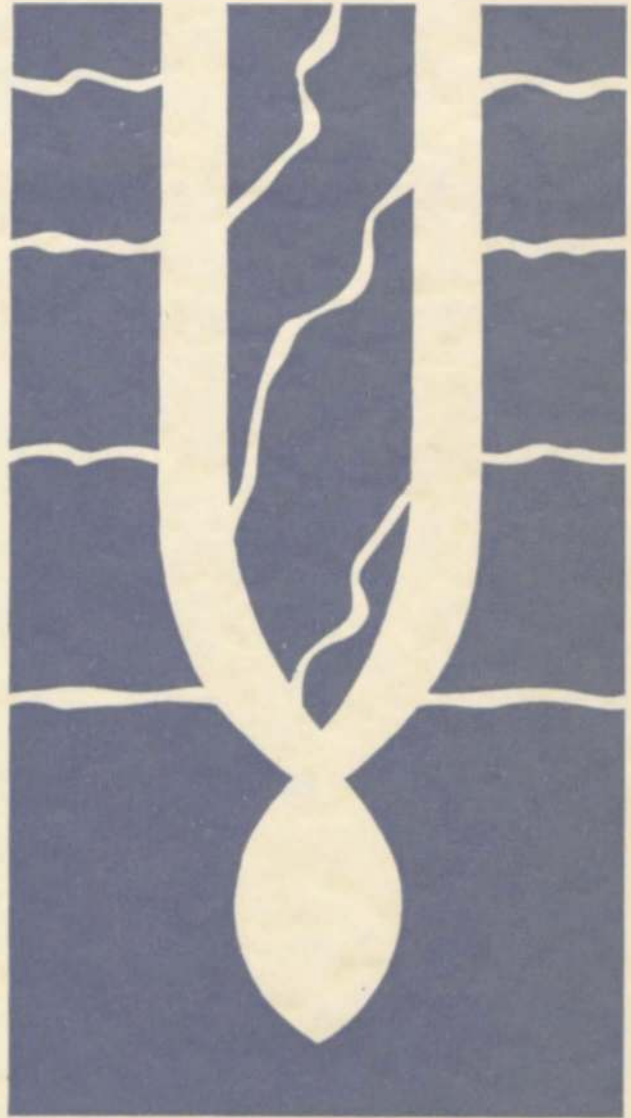


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survey papers

Nr. 1



THE PREPARATION OF
MAMMOTH-SIZED
THIN SECTIONS

by A. Jongerius and
G. Heintzberger

survey institute, Wageningen

RECTIFICATION

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SOIL SURVEY PAPERS

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No. 1

A. JONGERIUS

and

G. HEINTZBERGER

THE PREPARATION OF MAMMOTH-SIZED THIN SECTIONS

THE SOIL SURVEY INSTITUTE - WAGENINGEN
THE NETHERLANDS

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PREFACE

With the first number of this new series it is appropriate to state the object of adding yet another series of publications to the already crowded field of contributions to the knowledge of Soil Science.

The Netherlands Soil Survey Institute, at Wageningen, has up until now published its work in the following series: DE BODEMKARTERING VAN NEDERLAND, (Soil Survey of the Netherlands), BODEMKUNDIGE STUDIES, (Studies on Soil Science) and BOOR EN SPADE, (Auger and Spade), with the latter subtitled "Miscellaneous Contributions to the knowledge of the Soils of the Netherlands". All these publications are in Dutch, with a summary in some other language, usually English.

It has been found more and more appropriate however, that different problems around the soils of the Netherlands, and the ways in which they are solved, command a wider interest. Therefore it was considered desirable to make that part of the Institute's work of more than national or regional importance accessible to workers in other language areas. For this English — as the language capable in reaching most people — was chosen.

The title of these SOIL SURVEY PAPERS reflects the field of study, and the nature and scope of the problems treated. The word "survey" is used here in the wider sense of examination, while the word "papers" serves to indicate the separate nature of each subject. In other words, every number in this series deals with one special subject having as background some aspect of the soils of the Netherlands.

The Director,
F. W. G. PIJLS

THE PREPARATION OF MAMMOTH-SIZED THIN SECTIONS

De vervaardiging van mammoet-slijpplaten

by/door

A. JONGERIUS AND G. HEINTZBERGER

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the Netherlands

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2. Theory of the hardening process
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Summary

The method described here deals with the preparation of the so called mammoth-sized thin sections (15 × 8 cm large and only 15 microns thick) with the unsaturated polyester resin Vestopal-H. The sections can be made from loose as well as dense soil samples. The paper includes a brief survey of the development of the thin section technique. It treats the main aspects of the theory of the hardening process involved in the present method, in so far as a knowledge of it is considered necessary. The method is described in detail, with the pre-treatment of the samples, the resin, the catalyst and accelerator, the impregnation, the hardening, the sawing, the grinding, the polishing and the mounting of the slides each dealt with in turn.

In conclusion the safety precautions considered necessary for handling the polyester, the catalyst, the accelerator and the other materials used in preparing the sections are summed up.

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. They all however, involve the principle of impregnating (usually in vacuo) an air-dried sample of undisturbed soil. The material used for this must on the one hand be 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, they all have one or more properties limiting their suitability or adversely affecting the quality of the thin sections.

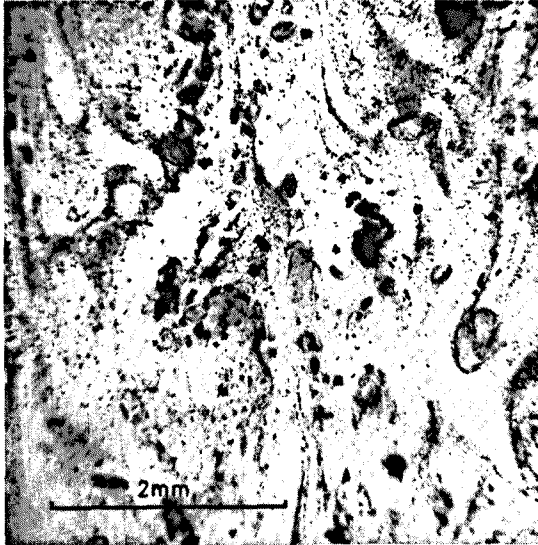
Thus, for example, disturbances may occur in the structure even during impregnation. Several hardeners are namely only sufficiently fluid at fairly high temperatures (of up to about 150°C). As a result the soil sample is "cooked" in the impregnant. This is often accompanied by violent foaming, which 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.

As well as brittleness, impregnants may have various other shortcomings such as insufficient hardness, lack of impregnating power, lack of cementing power or a low melting point. These may give rise to various difficulties during grinding, like the grinding away of sand grains, the contamination of thin sections by grinding powders, smearing, etc. Fig. 1 very clearly illustrates the last two defects. At the same time it is practically impossible to make flawless slides which are thinner than 25 to 30 microns, which is too thick for many investigations. Another feature of these deficiencies is that usually no thin sections larger than 3 cm square can be obtained. 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 with the different hardeners should serve to indicate that the percentage of inferior thin sections and failures is usually high, while the dimensions of the preparations are bound to limits which it is practically impossible to exceed. Sandy soils present the greatest difficulties (disruption of the microstructure, removal of sand grains, etc.) as do very dense soils (insufficient impregnation).

Over recent years however, great improvements have been obtained with unsaturated

polyester resins. Although HAGN (1953) and ALEXANDER and JACKSON (1954) amongst others had already used these materials, it was ALTEMÜLLER (1956, 1962) in particular who, by introducing the unsaturated polyester resin Vestopal-H, made a very important contribution towards the solution of many of the difficulties referred to above. The complete impregnation of dense samples however requires a very elaborate treatment



Photo/Foto Stiboka nr. 9810

Fig. 1 Extreme example of a failure. Thin section of soil hardened in vernicolor. During grinding the vernicolor softened by frictional heat. Soil components were grinded away and moved (see sand grain at the right side of the photograph). The silicium-carbide grinding powder (the fine-sized dark grit on the photograph) was oriented in more or less vertical streaks towards the direction of grinding (notice also the scratches on the left side of the photograph) and was cemented in the resin.

Extreem voorbeeld van een mislukt slijpplaatje van grond gehard in vernicolor. Gedurende het slijpen is de vernicolor zacht geworden ten gevolge van wrijvingswarmte. Daardoor zijn de bodemcomponenten losgerukt en verslept, wat onder meer zeer fraai te zien is aan de zandkorrel aan de rechterzijde van de foto. Het silicium-carbide-slijppoeder (het fijne donkere gruis op de foto) is verkit geraakt in de kunsthars en in min of meer verticale banen georiënteerd, d.w.z. in de slijprichting welke duidelijk herkenbaar is aan de krassen links op de foto.

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 large sized sections to be prepared. Initially this appeared impossible with Vestopal-H. ALTEMÜLLER's method was developed in the first place for sections 28×48 mm large. With it large sections (so called Grosschliffe, 6×9 cm) can also be made, but these have the

disadvantage of being much thicker (approx. 30 microns). Here again denser samples require repeated impregnation (ALTEMÜLLER, 1962).

Building on the work of ALTEMÜLLER and also using Vestopal-H, the authors have developed a method for preparing thin sections only 15 microns (or less if required) thick and as large as 8×15 cm. These sections can be made at room temperature and of any type of soil, including the heaviest. On the one hand they provide an immediate picture of the macrostructure of a horizon. At the same time such mammoth-sized thin sections allow a very detailed examination at the highest microscopic magnifications. Their only defect is that the hardened Vestopal-H has a refractive index somewhat higher (namely 1.552 ¹⁾) than that of Canada balsam.

Since the mammoth-sized thin sections are not only important in connection with soil-genesis and applied agricultural investigation (e.g. the effect of cultivation), but also for sedimentology, soil mechanics, archaeology and the ceramic industry, a very detailed description of the method for preparing them will be given in the following pages.

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

¹⁾ The refractive index of Vestopal-H was determined by DR. D. J. G. NOTA of the Regional Soil Science, Geology and Mineralogy Department of the Agricultural University, Wageningen.

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 with the content of these acids (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, styrene bridges between the polyester chains and a spatial net structure being formed (*cross-linking*).

This copolymerisation is not a spontaneous reaction, but it 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 ultra-violet 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. With simultaneous 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, 1954; CYWINSKI, 1960). This in spite, the term "catalyst" will be used in the remainder of the paper as it accords with general usage.

The oxygen bridge of the catalyst also requires energy (heat or ultra-violet light), or the addition of an *accelerator*, to be broken (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 accelerator. Thus tertiary amines (especially dimethyl aniline) are used to split benzoyl peroxide, and metal soaps, particularly soluble Co-soaps such as Co-naphthenate and Co-octoate, 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). This being due to the *exothermic* nature of the polymerisation. By following the temperature changes

²⁾ The authors want to record here their gratitude to MR. L. W. J. DAMEN, head of the plastics laboratory of Messrs. Noury and Van der Lande N.V., Deventer, for his valuable advice and criticism of this part of our paper, as well as for samples of catalysts and accelerators which he generously made available.

in the polymerising resin (which needs to be insulated for measurements at room temperature) and plotting temperature against time, the so-called *polymerisation curve* is obtained (BERNDTSSON and TURUNEN, 1954; MALTHA, 1957, CYWINSKI, 1960). The temperature in the plastic does not rise to begin with, as can be seen in figure 2. During this 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. 2, S)

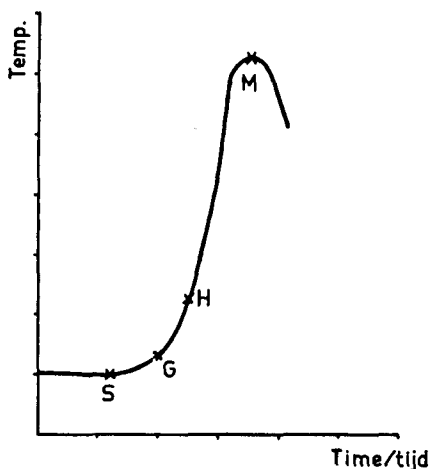


Fig. 2

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").

Theoretische voorstelling van een polymerisatiecurve bij 20°C. S = start van de polymerisatieactie; G = gelegeringspunt; H = tijdstip waarop het plastic hard is; M = tijdstip van de maximale warmte-ontwikkeling ("peak exotherm").

that polymerisation begins. This is revealed amongst other things by a rise in temperature, followed by gelling of the material (*gelling point*: fig. 2, G). This rapidly raises the reaction rate and therefore also the temperature, 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 can be reduced for a short time (BEYER, 1955; MALTHA, 1957). Only after the plastic has hardened (fig. 2, H), the maximum heat evolution occurs (fig. 2, 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 composition. 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, page 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 workers (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 any rate 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) raise the polymerisation rate. It is noticeable however that at higher concentrations of dimethyl aniline the polyester resin is not hardened (MALTHA, 1957). BERNDTSSON and TURUNEN (1954) found that the plastic Soredur 9155 B gelled more rapidly at room temperature with cyclohexanone peroxide with increasing additions of Co-naphthenate. But above a given concentration of accelerator (about 2 % of 6 % Co-naphthenate) polymerisation again proceeded more slowly. 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. At higher concentrations (of up to 12 % of accelerator) the gelling time remained constant. 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, when 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 both for 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). Both the kind of filler and its concentration (vol. %) are very important; the higher the concentration the slower the polymerisation proceeds. This 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 the plastic, in so far as it had penetrated into the soil did not harden at all, 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 styrene content. The liquid unsaturated polyester resins are usually fairly viscous. This can be substantially reduced by adding monostyrene 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 indicated as far as necessary in the following section.

3. THE METHOD

The samples

These are taken in brass tins $15 \times 8 \times 5$ cm large and then air-dried in the laboratory. The material, if it should not be very coherent, is then prehardened by impregnating it with a 1 in 1 mixture of cellulose varnish and acetone (JONGERIUS, 1957). A label showing sample number and its original direction is pinned to each sample.

The resin

The resin used is Vestopal-H, previously known as resin type 7004 (ALTEMÜLLER, 1956) and manufactured by Chemische Werke Hüls, Marl, near Recklinghausen, Western Germany. It is a yellow, syrupy product (viscosity about 15 poise) capable of unlimited dilution with monostyrene. At room temperature it can be stored for about six months, at least in the dark. In the polymerised state the resin is very hard, practically colourless, vitreous and resistant to numerous chemicals (for further details see ANONYMOUS 2, no date). Vestopal-H shrinks about 8.2 % during hardening. Although in the presence of fillers the shrinkage is reduced in proportion to the amount of these fillers (ANONYMOUS 2, no date). Consequently no shrinkage can be observed in hardened soil samples.

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 over 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. The former can be accurately formulated, while Co-naphthenate may also contain other Co-compounds (DAMEN and KEVER, 1961). For catalyst and accelerator we use products named as:

Cyclonox LTM-50: a solution containing at least 50 % of pure cyclohexanone peroxide.

Accelerator NL-49: a solution of Co-octoate with 1 % Co.

Manufactured by Messrs. Noury and Van der Lande, Deventer.

N.B. Other more highly concentrated Co-octoate solutions are also available but they are less suitable for our purpose, as the very small amounts used (see below under impregnation) are much more difficult to measure off at higher concentrations.

The impregnation

For the complete impregnation and proper hardening of soil samples as large as $15 \times 8 \times 5$ cm with Vestopal-H several conditions must be fulfilled (see also ALTEMÜLLER, 1962):

1. The impregnation should take place in a fairly high vacuum.

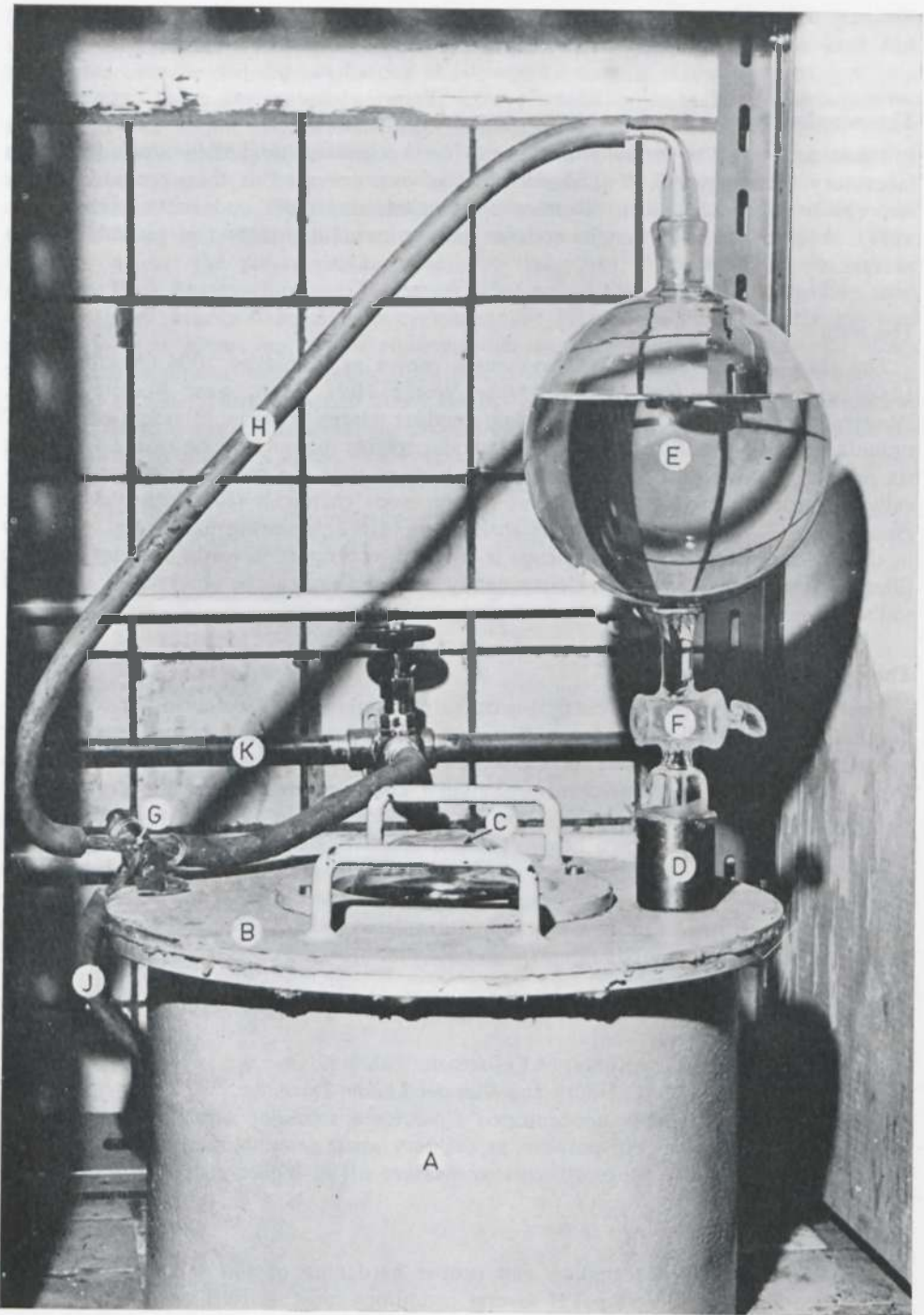


Fig. 3

Photo/Foto Stiboka nr. R22-6

2. The viscosity of the plastic has to be considerably reduced. This can be done by diluting with monostyrene.

3. The excess monostyrene has to be removed again from the samples in good time, since a high styrene content reduces the polymerisation rate of Vestopal-H and adversely affects various properties of the polymerisate, such as its resistance to various chemicals, modulus 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 structural elements in the thin sections. This makes such slides unsuitable for microphotography in particular (ALTE-MÜLLER, 1956).

4. In order to allow the excess monostyrene to evaporate and to make sure that the samples have been completely impregnated by the Vestopal resin a long gelling time is required. This is achieved by adding only low concentrations of catalyst and accelerator. This brings with it the advantage that the exothermic heat peak (see fig. 2) 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 induced particularly at higher temperatures (DAMEN and KEVER, 1961). Finally, research by the said authors 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 is 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 from 4 to 6 weeks to give the best results with all types of soil.

The procedure is as follows.

Four air-dried soil samples $15 \times 8 \times 5$ cm large, are placed alongside each other on their narrow longitudinal side in a cardboard box measuring $24 \times 17 \times 11$ cm and lined with plastic foil. Their labels should face upwards.

This has two advantages:

1. The bottom of the box is virtually evenly filled with soil, which is very important for a successful hardening. In fact, 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

Fig. 3 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) into 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 resp. the rubber lines H and J, with the line (K) of an electric vacuum pump.

Impregnatie-apparaat. Deze bestaat uit een stalen cilinder (A), waarop een deksel (B) met dikwandig, bolvormig kijkglas (C). In het deksel is een kleine ronde opening met opstaande rand (D), waarin de vacuümbol (E) uitmondt. De verbinding tussen cilinder en bol kan worden afgesloten door middel van een kraan (F). Via een driewegkraan (G) zijn zowel de bol als de cilinder door resp. de slangen H en J aangesloten op de leiding (K) van een elektrische vacuümpomp.

the plastic in the soil samples (see par. 2: the amount of polyester resin and the shape of the cast). As a result so much heat may be liberated that the polymerisation in the soil samples is accelerated as well, thereby causing the disadvantages enumerated above.

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 much glassware (taps, tubes, etc.). This applies much less to the procedure 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 it would also make the hardened blocks too large and heavy to handle. Also, the daily output of mammoth-sized thin sections by a single technical assistant is satisfied by the impregnation of four samples per day.

The box filled with the soil samples is then placed in a steel cylinder connected to a vacuum line, and with a flat, wide upper rim. The cylinder is closed with steel cover having a carefully planed underside. To obtain an air-tight seal the corresponding edges of cylinder and cover are generally greased beforehand with vaseline. The cover has a small round aperture with a raised edge, which holds a glass vacuum sphere of about 2 litres capacity (fig. 3).

The four samples are impregnated with a liquid of the following composition:

1500 cc Vestopal-H
1000 cc monostyrene
4 cc cyclonox LTM-50
2 cc accelerator NL-49.

With operating temperatures higher than about 22°C (as for instance in summer) only half the amount of both catalyst and accelerator are used, i.e. 2 cc and 1 cc respectively (see par. 2: operating temperature).

It is interesting to compare these figures for the impregnating liquid with those used by ALTEMÜLLER (1962) on small samples. A conversion to the above volume gives 1500 cc Vestopal-H, 1250 cc monostyrene, 8.25 cc 50 % cyclonox and 4 cc cobalt octoate. The main difference is the (relatively) much higher concentration of catalyst and accelerator. This will be discussed further below.

As shortly as possible before impregnation the liquid is prepared in a 3-litre beaker. The plastic is poured in first of all, followed by the monostyrene. Both are thoroughly mixed with a stirring rod. The accelerator measured off by a 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. The cyclonox is added last of all 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 can 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 and accelerator. This stock solution can be stored for a fairly long period at room temperature. For impregnation the required amount of solution is taken out and the corresponding volume of catalyst added.

The vacuum sphere — with the connecting tap to the cylinder (fig. 3) closed — is then filled with the impregnating liquid. The top of the sphere is then connected

through a ground glass joint to the vacuum line. The $\frac{1}{2}$ litre or so of liquid left over after filling the sphere is kept to the following day.

After that the cylinder and sphere are evacuated in order to remove as much air as possible from both the soil samples and impregnating liquid (the large amount of stirring can introduce a great many microscopic air bubbles in the latter). An Edwards electric vacuum pump is used for evacuation. 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 silicagel 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. An electric pump only takes about 5 minutes to reach the required vacuum of about 60 mm Hg. At this point the connecting tap between sphere and cylinder is opened sufficiently for the liquid to fall drop by drop into the box of samples. The box should be placed so that the plastic does not drip on but between the samples. This prevents any air still present in the soil from being occluded by the plastic. The plastic rises very slowly in the box — all the time under the same vacuum — until after about $7\frac{1}{2}$ hours all the liquid will have run out of the sphere. The level of the liquid in the box has then reached the top of the soil samples. The taps to the vacuum cylinder are closed and the impregnated soil samples left in it overnight. The vacuum is gradually reduced until it has disappeared by the morning. The cylinder is opened and the plastic left over from the previous day, which will have lost all its air after standing over for 24 hours, is poured into the box. The impregnating liquid then covers the samples to a depth of about 1.5 cm.

The equipment can be used for about five days running; after that however all glass parts should be cleaned with *P3-Nevol* in warm water.

It will be obvious from the foregoing why the impregnation vessel should be made of steel. A glass cylinder with a diameter of 30 cm (e.g. a vacuum exsiccator) would be crushed by such a high vacuum, which could lead to an explosion with a strong splintering effect.

The next stage in the work is the expulsion of the large excess of monostyrene from the plastic. To this end the box of impregnated samples is placed in a fume cupboard in which the air is drawn off by a large ventilator operating for 24 hours per day.

The evaporating monostyrene causes the liquid level to fall fairly rapidly and after some days the samples would show up above the liquid. This must be prevented as otherwise air might enter the samples, so that after two or three days an amount of undiluted plastic equal to the amount of evaporated monostyrene is poured into the box. This is repeated every two or three days until there is no further fall in the liquid level (usually after about three weeks). The plastic used for replenishing has the following composition:

- 1000 cc Vestopal-H
- 2.5 cc cyclonox LTM-50
- 2.5 cc accelerator NL-49.

ALTEMÜLLER (1962) replenishes with concentrated Vestopal containing 0,3 % cyclonox and 0,15 % accelerator. Comparing both methods it is clear that ALTEMÜLLER sought a solution to the problem of impregnation in a very high dilution of the plastic with monostyrene. Obviously he attached less importance to a very long gelling time. In his case the process takes 4-6 days against 6 weeks with our method. The fact that his denser samples had to be reimpregnated in several cases, while this was never necessary with our own samples of about 40 times the volume (except for

an occasional failure due to a sudden, very rapid rise in the operating temperature — par. 2), proves that a low viscosity of the polyester by itself is not sufficient to impregnate the soil completely with enough plastic. Some of the monostyrene probably penetrates fairly rapidly into the soil samples, while the plastic would follow it much more slowly: the monostyrene 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.*

It might be concluded from the foregoing that some of the excess monostyrene is absorbed in the soil without being released again afterwards and even that the gradual fall in the liquid level during the time in the fume cupboard is not caused by an evaporation of the monostyrene but by its continued absorption in the soil. If this laboratory. It is best to leave them there for at least some weeks to give the samples 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 monostyrene 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 originally added. From this it follows that the excess styrene initially present in the samples gradually disappears again.

Hardening

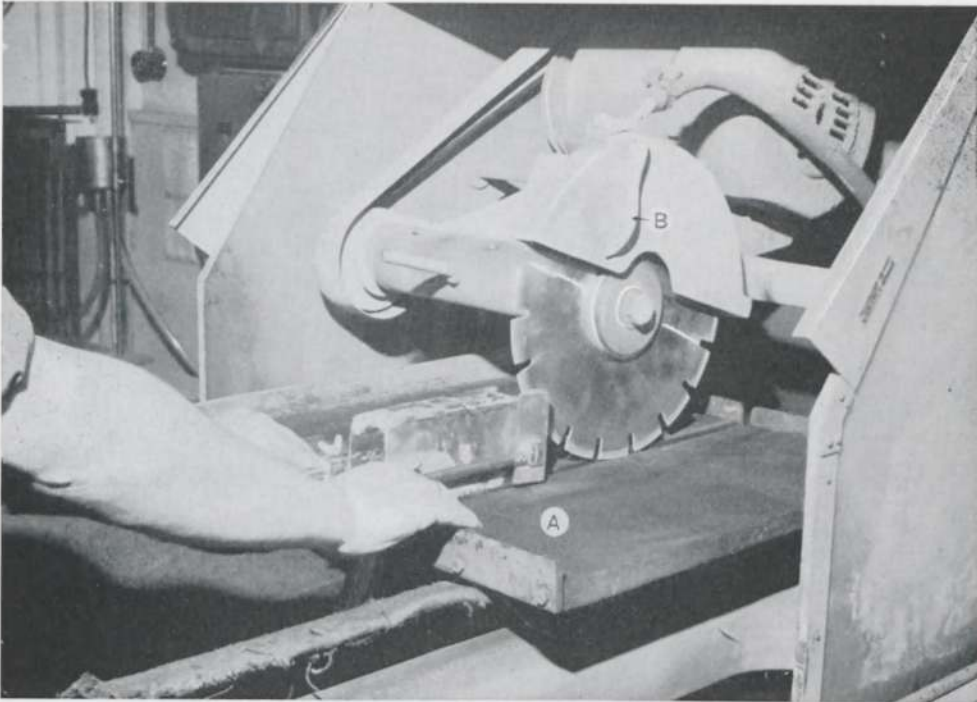
On completion of the impregnation process the boxes with samples remain a few more weeks in the fume cupboard. During this period there is a very gradual gelling followed by the hardening. When the samples have reached this stage the boxes are removed from the fume cupboard to be stored on shelves in a cool place in the laboratory. It is best to leave them there for at least some weeks to give the samples time to harden thoroughly. At first the topmost 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 bigger risk of stresses occurring in the hardened samples, than when some time is allowed to elaps before the final hardening. This is because the exothermic evolution of heat takes place in large part after the beginning of hardening. Where the final hardening is not delayed this heat cannot be removed and there is a big rise in temperature in the hardening plastic.

After the final hardening the block is removed from its box and the plastic foil pulled off. It is now ready for processing.

Sawing

The blocks are sawn first of all into smaller pieces. The size and hardness of the blocks makes a firm sawing machine with a large saw blade necessary. The Bruyaux type A *sawing machine* with a cutting disc 300 mm in diameter (fig. 4) is very satisfactory for this purpose. One can either use a disc with an uninterrupted diamond



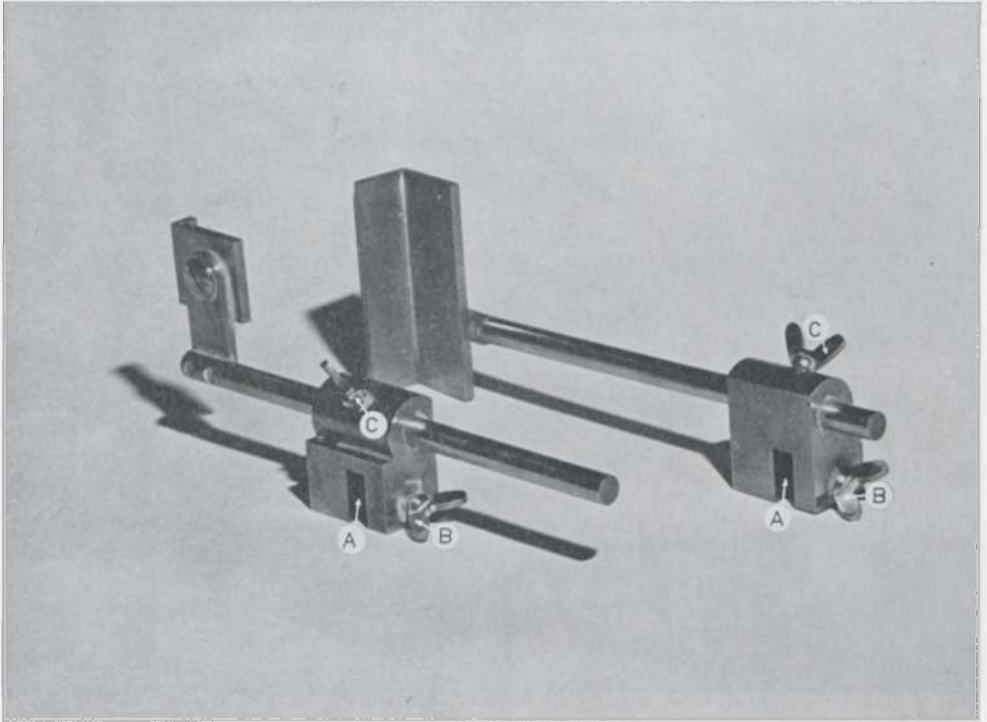
Photo/Foto Stiboka nr. R21-136

Fig. 4 The Bruyaux type A sawing machine with a cutting disc of 30 cm 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 V-0847 by means of tubing openings on either side of the disc (B). The sawing machine is enveloped in a plexi-glass cap for protection against the strongly atomized oil and sawing sludge. For reasons of safety the sample is anchored by two metal clamps.

Bruyaux afkortmachine met zaagblad van 30 cm diameter. Het getande blad heeft een gediamenteerde rand. Het monster wordt gezaagd door de brug (A) onder het draaiende zaagblad door te duwen. Er wordt gekoeld met Shell-olie V-0847 via een aan beide zijden van het zaagblad uitmondende geleiding (B). Met het oog op de sterk vernevelende olie en het zaagvuil is de zaag omgeven door een plexiglas kap. Om veiligheidsredenen is het monster vastgezet met twee metalen klemmen.

impregnated edge (bronze bonding; 12.3 crt) or one with diamond impregnated teeth (steel bonding; 12.7 crt); the former saws somewhat faster, while the toothed blade probably has better air-cooling. The machine must stand on a very stable table specially constructed for this purpose.

The objects to be sawn are placed on a bridge and this is pushed underneath the rotating cutting disc which can be adjusted to different heights. During the sawing the material is strongly cooled by means of tubing openings on either side of the disc (fig. 4 B). Water is not used for the cooling, but the volatile *Shell oil V-0847*, after the German micromorphologists (KUBIENA; ALTEMÜLLER, 1962). Water causes the equipment to rust and various soil components, such as clay minerals and organic matter to swell up, even in a completely impregnated and thoroughly hardened sample. With grinding this gives holes in the preparations. The same oil is also used in the grinding process (see below). This is directed as required to the grinding machine or



Photo/Foto Stiboka nr. R23-176

Fig. 5 The metal clamps used in cutting the sample. The grooves (A) are pushed on the raised edge of the bridge and screwed on by butterfly nuts (B). The length of the arm is adjustable with the butterfly nuts C. The retaining piece of the right clamp is much longer than that of the left one. Apart from clamping the right clamp has a bearing function, viz. to prevent the sawn off plate from being hurled away (see for position of the right clamp also fig. 4).

De beide metalen klemmen die bij het zagen worden gebruikt. De gleuven (A) dienen om ze op de opstaande rand van de zaagbrug te kunnen schuiven, waarna ze door aandraaien van de vleugelmoeren B worden vastgezet. De lengte van de armen is verstelbaar met de vleugelmoeren C. De bek van de rechter klem is veel langer dan die van de linker; de eerste heeft namelijk naast een klemmende tevens een dragende functie, te weten te voorkomen dat de afgezaagde plak grond wordt weggeslingerd (zie ook positie van de rechter klem in fig. 4).

the sawing machine from a central tank (fig. 6, E) by means of a small electric pump belonging to the grinding machine. The oil runs back again into the tank via a drain in the collecting vessels of the machines. Filters (fig. 6 F) in the lines keep the oil regularly remove the sludge and fine grinding powder.

The several samples in the block are now sawn out, with the faces as much as possible at right angles to each other. Where the samples are vertically oriented the topside 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. For this the soil is sawn into sections at least 8 mm thick. This is important as a thinner section would very soon

warp. Even a 8 mm thick one should be ground, polished, mounted on an object glass and weighted down (see under mounting) the same day if warping is to be avoided. In spite of the very long hardening time the polymerisation process may not be fully complete 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 (figs. 4 and 5). These are only suitable for use when the specimen are cut out at right-angles.

Grinding

This is done in two stages, viz. machine planing and 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) introduced however, the semi-automatic Klaiber *grinding machine* type HV II (fig. 6) for the preparation of small thin sections and we also found it very suitable for the grinding of larger slides¹⁾.

This machine, used in industry for very fine grinding work has a vertically mounted stone rotating above an electromagnetic clamping table 30×15 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 microns every time for sandy samples and about 75 microns for clay samples. This should be reduced to only a few microns every sweep in a thinly ground section.

Only certain types of stones are suitable for making thin sections. Feldmühle stones of ceramically bound Si carbide, grain size 240 and Lg hardness are quite satisfactory. For large preparations a stone 2 cm thick is preferred to the 3 cm one used by ALTEMÜLLER (1962) for his smaller sections. Although a 2 cm stone takes off a smaller area, it has been found that with the narrower stone there is less risk of the section fracturing, especially when it is fairly thin.

One of the sawn off sections is now placed on the clamping table, after the sample number has been scratched on the lower surface with a diamond pencil, and it is enclosed by two polished steel angles about 5 mm thick (fig. 7). The steel angles are drawn on to the table by the electromagnetic field so that they rigidly anchor the section. The top 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 mm thick.

The planed side of the soil section is then finely finished by hand on $500 \times 300 \times 12$ mm sheets of plexiglass. For this a series of aluminum-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

¹⁾ There are also fully automatic grinding machines with at the same time a considerably larger clamping table. These would save considerable labour and have a greater capacity. Prof. KUBIENA, for example, has been using for some time a fully automatic *Blohm-HF6* with a table of 60×30 cm for the production of thin sections of soil (GEYGER, 1962). Type HFS 15 even has a table measuring 162×30 cm. These grinding machines are however very expensive.

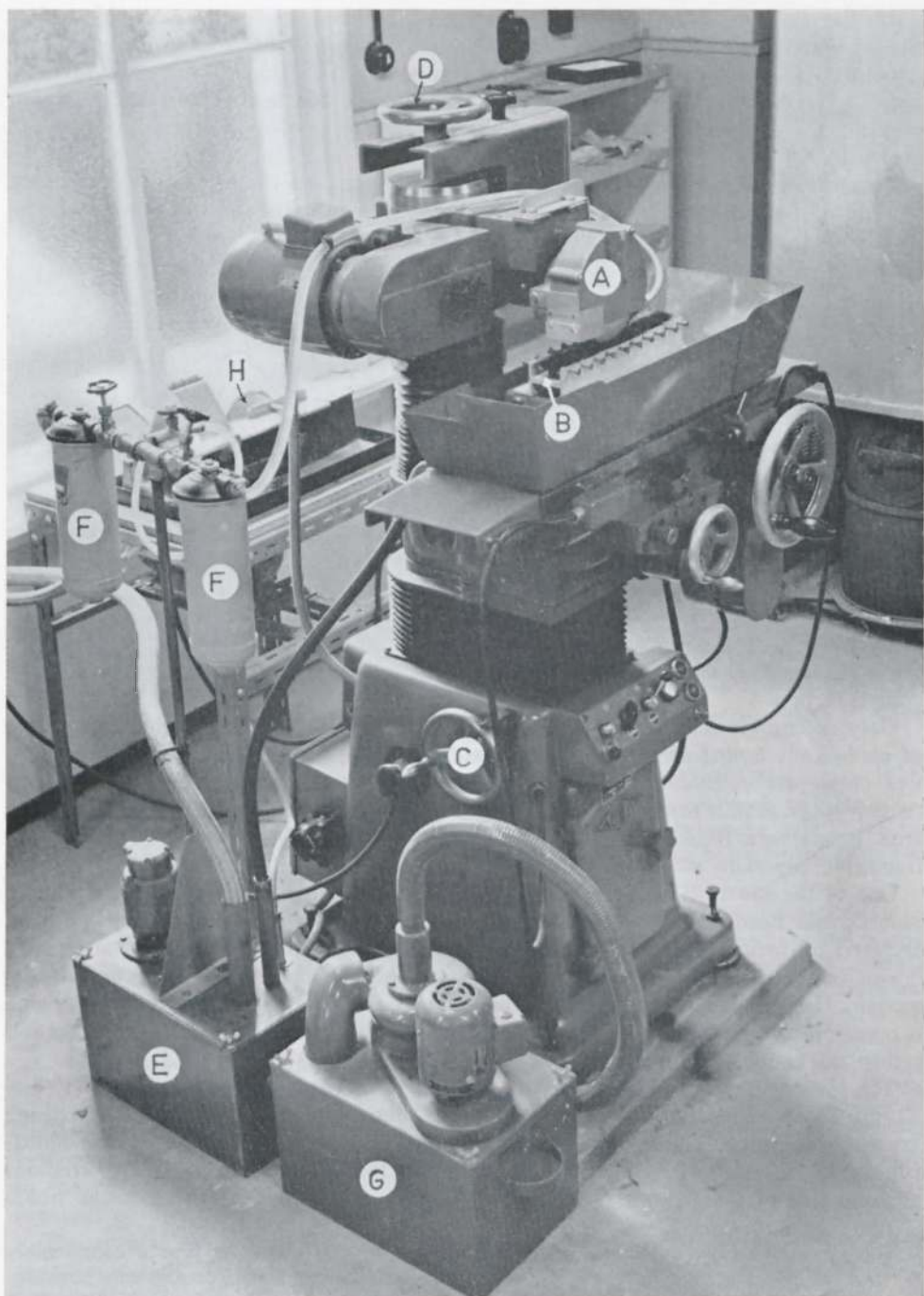
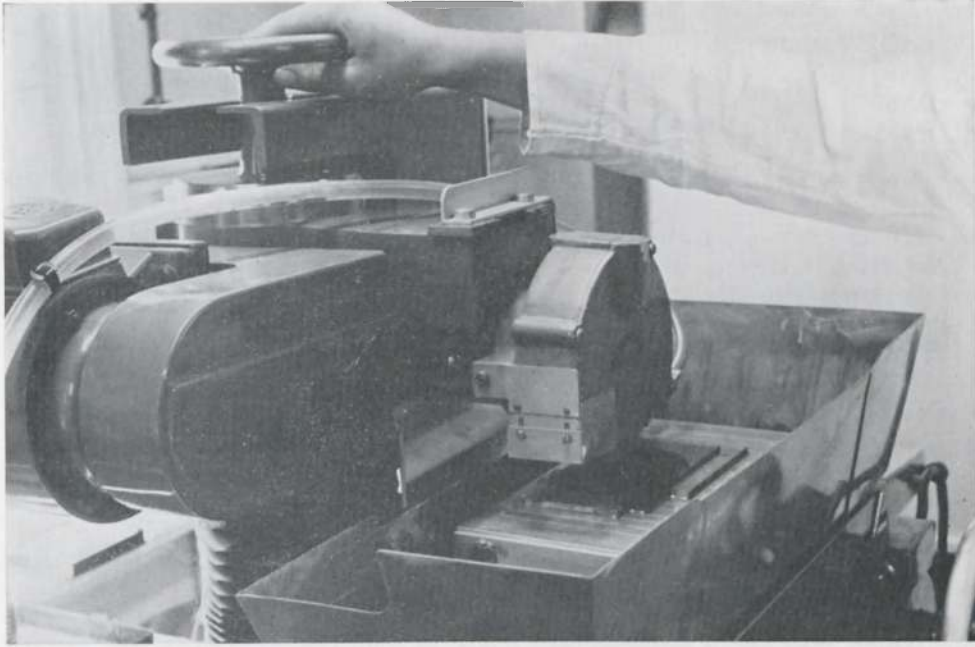


Fig. 6

Photo/Foto Stiboka nr. 21-146



Photo/Foto Stiboka nr. 21-137

Fig. 7 Detail of the grinding machine. On the electromagnetic clamping table is a soil section (hardened by Vestopal-H) of appr. 8 mm in thickness. The sawn off section is enclosed on the table by two polished steel angles (appr. 5 mm in thickness). On the picture the technical assistant is just twining the stone a bit lower.

Detail van fig. 6. Op de elektromagnetische opspantafel ligt een ± 8 mm dikke, met Vestopal-H geharde grondcoupe. Deze is met twee gepolijste, ± 5 mm dikke stalen winkelhaken vastgeklemd op de tafel. De laborant laat juist de slijpsteen wat zakken.

Fig. 6 Klaiber grinding machine. The grinding stone (A) rotates vertically above an electromagnetic clamping table (B), which moves to and fro automatically in the plane of the grinding stone. The table can be moved at right-angles to the stone rapidly by hand or slowly automatically. The rough vertical motion of the table is governed by the wheel C, the fine one by D. The cooling oil is directed as required to the grinding machine or the sawing machine from the central tank (E) by means of a small electric pump. Filters (F) keep the oil regularly remove the sludge and fine grinding powder. In foreground suction equipment for dry-grinding (G). Near the window a small Winter-sawing machine (H) used for preparing small thin sections. On the clamping table a jig for flattening small hardened samples.

Klaiber vlakslijpmachine. De slijpsteen (A) draait verticaal boven een elektromagnetische opspantafel (B), die automatisch horizontaal heen en weer beweegt in het vlak van de slijpsteen. Loodrecht daarop kan de tafel door handbediening worden bewogen of automatisch zeer langzaam onder de steen doorschuiven. De grove verticale beweging wordt geregeld met het wiel C, de fijne met D. Vanuit het oliereservoir E wordt de koelolie naar de slijpmachine of de zaagapparatuur gepompt. Door filters (F) wordt de olie gereinigd van slib en slijppoeder. Op de voorgrond een afzuiginstallatie voor droogslijpen (G). Tegen het raam staat een kleine Winter-zaagmachine (H), die gebruikt wordt bij het maken van kleine slijpplaatjes. Op de opspantafel bevindt zich een mal voor het vlakken van kleine geharde monsters.

are already ground very smooth. The entire series of powders successively used is:

BAR 30	(grain size approx. 36-26 microns)
BAR 23	(„ „ „ 26-18 „)
BAR 13	(„ „ „ 18-10 „)
BAR 7	(„ „ „ 10- 5 „)

When grinding clay and peat samples BAR 30 can be omitted. BAR 7 is not used on sections more than 30 microns thick (for special purposes such as the study of the macrostructure this is sufficient).

ALTEMÜLLER (1962) uses corundum powders 23 and 13, and in special cases also 15 mu diamond powder. Diamond powders produce a very flat and clean preparation but they are very expensive (see under polishing).

As for the machine planing, the liquid used for grinding by hand is Shell oil V-0847. 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 different plexiglass plates should be kept carefully separate, for example by using wooden or plexiglass partitions. After every stage of grinding the preparations should also be thoroughly cleaned in petrol with a soft brush. 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 coarser one or with a grain of sand broken out of the preparation, the relevant plexiglass plate should be completely cleaned at once, and the grinding resumed with pure powder.

Polishing

The ground surface is polished with diamond pastes to make it completely smooth. This also considerably reduces possible contaminations with fine grained materials.

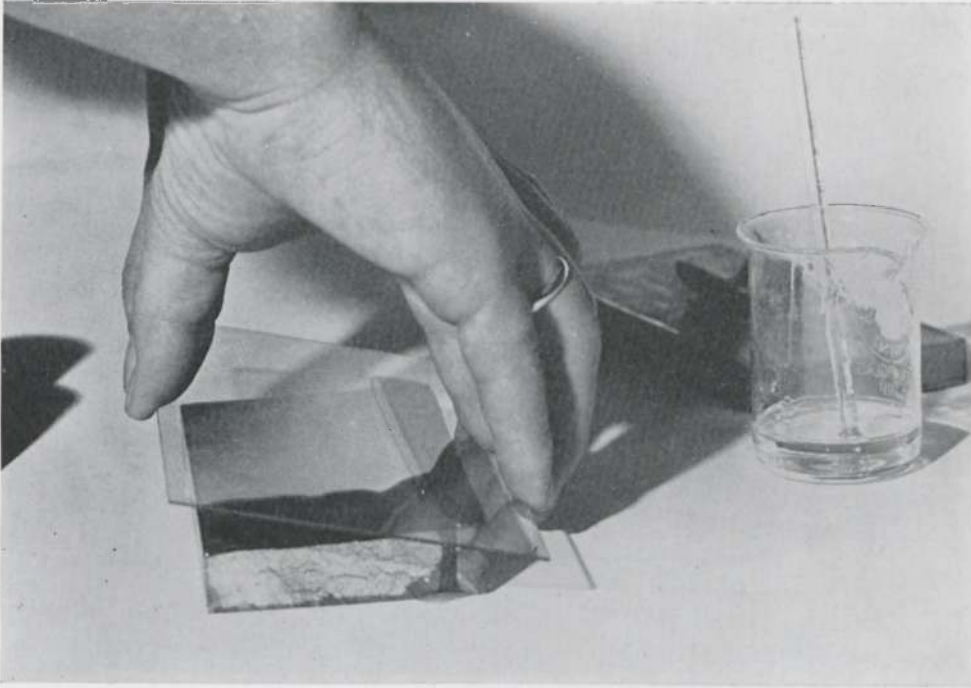
With smaller preparations excellent results were obtained on a Struers DP-IV *polishing machine*. This apparatus has a rotating PVC disc, 200 mm in diameter, mounted on double bearings and running at 125 and 250 r.p.m. These are much lower than those generally employed in industry for polishing (viz. up to about 2000 r.p.m.). The latter are designed to produce a smooth surface by plastic deformation under influence of frictional heat (BURKART, 1959). In this case, however, plastic deformation and hence a high speed are the very things to be avoided and the polishing with diamond pastes serves only to remove the last small unevennesses.

A cloth of acid-resistant synthetic material is stretched over the disc. This cloth is made up of three layers, viz. the polishing layer, a dense layer holding back the diamond, and a resilient layer resting on the disc. The dense layer prevents the diamond powder from penetrating deeply into the cloth, thus ensuring economic use of the pastes, and the resilient layer eliminates scratching by the diamond. The cloths are available in harder and soft forms (for a coarser and fine diamond respectively).

We also employ Struers' *diamond pastes* for their very narrow range of grain size. There are three types, viz. A, B and C with diamonds of approx. 3, 1 and 0.5 micron respectively. Each paste requires a separate cloth. Paste consumption is very low and 500 to 1000 small preparations can be polished with 2 cc of DP paste.

A lubricant or diluent is used in polishing. The Struers-DP liquid was not found satisfactory for our preparations as it weakens the hardened Vestopal-H again. Cleaning petrol gave good results however.

Unfortunately the very large preparations cannot be processed on the polishing



Photo/Foto Stiboka nr. 15901

Fig. 8 The mounting of a soil section on an object glass. The grinded and polished side of the section is upward and covered by a thin layer of plastic. After a thicker layer is applied to one of the long edges (the dark strip at the right) the glass is applied to the section in a hinge-like motion.

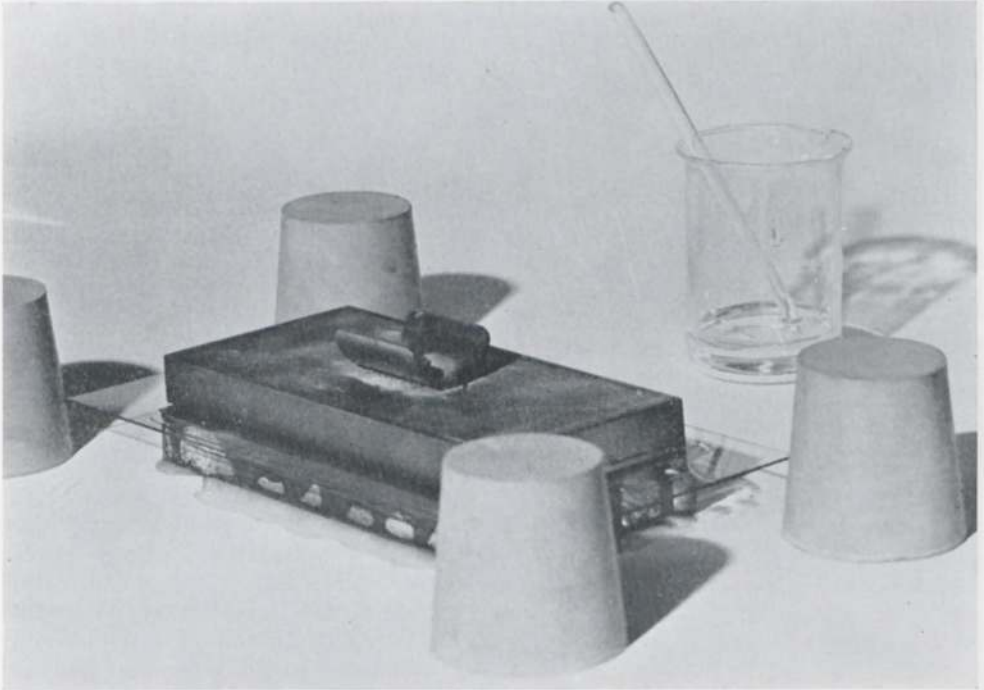
Het opplakken van een grondcoupe op een objectglas. De geslepen en gepolijste zijde van de coupe ligt boven en is bedekt met een dun laagje plastic. Nadat langs één der lange zijanten een dikkere laag is opgebracht (de donkere baan rechts) wordt het glas vanaf die zijde scharnierend op het preparaat gedrukt.

machine. Not only is the diameter of the disc too small, but the large dimensions of the surface to be polished may produce a considerable friction which makes it impossible to hold a mounted and thinly ground section on the disc (see below).

Large sections can therefore best be polished on 500×300 mm plexiglass plates covered with a few layers of synthetic (e.g. nylon, perlon, etc.) in a combination approaching as much as possible the principle of the polishing cloths. To minimize the risk of damage to the cloths it is desirable to bevel the edges of the preparations slightly beforehand.

ALTEMÜLLER (1962) recommends cleaning a polished preparation with a cloth and washing it with plenty of petrol on a rotating disc covered with a chamois leather. This cleaning operation is also considered helpful for mammoth-sized thin sections, although the authors used here also a covered plexiglass plate.

In conclusion it is noted that both polishing as well as the mounting and covering (to be discussed below) should be carried out in an atmosphere free from dust. This demands a small separate room with good exhaustion and specially finished floor, ceiling and walls.



Photo/Foto Stiboka nr. 15902

Fig. 9 Weighing down the object glass promotes an equal thickness of the cementing layer. The rubber stops prevent the glass from sliding over the section.

Door het objectglas met een gewicht te verzwaren krijgt men een gelijkmatige dikte van de kitlaag. De rubber stoppen voorkomen dat het glas over het preparaat schuift.

Mounting

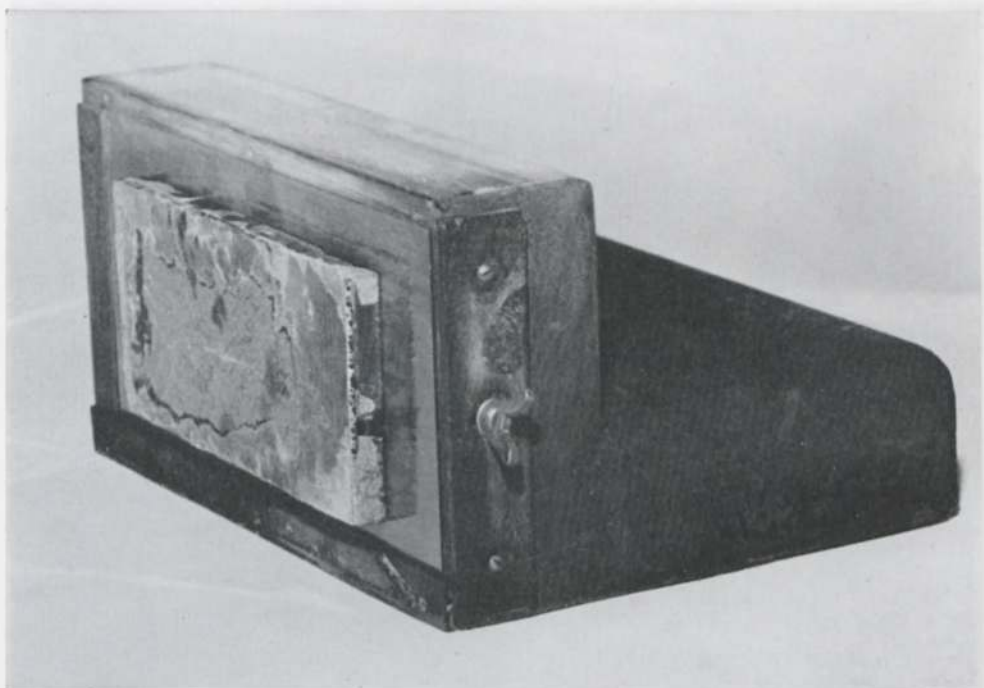
After the soil section has been properly dried (if necessary this can be speeded up by holding it about 1 metre away from a hot air blower) the polished side of the sample is mounted on an object glass size about 1.2 mm thick and 20 × 10 cm large. The glass should have the optical properties of the usual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

For mounting, the following materials are used per section:

- 10 cc Vestopal-H
- 0.2 cc cyclonox LTM-50
- 0.1 cc accelerator NL-49.

The gelling time of this mixture is about 1½ hours at room temperature. With an operating temperature higher than about 22°C half the amount of both catalyst and accelerator are used.

The mounting is done as follows: the section is placed on a table with the polished side upward, and a little of the mixture is poured on to the section to be evenly spread out with a stirring rod. Any cracks or small cavities in the surface can then be filled up with the plastic. A thin layer of the mixture is then applied to one of the long edges



Photo/Foto Stiboka nr. 15904

Fig. 10 Wooden jig for the sawing off of mounted soil sections. The sections are clamped to the jig by thin bakelite strips.

Houten mal voor het afzagen van opgeplakte grondcoupes. De preparaten worden door dunne strippen bakeliet op de mal geklemd.

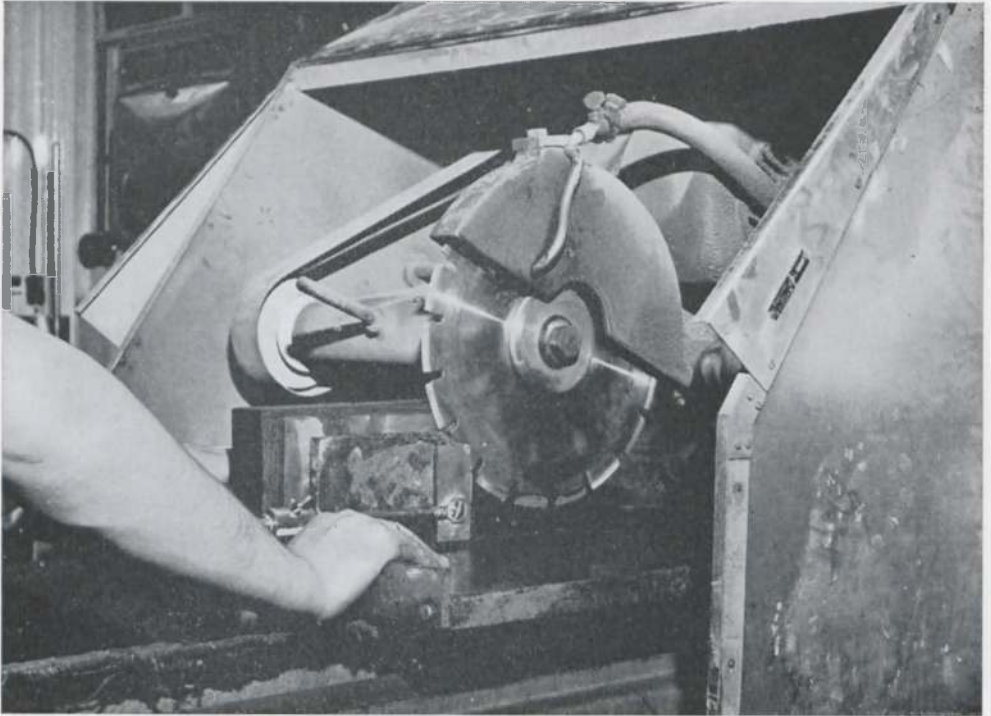
and after the air has disappeared from the plastic, the object glass is applied to the section in a hinge-like motion (fig. 8). This prevents any air bubbles from being trapped underneath the object glass.

The section is joined exactly parallel on to the glass by weighing it down with about 1450 g. The weight has a polished base of 7×14.5 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. 9). Covering the working table with a few layers of paper or plastic foil will prevent excess plastic from sticking the section to the table. The mounted specimen is left like this for about 24 hours.

Sawing the mounted preparation

With a special jig (fig. 10) the mounted section can be reduced to a thickness of about 2 mm with the circular saw, which eliminates about $1\frac{1}{2}$ 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



Photo/Foto Stiboka nr. R23-175

Fig. 11 The sawing off of the mounted section with the help of the wooden jig. A metal safety clamp prevents the sawn off plate from being hurled away.

Het dun afzagen van een opgeplakt preparaat met de houten mal. De metalen klem voorkomt dat de afgezaagde plak wordt weggeslingerd.

clamp with a long retaining piece (figs. 5 and 11) prevents the 5 to 6 mm thick plate 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: mounting) to prevent warping.

Thin grinding and polishing

The section is now reduced from about 2 mm to a thickness of about 40 microns 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 microns 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 microns. 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 8 mm thick sections. Grinding is continued with the finest powder (BAR 7) until a thickness of about 15 microns has been reached, which is indicated by the sand grains becoming grey under crossed nicols. After each grinding stage the section should of course be washed with cleaning petrol.

After grinding the preparation is polished with diamond pastes, in the same way as already described.

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 $160 \times 80 \times 0.2$ mm large is laid 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 (as under mounting) 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.

The completed thin sections

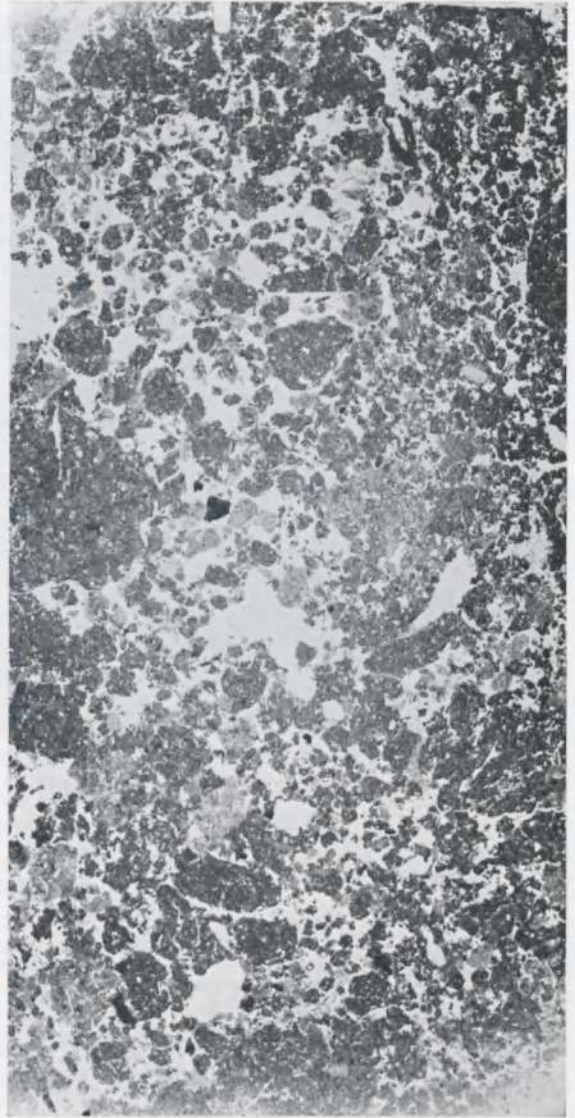
Labels giving various data (such as record number, place of origin, type of project, etc.) are attached to the free ends of the object glass. The slides are now ready for microscopic examination.

Figures 12 and 13 give some idea of the quality of the mammoth-sized thin sections while they also illustrate some of the possible applications of this kind of slides.

Fig. 12

Mammoth-sized thin section (full-size) of a river levee soil mixed by a planting hole auger (sample of 20-35 cm below surface). Structure and mixing are markedly demonstrated by this thin section (dark-grey elements: topsoil; light-grey elements: subsoil) Horticultural experimental field (Werkhoven, Utrecht).

Mammoet-slijpplaat op ware grootte van een met de plantgatenboor gemengde stroomgrond, 20-35 cm-m.v. De slijpplaat geeft een goed overzicht van de structuur en van de door de boor veroorzaakte menging (donkergrijze elementjes: bovengrond; lichtgrijze: ondergrond). Herkomst: Stichtse Fruittuin, Werkhoven (Utr.).



Photo/Foto Stiboka. Thin section/Slijpplaat G7

Fig. 13 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 below surface: Ter Aar (Western Netherlands).

Detail uit een mammoet-slijpplaat: inspoeling van meermolm in oude zeelei (de zeelei links op de foto: lichtgrijs). De fijngelaagde, verticaal gerichte opbouw van de inspoelingshuid, die uit baantjes bestaat van opeenvolgend kleiig, fijnzandig en organische-stofrijk materiaal, is tot in de kleinste details goed zichtbaar. Herkomst: Ter Aar (TAS 1), 50-65 cm-m.v.



Photo/Foto Stiboka nr. 12511. Thin section/Slijplaat G1

Fig. 13

4. 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 and petrol vapours.
2. Owing to the inflammable nature of the unsaturated polyester resins, the monostyrene, 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 to the body organism. Should they be swallowed by accident (e.g. in pipetting) one should immediately try to vomit, and drink copious draughts of sodium ascorbate dissolved in water. 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 intensely 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 nearly superfluous to say a physician should be called in immediately in the above cases.

Contact with the skin should be avoided. Especially when this occurs repeatedly a hypersensitivity may develop, 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 water and soap, and lanoline ointment rubbed on it.

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

Prolonged contact of the skin with Vestopal should also be avoided. The hands must be thoroughly washed with soap after work and any adhering residues removed with acetone. Hands should be well rubbed in with a barrier cream both before and after using Vestopal.

5. Shell oil V-0847 atomises strongly, especially during sawing and it is easily inhaled this way. A breathing mask should therefore be used during this work, while 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 (figs. 4, 5 and 11).

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 about 5×5 cm in size. This requires a fairly small diamond saw (e.g. a Winter saw of the type just visible in fig. 6) and a rotating grinding disc (e.g. Wirtz). 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. 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 yield the same possibilities as the mammoth-sized sections, they still provide much more information than the small "conventional" slides.

5. SAMENVATTING

Inleiding

Men kan van grond alleen slijpplaten maken door het materiaal te verharden. Hiervoor zijn in de loop der jaren vele stoffen gebruikt, doch deze bezitten alle één of meer ongewenste eigenschappen, zoals: brosheid, geringe kitkracht, onvoldoende hardheid, laag smeltpunt, te gering impregnerend vermogen, etc. Bij het slijpen van het geharde monster kunnen dan allerlei complicaties optreden (fig. 1), terwijl het maken van gave preparaten van meer dan 3×3 cm en minder dan 25 à 30 μ vrijwel onmogelijk is. Met de onverzadigde polyesterhars Vestopal-H gelukte het ons echter slijpplaten van 15×8 cm en niet meer dan 15 μ dikte te vervaardigen.

Theorie van het hardingsproces

Vestopal-H gaat via een gelfase in de vaste toestand over door copolymerisatie van polyester en styreen (ruimtelijke net-structuur), waarvoor activering van die monomeren nodig is (verbreking van hun C-dubbelbindingen). Deze activering kan bij kamertemperatuur geschieden door „katalysatoren” (organische peroxyden), die echter op hun beurt eerst geactiveerd moeten worden door versnellers (tertiaire aminen en Co-zepen). De copolymerisatie is een exotherm proces waarin enige fases zijn te onderscheiden (fig. 2). De snelheid waarmee het proces verloopt wordt door verschillende factoren bepaald t.w.: de soort plastic, de soort en concentratie van de katalysator en de versneller, de werkt temperatuur, de hoeveelheid polyesterhars en de vorm van het gietstuk, vulstoffen, het vochtgehalte van de vulstof, inhibitors en het styreengehalte.

Methode

Luchtdroge grondmonsters van $15 \times 8 \times 5$ cm worden in series van vier geïmpregneerd. Voor een goed resultaat is nodig: een vacuüm van ± 60 mm kwik, een sterke verlaging van de viscositeit van het plastic (± 15 poise) met monostyreen, en een lange geleringstijd (d.w.z. lage katalysator- en versnellerconcentraties). De samenstelling van de impregnatievloeistof is daarom: 1500 cc Vestopal-H; 1000 cc monostyreen; 4 cc katalysator (cyclohexanonperoxyde 50 %); 2 cc versneller (cobaltoctoaat 1 %). Bij een werkt temperatuur hoger dan $\pm 22^\circ\text{C}$ moeten de katalysator- en versnellerconcentraties worden gehalveerd. De impregnatie duurt $\pm 7\frac{1}{2}$ uur en geschiedt in het in fig. 3 afgebeelde apparaat. De doos met de vier geïmpregneerde monsters plaatst men vervolgens in een zuurkast met sterke afzuiging om de overmaat monostyreen zo snel mogelijk te doen verdampen (noodzakelijk, daar een hoog styreengehalte verschillende eigenschappen van het polymerisaat ongunstig beïnvloedt). Dit duurt ongeveer drie weken, gedurende welke tijd iedere twee à drie dagen de in die periode verdampte hoeveelheid monostyreen wordt vervangen door een gelijk volume onverzadigd plastic (samenstelling: 1000 cc Vestopal-H; 2,5 cc katalysator; 2,5 cc versneller). Na nog enige weken in de zuurkast te hebben gestaan zijn de blokken hard en worden bij kamertemperatuur op het laboratorium bewaard. Tegen de tijd dat ze verwerkt moeten worden, vindt een naharding van enige dagen bij maximaal 40°C plaats. De blokken worden vervolgens met een grote steenzaag (fig. 4) in plakken van minimaal 8 mm dikte gezaagd. Op een vlakslijpmachine met elektromagnetische opspantafel waarop de coupes met metalen mallen worden vastgeklemd (fig. 6 en 7), vlakt men daarna de plakken stuk voor stuk. De gevlakte zijde van de coupe slijpt men dan uit de hand,

op plexiglas platen met een serie korundpoeders van afnemende korrelgrootte, fijn af. Bij het gehele procédé wordt gekoeld met Shell-olie V-0847, terwijl het monster na iedere bewerkingsfase met wasbenzine grondig wordt gereinigd. Het geslepen vlak polijst men dan op met kunststof bespannen plexiglas platen met diamantpasta's (fijnste: 0,5 mu diameter) en wasbenzine als smeermiddel. De gepolijste zijde wordt met veel benzine ten slotte op zeemleer gewassen en daarna op een objectglas van $200 \times 100 \times 1,2$ mm geplakt met: 10 cc Vestopal-H; 0,2 cc katalysator en 0,1 cc versneller. Om de coupe planparallel op het glas te krijgen verzwaart men hem met een gewicht van ± 1450 g (fig. 8 en 9). Het opgeplakte preparaat wordt met behulp van een speciale mal op de zaagmachine tot ± 2 mm dikte afgezaagd (fig. 10 en 11), waarna het op de vlaklijpmachine tot ± 40 mu dikte kan worden afgenomen. Met de hand wordt op de reeds genoemde wijze tot 15 mu dikte afgeslepen en gepolijst. Het plastic voor het afdekken heeft dezelfde samenstelling als dat wat voor het opplakken wordt gebruikt. Het dekglas is $160 \times 80 \times 0,2$ mm. Ook het afgedekte preparaat wordt met een gewicht van 1450 g verzwaard. De figuren 12 en 13 geven een indruk over de kwaliteit en enige toepassingsmogelijkheden van de mammoet-slijpplaten.

Veiligheidsmaatregelen

Noodzakelijke veiligheidsmaatregelen bij de toepassing van de methode zijn vooral een goede ventilatie van de werkruimte, het vermijden van open vuur en vonkvorming en het goed verpakt, afzonderlijk bewaren van katalysatoren en versnellers op een brandvrije plaats. Vloeibare organische peroxyden kunnen ernstige beschadigingen in het organisme aanrichten (tegenmaatregelen: vomeren, veel in water opgelost natriumascorbaat drinken en de maag uitpompen. Onder meer kan cyclohexanonperoxyde bij contact met de ogen tot blindheid leiden (veiligheidsbril, wassen met 5 %-oplossing van natriumascorbaat resp. 2 % natriumbicarbonaat en minstens 15 minuten met water naspoelen). Altijd arts waarschuwen! Langdurig contact van de huid met organische peroxyden, vloeibaar plastic en koelolie kan leiden tot huidandoeningen (speciale handschoenen, huidcrèmes). Bij het zagen is het gebruik van een ademhalingsmasker (vernevelende olie!) en van veiligheidsklemmen (fig. 4, 5 en 11) geboden.

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