Soil Survey

Soil Survey Laboratory Methods

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SOIL SURVEY LABORATORY METHODS

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1 Introduction

B. W. Avery

This publication describes methods currently used in the Soil Survey of England and Wales to characterize soil materials in the laboratory. Most of the procedures are described in enough detail for them to be reproduced in other laboratories, but appropriate references are given to aid readers needing further information about the theory or technique of particular methods. Suppliers of special apparatus and materials are listed in the Appendix. Description and measurement of soil properties in the field, and procedures for collecting soil samples, are treated in the Soil Survey Field Handbook (Hodgson 1974).

Sampling units

The value of laboratory data in soil studies depends on effective sampling. Every sample should represent a definite body or class of soil that is as homogeneous as possible. The dimensions of the sampling unit depend on the purpose of the study and on the size of sample needed to obtain valid representation of the property determined. For example, if the proportion of stones is required, the sample must clearly be much larger than the largest stone present; otherwise the inclusion or exclusion of a single stone will affect the result significantly.

Samples of 1 g or smaller are sufficient for some analyses, and small but distinctive bodies of soil such as clay coats or mottles may be sampled as separate units in detailed pedological studies. For other purposes, samples may be taken from several sites to represent a much larger body of soil, such as the topsoil of a field or experimental plot, or that enclosed by a boundary on a soil map. The samples can be mixed for analysis, or analysed separately to characterize the body sampled in terms of the mean and variance of whichever parameters are determined. However, because of vertical and lateral variability, a very large number of analyses is needed to characterize many bodies of soil in this way.

Hence, in general soil surveys, mappable bodies of soil are characterized primarily by properties of the soil profile that can be measured or assessed in the field at numerous sampling points, using an appropriate classification (Avery 1973). Laboratory determinations are made on samples collected from selected 'bench-mark' soils to standardize and confirm field estimates of properties, such as particle-size distribution and organic-matter content, on which the classification is based, and to evaluate accessory properties not used for classification. For these purposes the basic sampling units are generally soil horizons, *i.e.* more or less homogeneous layers of soil delimited as elements of the soil profile at sites selected to represent a particular class of profiles, *e.g.* a soil series.

The Soil Survey of England and Wales uses undisturbed cores 15 cm in diameter extracted by a Proline soil coring machine (Plate I), or specially dug pits about 1·5 m long and 1 m wide, to identify and examine soil horizons at sampling sites. Horizon samples large enough for many laboratory determinations can usually be taken from Proline cores, but the shape and short-distance lateral variability of horizons, and important properties of individual horizons such as structure (pedality) and stone content, often cannot be assessed adequately. For this reason a pit is preferred when the objective is to characterize a soil profile as fully as possible.

Kinds of sample

Samples collected from soil horizons include (1) disturbed (fragmental or loose) samples for particle-size, chemical and mineralogical analyses; and (2) undisturbed samples for volumetric measurements and studies of moisture-release, structural and micromorphological characteristics. Undisturbed monoliths representing the whole soil profile are also taken, whenever possible with the Proline corer, for exhibition and comparative purposes.

Disturbed samples are normally air dried, screened through a 2 mm sieve, and subsampled for laboratory measurements, most of which are based on unit mass (weight) of oven-dry soil material < 2 mm, or of a smaller particle-size separate. However, the primary sampling units are volumes of soil, and interpretations of soil data are often more usefully expressed on a volume basis. As the mass of fine earth < 2 mm per unit volume can vary greatly, within profiles as well as laterally, the investigator needs to assess and record the contribution of all components of the soil to the total volume. This involves measuring or assessing the bulk density of the fine earth and the percentage by volume of stones coarser than 2 mm.

Stony soils

Very stony soils present serious sampling problems, partly because coring implements are ineffective, and partly because samples need to be large to get valid estimates of stone content. Ideally, samples for measuring stone content by volume or by weight should be 100 times larger than the largest size of stone present, as exemplified in the following Table.

TABLE 1

Minimum Sample Sizes for Estimating Stone Content

Effective diameter of largest particle mm	*Minimum volume of sample* l	*Minimum mass of sample (particle density 2·7)* kg
20	0·4	1·1
60	11	30
200	400	1000

Stone content is usually estimated by volume (or strictly by area) in the field by comparing the appearance of a vertical or horizontal face with figures illustrating various proportionate areas, or by superimposing a suitable grid and counting the number of grid intersections that fall on stones. For more accurate work, the stones in a sample of appropriate size can be separated by sieving and the proportion measured either by volume or by mass (Section 4.1.2). Percentages of a stone fraction by volume can be converted into percentages by mass and *vice-versa*, using the following formulae, where D_{bf} is the bulk density of the fine earth fraction and D_{bs} the bulk density of the stones (approximately 2·7 for most non-porous rocks).

$$\text{Mass \%} = \frac{D_{bs} \times \text{volume \%}}{D_{bf}(100 - \text{volume \%}) + D_{bs} \times \text{volume \%}} \times 100.$$

$$\text{Volume \%} = \frac{\text{mass \%} \times D_{bf}}{(100 - \text{mass \%})D_{bs} + \text{mass \%} \times D_{bf}} \times 100.$$

Table 2 gives mass % equivalents of volume % estimates for stones of specific gravity 2·7 in fine earth of various bulk densities.

Proportions of very small (2–6 mm) and small (6–20 mm) stones are difficult to estimate by volume in the field, and can be determined in the laboratory (Section 2.1) if a large enough sample (*c.* 2 kg) is taken.

Precision and accuracy

In formulating analytical procedures and using the results, both the accuracy and the precision of the data have to be considered. If an analysis of a soil sample is repeated and the result is the same each time, the determination is said to have high *precision*. The *accuracy* of the data is its correctness relative to some absolute standard. For many conventional soil analyses there are no absolute standards. Different methods give differing results, and one method may give more useful results for certain purposes or on certain soils than another, but the greater correctness of the data remains unproven. Hence the method by which a soil property is measured should always be specified, and soils can only be compared rigorously if the same method is used.

TABLE 2

Mass % Equivalents of Volume % Estimates for Stones

(sp. gr. 2·7)

% stones by volume	*% stones by mass when the bulk density (g cm^{-3}) of the fine earth is*					
	0·5	*1·0*	*1·3*	*1·5*	*1·7*	*2·0*
5	22	12	10	9	8	7
10	38	23	19	17	15	13
15	49	32	27	25	22	19
20	57	40	34	31	28	25
25	64	47	41	38	34	31
30	70	54	47	44	40	37
35	74	59	53	50	46	42
40	78	64	58	55	51	47
45	82	69	63	60	56	52
50	84	73	68	64	61	57
55	87	77	72	69	66	62
60	89	80	76	73	70	67
65	91	83	80	77	75	71
70	93	86	83	81	79	76
75	94	89	86	84	83	80
80	96	92	89	88	86	84
85	97	94	92	91	90	88

References

AVERY, B. W. (1973). Soil classification in the Soil Survey of England and Wales. *J. Soil Sci.* **24**, 324–38.

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2 Sample Preparation and Stone Content

C. L. Bascomb and P. Bullock

2.1 AIR-DRY < 2 mm SAMPLES

2.1.1 Mineral soil (< *c*. 30% organic matter)

Apparatus

Air drying cabinet maintained at 25–30°C, well ventilated
Sieves, 20 mm, 6 mm and 2 mm (B.S. 410)
Rukuhia soil crusher, 2 mm round hole screen (Waters and Sweetman 1955) (Appendix, p. 82)

Procedure

Spread the field sample on a drying tray, break down clods to about 3 cm and place in drying cabinet until air dry (24–48 hours).

If stone fractions < 20 mm are to be determined, record the mass (T) of air-dry sample. Hand sieve on 20 mm sieve and record mass ($S1$) of stones retained. When it is important to avoid comminution of soft rock fragments (*e.g.* chalk, shale), the < 20 mm sample must be hand-screened on a 2 mm sieve, gently breaking aggregates; *otherwise* transfer all material passing the 20 mm sieve to the drum of the Rukuhia soil crusher and revolve until all aggregated material has been reduced to < 2 mm and passed into collecting tray. Mix carefully and transfer to labelled box for storage. Remove from drum and place on 6 mm sieve. Record mass ($S2$) of 6–20 mm stones retained and mass ($S3$) of < 6 mm stones passed through.

If no determination of stone content is required, crush or hand-screen the air-dry sample as described above, without weighing, and discard material > 2 mm.

Calculations

$$\text{2–6 mm stones } \% = \frac{S3 \times 100}{T - S1}.$$

$$\text{6–20 mm stones } \% = \frac{S2 \times 100}{T - S1}.$$

References

British Standards Institution (1969). *Specification for test sieves* B.S. 410.

Waters, D. F. and Sweetman, I. C. (1955). The Rukuhia soil grinder. *Soil Sci.* **79**, 411–13.

2.1.2 **Peat soil** (> *c.* 30% organic matter)

Apparatus

Air drying cabinet maintained at 25–30°C, well ventilated
Sieve, 2 mm (B.S. 410)

Procedure

Take about two-thirds of the moist sieved material (Section 2.2 below) and spread it on a drying tray. Place in drying cabinet. Stir once each day until air dry. Break up by hand and pass through a 2 mm sieve, discarding firm woody pieces and roots > 2 mm. Store in labelled container.

2.1.3 **Subsampling**

Apparatus

Subsampling tray* (Plate IIa)
Sieve, 500 μm (B.S. 410)
Morrice mechanical agate pestle and mortar
Glen Creston Hammer Mill with 0·5 mm screen

Procedure

Place the subsampling tray on a sheet of paper. Spread the < 2 mm air-dry soil in an even layer in the tray. Carefully lift the tray off the paper and return the remaining contents of the tray to the sample box. Collect the subsample from the paper. If it is still too large, repeat the procedure.

If carbon, nitrogen or calcium carbonate are to be determined in mineral soil, grind not less than 20 g of the subsample in the agate mortar to pass 500 μm sieve. If the soil is peat, mill 10 g of the subsample in the Glen Creston machine to pass 0·5 mm screen. Store in a labelled tube in the same box as the < 2 mm sample.

Reference

BRITISH STANDARDS INSTITUTION (1969). *Specification for test sieves.* B.S. 410.

2.2 FIELD-MOIST PEAT SAMPLES

Apparatus

Sieve, 20 mm (B.S. 410)

Procedure

Samples are received in moisture-proof containers, *e.g.* sealed polythene bags. Each sample is gently hand sieved, using a 20 mm sieve and taking care that undecomposed plant structural units > 20 mm are not broken. The operation is done as quickly as possible to avoid loss of water. The sieved sample is returned to a moisture proof container.

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* Designed by K. Thanigasalam.

The layer of peat in contact with a gas-permeable polythene bag may undergo oxidation during prolonged storage in the moist state and should therefore be avoided when abstracting subsamples for analysis.

2.3 THIN SECTIONS

Undisturbed samples for micromorphological studies are collected whenever possible in rectangular boxes, termed Kubiena boxes, made of brass or aluminium. They are hinged in one corner to facilitate removal of the sample and have lids at top and bottom. The size of box varies with the size of the section required, which in turn depends on the purpose for which the study is being made.

If the sample is intended to represent a soil horizon identified and described in the field, samples large enough to make a section 15 × 8 cm are preferable. Alternatively, triplicate randomly sited sections of smaller (7·5 × 2·5 cm) size, or composite sections (Grossman 1964) may be used. For purposive sampling, when specific macroscopic features are selected for characterization in detail, the size of the sample will depend on the size of the feature.

References

Grossman, R. B. (1964). Composite thin sections for estimation of clay-film volume. *Proc. Soil Sci. Soc. Am.* **28**, 132–3.

Hodgson, J. M. (Ed.) (1974). *Soil Survey Field Handbook*. Tech. Monogr. Soil Surv. No. 5.

2.3.1 Impregnation of dried samples using Autoplax 110C

Apparatus

Air drying cabinet (Section 2.1)
Oven or freeze drier
Lightweight polythene boxes to fit samples
Mechanical stirrer
Vacuum desiccator
Resin reservoirs

Materials (Appendix, p. 82)

Autoplax resin 110C
Autoplax Styrene Monomer C
Catalyst powder 2
Accelerator 5
Uvitex OB fluorescent dye

Procedure

Preparation of samples. All samples must be dried because the polyester resin is immiscible with water. Samples with a small water-retention capacity are air dried in their containers for one week before impregnation. Those with a large water-retention capacity are freeze or oven dried, the former being recommended. If freeze dried, only the outer parts of the sample should be used for sections.

When dry, the samples are transferred to individual polythene boxes labelled with profile number, depth and orientation. Samples liable to collapse can either be left in the original sampling tin until after impregnation (which can spoil the tin for further sampling) or sprayed with a cellulose acetate solution until firm enough to be removed.

Impregnation with Autoplax 1*10C**. Autoplax 110C is a polyester resin which on addition of catalyst and accelerator hardens a soil sample so that sections can be prepared using rock sectioning equipment. The addition of fluorescent dye to the impregnating mixture enables voids to be clearly seen in the finished section when viewed under u.v. light and thus aids their measurement. The composition of the impregnating resin mixture is in Column 2, Table 3. The Autoplax resin and Styrene Monomer C are mixed for 5 min with a mechanical stirrer. The catalyst and fluorescent dye are then added and the whole mechanically stirred again. A litre of impregnating mixture is sufficient for four containers with samples.

Individual containers with samples are placed on rotatable trays in a vacuum desiccator (Fig. 1) and evacuated for 15 min before addition of the impregnating mixture.

TABLE 3

Composition of Resin Mixture used in Thin Section Preparation

	Impregnation	*Replenishment*	*Re-impregnation*	*Fixing of section and cover slip*
Autoplax resin 110C	660 ml	660 ml	200 ml	25 ml
Styrene Monomer C	340 ml			
Accelerator 5			2–3 ml	0·5 g
Catalyst powder 2	4 g	4 g	6 g	1 g
Uvitex OB dye	4 g	2 g	0·5 g	

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* Due to a current world shortage of styrene, a method based on Bakelite resin using acetone instead of styrene is being investigated as a possible replacement for Autoplax. Details of the Bakelite method can be obtained from FitzPatrick (1970), but it should be noted that the manufacturers have now replaced the resin BK 9001 by BK 9191, and this requires four times more catalyst than the original BK 9001.

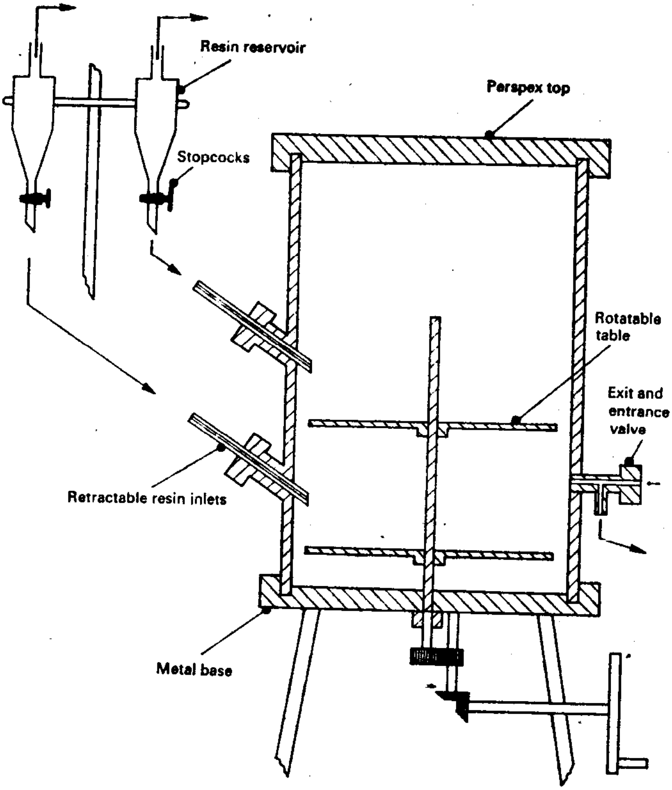

Fig. 1. Vacuum desiccator for impregnation with Autoplax 110C.

The resin mixture in two reservoirs above the desiccator is added to the evacuated samples individually by means of retractable inlet tubes and rotation of the table. When the resin has been added, the system is evacuated for a further 12 hours, at the end of which the vacuum is released slowly over a period of 5 min and the samples removed to a fume cupboard to polymerize.

Polymerization of impregnating mixture. Polymerization time can be adjusted by changing the amount of catalyst. Normally a period of at least 3 weeks, using the standard composition in Table 3, is advisable to reduce stresses and strains on gelling to a minimum.

During the early stages of gelling, the level of liquid in the containers falls due to evaporation of styrene. Samples must remain covered by liquid, otherwise air may re-enter. Thus the original level of the liquid should be regularly maintained using a replenishment mixture (Column 3, Table 3). After about 6 weeks, when gelling is complete except for a tacky outer surface, the samples are transferred to a drying oven at 40°C for final hardening.

2.3.2 Impregnation of moist samples using Carbowax 6000

Materials

Carbowax 6000
Crystal violet dye
1,1,1,trichloroethane

The use of Carbowax 6000 to impregnate moist samples of clays and organic soils has been demonstrated by Mitchell (1956), Morgenstern and Tchalenko (1967) and Greene-Kelly and Chapman (1970). It can also be used for soils containing as little as 20% clay. Because samples need not be dried first, shrinkage is reduced to a minimum and the method is therefore preferable to that using polyester resins when pore space is to be measured in thin sections. Its main disadvantage is that thin sections need to be mainly hand processed.

Procedure

Samples are immersed in a 10% aqueous solution of Carbowax for 5 min, followed by 2 hours in a 25% and 4 hours in a 50% aqueous solution. The temperature is then raised to 65°C and the sample left at this temperature overnight. The sample is then immersed in pure Carbowax which is changed twice during the impregnation period of 5 days. Fairly dry (pF 2–3) or loose samples can be placed directly in a 50% aqueous solution. When impregnation is complete the sample is removed and allowed to set after draining as completely as possible.

Cationic dyes such as crystal violet are useful for studying voids. The dye can be added during impregnation or used as a stain after grinding the section and before mounting the cover slip.

References

FitzPatrick, E. A. (1970). A technique for the preparation of large thin sections of soils and unconsolidated materials. In: *Micromorphological techniques and applications*. Tech. Monogr. Soil Survey No..2.

Greene-Kelly, R. and Chapman, S. (1970). The preparation of thin sections of soils using polyethylene glycols. In: *Micromorphological techniques and applications*. Tech. Monogr. Soil Survey No. 2.

Mitchell, J. K. (1956). The fabric of natural clays and its relation to engineering properties. *Proc. Highw. Res. Bd*, **35**, 653–713.

Morgenstern, N. R. and Tchalenko, J. S. (1967). The optical determination of preferred orientation in clays and its application to the study of microstructure in consolidated kaolin. *Proc. R. Soc. A*, **300**, 218–34.

2.3.3 Cutting, mounting and grinding

Apparatus (Appendix, p. 82)

Woco 220 Diamond saw
Hacksaw
Glass microscope slides
Dawe ultrasonic cleaner
Slide press
Jones and Shipman Model 540VS surface grinding machine
Metaserv Universal Polisher with twin lapping wheels
Carborundum papers
Metaserv lapping cloths
Dialap Diamond Compound

Materials

Shell fusus A lubricant oil (Appendix, p. 82)
Paraffin
Beeswax

Procedure

Preparation of sections impregnated with Autoplax 110*C.* Three slices, 1 cm thick, are sawn from each block with a Diamond saw using Shell fusus A oil as lubricant. The slices are cut according to the features to be examined and the orientation required for the section. If impregnation of the sample is poor, slices should be re-impregnated with the appropriate mixture.

A well impregnated slice is fixed to a planed steel block of about its size with beeswax. The block is placed on the magnetic chuck of a Jones and Shipman vertical spindle surface grinding machine and the upper face of the slice ground smooth. The slice is then removed from the block and the smoothed face polished on a lapping wheel with diamond paste. After thorough ultrasonic cleaning with deodorized paraffin, the slice is ready for mounting.