



ENVIRONMENT AGENCY

The preparation and pre-treatment of potentially contaminated soils  
prior to chemical analysis (2006)

*Methods for the Examination of Waters and Associated Materials*

This document was archived on 12/11/2018.

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## **The preparation and pre-treatment of potentially contaminated soils prior to chemical analysis (2006)**

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

This booklet contains details of the preparation and pre-treatment of soils, potentially contaminated land and similar materials prior to chemical analysis.

This document was archived on 12/11/2018.

Whilst this booklet refers to equipment actually used, this does not endorse these products as being superior to other similar products but serves solely as an illustrative example. Equivalent equipment is available and it should be understood that resulting performance characteristics might differ when other products are used. It is left to users to evaluate these procedures in their own laboratories.

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## About this series

### Introduction

This booklet is part of a series intended to provide authoritative guidance on methods of sampling and analysis for determining the quality of drinking water, ground water, river water and sea water, waste water and effluents as well as sewage sludges, sediments, soil (including contaminated soil) and biota. In addition, short reviews of the most important analytical techniques of interest to the water and sewage industries are included.

### Performance of methods

Ideally, all methods should be fully validated with results from performance tests. These methods should be capable of establishing, within specified or pre-determined and acceptable limits of deviation and detection, whether or not any sample contains concentrations of parameters above those of interest.

For a method to be considered fully evaluated, individual results encompassing at least ten degrees of freedom from at least three laboratories should be reported. The specifications of performance generally relate to maximum tolerable values for total error (random and systematic errors) systematic error (bias) total standard deviation and limit of detection. Often, full evaluation is not possible and only limited performance data may be available. An indication of the status of methods is normally shown at the front of these publications on whether the method has undergone full performance testing.

In addition, good laboratory practice and analytical quality control are essential if satisfactory results are to be achieved.

### Standing Committee of Analysts

The preparation of booklets within the series

"Methods for the Examination of Waters and Materials" and their continuing revision is the responsibility of the Standing Committee of Analysts. This committee was established in 1972 by the Department of the Environment and is now managed by the Environment Agency. At present, there are nine working groups, each responsible for one section or aspect of water quality analysis. They are

- 1 General principles of sampling and accuracy of results
- 2 Microbiological methods
- 3 Empirical and physical methods
- 4 Metals and metalloids
- 5 General non-metallic substances
- 6 Organic impurities
- 7 Biological methods
- 8 Biodegradability and inhibition methods
- 9 Radiochemical methods

The actual methods and reviews are produced by smaller panels of experts in the appropriate field, in co-operation with the working group and main committee. The names of those members principally associated with this booklet are listed at the back of this booklet.

Publication of new or revised booklets will be notified to the technical press. If users wish to receive copies or advance notice of forthcoming publications, or obtain details of the index of methods then contact the Secretary on the Agency's internet web-site ([www.environment-agency.gov.uk/nls](http://www.environment-agency.gov.uk/nls)) or by post.

Every effort is made to avoid errors appearing in the published text. If, however, any are found, please notify the Secretary.

Dr D Westwood  
*Secretary*

April 2004

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### Warning to users

The analytical procedures described in this booklet should only be carried out under the proper supervision of competent, trained analysts in properly equipped laboratories.

All possible safety precautions should be followed and appropriate regulatory requirements complied with. This should include compliance with the Health and Safety at Work etc Act 1974 and all regulations made under the Act, and the Control of Substances Hazardous to Health Regulations 2002 (SI 2002/2677). Where particular or exceptional hazards exist in carrying out the procedures described in this booklet, then specific attention is noted.

Numerous publications are available giving practical details on first aid and laboratory safety. These should be consulted and be readily accessible to all analysts. Amongst such publications are; "Safe Practices in Chemical Laboratories" and "Hazards in the Chemical Laboratory", 1992, produced by the Royal Society of Chemistry; "Guidelines for Microbiological Safety", 1986, Portland Press, Colchester, produced by Member Societies of the Microbiological Consultative Committee; and "Safety Precautions, Notes for Guidance" produced by the Public Health Laboratory Service. Another useful publication is "Good Laboratory Practice" produced by the Department of Health.

## Glossary

Air-dried sample	A sample that has undergone a simple air-drying process at less than 30 °C. See also assisted dried sample.
Air-dried solids (%)	Value derived from air-dried sample.
As-received or wet-weight sample	The sample as it is removed from a location within a site and then submitted to the laboratory in a suitable container.
Assisted-dried sample	A sample that has undergone a specified accelerated drying process. This may involve oven-assisted drying at a specified temperature, freeze-drying or some other process.
Assisted-dried solids (%)	Value derived from assisted-dried sample.
Contaminated land	Any solid non-agricultural sample (irrespective of the water content) taken from the ground including made-ground and sediments.
Determinand	Refers to the parameter or analyte being determined.
Dried weight at 105 °C	A sub-sample that is dried at 105 ± 5 °C for a minimum of two hours. This procedure is carried out to enable results to be expressed on a dry weight basis.
PAHs	Polycyclic aromatic hydrocarbons.
PCBs	Polychlorinated biphenyls.
Sample	A quantity of material submitted to the laboratory. This comprises the “as-received” or wet-weight sample.
Soils	A generic term that includes contaminated land and similar materials, but within the context of this booklet, does not refer to agricultural soils.
Sub-sample	A representative portion of the sample used in the analytical determination.
VOCs	Volatile organic compounds.

# The preparation and pre-treatment of potentially contaminated soils prior to chemical analysis

## 1 Introduction

The preparation and pre-treatment of soils prior to chemical analysis is a major consideration in the generation of reliable data. Notwithstanding the quality of the subsequent analysis, if an inappropriate sample has been taken and/or an associated sub-sample has been incorrectly prepared, then the results generated from that sub-sample will be of little value. Furthermore, it will be impossible to offer decisive or definitive interpretations to these results.

The aim of this booklet is to provide advice and guidance in order for a consistent approach to be adopted on the preparation and pre-treatment of contaminated soil samples. Ideally, this should ensure that prepared and treated samples are obtained that are fit-for-purpose, representative and/or homogenised. In addition, the use of correct pre-treatment and preparation procedures should also ensure that sub-samples are analysed without significant losses occurring to the determinand being analysed. The guidance in this document deals only with the preparation and pre-treatment of samples after they have been submitted to the laboratory. Advice and guidance on sampling strategies involving the manner in which samples are taken on-site, their respective numbers and locations is not described in this document and details of this may be found elsewhere<sup>(1 - 7)</sup>. Furthermore, details highlighting the problems that may be encountered on whether the sample taken is representative of the bulk of the material being sampled on-site are not included in this booklet.

### 1.1 Pre-treatment procedures

There are several basic approaches to the preparation of suitable sub-samples prior to analysis and additional guidance can be found elsewhere<sup>(8 - 10)</sup>. Each approach depends upon the stability and volatility of the determinands being analysed and for any given sample, it may be necessary to utilise or adapt, to suit individual circumstances, one or more of the approaches described. When samples are submitted to the laboratory, these may comprise either a single individual sample (on which all analytical determinations are to be carried out) or a series or number of separate samples (thus enabling specific determinations to be undertaken on individual samples within the series). For example, different samples for metal determinations and the analysis of volatile organic compounds (VOCs) may be submitted. For specific determinations, such as VOCs, specific sample containers (for example sealed vials) are required. The number of replicate samples for a given sample will ultimately depend upon individual circumstances and the determinands requiring analysis. Due to the extremely variable composition of soil samples, the approach and procedures used will depend on specific individual cases and it is up to the laboratory to demonstrate the procedures used are appropriate to the investigation. Appendix 1 lists examples of relative determinand stabilities and/or volatilities that will need to be considered before undertaking specific procedures.

#### 1.1.1 Unstable determinands

Unstable determinands are affected by sample preparation and pre-treatment procedures (such as drying and subsequent processes involving crushing, grinding and/or sieving etc) commonly used in laboratories. For samples requiring the analysis of these and similar determinands, it is impossible to prepare and pre-treat the samples using techniques

involving these processes without significant loss of determinands occurring. Hence, determinands that are unstable or easily degradable should be analysed on an as received or wet-weight sample.

### **1.1.2 Volatile determinands**

The analysis of relatively volatile organic determinands, requires specialised sampling equipment. For example, sealed vials or containers. In addition, specialised devices for the collection, transportation and handling of soil samples requiring VOC analysis may need to be used. These devices reduce VOC losses by minimising sample exposure to the atmosphere and maintain sample integrity from sample collection to delivery to the laboratory. In these cases, it may be appropriate to analyse the whole of the contents of the container submitted to the laboratory, rather than a sub-sample. These determinands are affected by sample preparation and pre-treatment procedures commonly used in laboratories and unless special precautions are taken, significant loss of VOCs will occur prior to analysis. Hence, volatile determinands should be analysed on an as received or wet-weight sample.

### **1.1.3 Stable and non-volatile determinands**

Determinands regarded as being stable and non-volatile are not subject (to any significant extent) to processes such as volatilisation or chemical or microbiological degradation, or undergo change when subjected to elevated drying temperatures and subsequent processes involving crushing, grinding and/or sieving etc. Hence, determinands that are stable and non-volatile may be analysed on a dried, crushed, ground and sieved homogenised sample.

### **1.1.4 Preserved determinands**

There are some determinands (for example, phenolic compounds) for which on-site preservation should be undertaken. Where the preservation or stabilisation procedures have been carried out on-site when the sample was taken, the analyst needs to be aware of the preserving or stabilising agents used. These agents may cause interference effects with the determinand being analysed or any pre-treatment and preparation procedure used. Whether all or part of the sample should be analysed would need to be considered.

## **1.2 Drying procedures**

For some determinands, the approach to be adopted is not easily defined and the drying conditions (including temperature) are often critical. The analyst should, therefore, demonstrate that the procedures used do not lead to a significant loss of the determinand of interest. When referring to dried samples (however they are prepared) it is essential to report the particular drying process and drying temperature used.

For the majority of samples, the difference in the weight loss (due to the loss of moisture) between an air-dried sample and a dried sample may not be significant, see Appendix 2. This appendix highlights the results obtained under different drying temperatures for a variety of samples. The differences observed will depend on the conditions of the drying process, such as the temperature and period of drying, as well as the constituents of the sample. Air-dried samples should be obtained at ambient temperatures not exceeding 30 °C whilst dried samples are prepared, for example, using oven-assisted facilities at elevated temperatures. Depending upon the matrix of the sample (soil type, particle size, moisture and volatile contaminant content, etc) the time taken to dry the sample will vary. The procedures employed

are, usually, undertaken for a specified period of time, or until an effective equilibrium state has been reached, or until a constant weight has been achieved. This difference in the loss of moisture (and possibly other volatile matter content) will, however, not be the same for all samples, particularly for those samples containing high levels of VOCs and other hydrocarbon compounds etc. Hence, when results are recorded there should be an awareness of how the results are to be reported. For example, whether the results are to be reported on an air-dried basis, an assisted-dried basis (i.e. whatever drying process or temperature has been used) or converted to a dry-weight basis (usually based on a drying procedure at 105 °C) then these details should be stated.

### **1.3 Extraneous material**

For many of the samples received within a laboratory the sample is considered representative of the area on site where the sample has been taken and therefore the whole sample should be processed. In some instances, it may be necessary to remove large particles and material not amenable to preparation or analysis. These extraneous constituents can include, for example,

- glass, metallic and plastic fragments,
- plant and fibrous material,
- large stones, etc.

If these constituents are not amenable to any sample preparation or pre-treatment process and are not considered to contain significant amounts of the determinands of interest, consideration should be given to removing them from the sample<sup>(11)</sup>. The removal of any material from the sample submitted to the laboratory prior to commencement of analysis should, therefore, be recorded and fully documented. Such details should include the nature and quantity of the material removed, as this may affect the interpretation of the results reported. Whatever sample preparation and pre-treatment procedures are used, including crushing, grinding and/or sieving etc. all details should be recorded. In addition, it should be noted whether the analysis undertaken relates to all or constituent parts of the sample, and whether any material removed also separately undergoes the same analysis as the conventional components of the sample. All procedures used should be fully documented and details provided with the results.

### **1.4 Sub-sampling**

#### **1.4.1 Sub-sampling for unstable determinands**

For the analysis of unstable determinands, it is necessary to obtain a sub-sample that is, ideally, representative of the as received or wet-weight sample submitted to the laboratory. This may not, normally, involve the removal of any material from the sample. Considering the complex nature of samples submitted to laboratories, the process of obtaining a sub-sample is not an easy operation to carry out. Careful judgement is required in order to ensure the portion taken for analysis is as representative (and homogeneous) as possible of the whole sample. Due to the large degree of uncertainty associated with the taking of a sub-sample under these conditions, greater confidence in this procedure (and the subsequent results generated) can be obtained by repeating the process and subsequent analysis. The greater the number of replicate sub-samples taken and analyses undertaken, the greater the confidence in the interpretation of the results reported.

### **1.4.2 Sub-sampling for volatile determinands**

Extreme care needs to be exercised when samples of contaminated soil are sub-sampled for the analysis of volatile determinands, and every effort should be made to ensure no losses occur. Wherever possible, the sample should be collected in an appropriate container that reduces contact of the sample with the atmosphere and that the whole of the sample submitted is analysed. To eliminate loss of volatile determinands, separate samples should be taken for each analysis and no sub-sampling undertaken.

### **1.4.3 Sub-sampling for stable and non-volatile determinands**

For the analysis of stable and non-volatile determinands, the approach to be adopted involves the preparation of homogeneous sub-samples, which may or may not involve removal of any material from the original sample submitted. The weight of the whole of the sample submitted to the laboratory should be recorded, and if any material is removed or rejected prior to drying then the corresponding amounts should also be weighed and details recorded. The sample should then be dried by a suitable process involving, for example, air-drying, drying at a suitable elevated temperature, freeze-drying or by some other process. The weight of the whole of the remaining dried sample should then be recorded. Details, including the amount, of any material removed after drying should also be recorded prior to crushing, grinding and/or sieving etc, as appropriate, including any other sample preparation or pre-treatment process. This dried and treated sample should be mixed thoroughly and transferred to a suitable container, which should then be tightly sealed. A portion of this prepared sample should then be used, as required, for the analysis of stable and non-volatile determinands.

### **1.5 Quantity of sample**

In order to ensure all relevant analyses are undertaken, including repeat analyses where necessary, it is essential that a sufficient quantity of sample be submitted to the laboratory. The actual amount submitted depends upon a number of factors, including the number and type of analyses required, and whether a single bulk sample is provided (for all determinations) or separate individual containers are submitted for specific analytical determinations. There should be good communication between the laboratory and the sample provider to ensure all requirements are identified and addressed. It may be much cheaper and more efficient to submit a greater quantity of sample to the laboratory than is actually required, rather than suffer the risk of having to repeat the sampling process. As the weight of sample submitted to the laboratory is reduced then difficulties may arise if the sample no longer remains representative of the site sampled. Further difficulties may be introduced in the production of suitable sub-samples and whether these remain truly representative of the sample. This will be exacerbated, for example, where high levels of determinands need to be determined and the amount of sub-sample taken for analysis becomes ever smaller.

### **1.6 Reporting of samples**

The laboratory report of the results should contain details of, or make reference to, the procedures used to obtain sub-samples, and any unusual aspects of the sample. Information on, for example, a description of the sample and major sample components, plus constituents should also be included. In addition, details of the sample preparation and pre-treatment procedures together with analytical methodologies should be available if requested.

## 2 Scope

This document highlights some of the actions that should be considered when contaminated land, mud, silt, sediment, and slurries are prepared and pre-treated prior to chemical analysis. Appendix 3 shows a schematic flow chart of the main procedures to be considered.

## 3 Hazards

Care should be taken when handling potentially contaminated samples. Skin contact with the sample should be avoided, and appropriate provision should be made for drying, crushing, grinding and/or sieving, and other processes involving the potential discharge of toxic fumes, dusts or fibres etc from the sample. Suitable personal protective equipment (for example gloves, masks and eye protection) should be used when handling samples. Samples known to contain or suspected of containing, contaminants such as asbestos should not be dried, crushed, ground or sieved etc unless specialist advice and equipment are available. Before sub-sampling is undertaken, samples should be inspected for the presence of visible/obvious hazards. Odorous samples should alert staff to the presence of potentially toxic volatile substances and hence the potential risks arising from their presence. However, care should be taken if further investigations are undertaken.

Samples may be hazardous simply by the presence of such contaminants as high levels of PAHs, cyanide, mercury, asbestos, fungal spores, microbial pathogens, explosives or radionuclides etc. In such cases, appropriate safety precautions should be considered and taken into account prior to any preparative or pre-treatment process. All samples should, therefore, be viewed as potentially hazardous. The presence of contaminants need not necessarily be confined to the requested suite of analyses, as other contaminants (not requested for analysis) may also be present.

## 4 Reagents

**4.1 Cleaning reagents.** Cleaning reagents, such as sand and solvents, should not contain significant amounts of the determinands of interest. Cleaning reagents are used for cleaning equipment such as crushers, grinders, sieves and glassware etc. These reagents may be used for audit control purposes to ensure significant cross-contamination does not arise between the processing of highly contaminated samples and samples which are not highly contaminated.

## 5 Apparatus

**5.1 Drying facility.** For air-drying purposes, this may be a clean designated area open to the atmosphere at ambient temperatures. The temperature should not be allowed to exceed 30 °C. For assisted-drying purposes, this may be an oven, or other facility, with for example thermostatic control, forced ventilation etc, capable of maintaining a constant temperature to within  $\pm 3$  °C. Other facilities, including freeze-drying, may also be applicable. The temperature used should not have any adverse effect on the determinands being analysed. Any freeze-drying procedure should also not adversely effect the determinands being analysed. An additional oven, maintained at  $105 \pm 5$  °C, may be required to enable results to be expressed on a dry weight basis.

**5.2 Crusher, grinder etc.** This may be a mechanical or manual device, with or without the facility for sieving, and should be made of material that does not significantly contaminate the

samples with the determinands under investigation.

**5.3 Balance.** Capable of weighing to an appropriate tolerance.

**5.4 Sieve.** This should be of an appropriate size and material so as to avoid contaminating the sample being analysed. The size of the sieve may depend upon the analytical technique used. For example, X-ray fluorescence techniques may require a smaller particle size than inductively coupled plasma techniques. A sieve capable of producing a particle size of less than 250 µm has been found useful for most matrices and analytical techniques. It may not be necessary to specifically sieve every sample, provided the crushing and grinding technique has been validated for different sample matrices and demonstrates that a sample of the required size is produced.

**5.5 Riffle box.** A device used to facilitate homogenisation of dried samples only, the use of which, produces sample material similar to that produced by the cone and quartering technique (used on as received or wet-weight samples, or indeed dried samples). When used repeatedly, a homogeneous sample is produced.

## **6 Sample collection and storage**

For the analysis of potentially contaminated land samples, it is essential that the sample submitted to the laboratory is representative of the material or location under investigation<sup>(1-7)</sup>. In cases where a single sample is submitted to the laboratory and this sample is to be used for all analyses that need to be undertaken, then as well as sufficient sample being provided only appropriate analyses should be undertaken. For most purposes 1 kg should be adequate unless, for example leachability tests may be required.

Suitable equipment and containers should be available for taking and storing samples. Specific guidance on details of sampling, sample containers and storage conditions are usually given in the individual methods describing the analysis of specific determinands. Samples should be stored under appropriate conditions throughout the whole process including storage prior to and during transportation to the laboratory and whilst in the laboratory.

## **7 Preparation and pre-treatment procedures**

A schematic flow chart of these procedures is shown in Appendix 3.

### **7.1 Sample description**

A brief description of the sample should be recorded, for example giving details of the sample submitted and its quantity, soil type, colour, particle size and possibly odour. If the odour is to be described, this assessment should be undertaken with care, and appropriate health and safety requirements satisfied. The presence of vegetation, extraneous matter and other noticeable or relevant features may also need to be reported. This description should be recorded before any preparative or pre-treatment processes are carried out.

### **7.2 Homogenisation**

Depending upon the determinand being analysed, the process of homogenisation may need to be carried out before any drying procedure is considered (i.e. for unstable or volatile

parameters) or after the drying stage (i.e. for stable and non-volatile compounds). Ideally, the whole sample should be homogenised. However, it is recognised that this may not be practicable or even appropriate. The sample should be homogenised using the most appropriate means available and a portion should be taken at this stage, and correctly stored for possible future reference purposes or analysis.

For contaminated land samples, it is important that the sub-sample analysed is, ideally, homogeneous and representative of the whole sample submitted to the laboratory. For sub-samples analysed on an as received or wet-weight basis this may be problematical, due to the heterogeneous nature of the sample submitted to the laboratory, and the difficulties of obtaining a representative sub-sample of the sample. The uncertainty associated with analyses performed on as received or wet-weight samples is very high and in order to reduce this uncertainty, greater confidence will be obtained if replicate sub-samples are taken for analysis. For dried sub-samples where stable or non-volatile determinands need to be analysed, this should not be a cause for concern, as homogeneous samples can easily be prepared.

The removal of extraneous constituents (namely, large stones, pieces of plastic or other debris etc) prior to drying and sub-sampling may adversely affect the resulting analysis by significantly reducing the contamination under investigation, and hence, the determinands of interest. Thus it is of paramount importance to record details of any actions taken, and whether any material removed undergoes analysis.

Certain determinands need to be analysed on an as received or wet-weight basis in order to prevent significant loss or degradation of unstable (or volatile) compounds that may occur during the preparation or pre-treatment processes, such as drying, crushing, grinding and/or sieving etc. It is, therefore, essential to thoroughly mix the sample to enable a representative sub-sample to be obtained for analysis. Even during these procedures, care should be taken to minimise loss of unstable, easily degradable substances or volatile compounds from the sample. In addition, where the sample contains for example, large particles greater than 10 mm, it will be necessary to break down these particles to a more manageable size, whilst at the same time minimising loss of determinands of interest. If these large particles (for example, large stones or rock fragments) are considered to be inert, then they may be removed and details recorded. It may be that mechanically breaking down these particles will generate significant quantities of heat and lead to consequential losses of unstable or volatile determinands.

Examples of mixing procedures<sup>(12)</sup> include cone and quartering, kneading (for example with clay soils) and the use of a paddle mixer or other electric device. The cone and quartering technique involves placing the sample on a flat surface and repeatedly moving the outer edges of the sample (with a suitable device, for example, a large spatula or scoop) into the shape of a pyramid or cone. The cone should then be flattened and the sample divided into four segments. The two opposing quarters should be mixed together. These two portions should then be combined and thoroughly mixed together. The whole procedure should then be repeated several times. At all times, care should be taken to minimise as much as possible the loss of volatile or unstable compounds from the sample.

After homogenisation, a sufficient portion of the well-mixed sample should be transferred to a suitable container and stored under appropriate conditions. This portion should be retained for future reference and further analyses, if required. The remaining sample should be placed in an airtight container, sealed and used as required.

As an aid to the production of a homogeneous sample, a riffle box may be used on the dried sample. The dried sample should be passed through the riffle box, and the separated fractions of the sample collected and mixed together. The whole process should then be repeated until the sample is homogeneous. This technique is equivalent to that of the cone and quartering process.

### **7.3 Sub-samples for volatile or unstable determinands**

The analysis of volatile and/or unstable determinands should be undertaken on an as received or wet weight basis. Either the whole of the sample or, as far as possible, a representative sub-sample (see section 7.2) should be taken for analysis. If, for any reason, for example repeat analysis, the amount of sample or sub-sample taken for analysis needs to be reduced, then care should be taken to ensure the amount remains representative of the sample. Alternatively, replicate samples may be considered and collected at the time of sampling to pre-empt this situation. For example, this could occur in cases where repeat analyses need to be carried out as a result of detecting the presence of high levels of determinands. In order to increase confidence in the reported results, it may be necessary that extra sub-samples need to be taken and analysed.

#### **7.3.1 Chemical Drying**

When as received or wet-weight contaminated soils need to be solvent extracted (for example in the analysis of certain organic compounds) the addition of a desiccating agent, such as anhydrous sodium sulphate, will improve the extraction efficiency. However, it is important to add sufficient desiccant to the soil to achieve a free-flowing mixture. Usually, depending upon the moisture content of the contaminated soil, equal volumes of desiccant and soil are required. This technique is normally described in the relevant section of the method of analysis for the particular determinand. The benefits of using a technique such as this should be balanced against potential losses (in terms of the extraction) of the determinand of interest when carrying out this procedure.

### **7.4 Sub-samples for stable and non-volatile determinands**

#### **7.4.1 Drying**

For the analysis of stable and non-volatile compounds, a portion (at least 100 g) of the well-mixed sample (see section 7.2) should be transferred to a clean non-absorbent tray. The tray material should not adversely affect the sample and vice versa. The sample should then be spread or distributed into a thin layer, for example not greater than 15 mm, and the thinner the sample is spread the quicker the drying process is completed. If the determination of the loss on drying is required, then the weight of sample transferred to the tray should be recorded. The tray and its contents should then be placed in a suitable drying facility (5.1) at a specified temperature until the sample is dried (for example, for 24 hours). Depending upon individual sample matrices, this period of time may need to be increased or decreased. For example, clay soils or soils containing high levels of organic matter may require longer drying times. When the sample has been dried, the weight of the dried material should be recorded in order to determine the loss in weight due to the drying procedure.

For results to be expressed on a dry weight basis, a second sub-sample should be dried at 105 °C to enable a suitable factor to be calculated.

## 7.4.2 Crushing, grinding, sieving etc

The dried material can now be crushed, ground and/or sieved to produce a finely powdered homogenised material of the requisite particle size (typically less than 250 µm) - sub-samples of which should be taken for analysis of specifically identified determinands. Care should be taken that excessive heat should not be generated by the grinding equipment that may lead to losses of volatile or unstable determinands. It is important that all the crushed and ground material should pass through the sieve, although not all samples require sieving (5.4). Each procedure may be undertaken in a suitable mechanical apparatus or carried out manually. In addition, the procedures may be undertaken either consecutively or as a combined operation. Details of any extraneous constituents or conventional components not included in the finally prepared homogenised material should be recorded, as should the size of any sieve used. It may be necessary to determine whether the processes of crushing, grinding and/or sieving etc contribute to any significant loss of determinands being analysed or whether any material removed, i.e. not included in the finally prepared material, undergoes analysis. Periodic checks should be made to ensure that there is no significant contamination of the sample resulting from the crushing, grinding and sieving processes.

If the analysis of more than one determinand is required, then sufficient material should be prepared to allow all analyses, including repeat analyses, to be performed. The crushed, ground and/or sieved material should be thoroughly mixed then transferred to a suitably labelled container and tightly sealed.

In order to reduce cross contamination, any equipment used in the crushing, grinding and/or sieving processes should be thoroughly cleaned before being used again on other samples. After the equipment has been used on a particular sample known or suspected of containing a high level of a determinand, it should be thoroughly cleaned. The cleanliness of the equipment should then be checked using uncontaminated sand, which is treated in the same manner as the sample. The sand should then be analysed and the resulting analysis should confirm the absence of significant quantities of determinand in the sand. This should minimise any "carry-over" effects.

## 8 Report

When results are reported, details should be provided to ensure the result can be related to all or constituent parts of the sample submitted to the laboratory. These details should include, for example, the following information<sup>(13, 14)</sup>.

- a brief description of the sample,
- brief descriptions of all procedures used to prepare the sample, and if the analysis is performed on a dried or as received basis,
- details (including amounts and descriptions) of any portion of the sample or sub-sample removed prior to analysis,
- details of whether any portion removed undergoes analysis,
- details of the drying conditions used to prepare the dried material,

- the percentage loss in weight resulting from the drying process,
- any other relevant information.

## 9 References

- 1 Standing Committee of Analysts, The sampling and initial preparation of sewage and waterworks' sludges, soils, sediments, plant materials and contaminated wild life prior to analysis 1986 (Second edition), *Methods for the Examination of Waters and Associated Materials*, ISBN 0117518859, in this series.
- 2 BS 2000:10175 - Code of practice for investigation of contaminated sites.
- 3 ISO/FDIS 2002:10381-1 – Soil Quality – Sampling - Part 1 – Guidance on the design of sampling programmes. This document is currently under revision.
- 4 ISO/FDIS 2005:10381-5 – Soil Quality – Sampling - Part 5 – Guidance on the procedure for the investigation of urban and industrial sites with regard to soil contamination. This document is currently under revision.
- 5 Guidance for obtaining representative laboratory analytical sub-samples from particulate laboratory samples, R W Gerlach and J M Nocerino, United States Environmental Protection Agency, GS-35F-4863G (Task Order Number 9T1Z006TMA).
- 6 Contaminated Land Research Report: Sampling strategies for contaminated land, 1994, Department of the Environment, CLR Report number 4.
- 7 The statistical basis for spatial sampling of contaminated land, C C Ferguson, Ground Engineering, 1992, **25** (5), pp34-38.
- 8 ISO/DIS 2004:11464 – Soil quality - Pre-treatment of samples for physico-chemical analysis. This document is currently under revision.
- 9 ISO FDIS 2003:14507 – Soil quality - Pre-treatment of samples for determination of organic contaminants. This document is currently under revision.
- 10 ISO/FDIS 2004:16720 – Soil quality - Pre-treatment of samples by freeze drying for subsequent analysis. This document is currently under revision.
- 11 The Analysis of Agricultural Materials, Reference book 427, Ministry of Agriculture, Fisheries and Food, A manual of the analytical methods used by the Agricultural Development and Advisory Service, 1986, ISBN 0112403522.
- 12 Initial sample preparation, M Allen, in Chemical Analysis of Contaminated Land, (Eds) K C Thompson and C P Nathanail Blackwell, 2003, ISBN 1841273341.
- 13 MCERTS Performance Standard for Laboratories Undertaking Chemical Testing of Soil, Environment Agency, February 2003, Version 2. (Version 3 in preparation).
- 14 ISO/IEC 17025:2005 General requirements for the competence of testing and calibration laboratories.

## Appendix 1      Relative stabilities and/or volatilities

These lists provide a guide to the relative stability/volatility for certain common determinands. Analysts should ensure that the preparation and pre-treatment procedures, and the storage conditions used in their laboratories are fit for purpose and do not lead to significant losses of the determinands of interest.

### **Unstable and/or volatile determinands**

organic/elemental mercury  
cyanide and similar compounds  
hexavalent chromium  
volatile organic compounds  
explosives  
phenols  
sulphides  
organo-lead compounds  
hydrocarbon compounds below C<sub>11</sub>  
ammonia

### **Borderline determinands**

C<sub>11</sub> - C<sub>19</sub> hydrocarbon compounds  
semi-volatile compounds  
inorganic mercury  
low molecular weight PAHs and PCBs

### **Stable and non-volatile determinands**

metals, except mercury  
chloride  
sulphate  
water soluble boron  
organo-tin compounds  
elemental sulphur  
higher molecular weight PAHs and PCBs  
hydrocarbon compounds above C<sub>19</sub>  
dioxins  
furans

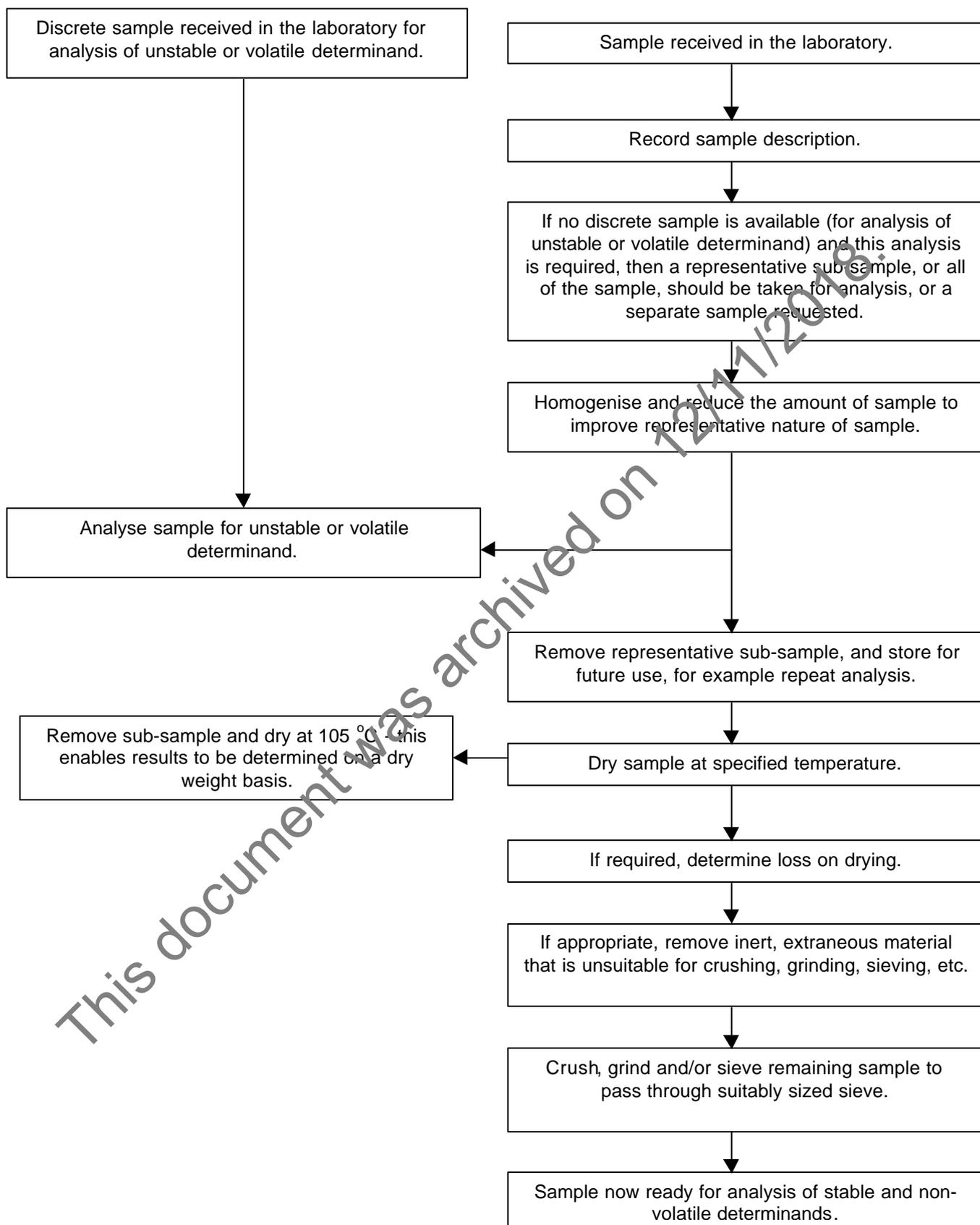
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## Appendix 2 Comparison of loss of weight values obtained at different temperatures

Description of sample	Loss in weight at 40 °C (%)	Loss in weight at 105 °C (%)
Sandy	9.9	10.3
Silty sand	13.4	13.9
Silty sand	12.3	12.6
Silty sand	11.5	12.1
Silty sand	11.1	11.2
Silty sand	15.1	15.8
Silty sand	13.2	13.5
Silty sand	21.2	21.3
Oily/tarry*	30.5	37.8
Oily/tarry*	37.5	41.4
Silty clay/oily odour*	19.8	23.7
Clay	31.5	31.8
Clay	43.2	43.7
Silty clay	22.7	23.2
Silty clay	19.1	19.2
Silty clay	18.6	18.8
Silty clay	20.7	21.8
Clay	25.5	26.2
Clay	35.7	36.1
Clay	35.2	35.6
Clay	27.8	29.9
Silty Clay	20.1	21.5
	Loss in weight at 30 °C (%)	Loss in weight at 105 °C (%)
Sand	0.2	0.5
Silty Clay	6.1	6.6
Silty clay	13.6	15.8
Clay	32.8	34.2
Loamy soil	8.8	10.2
Sandy clay	7.7	8.2
Sandy clay	6.1	6.9
Silty clay	9.4	9.9
Silty clay	13.1	14.7
Silty clay	11.3	12.8

\* The loss in weight from samples containing visible oily/tarry material may include certain organic compounds, not simply the water content.

### Appendix 3 Schematic flow chart for sample preparation and pre-treatment



## **Address for correspondence**

However well procedures may be tested, there is always the possibility of discovering hitherto unknown problems. Analysts with such information are requested to contact the Secretary of the Standing Committee of Analysts at the address given below. In addition, if users wish to receive advanced notice of forthcoming publications, please contact the Secretary.

Secretary  
Standing Committee of Analysts (National Laboratory Service)  
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56 Town Green Street  
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Leicestershire  
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[www.environment-agency.gov.uk/nls](http://www.environment-agency.gov.uk/nls)

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