

The Sampling and Initial Preparation of Sewage and Waterworks' Sludges, Soils, Sediments and Plant Materials Prior to Analysis 1977

Warning to users

Methods for the Examination of Waters and Associated Materials

This booklet consists of two parts:

- A Sampling of Sewage and Waterworks' Sludges and Related Solids, and Plants for subsequent Analysis
- B Initial Preparation of Soils, Sediments and Plant Materials prior to Analysis

The methods described should be used in conjunction with the appropriate methods for specific determinands now being issued as part of this series.

Contents

Warning to users	2
About this series	3
A Sampling of Sewage and Waterworks' Sludges and Related Solids, for subsequent Analysis	4
1 Introduction	4
2 Notes on Sampling	4
3 Hazards	5
4 Sampling from Tanks	5
5 Sampling from Pipes	6
6 Sampling from Open Channels	7
7 Sampling of Sludge from Heaps and Stockpiles of Sludge Cake	7
8 Sampling of Soil	7
9 Sampling of Sediments	8
10 Sampling of Plants	9
11 Anomalous Material	9
12 References	10
B Initial Preparation of Soils, Sediments and Plant Materials prior to Analysis	11
1 Introduction	11
2 Precautions	11
3 Procedure for Soils	11
4 Procedure for Plant Materials	12
5 References	13
Address for Correspondence	13
Membership responsible for this method	inside back cover

Warning to users

The analytical procedures given in this booklet should only be carried out by competent trained persons, with adequate supervision when necessary. Local Safety Regulations must be observed. Laboratory procedures should be carried out only in a properly equipped laboratory. Field operations should be conducted with due regard to possible local hazards, and portable safety equipment should be carried. Care should be taken against creating hazards for others. Lone working, whether in the laboratory or field, should be discouraged. Reagents of adequate purity must be used, along with properly maintained apparatus and equipment of correct specification. Specifications for reagents, apparatus and equipment are given in manufacturers' catalogues and various published standards. If contamination is suspected, reagent purity should be checked before use.

There are numerous handbooks on first aid and on laboratory safety. One such publication is 'Code of Practice for Chemical Laboratories' issued by the Royal Institute of Chemistry, London. Where the Committee has considered that a special unusual hazard exists, attention has been drawn to this in the text so that additional care might be taken beyond that which should be exercised at all times when carrying out analytical procedures. It cannot be too strongly emphasised that prompt first aid, decontamination, or administration of the correct antidote can save life, but that incorrect treatment can make

matters worse. It is suggested that both supervisors and operators be familiar with emergency procedures before starting even a slightly hazardous operation, and that doctors consulted after any accident involving chemical contamination, ingestion, or inhalation, be made familiar with the chemical nature of the injury, as some chemical injuries require specialist treatment not normally encountered by most doctors. Similar warning should be given if a biological or radiochemical injury is suspected. Some very unusual parasites, viruses and other micro-organisms are occasionally encountered in samples and when sampling in the field. In the latter case, all equipment including footwear should be disinfected by appropriate methods if contamination is suspected.

The best safeguard is a thorough consideration of hazards and the consequent safety precautions and remedies well in advance. Without intending to give a complete checklist, points that experience has shown are often forgotten include: laboratory tidiness, stray radiation leaks (including ultra violet), use of the correct protective clothing or goggles, removal of toxic fumes and wastes, containment in the event of breakage, access to taps, escape routes, and the accessibility of the correct and properly maintained first aid, fire-fighting, and rescue equipment. If in doubt it is safer to assume that a hazard may exist and take reasonable precautions than to assume that no hazard exists until proved otherwise.

About this series

This booklet is one of a series intended to provide recommended methods for the determination of water quality. In the past, the Department of the Environment and its predecessors, in collaboration with various learned societies, has issued volumes of methods for the analysis of water and sewage culminating in 'Analysis of Raw, Potable and Waste Waters'. These volumes, inevitably, took some years to prepare, so that they were often partially out of date before they appeared in print. The present series will be published as individual methods, thus allowing for the replacement or addition of methods as quickly as possible without need of waiting for the next edition. The rate of publication will also be related to the urgency of requirement for that particular method, tentative methods being issued when necessary. The aim is to provide as complete and up to date a collection of methods and reviews as is practicable, which will, as far as possible, take into account the analytical facilities available in different parts of the Kingdom, and the quality criteria of interest to those responsible for the various aspects of the water cycle. Because both needs and equipment vary widely, where necessary, a selection of methods may be recommended for a single determination. It will be the responsibility of the users - the senior analytical chemist, biologist, bacteriologist etc, to decide which of these methods to use for the determination in hand. Whilst attention of the user is drawn to any special known hazards which may occur with the use of any particular method, responsibility for proper supervision and the provision of safe working conditions must remain with the user.

The preparation of this series and its continuous revision is the responsibility of the Standing Committee of Analysts (to review Standard Methods for Quality Control of the Water Cycle). The Standing Committee of Analysts is one of the joint technical committees of the Department of the Environment and the National Water Council. It has nine Working Groups, each responsible for one section or aspect of water cycle quality analysis. They are as follows:

- 1.0 General principles of sampling and accuracy of results
- 2.0 Instrumentation and on-line analysis
- 3.0 Empirical and physical methods
- 4.0 Metals and metalloids
- 5.0 General non-metallic substances
- 6.0 Organic impurities
- 7.0 Biological methods
- 8.0 Sludge and other solids analysis
- 9.0 Radiochemical methods

The actual methods etc are produced by smaller panels of experts in the appropriate field, under the overall supervision of the appropriate working group and the main committee. The names of those associated with this method are listed inside the back cover.

Publication of new or revised methods will be notified to the technical press, whilst a list of Methods in Print is given in the current HMSO Sectional Publication List No 5, and the current status of publication and revision will be given in the biennial reports of the Standing Committee of Analysts.

TA DICK
Chairman

LR PITTEWELL
Secretary

4 November 1976

Part A Sampling of Sewage and Waterworks' Sludges and Related Solids for Subsequent Analysis (1977 version)

1 Introduction

Sampling and the determination of the physical and chemical properties of sludges and related solids are normally carried out for a specific purpose within an organisation. The analysis is not normally the subject of a potential inter-organisation dispute and a rigid adherence to a precise procedure is not always necessary provided that the analyst is aware how deviations can effect the final results obtained. Where an analytical method is applicable to a particular type of solid this is stated, otherwise the methods are suitable for general use but do not exclude modification in the light of any special factor known to the analyst. For particular sampling and analytical problems and where more detailed information is required an appropriate body such as the Water Research Centre, Water Authorities, or Government Department should be contacted.

Reference should be made to the publication in this series 'General Principles of Sampling and Accuracy of Results'.

The importance of using a valid sampling technique cannot be overemphasised if the subsequent analysis is to be worthwhile. It is essential that the personnel taking and analysing the sample are made fully aware of its nature and the purpose for which the analysis is required *before* embarking on a work programme.

2 Notes on Sampling

The sampling of sludges and similar solids may be required for a variety of reasons including:

- 2.1 Process control in potable and waste water treatment including:
 - 2.1.1 Addition or withdrawal of solids
 - 2.1.2 Addition or withdrawal of liquid
- 2.2 Environmental aspects of the disposal of sewage and waterworks sludges and related solids
- 2.3 Monitoring of river, estuarine and marine sediments
- 2.4 Special investigations into the performance of new equipment and processes

The types of sample which may be required include:

- 2.4.1 Instantaneous
- 2.4.2 Continuous: (a) constant rate sampling (b) flow related sampling
- 2.4.3 A programmed series of discrete sub-samples for preparing a composite sample

Individual samples should be retained until the composite has been analysed. Where there is a requirement for mass flow information then it is necessary to measure the associated flow.

The methods of sampling and analysis are both very time dependant and where there is an immediate operational requirement for information, a certain loss of accuracy may be acceptable.

It is most important that the sample be representative of the solid concerned. For some types of sample, particularly raw sewage sludge, gross atypical solids should be removed by passing the sample through a 5 mm sieve. Atypical solids may be needed for further examination and should be retained. Some samples may change significantly because of biological activity and it is therefore important that such samples should be analysed

3 Hazards

as soon as possible after sampling. If samples have to be stored or transported prior to analysis their temperature should be maintained between 0 and 5°C; in some cases preservatives may be added. Special precautions may be needed for certain analyses and details of these are given in the appropriate method booklets.

It may be necessary to take samples from a sewer system as well as a sewage treatment works, but in either case certain risks or hazards are likely to be present. These include:

- Physical injury and bacterial infection
- Dangerous atmospheres (oxygen deficiency, toxic gases and vapours, flammable and explosive gases and vapours)
- Radioactivity
- Flooding

Personnel engaged in sampling must make themselves familiar with the safety equipment and procedures available to avoid physical injury etc, and the operation of gas/vapour testing equipment that is used to assess the quality of the atmosphere in restricted spaces; eg wet wells of pumping stations, sewers and manholes, sludge digestion plant etc.

The provisions of the Health and Safety at Work Act and the detailed information given in reference ⁽¹⁾ should be carefully studied and put into effect.

4 Sampling from Tanks

The performance of tanks used for sedimentation or consolidation of water works or sewage sludges, digesters, and other tanks, cannot always be gauged from samples taken from the inlet and outlet pipelines because of the segregation of the solids that can occur. This can be detected by sampling different sections and depths of a tank.

4.1 If suitable sampling points are not built into a tank, samples will have to be taken from the top of the tank. For most applications a commercial fixed volume depth sampler can be used. Alternatively the vacuum sampler illustrated in figure 1 has been used successfully by the Water Research Centre. Aluminium pipe, earthed to the tank of 25 mm bore, in 2 m sections joined by screw connections which do not reduce the bore, is connected via a flexible pipe and valve to a 10-litre glass bottle which must be surrounded by an adequate guard to prevent injury should it collapse; it may be evacuated either by a hand or by a vacuum pump fitted with a flame-proof motor. It is necessary to obtain a good vacuum in the bottle, before suddenly opening the valve to the sampling line. Before taking a sample at each position, withdraw some sludge into another clean

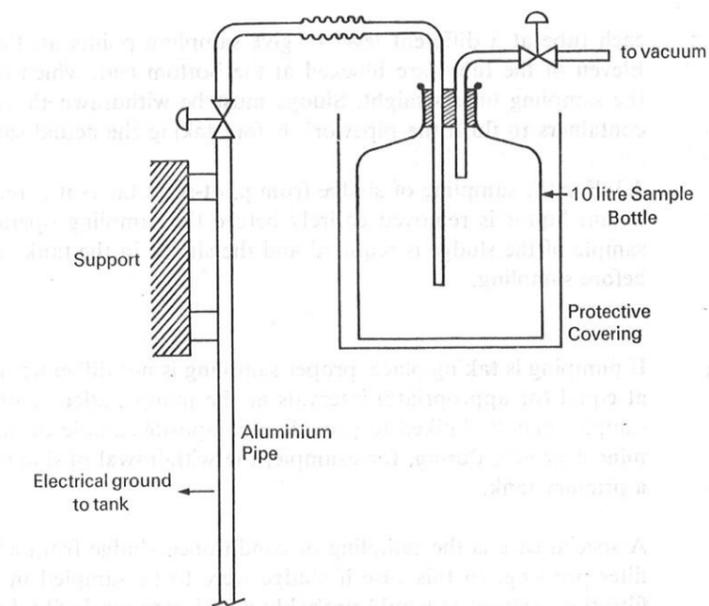


Figure 1 Vacuum sampler

10-litre bottle to flush the pipe out. This method is particularly suitable for sampling from digesters, either through a port on the roof or the sludge seal. It is important to remove encrusted sludge from the sampling point before inserting the aluminium pipe. Only suitably protected electrical equipment should be used for sampling from digesters, these and all other metal components must be earthed electrically.

4.2 For sampling thin sludges, as for example in blanket clarifiers or final settlement tanks, a suitable commercial sampler using small-bore plastic tube can be used. Depth profiles of solids concentration in final settlement tanks have been successfully determined by the Water Research Centre using a modified commercial 12-bottle vacuum sampler to take simultaneous samples as shown in figure 2. A small plastic T-piece is inserted in

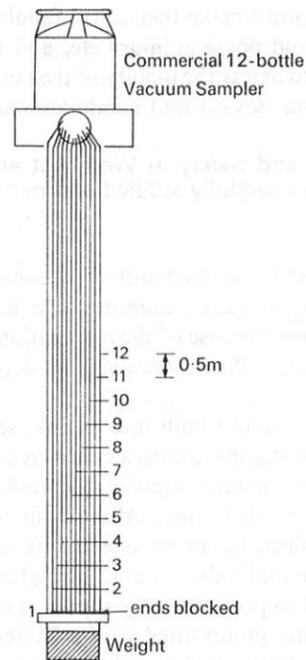


Figure 2 Multiple depth sampler

each tube at a different level to give sampling points at, for example 0.5 m intervals. Eleven of the tubes are blocked at the bottom end, which is heavily weighted to keep the sampling lines straight. Sludge must be withdrawn through the tubes into dummy containers to flush the pipework before taking the actual samples.

4.3 For the sampling of sludge from pilot-scale tanks it is recommended that the supernatant liquor is removed entirely before the sampling operation. Usually a composite sample of the sludge is required and the sludge in the tank should be thoroughly mixed before sampling.

5 Sampling from Pipes

If pumping is taking place, proper sampling is not difficult; sub-samples should be taken at equal (or appropriate) intervals at the pump outlet or other convenient place. The samples may be bulked to provide a composite sample or analysed separately to determine a profile, during, for example, the withdrawal of sludge from a blanket clarifier or a primary tank.

A special case is the sampling of conditioned sludge from a high-pressure line prior to filter pressing. In this case if sludge were to be sampled in a conventional manner its filtration properties would probably deteriorate markedly due to shear in the sampling valve. To sample a conditioned sludge with the minimum of shear, the simple apparatus shown in figure 3 should be used.

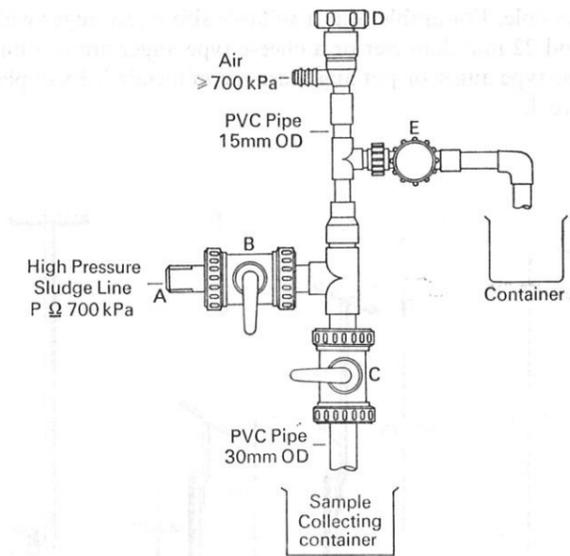


Figure 3 High pressure sampler

The apparatus is connected to the high pressure line at point A. All the valves are closed.

- 1 Open valve D and admit compressed air until the pressure in the apparatus is equal to the operating pressure of the filter press.
- 2 Close valve D and open valve B.
- 3 Slightly open valve E to allow air to escape and sludge to be sampled through the open valve B.
- 4 When sludge appears at the outlet of valve E the sampling compartment is full of sludge. Close valve E.
- 5 Close valve B and open valve E to reduce the pressure to atmospheric.
- 6 Open valve C and withdraw the sludge sample.

If the dead volume of sludge in the high pressure sampling line A is significant, then the above procedure should be repeated as necessary to ensure that fresh sludge is drawn off as the sample.

6 Sampling from Open Channels

It is normally satisfactory to use a weighted bucket or a suitable pump to sample from an open channel provided that in the latter case the velocity in the suction pipe is sufficient to keep all the heavy particles in suspension. Samples should be taken across the width and depth of the channel to ensure that a representative composite sample is obtained after mixing the individual samples. It should be borne in mind that the physical characteristics of the sludge may change in passing through a pump.

7 Sampling of Sludge from Heaps and Stockpiles of Sludge Cake

When sampling heaps of air-dried sludge lifted from drying beds or stockpiles of sludge cake it is important to obtain portions of sludge from throughout the heap and not just from the surface layer. Where sludge cake is conveyed by belt from the drying beds or other dewatering process area to the heap, pieces of cake should be taken from the belt at random.

A core should be taken through the depth of each separate piece of cake from the heap/stockpile and a composite sample prepared from, say, 25 such cores. Standard procedures such as quartering⁽²⁾ should be used for sub-sampling.

8 Sampling of Soil

Soil sampling may be carried out to monitor the effect of the agricultural use of sludge. The soil sampling methods are based on those recommended by the Ministry of Agriculture, Fisheries and Food⁽³⁾. It is essential that a proper sampling tool be used where-

ever possible. For arable soils a suitable size screw auger with a screw bit about 180 mm long and 22 mm diameter or a cheese type auger are recommended. For grassland soils a cheese type auger or pot auger are recommended. Examples of these augers are shown in figure 4.

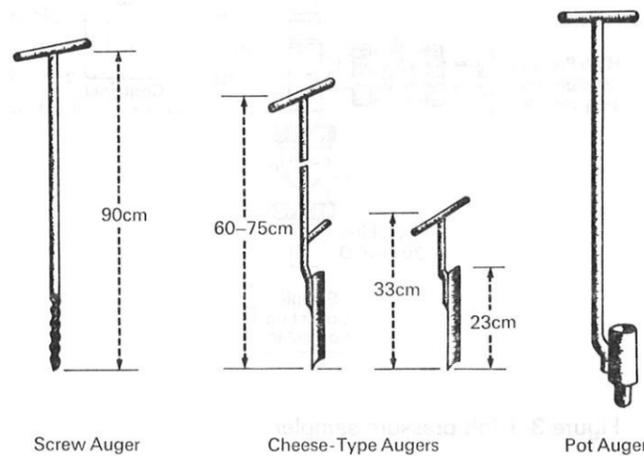


Figure 4 Augers

The sampling area should not be more than approximately 4 ha. Thus if a large area of, say, 12 ha is to be sampled for soil analysis, 3 separate areas of 4 ha should be sampled. At least 25 locations in each area irrespective of its size should be sampled in a W-shaped path as shown in figure 5. Do not take the sub-samples from any obviously unusual

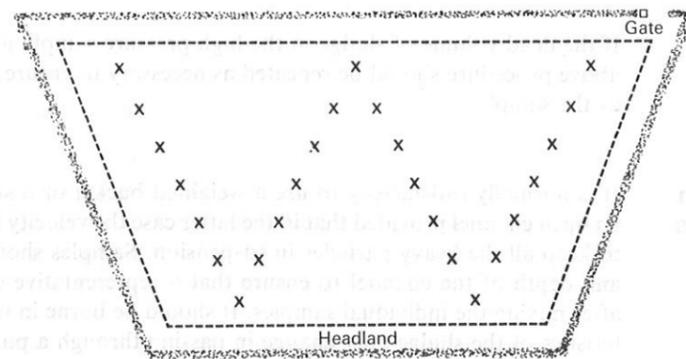


Figure 5 Field sampling pattern, Showing sampling path across field. X marks points where samples taken.

areas of the field (but see also Section 11). The depth of the core taken should be 150 mm for arable soils and short-term leys and 75 mm for permanent grassland and long-term leys. Standard procedures such as quartering⁽²⁾ should be used for sub-sampling.

9 Sampling of Sediments

The sampling of benthos has been reviewed by Kajak⁽⁴⁾. For intact samples from solid beds and fairly stable flocculant beds a tube corer should be used. A construction drawing of a simple type used by the Water Research Centre is shown in figure 6.

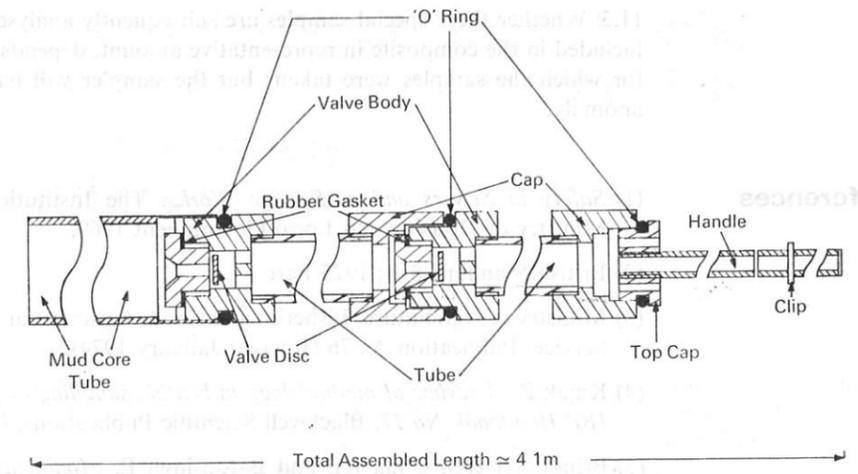


Figure 6 Tube corer

This is suitable for use in water depths up to about 2 m. The corer is pushed vertically into the bed, by hand, to the required depth. As the corer is withdrawn the internal valves help to keep the sample intact. Just before the bottom of the corer breaks the water surface a rubber bung is inserted in the bottom of the tube. The bottom section of the tube containing the core can be detached and the appropriate analysis carried out on the sample. Other designs of corer are described by for example Kajak⁽⁴⁾, Brinkhurst⁽⁵⁾ and for estuarine muds by Holme and McIntyre⁽⁶⁾. For loose gravel sediments, samples can be taken using an Ekman or similar grab sampler⁽⁴⁾ (see also Section 11).

10 Sampling of Plants

A composite sample should be prepared which is representative of the plant population and be free of contamination. Plants may be sampled for the examination of the leaves and/or the root system, but the aerial parts must be packed separately from root samples for transport to the laboratory. The aerial parts must be obtained by cutting with non-rusty steel shears or secateurs at least 3 cm above the level of the soil; the exact distance depending on the form and growth habit of the plant. When sampling root crops the whole root should be taken and transferred to the laboratory for the removal of adhering soil by washing prior to sub-sampling. The containers used for transporting and storing samples must be made from materials which will not contaminate them.

Contamination of the plant may arise as the result of spraying or the application of fertilizer to the plant or by spray and dust drift from neighbouring areas. It is advisable to ascertain whether such treatment has been given before commencing a sampling programme. The extent of contamination by soil depends on the plant form, weather conditions prevailing prior to sampling and whether or not in the case of pasture herbage, grazing has occurred. It is recommended that at least 2 weeks should elapse from the cessation of grazing or at least 10 cm of new growth has appeared before sampling is carried out.

It is recommended that before commencing a particular programme of plant sampling it should be discussed with the appropriate Government Department. Further details regarding plant sampling procedure may be found in references⁽⁷⁾ and⁽⁸⁾ (see also Section 11).

11 Anomalous Material

11.1 If the material, area, or crop to be sampled contains some obviously unusual material, this should be sampled separately and kept apart from the main samples in clearly marked containers. If several different types of anomaly occur, the various special samples should be kept separate from each other.

11.2 The sampler must submit a written report on the nature, size and location of each anomaly sampled and the location of the extra separate sample points, which report should accompany both the main and special samples.

11.3 Whether these special samples are subsequently analysed separately, discarded, or included in the composite in representative amount, depends on the problem or control for which the samples were taken; but the sampler will have drawn attention to the anomaly.

12 References

- (1) *Safety in Sewers and at Sewage Works*. The Institution of Civil Engineers and Ministry of Housing and Local Government 1969.
- (2) British Standard 812: 1975 Part 1.
- (3) Ministry of Agriculture, Fisheries and Food, Agricultural Development and Advisory Service. Publication As 76 (Revised January 1974).
- (4) Kajak Z. *A review of methodology in benthic sampling* in Edmonson WT (Ed), 1969. *IBP Handbook No 17*, Blackwell Scientific Publications, Oxford.
- (5) Brinkhurst RO, Chua KE and Batoosingh E. *Modifications in sampling procedures as applied to studies on the bacteria and tubificid oligochaetes inhabiting aquatic sediments*. J. Fish. Res. Bd. Canada, 1969, **26**, 2581-2593.
- (6) Holme NA and McIntyre AD. *Methods of study of marine benthos*, 1971. *IBP Handbook No 16*, Blackwell Scientific Publications, Oxford.
- (7) Scott RO, Mitchell RL, Purves D, Voss RC. *Spectrochemical Methods for the Analysis of Soils, Plants and other Agricultural Materials*. The Macaulay Institute for Soil Research, Aberdeen. Consultative Committee for Development of Spectrochemical Work, *Bulletin 2*, 1971.
- (8) Mitchell RL. *Contamination problems in soil and plant analysis*. J. Sci. Fd. Agric. 1960, **10**, 553-560.

Part B The Initial Preparation of Soils, Sediments and Plant Materials Prior to Analysis (1977 version)

1 Introduction

It is most important that the sub-sample analysed represents the original sample otherwise the analytical result will be of little value. Each sample must be treated according to the analysis required. The following paragraphs should be regarded as a guide covering the initial preparation of samples which is applicable to most samples. It is not a comprehensive set of instructions. The initial preparation techniques are based on those recommended by the Ministry of Agriculture, Fisheries and Food⁽¹⁾.

2 Precautions

Contamination problems can arise during the preparation and analysis of soils and plants. Sources of trace element contamination can be soil (on plant material), atmospheric dust, laboratory equipment and reagents used during analysis and the analyst should take suitable precautions to reduce these to a minimum. Some of these problems have been reviewed by Mitchell⁽²⁾.

Special precautions are necessary during the initial preparation of soils and plants for certain analyses, eg boron, mercury and selenium and details of these precautions are given in the appropriate analytical method booklets in this series.

For volatile or labile determinands, special attention should be given to methods of drying or reduction of the sample. Under certain circumstances it may be preferable to determine moisture content by a method to be published later in this series and homogenise the sample with a blender or similar device.

3 Procedure for Soils

3.1 Principle

For most analyses, soil is brought to the air dried condition. This term refers to soil conditioned to ambient temperature and humidity, although artificial heating at a temperature not exceeding 30°C may be used in the drying process^(a). The soil is ground to pass through a sieve with round holes 2 mm in diameter, every effort being made to avoid metallic contamination. The sieve should meet the requirements of the British Standards Institute specification BS410.77. The special initial preparations required for certain analyses are given in the appropriate analytical method booklets in this series.

3.2 Drying

Place the soil sample in a shallow aluminium tray, freshly lined with either polyethylene sheeting or greaseproof paper to avoid metal contamination^{(b)(c)}. Break any clods between polyethylene gloved fingers and, as far as possible, remove stones. Air dry the soil by exposure to the atmosphere at a temperature not exceeding 30°C^(d). The drying time required will depend on the type of soil and drying method, but in any event should not be less than 24 hours^{(a)(e)}.

3.3 Sieving

Crumble the air-dried sample in a suitable mortar, or on a polyethylene sheet with a wooden roller, under conditions such that the soil aggregates are disintegrated but the crushing of the mineral particles of soil is avoided. Proceed until the entire sample, excluding stones and fibrous material, passes through the sieve described in Section 3.1.

For special determinations and when it is necessary to weigh amounts of less than 1 g,

the whole of the 2 mm sieved sample should be coned and quartered to produce a 20 g sub-sample which is then ground in an agate mortar to pass a 0.5 mm sieve.

3.4 Storage

The air-dried, sieved sample should be stored in a closed polyethylene container except when trace organic determinations are required, in which case glass containers should be used.

4 Procedure for Plant Materials

4.1 Principle

Some analyses must be made on the fresh plant material. However most analyses are carried out after the fresh sample has been sub-sampled, dried and ground. At all stages of the preparation, suitable precautions must be taken to avoid metallic and soil contamination. Where trace element analyses are required, samples must be taken with great care to avoid soil contamination. Actively growing, fresh material free from dust or surface contamination should be sampled.

4.2 Washing

If it is necessary to remove soil and other contaminants, plants can be washed with distilled water or in a 0.1 to 0.3% solution of a nonionic detergent in distilled water, followed by rinsing with distilled water and drying with a cloth or paper tissue. If trace element analysis is required it should be noted that paper tissues may give rise to contamination. Washing should be carried out as rapidly as possible to minimise loss of soluble constituents. For trace element analysis, washing of freshly cut plant material is undesirable: the samples should preferably be taken under conditions where soil contamination can be avoided.

4.3 Sub-sampling

A minimum of 200g of sub-sample, which is representative of the whole should be taken. This must be carried out as quickly as possible to avoid loss of moisture. The method of sub-sampling is dependent on the type of plant material and the analyst should consult the Ministry of Agriculture, Fisheries and Food publication⁽¹⁾.

4.4 Drying

4.4.1 Place at least 200g of the sample or sub-sample into a clean aluminium tray, freshly lined with greaseproof paper^(b) and dry for 12 hours in a gentle current of air, which has been preheated to a suitable temperature.

4.4.2 To avoid possible losses from samples required for the determination of fluorine, selenium and boron and to prevent loss of tissue fluids and caramelisation, such samples should not be dried at a temperature in excess of 50°C⁽¹⁾.

4.5 Grinding

Grinding of the freshly oven-dried material should be carried out in a mill such as a 200 mm mill fitted with a 1 mm sieve and plastic feeding chute^(f). The entire dried sample must be ground and collected from the mill.

4.6 Storage

The ground sample should be stored in glass jars with plastic screw caps away from direct sunlight but samples for trace element analysis are preferably stored in polyethylene containers.

4.7 Drying prior to Storage

Ground plant materials may absorb moisture and it is therefore necessary to dry a sub-sample of the prepared material for 1 hour at 105°C immediately before weighing for analysis to enable the results to be expressed on an oven-dry basis. In the special cases described in Section 4.4.2, an additional sub-sample must be used for moisture determination and the appropriate correction applied.

Notes:

- The length of time required to dry a sample to produce a friable material for subsequent sieving will depend on the nature and type of soil.
- Greaseproof paper or polyethylene sheeting may be used to line the drying trays rather than brown paper or cardboard to avoid contamination of the sample with boron which is present in the other types of paper. Polyethylene sheeting should not be used when drying plant materials.
- Stainless steel drying trays are not recommended even if they are lined with greaseproof paper. Experience has shown that samples can become contaminated with nickel and chromium if stainless steel is used.
- If the ambient temperature exceeds 30°C, air dry the sample at a convenient constant temperature as close as possible to the ambient temperature and record the value.
- Constant moisture content cannot be obtained by this method; if accurate results are required the moisture content must be determined by the method to be published later in this series.
- The materials of construction of the mill, sieves and trays must not give rise to any contamination of the sample.

5 References

- Ministry of Agriculture, Fisheries and Food, *Technical Bulletin 27, The Analysis of Agricultural Materials*, HMSO 1973.
- Mitchell RL, *Contamination problems in soil and plant analysis*, J. Sci. Fd. Agric. 1960, **10**, 553-560.

Address for Correspondence

However thoroughly a method may be tested, there is always the possibility of a user discovering a hitherto unknown problem. Users with information on this method are requested to write to:

The Technical Secretary
The Standing Committee of Analysts
The Department of the Environment
2 Marsham Street
LONDON SW1P 3EB
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Standing Committee of Analysts

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The Sampling and Initial Preparation of Sewage and Waterworks' Sludges, Soils, Sediments and Plant Materials Prior to Analysis 1977

6

Methods for the Examination of Waters and Associated Materials

The Sampling and Initial Prep. of Sewage and Waterworks' Sludges, Soils, Sediments and Plant Materials Prior to Analysis 1977

HMSO

ISBN 0 11 751251 6

London Her Majesty's Stationery Office 90p net