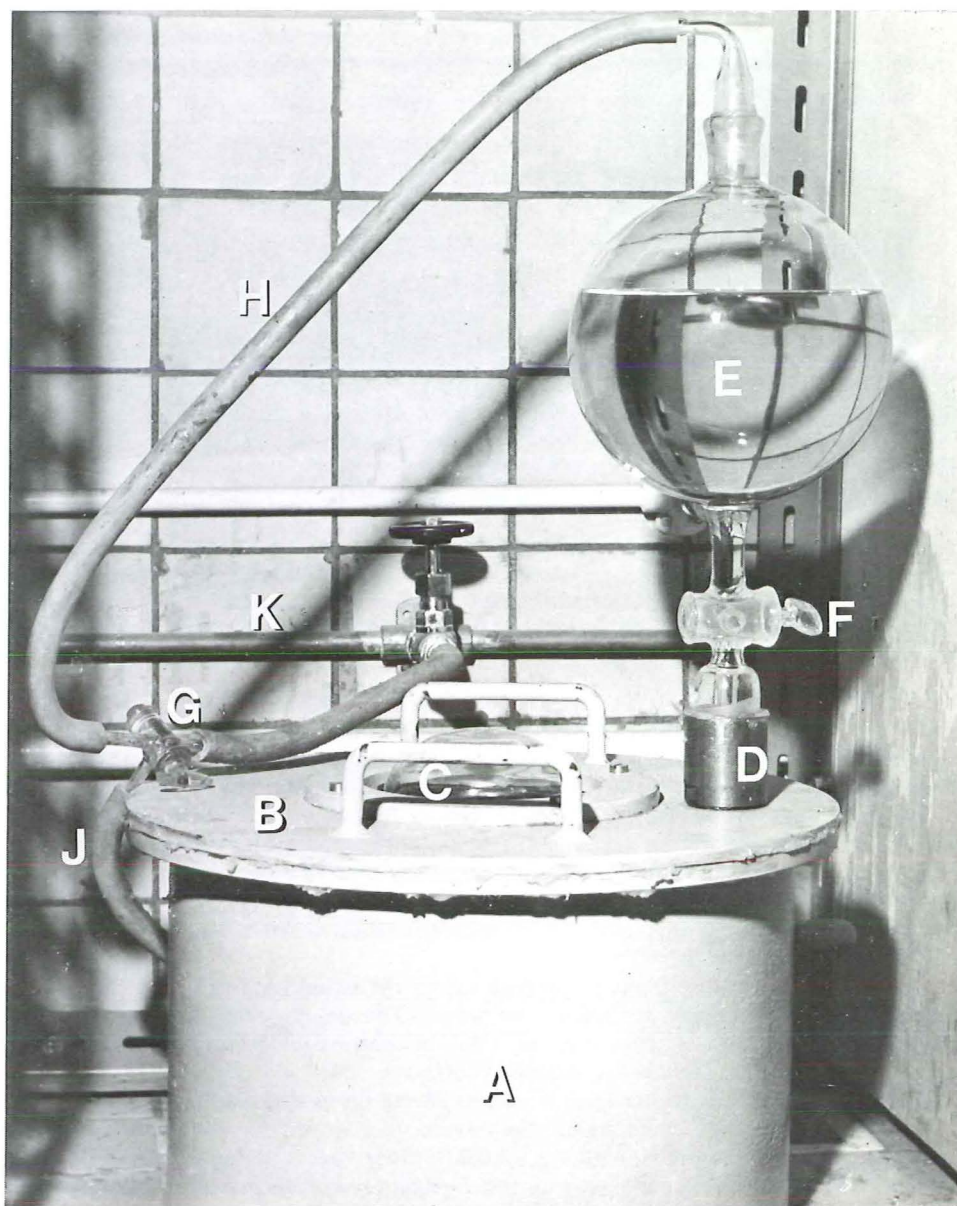


Fig. 10. Impregnation equipment consisting of a steel cylinder (A) and a cover (B) with a thick spherical observation glass (C). In the cover is a small round aperture with raised edge (D) which holds a glass vacuum sphere (E). The connection between cylinder and sphere can be closed by a tap (F). By tap G both sphere and cylinder are connected via the rubber lines H and J resp., with the line K of an electric vacuum pump.



Altemüller's. This remarkable difference could be explained as follows. Some of the monostyrene or acetone probably penetrates fairly rapidly into the soil samples, while the plastic would follow much more slowly: the monostyrene or acetone as it were prepares the way for the plastic. This is supported on the one hand by the fact that the samples are already moistened to the core a comparatively short time after the beginning of impregnation, while on the other, in a sample with the plastic polymerising too rapidly from a steep rise in the operating temperature (e.g. during hot weather) only the edges are found to be penetrated with sufficient plastic. A very long gelling time is therefore indispensable.

Experiments have shown that the length of the gelling time cannot be decreased by applying a high pressure. Also, in this case, shorter gelling times resulted in an insufficient consolidation of the cores of the samples. Nevertheless, application of high pressure is advisable as it facilitates the penetration of monostyrene or acetone, and in this way indirectly leads to a better and more complete consolidation of, in particular, very dense soil materials.

It might be concluded from the above that some of the excess monostyrene or acetone is absorbed into the soil without being released again afterwards and even that the gradual fall in the liquid level during the time in the cupboard is not caused by an evaporation of the monostyrene or acetone but by its continued absorption in the soil. If this happened, however, the concentration of the diluting agent in the samples would become so high that it would prevent proper hardening of the blocks. Daily weighings of a box of impregnated samples showed, moreover, a fairly considerable decrease in weight every day it was in the fume cupboard. This accords with the weight of a volume of diluting agent equal to the daily decrease in volume. The total decrease in weight throughout the period in the fume cupboard about equals the weight of the excess monostyrene or acetone originally added. From this it follows that the excess styrene or acetone initially present in the samples gradually disappears again.

Using the method described above, much bigger soil samples, i.e. 50 x 30 x 30 cm and even larger, can also be successfully consolidated. However, about two days are required for the impregnation in this case. For the further processing of such blocks to sections (the mastodont-sized sections) see Chapter 5.

Though the application of high pressure is advisable, most soil samples of 15 x 8 x 5 cm or smaller can be impregnated satisfactorily if the procedure is performed under vacuum only. The equipment needed in this case is much simpler (Fig. 10). For the impregnation under vacuum of four 15 x 8 x 5 cm soil samples in one box a steel cylinder with a diameter of 30 cm, which could be closed, was used (JONGERIUS and HEINTZBERGER, 1963).

The corresponding edges of cylinder and cover are greased beforehand with vaseline. The cover has a small rounded aperture with a raised edge, which holds a glass vacuum sphere. This device is manufactured by the Technical and Physical Engineering Research Service, Wageningen, The Netherlands. An impregnation vessel of such a size should be made of steel. A glass cylinder with a diameter of 30 cm (e.g. a vacuum exsiccator) would be crushed by the high vacuum, which could lead to an explosion with a strong splintering effect. Smaller vessels (20 cm diameter or less) can be made of glass. If the impregnation takes place in a vacuum vessel, only one electric vacuum pump is needed. A water-jet pump is not advised here. A minor drawback of this being that water vapour may enter the vacuum equipment and the samples may absorb some of it. This can be overcome, however, by providing a vessel of silica gel in the vacuum line. A worse feature of this pump, however, is that a several hours' evacuation is required to reach a sufficiently high vacuum in the cylinder.

## 4.5 Hardening

On completion of the impregnation process, the boxes with samples remain for a few more weeks in the fume cupboard. During this period there is a very gradual gelling followed by the hardening. When the samples have reached this stage the boxes are removed from the fume cupboard to be stored on shelves in a cool place in the laboratory. It is best to leave them there for a few weeks to give the samples time to harden thoroughly. At first the uppermost millimeters of the hardened block are still soft owing to air inhibition, i.e. inhibition of the polymerisation by the atmospheric oxygen (MALTHA, 1957). But this is only a minor matter, hardly affecting the evaluation of the state of the polymerisation in the block.

Towards the time that the samples are ready for processing, the relevant box is hardened for an additional few days in a drying oven at a temperature not exceeding 40° C. This ensures that the polymerisation reaction is as complete as possible. If necessary the box can be transferred directly from the fume cupboard to the drying oven, but this brings with it a considerably higher risk of stresses occurring in the hardened samples, than when some time is allowed to elapse before the final hardening. This is because the exothermic evolution of heat mainly takes place after the beginning of hardening. When the final hardening is not delayed this heat cannot be removed and there is a large rise in temperature in the hardening plastic.

After the final hardening the block is removed from its box. It is now ready for processing.

## 5. FURTHER PROCESSING

### 5.1 Sawing

The blocks are first of all sawn into smaller pieces. The size and hardness of the blocks make a firm sawing machine with a large saw blade necessary.

The Diamant Boart type A sawing machine (Fig. 11) with a cutting disc of 300 mm

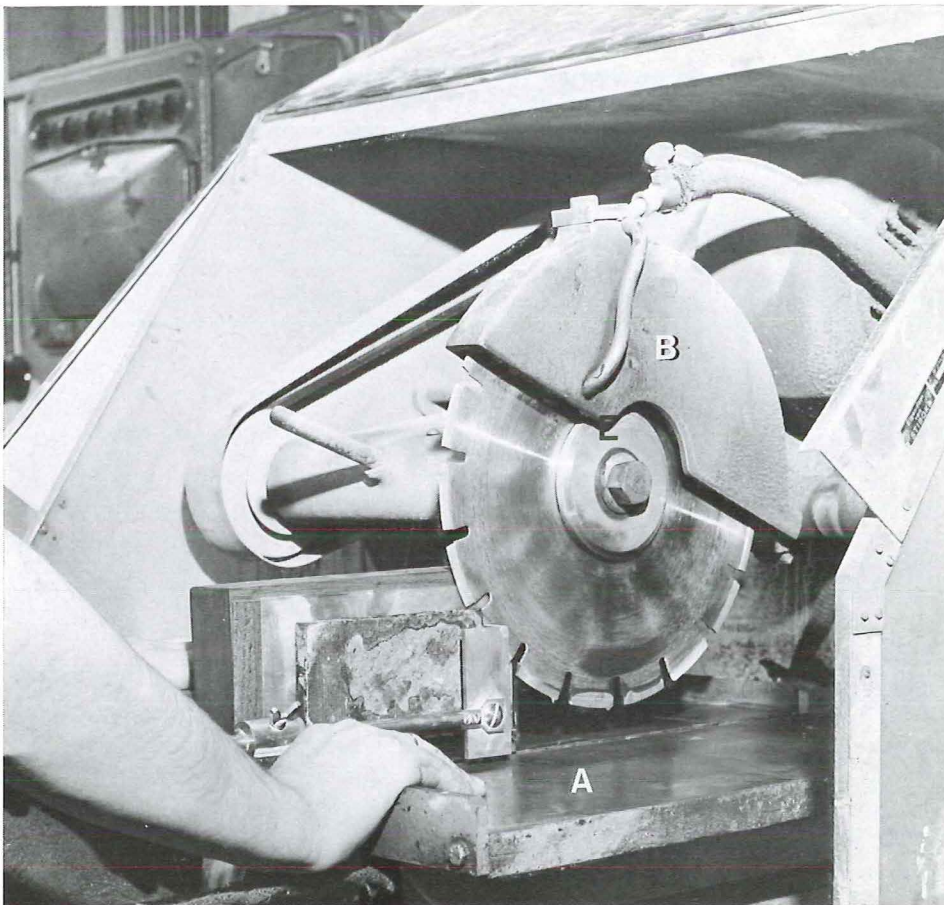


Fig. 11. The Diamant Boart type A sawing machine with a cutting disc of 300 mm in diameter. The objects to be sawn are placed on a bridge (A) and this is pushed underneath the rotating cutting disc. The material is cooled by Shell oil S - 4919 by means of tubing openings on either side of the disc (B). The sawing machine is enveloped in a plexiglass cap for protection against the strongly atomized oil and sawing sludge. For reasons of safety the sample is anchored by two metal clamps. Mounted soil sections are sawn off by means of a wooden jig (see Fig. 13).



in diameter is very satisfactory for this purpose. Either a disc with an uninterrupted diamond impregnated edge (bronze bonding: 12.3 carat) or one with diamond impregnated teeth (steel bonding: 12.7 carat) can be used; the former saws somewhat faster, while the toothed blade probably has better air-cooling. The machine must stand on a very firm table specially constructed for this purpose.

The objects to be sawn are placed on a bridge that is pushed underneath the rotating cutting disc which can be adjusted to different heights. During sawing the material is cooled by means of tubing openings on either side of the disc (Fig. 11B). Water is not used for the cooling, but the volatile Shell oil S-4919. Water causes the equipment to rust, and various soil components, such as clay minerals and organic matter to swell, even in a completely impregnated and thoroughly hardened sample. During grinding

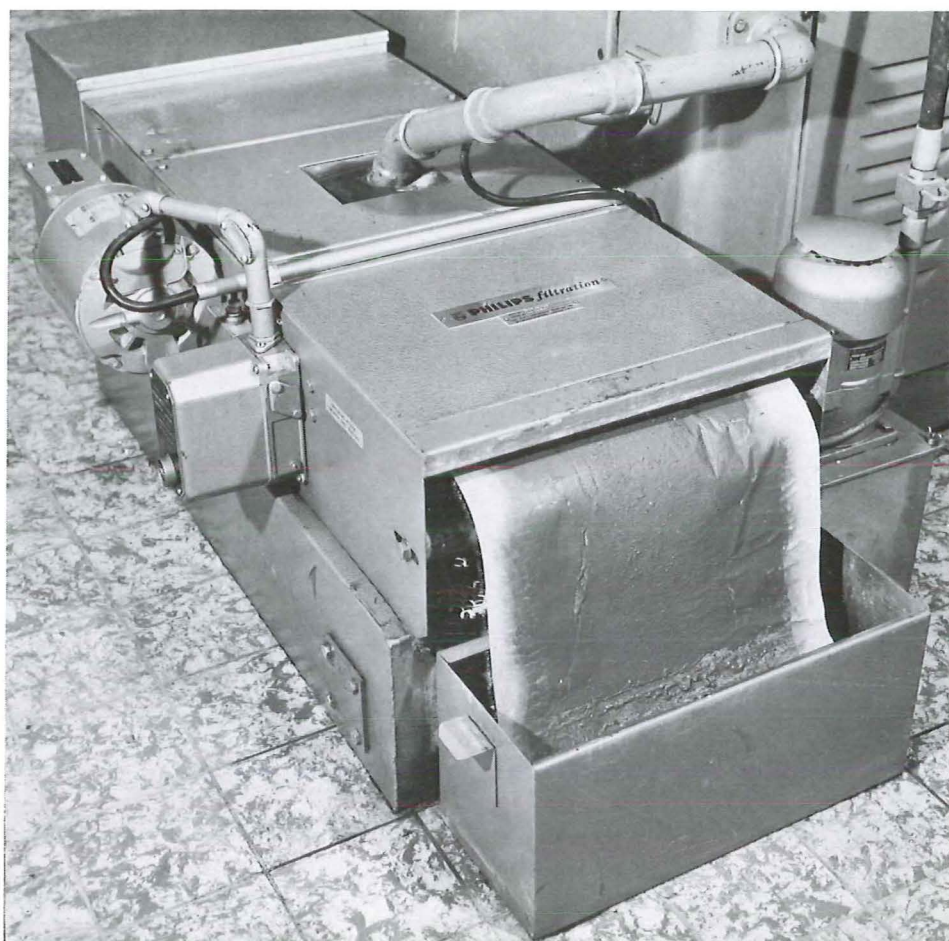


Fig. 12. Philips filtration equipment type no. E 7744/25. The apparatus is filled with a roll of filtration paper. When the paper is clogged by sludge and fine grinding powder, it is automatically moved on, so that clean paper for filtration becomes available.

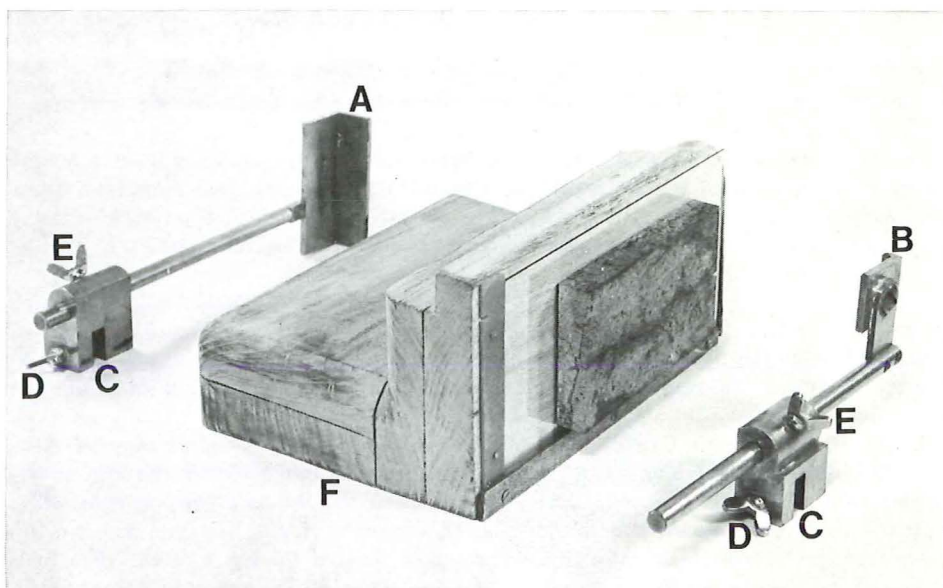


Fig. 13. The metal clamps (A and B) used in cutting the hardened soil material with the Diamant Boart sawing machine. The grooves (C) are pushed on the raised edge of the bridge and screwed on by butterfly nuts (D). The length of the arm is adjustable with the butterfly nuts E. The retaining piece of the left clamp is much longer than that of the right one. Apart from clamping the left clamp has a bearing function, viz. to prevent the sawn off plate from being hurled away. F: Wooden jig for the sawing off of mounted soil sections. The sections are clamped to the jig by thin bakelite strips.

this causes holes to appear in the preparations. The same oil therefore is also used in the grinding process (see below).

The used oil runs via a drain into a Philips filtration equipment type no. E 7744/25 (delivered by the M.E.L. Equipment Company, London) (Fig. 12). In this automatic equipment the sludge and fine grinding powder which appear in the oil are removed by means of Philips filtration paper E 7603/03D. With the aid of a small electric pump, which is part of the filtration equipment, the cleaned oil is led back to the sawing machine. In this way the oil can be used again and again for rather a long time.

The sawing machine is covered by a plexiglass cap for protection against the strongly atomized oil and sawing sludge.

The several samples in the block are now sawn out, with as far as possible the faces at right angles to each other. Where the samples are vertically oriented, the top is indicated by a saw-cut. Each sample is then placed in a soil sample bag showing sample number, place of origin, and depth.

The samples are now processed one by one to thin sections. To do this, the soil is sawn into sections of at least 8 mm thick. This is important as a thinner section would very soon warp. Even an 8 mm thick one should be ground, polished, mounted on an object glass and weighted down (see 5.3) the same day if warping is to be avoided. In spite of the very long hardening time, the polymerisation process may not be fully completed at the time of sawing, or the warping may be due to stresses in the hardened



plastic. This phenomenon does not however affect the quality of the thin section in the least.

For reasons of safety metal clamps specially constructed for this purpose are used for sawing the samples (Fig. 13). These are only suitable for use when the specimen is cut out at right-angles.

To saw very large hardened blocks, a Woco 220/700 universal sawing machine, which is manufactured by the Wolfgang Conrad Factory, Clausthal-Zellerfeld, West-Germany (Fig. 14) was recently put into use. Because its speed of rotation is variable, this machine can be equipped with diamonded discs of different sizes, i.e. varying from 350-700 mm diameter.

The disc, which is vertically mounted directly underneath the bench of the machine, protrudes to a length which is dependent upon its diameter. For a disc of 700 mm for instance, this length is 280 mm. That means that, as the sample turns during the sawing procedure, with a 700 mm disc samples of a diagonal length of 560 mm maximum can be processed.

The sample is fixed in a revolving clamping device which is connected to a swivelling arm. During sawing this arm gradually moves down. The movement of the arm as well as the rotation of the clamping device occur automatically, and are variable within rather wide limits. However, both can also be moved by hand, and the arm can also be moved downwards by means of a weight. The desired thickness of the slice to be sawn off can be chosen by adjusting the arm sideways. With such a machine, for instance, slices of 350 x 450 mm and not more than 3 mm thickness can be sawn off. The sawn surface of such a slice is very flat, i.e. the height differences do not exceed 0.02 mm.

Furthermore, by means of a suction plate device which is part of the Woco machine, slices mounted on glass plates (see below) can be sawn off to thicknesses of 0.2 mm and less.

This machine can also be used for grinding, although the authors have not used it for this purpose. The cooling liquid is again Shell oil S-4919, and for reasons of protection the Woco-machine is also covered by a (closed) plexiglass cap.

## 5.2 Grinding of a sawn surface

This is done in two stages, viz. (1) machine planing and (2) grinding by hand.

Since the soil sections are very hard and large, machine grinding cannot be done on the usual rotating discs. Kubiena and Von Buch (ALTEMÜLLER, 1962), however, introduced the semi-automatic Klaiber surface-grinding machine type HV II for the preparation of small thin sections which the authors also used for the grinding of larger slides (JONGERIUS and HEINTZBERGER, 1963). Nowadays because they save considerable labour and have a greater capacity, even much larger surface grinders are in use, such as the fully automatic Blohm HFS 6 with an electromagnetic clamping table of 60 x 30 cm (VON BUCH, 1967).

The authors use a semi-automatic Matra MF 80/40 (manufactured by Matra-Werke, Frankfurt/M, West-Germany. This machine (Fig. 15), which like other surface grinders is used in industry for very fine grinding work, has a vertically mounted stone rotating above an electromagnetic clamping table 80 x 40 cm large. The table, which should be exactly level in all directions moves to and fro automatically in the plane of the grinding stone; it can be moved at right-angles to the stone rapidly by hand or slowly automatically. After each grinding the stone is twined a bit lower, initially about 50  $\mu$ m



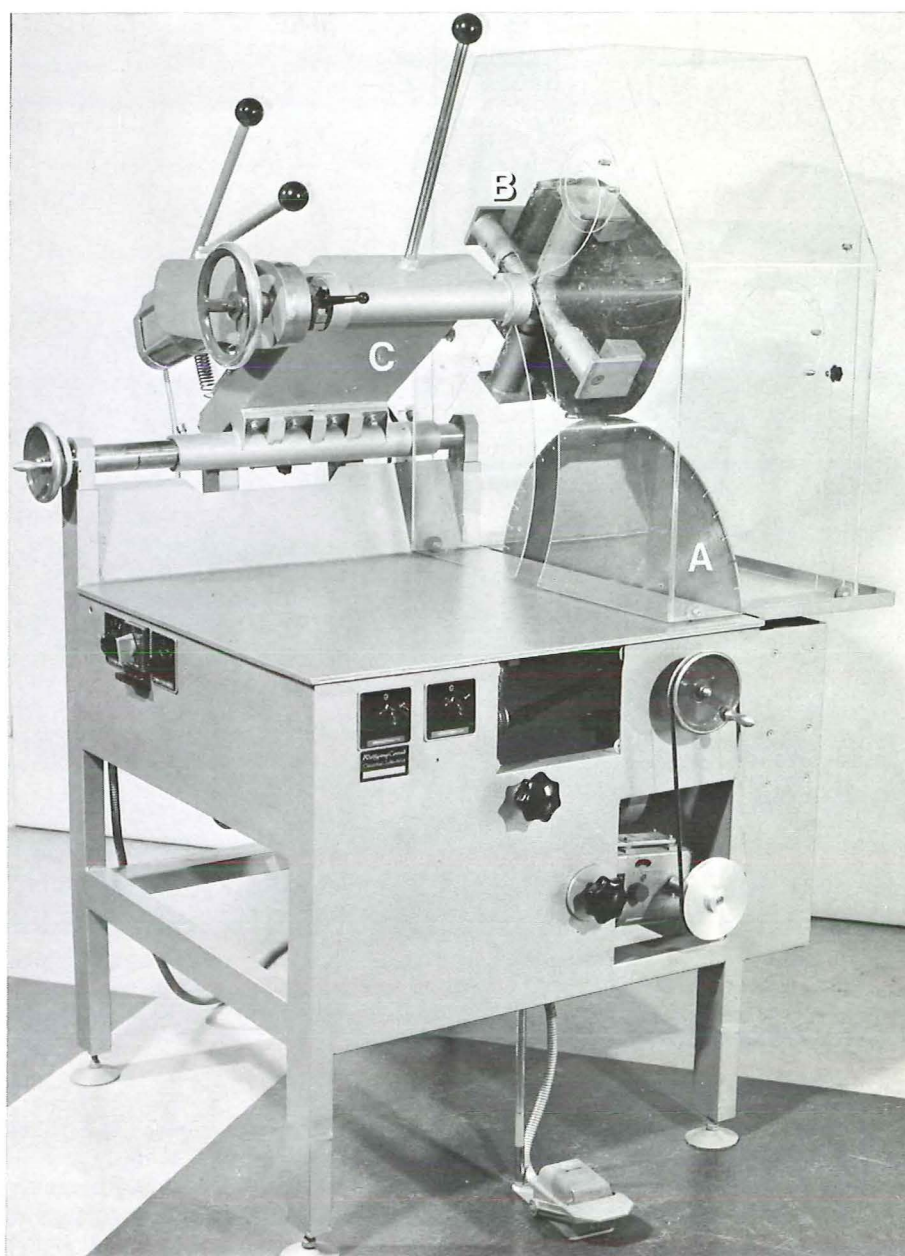


Fig. 14. The Woco 220/700 universal sawing machine. On the picture the machine is equipped with a sawing disc of 700 mm in diameter (A). The sample is fixed in a revolving clamping device (B), which is connected to a swivelling arm (C). This arm can move downwards and sideways.

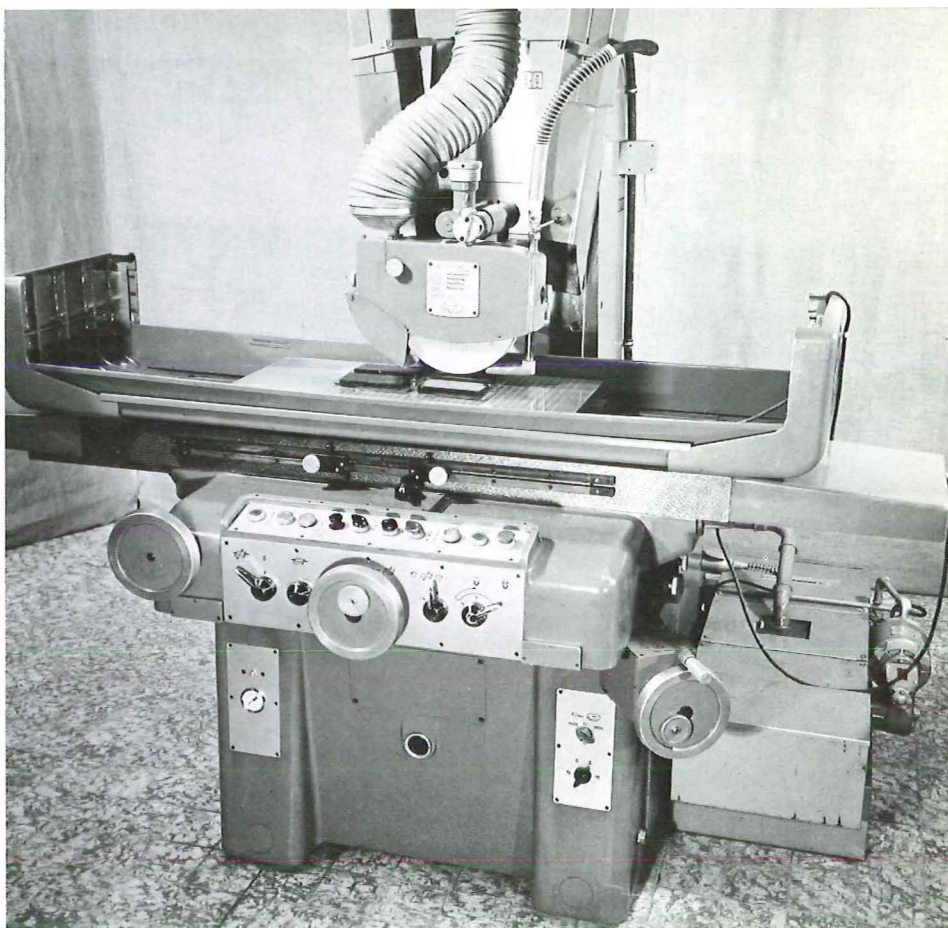


Fig. 15. Matra MF 80/40 grinding machine. The grinding stone rotates vertically above an electromagnetic clamping table, which moves to and fro automatically in the plane of the grinding stone. Because the cooling liquid Shell oil S - 4919 atomizes strongly, the machine is equipped with three exhaust chutes (top of the photo).

every time for sandy samples and about  $75\text{ }\mu\text{m}$  for clayey samples. This should be reduced to only a few micrometers every sweep in a thinly ground section.

Grinding stones of ceramically bound Si carbide, grain size 220 and Lg hardness (a.o. manufactured by Jannink and Co, Enschede, The Netherlands) are quite suitable for making thin sections. In an earlier publication (JONGERIUS and HEINTZBERGER, 1963) it is stated that (when a Klaiber machine is used) for the preparation of larger thin sections a stone of 2 cm thickness is preferable to a 3 cm one, which is used by ALTE-MÜLLER (1962) for smaller sections. This is because with a narrower stone there is less risk of the section fracturing, especially when it is fairly thin. On the big Matra machine, however, stones of 5 cm thickness can be used without any difficulty. This is probably due to the minimum vibration and the high stability of that machine.



One of the sawn off sections is now placed on the clamping table, after the sample number has been scratched onto the lower surface with a diamond pencil, and it is enclosed by two polished steel angles of about 5 mm thick. The steel angles are drawn onto the table by the electromagnetic field so that they rigidly anchor the section. The upper side of the section is then planed down. Initially the grinding machine is operated manually after which it is switched to fully automatic grinding. The section is then still about 8-10 mm thick.

The planed side of the soil section is then finished by hand on 500 x 300 x 12 mm glass plates. For this purpose some aluminium-oxide powders of decreasing grain size are used. The corundum powders sold by Messrs. Feldmühle of Düsseldorf-Oberkassel, Western Germany, are recommended for this as the very small variations in grain size of these powders minimize the risk of scratches on the surfaces, which are already ground very smooth. The powders successively used are: BAR 30 (grain size approx. 36 - 26  $\mu\text{m}$ ) and BAR 23 (grain size approx. 26 - 18  $\mu\text{m}$ ). When grinding clay and peat samples BAR 30 can be omitted.

ALTEMÜLLER (1962) uses corundum powders 23 and 13, and in special cases also 15  $\mu\text{m}$  diamond powder. Diamond powders produce a very flat and clean preparation but they are very expensive.

As for the machine planing, the liquid used for grinding by hand is Shell oil S-4919. Cleaning petrol may also be used. Several precautions must be taken to prevent contamination of the powders used in the various stages of grinding (the risk of scratching!). In the first place the glass plates should be kept carefully apart for example by using wooden or plexiglass partitions. After every stage of grinding the preparations should also be thoroughly cleaned in petrol. This can be done with a soft brush, but much better results are obtained with the Ultramet II Sonic Cleaner, manufactured by Buchler Ltd, Evanston, Ill., U.S.A. (Fig. 16). Fire or explosion risk make a high flash-point petrol advisable for this purpose (ALTEMÜLLER, 1962). Provided the necessary precautions are observed, however, ordinary cleaning petrol is also very suitable for cleaning. Should one of the grinding powders nevertheless be contaminated with a coarser one or with a grain of sand broken out of the preparation, the relevant glass plate should be completely cleaned at once, and the grinding resumed with pure powder.

If the slices are to be processed to thin sections, the ground surfaces are usually not polished with diamond (paste). Experience has shown that a polished surface of 15 x 8 cm mostly does not usually fix very strongly to the object glass onto which it is mounted (Cf. 5.3), so that the section can be damaged or even destroyed during the thin grinding of the preparation (Cf. 5.5). However, polishing of the ground surface is not essential to obtain a thin section of high quality. Only in some cases high polishing with diamond is necessary, e.g. if slices or thin sections are made for incident light investigation, and if preparations should serve for energy-dispersive X-ray analysis (BISDOM, HENSTRA, JONGERIUS and THIEL, 1975). The polishing technique is described below.

### 5.3 Mounting

After the soil section has been properly dried (if necessary this can be speeded up by holding it about 1 metre away from a hot air blower) the polished side of the sample is mounted onto an object glass of about 1.2 mm thick and 20 x 10 cm large. The glass should have the optical properties of the usual smaller object glasses. The sample number is first scratched with a diamond pencil into one end of the glass and the glass





Fig. 16. Ultramet II Sonic Cleaner. The cleaning liquid is petrol.

is then thoroughly cleaned with xylene or acetone.

For mounting, the following materials are used per section:

10 cc Synolite 544

0.1 cc Cyclonox LNC

0.05 cc Cobaltoctoate 1 %.

The gelling time of this mixture is about  $1\frac{1}{2}$  hours at room temperature. With an operating temperature higher than about  $22^{\circ}\text{C}$  half the amount of both catalyst and accelerator are used.

The mounting is done as follows (Fig. 17): the section is placed on a table with the ground side upwards, and a little of the mixture is poured onto the section and evenly spread out with a stirring rod. Any cracks or small cavities in the surface can then be filled up with the plastic. A layer of the mixture is then applied to one of the long edges and after the air has disappeared from the plastic, the object glass is applied to the section in a hinge-like motion. This prevents any air bubbles from being trapped underneath the object glass.

The section is joined exactly parallel onto the glass by weighing it down with about 1450 g. The weight has a polished base of  $7 \times 14.5$  cm, and it consists of a copper jacket filled with lead. To prevent the glass from sliding over the preparation it is secured at the corners, for example by means of large rubber stops (Fig. 18). Covering the working table with aluminium foil will prevent excess plastic from sticking the section to the table. The mounted specimen is left like this for about 24 hours.

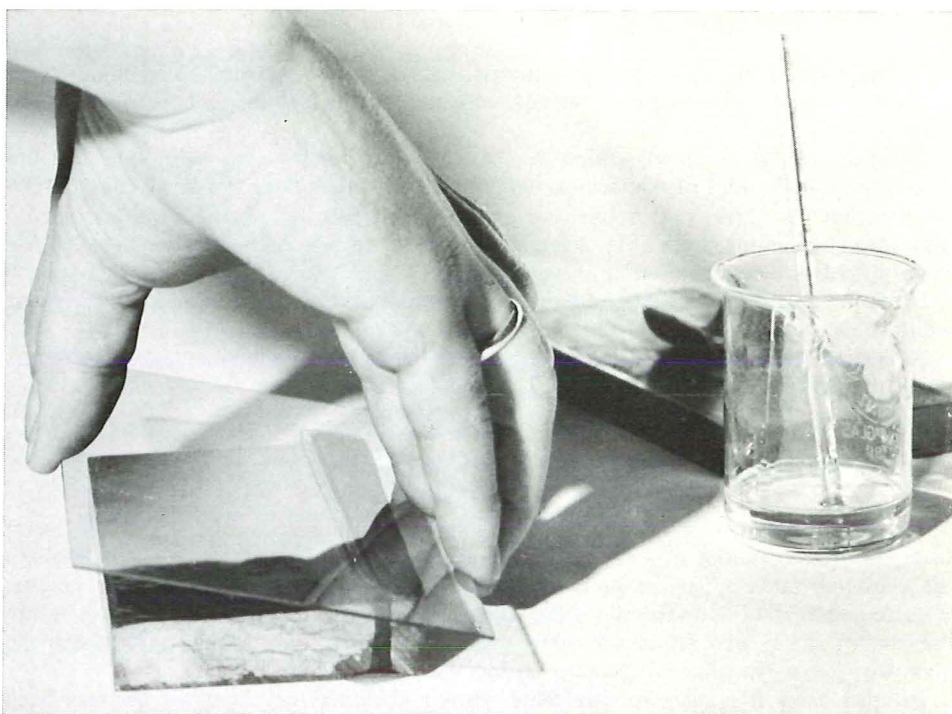


Fig. 17. The mounting of a soil section on an object glass. The grinded and polished side of the section is face upwards and is covered by a thin layer of plastic. A thicker layer is then applied to one of the long edges (the dark strip at the right) and the glass is applied to the section in a hinge-like motion.

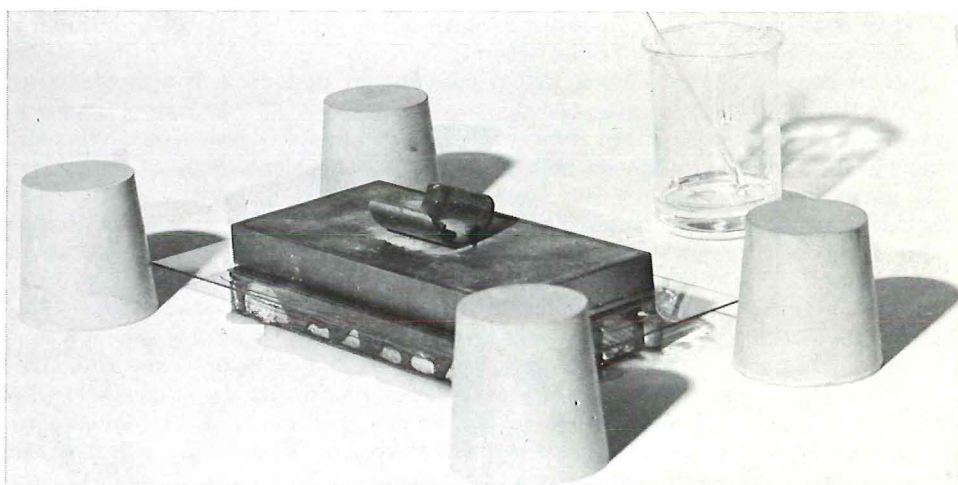


Fig. 18. Weighing down the object glass promotes an equal thickness of the cementing layer. The rubber stops prevent the glass from sliding over the section.

#### 5.4 Sawing the mounted preparation

With a special jig (Fig. 13) the mounted section can be reduced to a thickness of about 2 mm with the Diamant Boart saw, which eliminates about 1½ hours of machine grinding.

The glass of the mounted section is inserted into a narrow slot made with bakelite strips along the bottom of the vertical side of the jig. Strips along the short edges of the side prevent the glass from being torn out of the jig during sawing. A safety clamp with a long retaining piece (Fig. 13) prevents the 5 to 6 mm thick slice of soil being sawn off the section from being hurled away and thus damaging the section.

If the processing of the sawn section is interrupted at this stage it should be weighted while being stored (see 5.3) to prevent warping.

As already described, with a Woco machine the mounted sections can be sawn off much thinner, e.g. to a thickness of 0.2 mm and even thinner.

#### 5.5 Thin grinding and polishing

The section sawn with a Diamant Boart machine is now reduced from about 2 mm to a thickness of about 40  $\mu\text{m}$  on the grinding machine. The object glass is fixed to the clamping table by means of two 1 mm thick and very flat, polished steel angles. Both the table and the underside of the object glass should be thoroughly cleaned before the preparation is clamped to the table; even one minute grain of sand underneath the glass may cause the slide to fracture during the grinding at this stage.

In most cases it is easy to determine when a thickness of about 40  $\mu\text{m}$  has been reached as the section then becomes somewhat transparent and there is often a distinct colour change to yellowish tints. It can be mentioned here that much thinner grinding is possible on the grinding machine, even to less than 20  $\mu\text{m}$ . This is however not advisable as at this stage the sand grains begin to crack.

The fine finishing of the slides is performed by hand in the same way and with the same grinding powders as already described above for the grinding of the slides. Grinding is continued with the powder BAR 13 (grain size approx. 18-10  $\mu\text{m}$ ) until a thickness of about 20  $\mu\text{m}$  has been reached.

The last phase of the grinding as well as polishing are performed on a special Wirtz machine (Fig. 19) which is equipped with two rotating discs of 350 mm in diameter, the speed of which can be varied from 0-500 r.p.m. and 0-250 r.p.m. respectively. The grinding disc consists of diamond impregnated synthetic material (diamond grain size 8-15  $\mu\text{m}$ ). On this disc grinding (Fig. 20) is continued until a thickness of about 15  $\mu\text{m}$  has been reached, which is indicated by the quartz grains becoming grey under crossed nicols. For cooling, again Shell oil S-4919 is used.

The polishing disc is made of copper. For the polishing AB Metadi diamond paste, grain size 1  $\mu\text{m}$  (manufactured by Buchler Ltd., Evanston, Ill., U.S.A.) is used. Polishing takes place at about 50 r.p.m. This speed is much lower than that generally employed in industry for polishing (viz. up to about 2000 r.p.m.). In the latter case, however, a smooth surface is produced by plastic deformation under the influence of frictional heat (BURKART, 1959), whereas in thin section preparation plastic deformation and hence a high speed are the very things to be avoided: the polishing with diamond paste only serves to remove the last small irregularities.

Mastodont-sized sections are not thinly ground and polished, and they are also not covered with glass.





Fig. 19. A Wirtz machine for fine grinding and polishing. The machine is equipped with rotating Triefus discs of 350 mm diameter. The speed of the left-hand disc can be varied from 0 – 250 r.p.m., that of the right-hand one from 0 – 500 r.p.m. Discs for fine grinding consist of diamond impregnated synthetic material, and a copper disc is used for polishing.

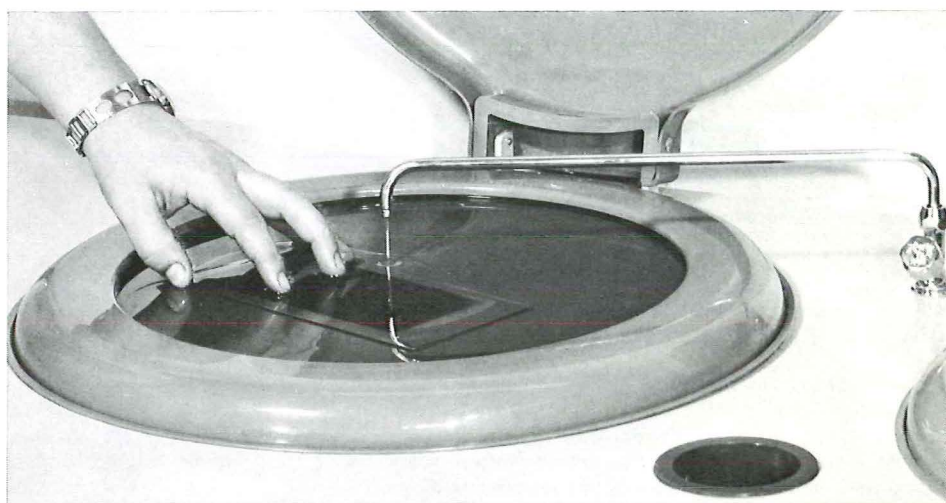


Fig. 20. Fine grinding on the Wirtz machine. The cooling liquid is Shell oil S – 4919.

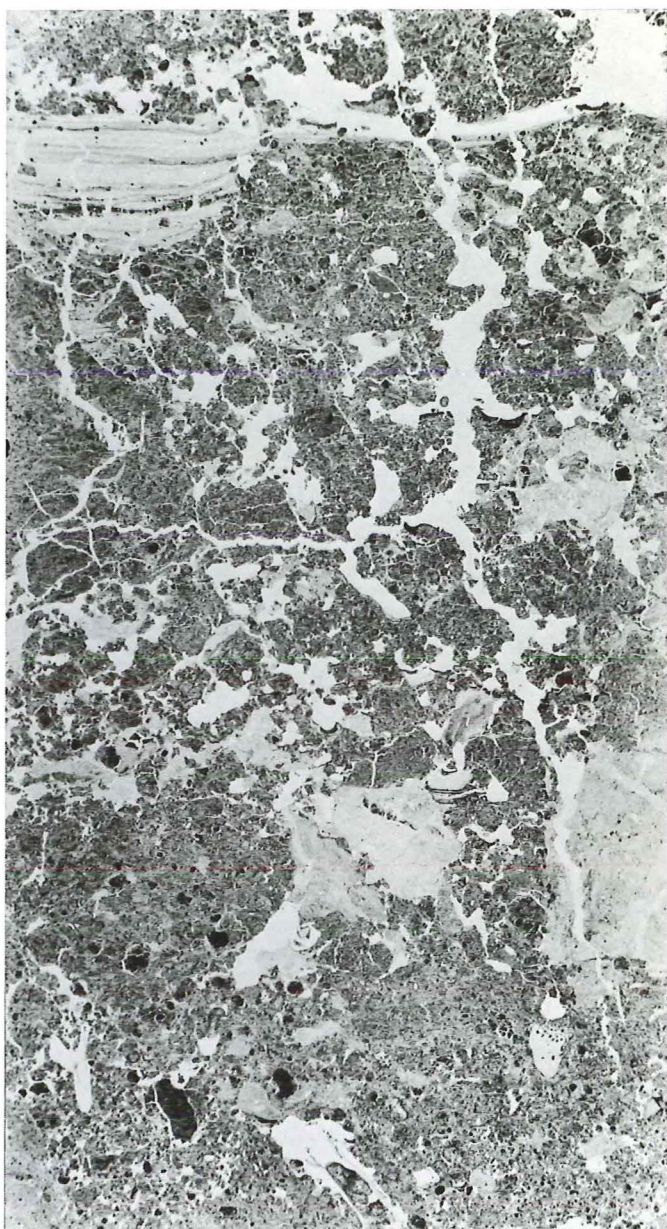


Fig. 21. Mammoth-sized thin section (full-size) of a soil thoroughly mixed by machinery: an Aquoll developed in marine silty material (sample of 0 – 15 cm depth). Structure, mixing and internal slaking are markedly demonstrated by this thin section (black and light-grey rounded elements: subsoil; dark-grey groundmass: topsoil; dark and greyish cutans and horizontally laminated material: accumulations due to slaking). Magn. 1.1.



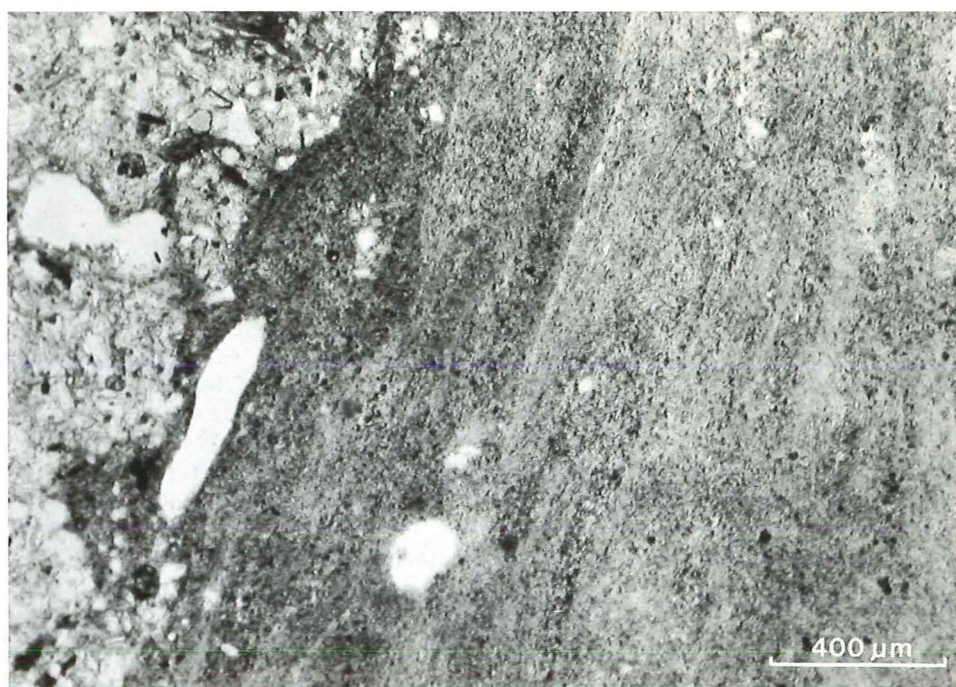


Fig. 22. Detail from a mammoth-sized thin section: illuviation of a peat mud in old marine clay (the marine clay at the left of the picture: light grey). The laminated vertically oriented structure of the illuviation cutan, consisting of a succession of bands rich in clay, fine sand and organic matter respectively, is distinctly visible in every detail. Sample of 50 – 65 cm depth; Ter Aar (Western Netherlands).

## 5.6 Covering

After the thin section has been properly dried, which takes at least half a day at room temperature, the section is coated along its long axis with a layer of plastic of the same composition as that used for mounting. After waiting for the air to escape from the plastic, a cover glass 160 x 80 x 0.2 mm is placed on the slide. By pressing the plastic from the centre outwards all the air under the cover glass is expelled. The covered preparation is weighted down (Cf. 5.3) to ensure that the coating layer is of uniform thickness. As soon as the plastic gels, the excess along the edges of the cover glass is removed with a razor blade. After it has hardened properly the finished section is thoroughly cleaned with acetone.

## 5.7 The completed thin sections

Labels giving various data (such as record number, place of origin, type of project, etc.) are attached to the free ends of the object glass. The slides are now ready for microscopic examination.

Figures 21 and 22 give some idea of the quality of the mammoth-sized sections, while they also illustrate some of the possible applications of these kinds of slides.



## 6. SAFETY PRECAUTIONS

In using the method described above the following points should be observed (ANONYMOUS 2, no date; ANONYMOUS 4, 1959):

1. The working space must be well ventilated for the rapid extraction of monostyrene, acetone and petrol vapours.
2. Owing to the inflammable nature of the unsaturated polyester resins, the monostyrene, the acetone, the organic peroxides and the petrol, all forms of open flame must be avoided. The working space should be explosion free (spark formation!) illuminated.
3. In view of the explosive nature of the catalysts they should not be mixed directly with accelerators. For this reason they must also be well packed and stored separately in a cool, dark and fireproof place, away from other inflammable chemicals.
4. Great care should be taken when working with liquid organic peroxides in particular. Although they are not serious poisons they have properties liable to cause serious injury. Should they be swallowed by accident (e.g. in pipetting) vomiting should be induced immediately and large amounts of sodium ascorbate dissolved in water should be drunk. The stomach should then be emptied with a stomach pump. Since several organic peroxides (amongst them cyclohexanone peroxide) may cause blindness if they come into contact with the eyes, it is essential to wear safety glasses. Should some still enter the eye it must be intensively washed with water, or preferably with a 5 % solution of sodium ascorbate or a 2 % sodium bicarbonate solution. In the latter cases the eye should afterwards be rinsed with water for at least 15 minutes (never with oil or liquids containing oil). The sodium ascorbate solution is the most active antidote, but it should be freshly prepared every month. It is almost superfluous to say that a physician should be called in immediately in the above cases.

Contact with the skin should be avoided. Hypersensitivity may develop, especially if this occurs repeatedly, which gives rise to prolonged eczema. These unpleasant consequences can be avoided by wearing special gloves, for example plastic gloves lined with cloth. Should the peroxides come into contact with the skin despite this precaution they must be removed with alcohol and the area then washed with soap and water. Lanoline ointment should then be applied.

Splashes on clothing are also dangerous, as they may cause spontaneous combustion.

Prolonged contact of the skin with Synolite should also be avoided. The hands must be thoroughly washed with soap after work and any adhering residues removed with acetone. A barrier cream should be used on the hands both before and after using Synolite.

Safety glasses, special gloves and a plastic apron should be used during quick-freezing.

5. Shell oil S-4919 atomizes strongly, especially during sawing and it is easily inhaled this way. A breathing mask should therefore be used during this work, and the provision of suction fans near the sawing and grinding equipment is also very desirable. Eczema on the hands and arms can be prevented by covering with protecting ointment such as Silicoderm-F (Bayer) both before and after sawing, and regularly washing with disinfectant soap. The use of plastic aprons too is very advisable.

6. Safety clamps should be used during sawing. On Woco machines such clamps are not needed.

The authors do not want to end without touching on the one drawback of the method discussed, namely the fairly expensive equipment involved. This makes it unsuitable or less attractive for many workers. At the same time the authors believe that the method

described can be adopted to make thin sections of about 5 x 5 cm. This requires a fairly small diamond saw and a rotating grinding disc. Working with smaller samples allows the impregnating equipment to be much smaller and made of glass, and the air can be evacuated with a water-jet pump, though this is not advisable (Cf. Chapter 4). The hardened specimen is sawn up into slices on the small sawing machine, ground on the revolving disc, worked up by hand, mounted, then brought down on the small saw again, finished by hand, and covered. Although these thin sections do not offer the same possibilities as the mammoth-sized sections, they still provide much more information than the small 'conventional' slides.

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