

# **The Sampling and Initial Preparation of Sewage and Waterworks' Sludges, Soils, Sediments, Plant Materials and Contaminated Wildlife Prior to Analysis 1986 (Second Edition)**

**Methods for the Examination of Waters and Associated Materials**

# The Sampling and Initial Preparation of Sewage and Waterworks' Sludges, Soils, Sediments, Plant Materials and Contaminated Wild Life Prior to Analysis 1986 (Second Edition)

This booklet replaces the 1977 booklet of similar name which is now withdrawn.

## 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, Plants, Soils, Sediments, and Wild Life 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 given in other parts of this series.

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# 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 properly equipped laboratories. 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 whether for one's self, one's colleagues in the laboratory, outsiders or subsequently for maintenance workers. 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 specifications. 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 laboratory safety. Among such publications are: 'Code of Practice for Chemical Laboratories' and 'Hazards in the Chemical Laboratory' issued by the Royal Society of Chemistry, London; 'Safety in Biological Laboratories' (Editors Hartree and Booth), Biochemical Society Special Publication No 5, The Biochemical Society, London which includes biological hazards; and 'The Prevention of Laboratory Acquired Infection', 'Public Health Laboratory Service Monograph 6', HMSO, London.

Where the Committee have considered that a special unusual hazard exists, attention has been drawn to this in the text so that additional care must be taken beyond that which should be exercised at all times when carrying out analytical procedures. It cannot be too strongly emphasized 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 radio-chemical 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. If calling an ambulance or sending someone to hospital after a known or suspected poisoning, mention this at the time of the call as such cases are usually sent to the nearest specially equipped hospital.

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 correct protective clothing and 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. Hazardous reagents and solutions should always be stored in plain sight and below face level. Attention should also be given to potential vapour and fire risks. If in doubt, it is safer to assume that the hazard may exist and take reasonable precautions, rather than to assume that no hazard exists until proved otherwise.

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# About this Series

This booklet is part of a series intended to provide both recommended methods for the determination of water quality, and in addition, short reviews of the more important analytical techniques of interest to the water and sewage industries. In the past, the Department of the Environment and its predecessors, in collaboration with various learned societies, have 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 determinand. 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 the 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 a committee of the Department of the Environment set up in 1972. Currently it has seven 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.
- 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
- 9.0 Radiochemical methods.

The actual methods and reviews 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.

Whilst an effort is made to prevent errors from occurring in the published text, a few errors have been found in booklets in this series. Correction notes and minor additions to published booklets not warranting a new booklet in this series will be issued periodically as the need arises. Should an error be found affecting the operation of a method, the true sense not being obvious, or an error in the printed text be discovered prior to sale, a separate correction note will be issued for inclusion in that booklet.

L R PITTWELL  
*Secretary*

*31 October 1983*

# Sampling of Sewage and Waterworks' Sludges and Related Solids, Soils, Sediments, Plants and Wild Life for Subsequent Analysis (1986)

## 1 Introduction

An analytical result will be no more accurate than the representativeness of the sample analysed will allow; but intensive sampling programmes may prove expensive and only marginally increase the overall accuracy, as accuracy usually tends to increase only proportionally to the square of the number of samples taken (see Ref 22) while for the investigation of the nature of an anomaly, a single properly selected grab sample sometimes suffices. For some purposes systematic sampling is of more use than random. There are a number of both naturally occurring and industrial processes where, once the pattern of variation has been determined by a very thorough sampling investigation programme, accurate results or adequate quality controls are attainable by relatively few programmed samples (such as for example every six hours or one sample each near the beginning, middle and end of a batch) provided that spot of random checks are made to ensure that the pattern is not changing. The pattern used will be dependent on the earlier findings. The art and science of sampling is neglected at the peril of wasted effort.

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-organisational dispute and a rigid adherence to a precise procedure is not always necessary provided that the analyst is aware how deviations can affect 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'.<sup>(1)</sup>

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. This purpose, the nature of the determinand, and the funds available determine the sampling methods used.

## 2 Notes on Sampling

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

Process control in potable and waste water treatment including:

Addition or withdrawal of solids

Addition or withdrawal of liquid

Environmental aspects of the disposal of sewage and waterworks sludges and related solids.

Monitoring of river, estuarine and marine sediments.

Special investigations into the performance of new equipment and processes.

Investigation of contaminated and uncontaminated land.

2.2 The types of sample which may be required include:

Instantaneous

Continuous:

(a) constant rate sampling

(b) flow related sampling.

A programmed series of discrete sub-samples for preparing a composite sample.

A programmed series of discrete samples analysed individually as part of a mapping project.

2.3 Individual samples should be retained at least until any composite prepared from them has been analysed.

2.4 Where there is a requirement for mass information then it is necessary to measure the associated flow rate or batch size.

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

2.6 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; but see 8.2, 9.10. 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 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.

2.7 Little quantitative information has been given as to the size of samples or number of increments. These criteria are dependent on the variability of the sampled material. See for instance refs 1, 22 and 28.

### **3 Hazards**

#### **3.1 Sampling from Sewers and Works**

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

Physical injury

Bacterial and parasitic 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 2 should be carefully studied and put into effect.

#### **3.2 Sampling Soils and Sediments**

3.2.1 Weather and if applicable tide conditions should be ascertained prior to sampling. In cold weather warmth and shelter should be available if the work is in exposed windy places or there is risk of falling into water.

3.2.2 Quick sands and bogs are a hazard, hence if ground is not known to be firm, it should be tested for firmness before being walked on or driven over. Safety lines and harness should be used when there is any doubt and life jackets should be worn when near or on water. Duckboards, sand trays and similar devices may also be a help. Attention should also be given to unstable slopes and overhanging rock or structures.

3.2.3 Information on ones whereabouts and an approximate timetable should be left with a reliable person prior to starting. Always ensure that this person is notified on your return to avoid placing the rescue services needlessly at risk. In hazardous situations provision of a "safety man", who stays in a known safe place to keep watch on and for the sampler is recommended. Such a person should always summon help prior to going to the assistance of a sampler in distress lest both should get into difficulties and be unable to summon help. A radio or telephone is useful.

3.2.4 If deep holes or trenches are dug, observe normal civil engineering safety precautions (see Ref 3). The top of any large hole made must be fenced in, especially if left unattended, suitable warning lights placed around it and maintained until the hole is filled in.

3.2.5 In addition to the usual risks of being caught by rising flood or tidal water, sinking into soft deposits and the collapse of trenches and slopes,<sup>(3)</sup> it must not be forgotten that exceedingly dangerous compounds ranging from toxic wastes, to inflammable oils, high explosives and white phosphorus have been found both in land and river bed deposits and that flammable gases such as methane and phosphines, alkyl mercury and arsenic and compounds such as chloromethyl sulphonium salts can be synthesized by natural organisms. Proper protective clothing and first aid equipment should be used when necessary.

3.2.6 Some soils are contaminated by pathogens from animal and human excrement and urine. Pathogenic fungal spores are also known. Use of gloves, dust mask and other protection is advised if such a risk is possible.

#### **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 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 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 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.



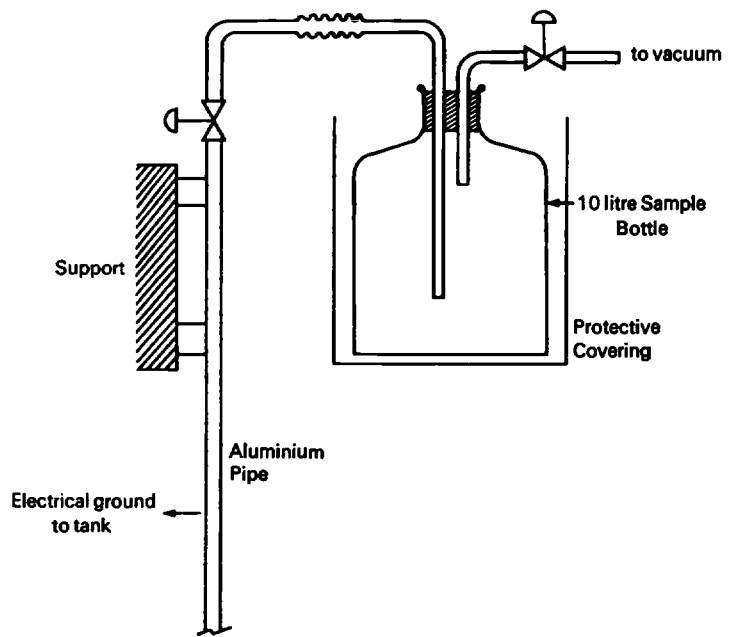


Figure 1 Vacuum sampler

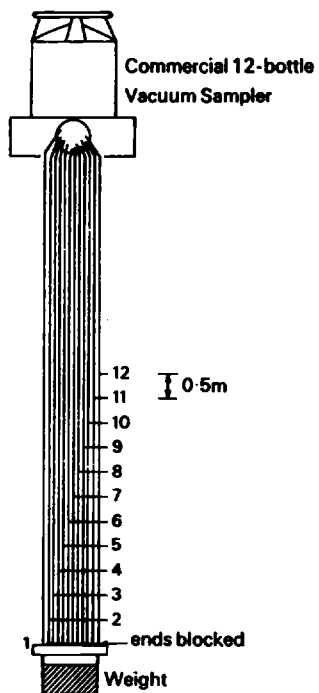


Figure 2 Multiple depth sampler

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.

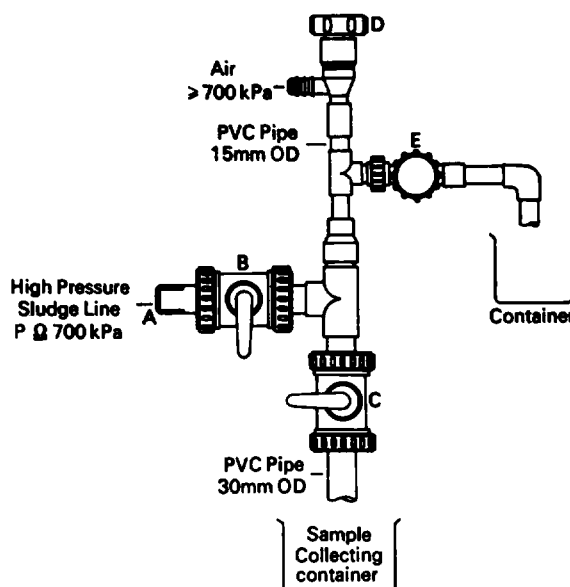


Figure 3 High pressure sampler

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.

See also Sampling of Oils, Fats, Waxes and Tars in Aqueous and Solid Systems 1983 (ref 4) Chapter 2, for additional useful information on sampling in homogeneous flows.

## **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.

See also Sampling of Oils, Fats, Waxes and Tars in Aqueous and Solid Systems 1983 (ref 4) Chapter 2, for additional useful information on sampling in homogeneous flows. For the sampling of scums and mousse see Ref 4 section 1.5.

## **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 areas to the heap, see Section 8 which follows.

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<sup>(5)</sup> should be used for sub-sampling, and for sample size reduction, see also Part B Section 3.4. The procedures given in Section 3.4 of Part B can be adapted to the sampling of heaps and stockpiles.

## **8 Sampling from Belt Conveyors**

### **8.1 Routine Control Sampling**

Mixtures tend to segregate by size and density when agitated. Hence if a representative sample is required of material on a belt conveyor, a complete cross-sectional sample must be taken, including fines. This is best done by a collecting device which temporarily collects the whole flow at a transfer station or conveyor discharge. The device may either collect the whole flow for a brief interval or make a complete traverse into and across the flow, going out on the opposite side, and emptying before returning.

Such devices are most useful for taking representative samples when dried sludges are being loaded for sale. If such a collection device is not possible at a transfer station or discharge an alternative procedure is to stop the conveyor periodically and treat the material on it as a long pile. (Part B Sections 3.4.1.3 and 3.4.1.4).

### **8.2 Random Sampling**

If the material is free of fines and of approximately uniform size, lumps may be hand picked from the belt at random. Alternatively, and especially if particle size is not uniform, the routine procedures may be used but with samples taken either at random times or at random distance intervals along the belt. If the material varies greatly in size care is needed as experience has shown that large lumps often have a different composition from fines.

## **9 Sampling of Soils, Sediments and Land**

### **9.1 Introduction**

There are several reasons why soils, sediments and land are sampled for analysis. The commonest are:

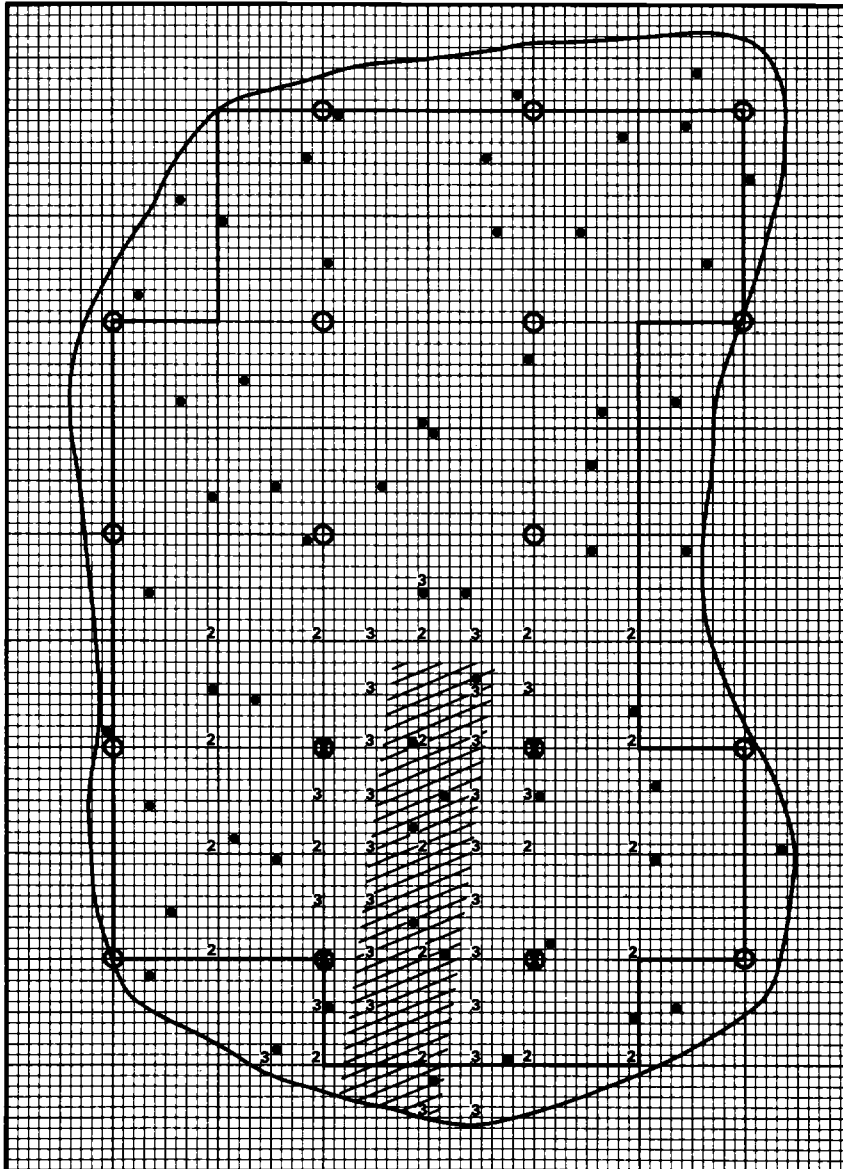
Monitoring the effect of the agricultural use of sludge or fertilizers, which involves analysis both before and after applications have been made.

Assessing whether land has been polluted, and if so how badly and the location of the pollution.

Detection of mineral or other naturally occurring deposits which may affect land or water use, or be of economic interest.

Assessing whether material, at present static, may, if mobilized by a flood or other changed circumstances, pose a threat to areas downstream.

Samples need to be representative. There are several methods of sampling land, hence it is wise to evaluate the possible sampling methods prior to use. In some instances, as when mapping land for subsequent reuse after possible contamination, other systems than the W or X path methods (Fig 5) of regular sampling are preferred, while for many purposes a system of random sampling, which completely covers the area, is used. In many instances careful site inspection with local grab samples is useful and also the cheapest method of making an initial assessment. Whenever possible, dependent on the nature of the material sampled and the determinand, it is advisable to take more sample than necessary and reduce the sample volume later.



**Figure 4**

- i) Example of a field or bank gridded and sampled by random number selection (in this case approximately one sample for each 100 small grids), sample site indicated by dot in small square sampled.
- ii) Example of the same area sampled by a systematic square pattern, primary sites indicated by circles. If the 4 sites marked also by crosses indicated an anomaly, sites indicated by Figure 2s might be sampled. Additional sites, marked 3, would further delineate the main anomaly (shaded).

### Figure 4 Examples

Comparison of Reported Results using different methods of sampling (based on the illustration in Figure 4 and assuming the following analytical pattern).

Random Sampling (sites shown by dots in squares)

Results are listed by site progressing by row from bottom to top and from left to right in Figure 4.

0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 1, 0, 0, 3, 2, 0, 0, 0, 4, 0, 0, 1, 8, 9, 8, 1, 6, 1  
7, 9, 4, 6, 0, 0, 1, 0, 1, 2, 1, 0, 1, 1, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0

Individual analysis of the random sites would have revealed the anomaly. A composite analysis on these sites would give a value of 1.42.

W and X Grid Sampling

In this example a W grid as in Section 9.1 might have given a composite value of 0.7 or 1.2 depending on which way the W was laid out while an X grid might have given 2.6. However, instances are known where W and X grid sampling patterns have missed important anomalous areas.

Systematic sampling (sites shown by circles at grid intersections) would have given an initial pattern of:

-, 0, 0, 0  
0, 0, 1, 0  
0, 0, 3, -  
0, 2, 3, 0  
0, 4, 1, 0 (arranged as in Figure 4)

At the second and third stages a more detailed pattern emerges (here turned sideways, third stage results are given in brackets), as this example shows. Access may restrict ability to sample all desired points.

bottom		0		0		0		0		0		top of	
of figure		1		0		0		0				figure	
	(3)												
	4	(4)	4	(3)	3	(2)	2		1	0		0	
		(7)	(6)	(6)	(6)	(5)	(4)	(3)	(2)				
(6)	7		8		9		8		5	(4)			
(2)	(3)	(4)	(5)	(6)	(6)	(6)	(6)	(7)	(7)				
	1		1		2	(2)	3	(3)	3	3		1	0
	0		0		0		0		0				0
			0				0					0	0

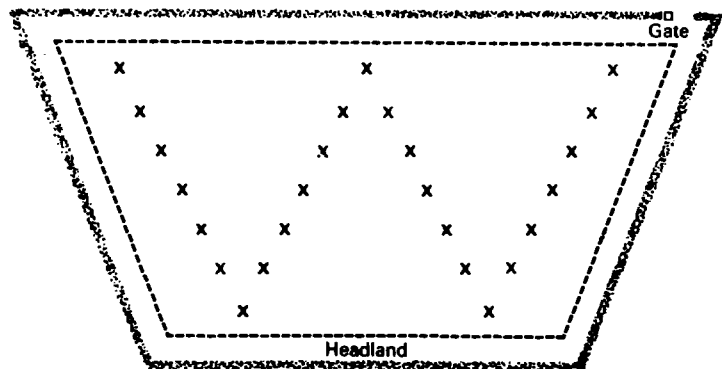


Figure 5 Field sampling pattern, showing sampling path across field. X marks points where samples taken.

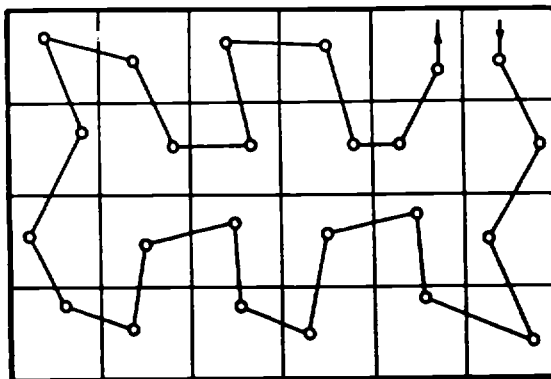


Figure 6 Example of Alternative Sampling Scheme  
(courtesy of the Macauley Institute for Soil Research)

Often, in order to reduce the laboratory work-load when a large number of samples has been taken, a composite sample may be prepared.<sup>(6)</sup>

Section 9.7 recommends depths to which core sample of soil should be taken. Ref 4, Section 1.4 mentions instances where deeper samples are necessary. There can be no single recommended depth limit for soil sampling. How deep a sample is taken depends on the problem; and for assessment of buried contamination, of downward penetration of surface applied materials and for the location of natural concentrations, progressive discrete sampling with depth may be desirable if it is necessary to determine the extent of the anomaly. This booklet contains information on all the above topics and some additional information on hazards and safety. It cannot be stressed too strongly that if the subsequent analysis is concerned with problem solving or with the prevention of problems arising, and not just with determining representative composition, the separate sampling of anomalous material can be important (see Section 12). See also the examples given in the appendix based on Figure 4. For general background reading see books on geochemical exploration such as Ref 28, and analytical principles as for instance Ref 22.

## 9.2 Sampling to a Regular Pattern

Long established practice in some but not all parts of Britain has been to locate sample stations along either a W-shaped path as shown in Figure 5 or an X-shaped path (7). The total area so sampled should not exceed 4 ha. If larger areas are to be sampled, they should be broken down into sub areas each with its own W or X-shaped path. At least 25 sampling stations should be located at regular intervals along the paths. It is advisable to over sample and reduce the sample size later, see Part B Section 4 and Reference 5. Do not sample any obviously unusual areas unless anomalies are being sought (see Section 12). Both path patterns do not give good coverage of a field if it varies markedly in soil composition. The X-pattern tends to over sample the centre, both patterns can leave large areas unsampled. For sampling contaminated land and other projects where marked variation is suspected the procedures which follow are preferred.

## 9.3 Site Examination

A proper site examination, firstly from maps, plans and photographs, and secondly on the site (which can include inspection from the air) may indicate areas where more detailed sampling is necessary compared with the site as a whole. Old maps, plans, photographs, records and even verbal recollections can, when compared with present data, help identify areas of potential interest. Vegetation type and vegetation coloration have often been used to distinguish changes in soil composition. Both the Random and Systematic Procedures which follow may be applied for sampling such limited areas of special interest. Single "grab" samples may be taken at random or taken at an obvious anomaly. The special case of river sediment deposits is dealt with in Section 9.7.

## 9.4 Random Square Sampling

9.4.1 Dependent on the site or volume to be sampled and the amount of sample required (though sample size reduction may subsequently be used if an initially large sample is essential to ensure representativeness), divide the surface to be sampled by a system of regularly spaced grid lines into equal sized squares. There may be irregular shapes at the edges (See Figure 4.)

9.4.2 At random, select one sample point in each square. If there is a risk that personal preference may affect this selection, each point in each square may be chosen by superimposing a similar much smaller grid on each square, each sub-grid intersection being numbered, then draw a number at random and sample at the spot indicated. A similar sub-grid selection procedure is then used to take samples from the irregular border areas, the number of samples so taken being determined by the total such area relative to the area of primary grid square. (See Figure 4.)

9.4.3 The samples are usually composited if only a representative sample is required.

## 9.5 Systematic Square Sampling of Fields, River Beds and the Like

For some purposes, especially when distribution is a criterion in addition to composition, systematic sampling can be preferable to random sampling. The following procedure has proved useful during initial investigations, supplemented by extra selected site sampling later, often using the same procedure but with closer sample points in the areas of interest.

9.5.1 Proceed as in 9.4.1 above.

9.5.2 Take samples at the intersections of each grid line, marking each by its grid reference. In practice problems of access may cause considerable departures from the regular grid. If the ideal spot is impracticable, relocate that sample by a known distance in a suitable known direction. Initial site surveys may also suggest variations in the pattern and initial sampling densities.

9.5.3 Analyse these samples separately.

9.5.4 If extra detail is required in certain areas, repeat the whole process with a smaller grid in these areas. (See Figure 4.)

9.5.5 Sampling in squares is sometimes replaced by sampling in circles. For example sample at one spot, then repeat at points 1 m north, north-east, east, south-east, south, south-west, west and north-west, then at points 2 m from the centre in the same 8 directions. Bulk the 17 samples.

## 9.6 Alternative Random Field Sampling Method

Soil is a very heterogeneous material and large variations can occur in the chemical and physical composition of random samples taken from a small area of apparently uniform soil. Sampling can introduce errors that are quite large in relation to the normal analytical errors; hence the samples must be as representative of the areas under consideration as possible. In practice, it is scarcely feasible to meet all the statistical requirements for random sampling, but an acceptable compromise can be reached by taking a composite sample in accordance with the following recommendations which are based on the method used by the Macaulay Institute for Soil Research (pages 8 and 9 of Ref 8).

9.6.1 Wherever there are likely to be differences in soil type or condition (eg drainage), crop growth or appearance, plant species or varietal distribution in pasture, previous cultivation or treatment, the field should be sub-divided according to these differences and a separate composite sample taken from each area.

9.6.2 Large fields or extensive unenclosed areas, even if apparently uniform, should be divided into smaller sampling units, each preferably of not more than 4 ha size. These should be further subdivided into units of one tenth to one quarter hectare, one sample being taken from each unit.

9.6.3 However small the plot of land being sampled, some 20 sub-samples should be taken in order to provide a representative composite sample.

9.6.4 It is recommended that sub-samples should be taken by traversing the area in a zig-zag manner similar to that indicated in figure 6.

9.6.5 There are many ways of locating the actual points in each sub-sample, ranging from the whim of the collector to systems of taking a variable number of paces sideways and another number of paces forward, varying the numbers for each sample.

9.6.6 Any obvious patches resulting from isolated operations (eg lime dumps, fires, storage pits) should be avoided, as should access areas and end-rigs; but see Section 12.

9.6.7 Sampling should be carried out where possible with a 30–40 mm diameter auger or corer; a spade is less satisfactory. The sub-samples should be of a uniform depth and also of a uniform cross-sectional area. See Section 9.8.

9.6.8 Sampling on arable land should normally be to plough depth, but on established pasture a shallower depth may be adequate to include the main root system of the herbage; but see also Section 9.9.

9.6.9 For specific purposes, a sub-soil sample may be required. This should be obtained by clearing the surface soil from a small area at each sampling point and sub-sampling the under-lying sub-soil as described above.

9.6.10 Sampling immediately following lime or fertilizer application is undesirable; adequate time should be allowed for the incorporation of such materials into the soil.

9.6.11 The total amount of the composite sample should, for mineral soils, be not less than 1 kg. With organic soils, larger quantities of fresh material are required. The objective is to provide at least 500 g of sieved air dry soil.

9.6.12 Where a problem arises only in specific areas, soil samples from such areas should be accompanied by samples from comparable normal sites. Where part of a crop in any field is unsatisfactory, comparative samples should be taken from the affected and from the normal portions, as directly comparable analytical findings can facilitate diagnostic interpretation.

## 9.7 River Sediment Examination

Examination of sediment in the beds of rivers has been used in geological prospecting (9, 10, 11, 12) but can also be used to detect the effects of pollution, both water and air-borne. It has even been used to determine past climatic and biological conditions.

In order to understand how to take best advantage of an examination of banks and beds it is useful to know how such materials are formed and eroded, and the effect of water conditions and other variables on the state of the sample site.

Deposits are usually composed of materials brought down from upstream, though tidal effects, flood surges, eddies and wind can transport material upstream. This is especially true at stream junctions and in estuaries. Rivers which flood over their banks may also build deposits on the flood plain and form levees or naturally raised banks along the outer limits of their channels. Banks and bars may also be deposited in the bed of a stream at places where the speed of flow drops, or in the lee of an obstruction. Erosion is due to increase in water velocity or mechanical abrasion at the outer edge of a bend in a river. Some areas are subject to alternation between deposition and erosion due to variations in water velocity or direction of flow. In such places, knowledge of the composition of deposited material may warn of potential hazards to downstream users and residents when erosion next occurs. Information can also be gained from such an examination both on the conditions upstream, and those which may be met with at the sample site itself under other conditions of flow.



Sometimes mud banks may be formed by precipitation downstream from the point of mixing of two dissimilar waters. This is especially true if a relatively clean water substantially dilutes a heavily mineralised one causing hydrolysis or reduction in complexant concentration. However, on alkaline or salt flats, some muds are stabilised by alkali metal ions, so that any leaching by fresh water may result in peptization of the mud.

In regions where there is seasonal variation in climate or vegetation, sediment layers may exhibit visible annual layering, similar to the growth rings on trees. These are known as varves. If these layers can be sectioned undamaged, time scales can sometimes be worked out by experts, but a reference point in time is necessary and care must be taken against discontinuities in the record due to erosion. Pollen grains, not only from aquatic plants, but also from nearby trees and plants can also accumulate in sediments. When these can be isolated and identified, they may provide an indication of previous climate. Sediments can also be rich in biota which require special sampling methods, dealt with in more detail in other booklets in this series. A careful sampling strategy may be necessary to ensure that sampling for one purpose does not invalidate sampling for other purposes by damaging or destroying the potential sample prior to it being sampled.

By using under water coring devices or grabs (Ref 4) and suitable station locating equipment the above procedures can be extended to the sea and river beds.

### 9.8 Taking Samples by Augers

It is essential that a proper sampling tool be used wherever 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 7. For routine purposes, 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. See also Section 9.6. Standard procedures such as quartering<sup>(15)</sup> should be used for sub-sampling, and sample size reduction, see also Part B Section 3.4. For depth profiles see Section 9.9 and 9.11 which follow.

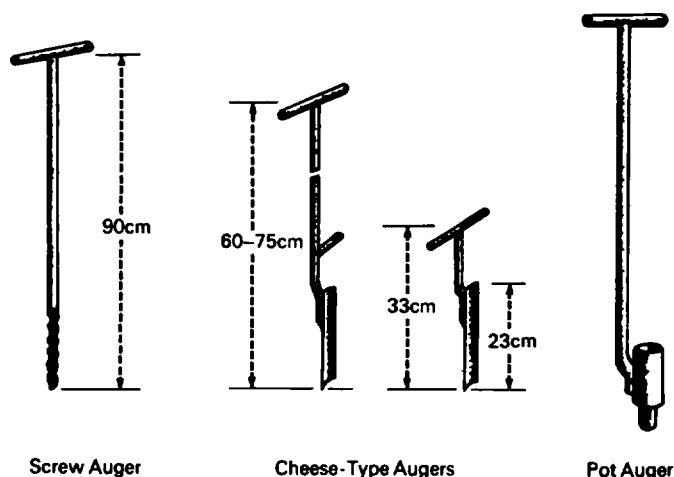


Figure 7 Augers

### 9.9 Depth Profile Sampling of Solids and Semi-Solids that Tend to Flow under Pressure (see Section 3 on Hazards)

9.9.1 It is sometimes necessary to obtain samples of materials which are too solid to pump, but which tend to flow if a shovel or auger is used. This problem becomes acute

when separate samples are required from different depths. Sandy soils, mud, sludges and porous materials with occluded liquids or gases are important examples. The exact solution to this sampling problem is dependent on local conditions; but in principle, a length of a strong inert pipe with a cutting edge at the bottom (or a circular bit) is driven down into the material to be sampled by impact and or rotation, to contain the sample. The top of the tube is closed and the tube together with the sample core is withdrawn. Occasionally, for very runny samples, it may be necessary to devise some way of closing the bottom of the tube by digging down beside it, or of freezing the core prior to withdrawal. The core can then be extruded from the tube onto a clean surface by means of a close fitting plunger and then sub-divided by depth as required. If necessary the sample can be frozen or solidified in other ways prior to extrusion. Provision of an inert lubricant coating inside and or outside the tube sampler may also be necessary. (See for example Reference 13.)

9.9.2 For materials too hard for such methods, rotary core drills of the geological sampling type may be needed. Take specialist advice.

9.9.3 In some river gravels and similar geological strata large stones may occur and be forced downwards by the corer, or even block it completely. Recent work has shown that screw augers (Section 9.7) can also cause contamination of material from lower layers by upper layers when a depth-concentration profile is being made. If the material is in anyway adhesive to the corer, even tube corers can cause such cross contamination. Two techniques have been found satisfactory, depending on conditions.

9.9.3.1 A large diameter core sampler is used. After removal from the hole, the core sampler is laid horizontal, and, if necessary, the core is frozen or otherwise made solid. Then the outer surface which has been in contact with the corer is carefully removed and only the central core is examined as sample.

9.9.3.2 A solid metal or wooden frame of suitable depth, preferably over 30 cm wide, is placed on the ground to be sampled. The material inside the frame is carefully scooped out to the required depth, and the initial sample is put into a labelled sample jar, and the jar sealed. The frame is then pressed downwards to line the hole, any material falling in during this process is carefully removed and discarded. The next sample is taken in the same way. If further samples are needed an additional identical frame is placed on top of the earlier one and pushed downwards as before thus lining the hole. The process is repeated as often as necessary.

## 9.10 Inclusion of Stones and Gravels, Leaves, Wood and other Extraneous Material

Dependent on the reason for the analysis, materials other than soil or sediment may be excluded from the sample considered. What is included, what is excluded and what is segregated for separate examination depends on the reason for the sample being taken and the determinands sought. However, when reporting results it should be made clear what has been excluded and what included, together with at least an approximate assessment of the proportions if the distribution is too variable for more precise measurement.

Stones and gravels are not usually included in samples unless they are likely to be leached or react under the conditions being investigated. If rejected they are removed by suitable screening, sieving (see Section 2.6) or by hand picking. A 2 mm aperture sieve is often used. If they are to be included see Part B Section 3.4.5.

Similarly for some purposes, plant debris such as dead leaves and wood may be excluded or segregated and treated separately because they may require different analytical procedures. The same may also apply to items such as bricks, metal objects, plastic and bottles. The sampler, analyst, and the originator of the project should agree on these matters prior to taking samples.

## 9.11 Sampling by Pedogenetic Horizons (see also ref 14)

First a pit is dug to bedrock or to the desired depth and secured against collapse. The sampling horizons are marked with small pieces of clean wood (eg matchsticks).

Samples are taken from each horizon, usually from the centre, using 2 stainless steel trowels to form an auger; samples are taken in order from bottom to top of the pit. Each sample is placed in a sample bag and sent to the top for immediate labelling before the next sample is taken. This method may also be used for regular interval sampling with depth.

### 9.12 Sampling Sediments

The sampling of benthos has been reviewed by Kajak<sup>(15)</sup>; see also refs 18 and 19 in this series. 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 8.

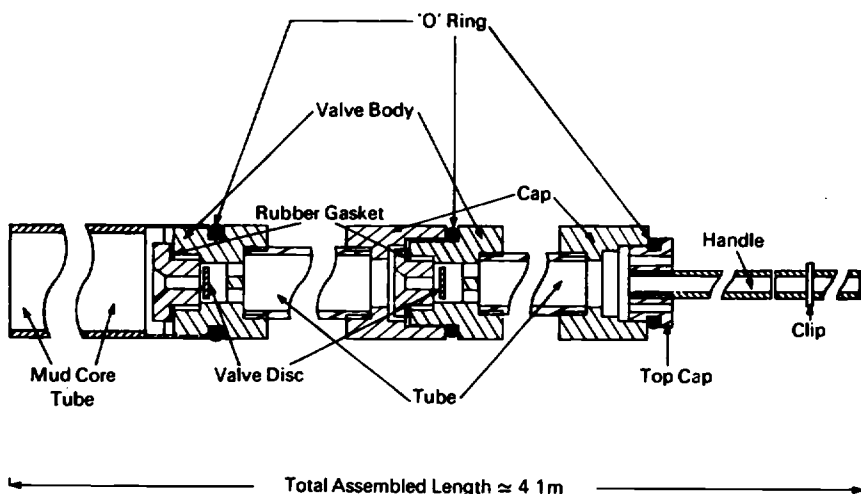


Figure 8 Tube corer

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<sup>(15)</sup>, Brinkhurst<sup>(16)</sup> and for estuarine muds by Holme and McIntyre<sup>(17)</sup>, also Shipek (ref 4 Section 1.1.3 and Figure 8). For loose gravel sediments, samples can be taken using an Ekman or similar grab sampler<sup>(15)</sup> but care is necessary to avoid loss of material if stones jam in the jaws preventing them closing properly (see also Section 9.9).

### 9.13 Sampling Beaches

Sampling beaches for solid components may be carried out as for soils and sediments detailed above. Sampling for oils and tars is already detailed in Ref 4 Section 1.2. Sampling for liquid or water soluble materials should be carried out as for oil, taking care to collect interstitial water, trying to avoid the ingress or loss of this water in the process, unless it is desired to collect such water separately. Techniques for sampling sediments using grabs are not ideal for shingles and gravels which tend to jam open the jaws and cause loss of liquid.

### 9.14 Sampling Rock and Included Ground Water

See Ref 4 Sections 1.4 and 1.3 respectively.

### 9.15 Sampling Benthos

See Refs 18, 19, 20 and 21. Other methods, also in this series, describe sampling methods for free living biota.

## 9.16 Contamination due to Roads

Unless pollution from highways is being assessed, if possible sample at least 100 m away from roads; for stream sediments sample at least 100 m upstream of any bridge. Lead is the chief pollutant along with hydrocarbons and asphalt materials.

## 10 Sampling of Plants

As certain wild plants are protected by the Wildlife and Countryside Act 1981 and consequent legislation, it is recommended that before commencing a particular programme of plant sampling it should be discussed with the appropriate Government Department. 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.

Further details regarding plant sampling procedure may be found in references 8, 23 and 25 (see also Section 12).

## 11 Sampling Wild Life

Ingested substances distribute differently between organs, both by substance and by species. Expert help is needed if specific organ analyses are carried out on dead animals or birds.

Contaminated birds and animals are often difficult to handle as their size precludes the use of most glass containers. Plastic bags, on the other hand, can cause contamination particularly if organic solvents are used to recover smears from the bag. Removal of the oil and similar materials from the bird's feathers may also prove difficult and natural oils from the plumage may cause contamination. Use of large clean metal containers may be more suitable. Unfortunately, experience has shown that the decay organisms and enzymes in body fluids can substantially alter organic materials, especially once putrefaction has set in and so nullify the analysis. Natural skin and body oils and fats from the bird or animal can also complicate the identification. Collection of rotting specimens for subsequent oil removal etc is rarely worthwhile. Likewise care should be taken not to cause rupture of the skin. Animal Welfare experts familiar with this problem recommend that the sample consist of contaminated feathers, hair or fur, clipped off with strong clean unlubricated clippers or scissors or shaved off with a razor without breaking the skin or disturbing the feather or hair roots. Such a sample can be stored in a glass jar. If necessary, uncontaminated feathers, hair or fur may also be sampled separately for identification of the creature's own oils and fats etc in the sample. However, birds and other wildlife are protected under the Wildlife and Countryside Act 1981 and corresponding Scottish and Northern Ireland legislation. The Nature Conservancy Council<sup>(a)</sup> in Britain and the Wildlife Branch of the Department of the Environment in Northern Ireland should be informed if oiled birds or animals are encountered. Expert advice and assistance on the handling of live birds and animals can be obtained from the Royal Society for the Prevention of Cruelty to Animals<sup>(b)</sup> in England and Wales, the Scottish Societies for the Prevention of Cruelty to Animals<sup>(c)</sup> and the Ulster Society for the Prevention of Cruelty to Animals<sup>(c)</sup>. Qualified veterinarians and the Royal Society for the Protection of Birds may also be able to help.

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For key to a, b and c see p. 20 top.

- (a) Nature Conservancy Council, Water and Energy Policy Branch, 20 Belgrave Square, London SW1X 8PY (01-253 3241 or 4150).
- (b) See entries under Royal Society for the Prevention of Cruelty to Animals in the local telephone directory or under Animal Welfare in the Yellow Pages.
- (c) See entries under Animals in either the local telephone directory or the Yellow Pages.

## **12 Anomalous Material**

12.1 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.

12.2 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.

12.3 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.

## **13 Sample Containers and Tools**

Containers need to be chosen with care. See Part B Section 3.5. Tools should be chosen to avoid contamination by sought substances and kept clean and rust free. Avoid high alloy steels if trace metals are to be determined.

## **14 Sample Site Location**

It is essential to locate and refer to sample sites in the field accurately. Normal map or plan reading and standard survey procedures usually suffice, but attention is drawn to the Section on the use of Optical Squares in Ref 25. This instrument can be very useful for locating sites in open locations where conventional measurement is difficult.

### 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.<sup>(24)</sup>

### 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<sup>(23)</sup>.

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 published later in this series<sup>(26)</sup> and homogenise the sample with a blender or similar device.

### 3 Procedure for Soils and Dry Sludge (notes a–f follow Section 4)

#### 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<sup>(a)</sup>. 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 BS 410.77. The special initial preparations required for certain analyses are given in the appropriate analytical method booklets in this series. If the sample is to be used for trace element analysis, it is recommended that nylon mesh sieves be used. For samples to be analysed for organic determinands absorbed by nylon, metal sieves may be used but care should be taken to use a metal not reacting with the determinand in any way<sup>(f)</sup>.

#### 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<sup>(b)(c)</sup>. 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<sup>(d)</sup>. 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<sup>(a)(e)</sup>.

### 3.3 Sieving (see also Sections 3.1 and 3.4.5)

Crumble the air-dried sample in a suitable mortar, or on a polyethylene sheet with a roller, preferably of glass, 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. Clay soils should not be dried completely as they set to a brick hard solid mass. It is best to dry them uniformly until friable and then determine the residual moisture content.<sup>(7, 26)</sup>

Be careful not to grind particles of soft rocks such as chalk, shales and some sandstones if only a soil sample is required.

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 Sample Volume Reduction

In order to obtain a truly representative sample of a large amount of material it is often advisable to take more material than is needed. Several individual samples from different places may then need to be combined and the total volume reduced. If the material being sampled comes in large lumps or a mixture of sizes it will be necessary prior to chemical analysis also to reduce the particle size. This should be done before any variation in composition with particle size becomes significant due to the reduction in sample volume. All equipment mentioned in this section is commercially available.

#### 3.4.1 Long Piles and Quartering

##### *Either (Long Pile)*

3.4.1.1 Mark out the storage area, or bin.

If convenient, the dividers (at stage 3.4.1.3) may be put in place prior to stage 3.4.1.2.

3.4.1.2 Pour the material into the pile or bin in such a way that it is distributed uniformly all over the pile.

3.4.1.3 Insert pairs of braced dividers into the pile at intervals along its length which should make good contact with the base. Remove all material between the pairs of dividers including fines at the bottom. This is the sample. The bracing is necessary to prevent the dividers being forced together during the removal of the sample.

3.4.1.4 After removing the dividers the pile will not be quite as uniform as before. Sufficient sample should if possible be removed at the first sampling for all requirements including checks.

##### *Or Quartering*

3.4.1.5 The sample is poured into a conical mound which has been divided into four using flat dividers larger than the pile. Diametrically opposite quadrants are removed (including fines) and combined. This process is repeated until the sample is sufficiently reduced in volume.

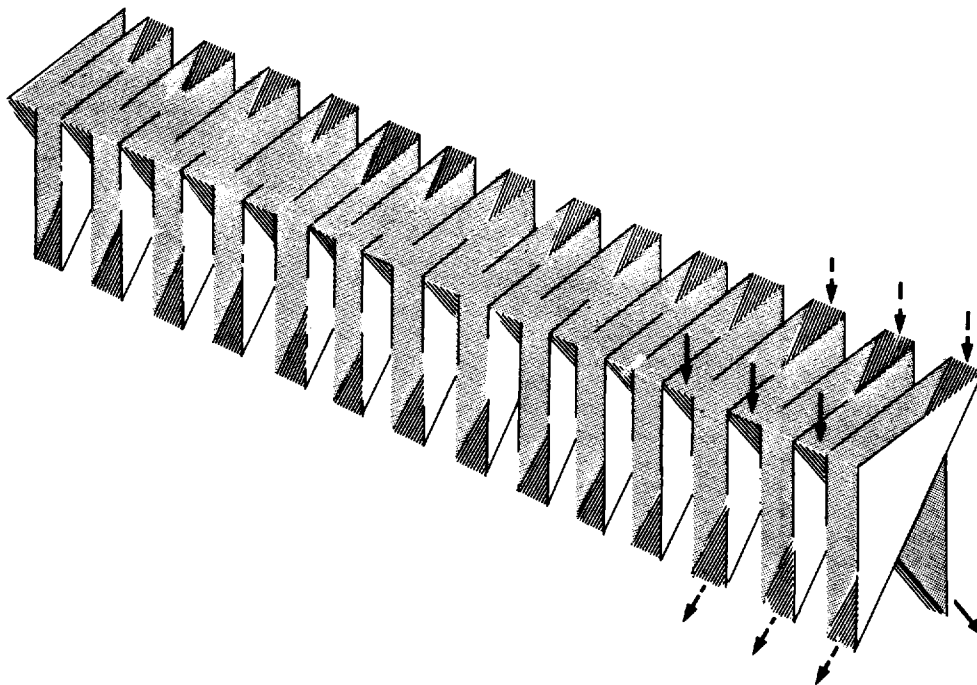
3.4.1.6 In the laboratory, quartering is often performed on a plastic sheet which can then be used for mixing the material and forming a new mound ready for the next reduction.

3.4.1.7 Conveyor belt samples and mechanical dividers have replaced these manual techniques for routine sampling.

#### 3.4.2 Riffing

Unless riffles are made from material not being determined and can be thoroughly cleaned they should not be used.

Riffles consist of a series of equal sized rectangular chutes alternately sloping in opposite directions with no gaps between the chutes. There are an equal number of chutes delivering to right and left. The riffle is mounted above 2 rectangular sample containers so that material tipped into the riffle is divided approximately equally between the 2 containers. A third similar container is used to hold the initial sample. The top of the riffle is fitted with an outward sloping rim so that sample containers can be emptied into the riffle without spillage. The chute apertures should be larger than the largest sample particle. (See Figure 9).



**Figure 9 Explanation of Division by Riffles**

3.4.2.1 Pour the sample into one of the rectangular containers and shake to distribute uniformly.

3.4.2.2 Place one empty container under each set of chutes.

3.4.2.3 Tip the sample into the riffle. If any chute blocks, stop adding sample and clear it at once with a spatula before adding more sample.

3.4.2.4 If further quantity reduction is required, replace one or both lower containers by clean empty containers and tip one of the now filled lower containers back through the riffle. Repeat as necessary.

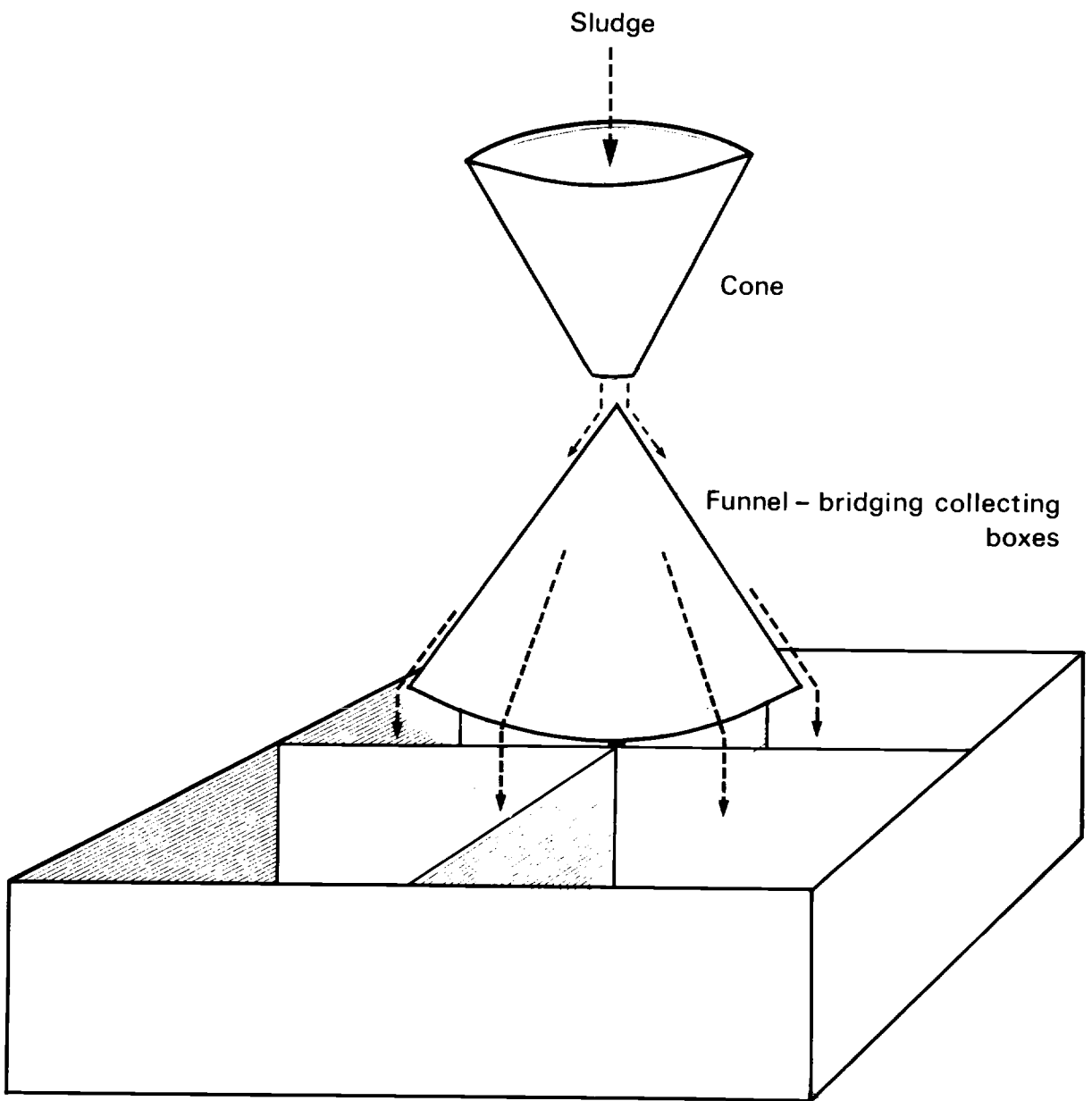
3.4.2.5 Samples should be ground to a relatively fine particle size before considerable reduction in volume has taken places.

### **3.4.3 Cone quartering**

Ensure that all equipment is clean before use.

Place 4 rectangular sample boxes together, with a common corner, and cover all the edge junctions to prevent sample from slipping between the boxes when in use. Mount a large funnel symmetrically over the common corner point. The aperture of the funnel should be greater than twice the diameter of the largest sample particles. Pour the sample through the funnel into the boxes below. Change boxes and repeat if necessary using the contents either of one of the boxes or of two diametrically opposing boxes for further sub-division. See Fig. 10.





**Figure 10 Explanation of Division by Cone Quartering and Similar Sample Dividers**

As with volume reduction using riffles, samples should be finely ground at a point in the process well before risk of an unrepresentative sample can occur.

#### **3.4.4 Rotating Pie Wedge Sampler**

Ensure that all equipment is clean before use.

An even number of containers, shaped like segments of a circle, are mounted on a rotating turntable in a tight circular cluster (like slices of cut circular pie), with all edge junctions covered to prevent material falling between boxes. A funnel is mounted over this device. In some models the funnel is set at about half way from the centre to the outer edge, in other models it is mounted in the centre of the circle, the centre of the boxes being shielded by a small cone (apex up). The turntable is set rotating and the sample is poured through the funnel into the boxes below. Material collecting in alternate boxes is collected, the remainder being rejected. The collected material is blended and sub-divided as before until a satisfactory sample size has been obtained. The procedure is similar to that described in the preceding section except that there are more boxes and that they rotate.

### **3.4.5 Notes on Particle Size Reduction<sup>(27)</sup>** (see also Section 3.3)

There are many different ways of crushing or grinding a sample dependent on the material and the determinand.

If it is necessary to determine variation in composition with particle size, the initial separation by screening etc should be made on the sample as received.

3.4.5.1 If the sample varies greatly in particle size never sub-divide to the level at which differences in particle distribution ratio start to occur spontaneously.

3.4.5.2 If grinding of larger particles is necessary, try to ensure that any material abraded from the grinder or crusher does not significantly affect the analytical result.

3.4.5.3 Never reject unground material. They may cause a change in sample composition, especially if hardness varies with composition. If necessary such uncrushable material may need to be treated separately by solution and aliquotting.

## **3.5 Storage**

Samples should be identified and packed so as to avoid any possibility of loss, contamination or intermixing.

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.

Except for samples to be analysed for organic materials, use double polyethylene bags for soil samples. Place the sample in the inner bag and seal it, place this sample bag and the label inside the outer polyethylene bag and seal that. Cloth bags are unsuitable as they are neither waterproof nor dustproof.

## **3.6 Preparation of Composite Samples**

Individual samples from which a composite sample is prepared must be as homogeneous as possible prior to the start of preparation.

Composites need to represent the whole period or area sampled. If each individual sample represents about an equal amount or area of the total being represented by the composite, weigh out or measure out an equal amount of each sample, pour into a suitable mixer jar or mortar and mix thoroughly. If the individual samples do not equally represent the original, attempt to proportionate each amount weighed out to make the composite to the fraction that sample is of the whole under consideration. See the next section for assessment of mixing efficiency.

## **3.7 Solid and Semi-Solid Sample Blending Homogeneity Tests**

It is often necessary, in order to reduce the amount of laboratory work, to make a composite sample from a number of individual samples. Such a composite must be as representative as possible.

3.7.1 Solids and sludges of varying density can prove extremely difficult to mix thoroughly. Usually, end over end as well as side to side and to and fro action is necessary and rollers or blenders with bottles mounted eccentrically, but symmetrically for balance, on a sloping rotating shaft are often used.

3.7.2 Whatever method is used, it is often difficult to know when satisfactory mixing has been obtained. One method of determining this is to add a small amount of some easily determined substance which does not react with the sample, and preferably does not interfere with the analysis, to one corner of the top of a trial sample, mix and then sample at the top, centre and bottom of the bottle, after suitable intervals of time, until uniform mixing is obtained. If possible, and especially if there is no opportunity for a trial sample, the added substance should be easily observed by colour or fluorescence at the concentration which would be obtained in a uniformly

mixed sample. If there is no suitable non-interfering substance, preliminary tests should be conducted as above to ascertain optimum mixing conditions prior to mixing actual samples without any addition.

#### **4 Procedure for Plant Materials (notes a–f follow this section)**

##### **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 200 g 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<sup>(24)</sup>.

##### **4.4 Drying**

4.4.1 Place at least 200 g of the sample or sub-sample into a clean aluminium tray, freshly lined with greaseproof paper<sup>(b)</sup> and dry for 12 hours in a gentle current of air, which has been preheated to a suitable temperature. (See also 4.7)

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<sup>(24)</sup>.

##### **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<sup>(f)</sup>. The entire dried sample must be ground and collected from the mill.

##### **4.6 Storage**

Plant samples are best collected and stored in strong new paper bags, unless boron is being determined. Many glasses also contain boron. Aluminium screw capped tins may be suitable for the sample for the boron determination.

For the determination of organic materials which may absorb to or diffuse through polyethylene or paper, or for very wet samples, glass jars such as kilner jars are suitable.

Cloth bags are unsatisfactory as they are neither waterproof nor dustproof.

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:

- (a) 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.
- (b) 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.
- (c) 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.
- (d) 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.
- (e) 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.
- (f) The materials of construction of the mill, sieves and trays must not give rise to any contamination of the sample.

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