

Standing Committee of Analysts

The preparation and pre-treatment of potentially contaminated soils and associated materials (2015)

Methods for the Examination of Waters and Associated Materials

The preparation and pre-treatment of potentially contaminated soils and associated materials prior to chemical analysis (2014)

Methods for the Examination of Waters and Associated Materials

This booklet contains details of two methods for preparing soil and associated materials prior to chemical analysis, one using the whole sample, and one using the < 2 mm fraction.

Whilst this booklet refers to equipment actually used, this does not endorse these products as being superior to other similar products. 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. Only limited performance data are presented.

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About this series

Introduction

This booklet is part of a series intended to provide authoritative guidance on recommended 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, soils (including contaminated land) 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 evaluated 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 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.

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 Associated Materials" and their continuing

revision is the responsibility of the Standing Committee of Analysts (established 1972 by the Department of the Environment). At present, there are seven 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, Inorganic and physical methods, Metals and metalloids
- 4 Solid substances
- 5 Organic impurities
- 6 Biological, biodegradability and inhibition methods
- 7 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 these methods are listed at the back of this booklet.

Publication of new or revised methods will be notified to the technical press. If users wish to receive copies or advanced notice of forthcoming publications or obtain details of the index of methods then contact the Secretary on the SCA's web-page:-
<http://www.standingcommitteeofanalysts.co.uk/Contact.html>

Every effort is made to avoid errors appearing in the published text. If, however, any are found, please notify the Secretary. Users should ensure they are aware of the most recent version they seek.

Rob Carter
Secretary
June 2017

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.

A1.0 The preparation and pre-treatment of potentially contaminated soils and associated materials prior to chemical analysis

A1.1 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 potentially contaminated soil/associated materials. 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 requiring analysis.

Requirements of the industry fall into two categories:

- (1) Data for the whole sample for general site assessment and waste categorisation or classification
- (2) Data to be used for human health risk assessment, which is performed on the < 2 mm fraction of a sieved sample

Methods are therefore provided to satisfy both these scenarios, but it is crucial that the client/supplier of the samples is aware of the default laboratory method and specifies the requirements for each batch of samples submitted. This should be communicated at the Contract Review/Quotation stage.

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 and their respective numbers and locations is not described in this document and details of this may be found elsewhere^(see references). However, brief guidance notes for site sampling staff regarding the manner in which samples should be taken and transported are included in Appendix 3.

This booklet is not intended for use with soils tested for agricultural purposes.

Pre-treatment procedures

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). This will depend upon the determinands required (see Section A1.10)

A more detailed list of typical containers for specific determinands can be found in Appendix 1, depending upon the stability of the determinands.

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 on site will depend on specific individual cases, and it is up to the client/supplier to ensure the sampling procedures are appropriate for supplying representative samples and subsamples for the investigation (see Appendices 1 and 3). Similarly, in the laboratory the analytical staff are responsible for checking that the samples as received are suitable, and ensuring that the preparative and analytical procedures used are appropriate for the determinands in question.

Unstable and volatile 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 and it is impossible to prepare and pre-treat the samples using these techniques without significant losses. Hence, determinands that are unstable or easily degradable should be analysed on an as received sample, and a subsample from this should be prepared with minimal handling after samples are received into the laboratory.

If separate samples specifically for VOCs are not taken on site, or if a significant headspace is present in the sample jar, these should be classified as deviating samples by the laboratory, in accordance with TPS 63, and the data on the final report will be flagged as potentially compromised.

However, if subsamples are to be taken from a bulk sample by the laboratory, a duplicate sample is required, as once the jar is opened and a subsample removed, volatiles will migrate into the created headspace and be lost, so repeat or further analysis is not possible.

Once taken, the samples should be stored in a cool (typically 5 +/- 3°C), dark environment, at the laboratory prior to analysis.

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.

Drying procedures

When referring to dried samples (however they are prepared) it is essential to report the particular drying process and drying temperature used. Typically, samples are dried overnight between 30°C and 40°C and the weights of the sample before and after drying recorded, and this loss in weight is used to correct reported results for determinands performed on as received samples. This can be reported as a % of the as received weight, or as a % of the dry weight to give the natural moisture content. However, the

temperature for drying will be dependent upon the analyte of interest, and minimal loss on drying should be proven during the validation process

Air-dried samples should be obtained at ambient temperatures not exceeding 30°C, but it may take several days for samples to dry. This approach is not commonly used.

However, for a true moisture content, the sample should be dried at 105°C (see references).

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, then these details should be stated in the report.

Extraneous material

For most samples received within a laboratory the sample should be considered representative of the area on site where the sample has been taken and therefore the whole sample should be processed. In some rare instances, it may be necessary to remove large fragments and material not amenable to preparation or analysis, or the client/supplier may request certain removals. These extraneous constituents can include, for example:

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

However, if the material on site is being assessed for waste categorisation, leaching procedures, or a general assessment of any contamination present, then it is important that all material is included in the analysis. It is important to note that bricks/stones/gravel/concrete etc. may be impregnated with hydrocarbons or other chemicals and should not be considered inert material, or removed. In some instances, however, they may require separate analysis.

However, if there are any constituents which are not amenable to any sample preparation or pre-treatment process and are not considered to contain significant amounts of the determinands of interest, or may be especially hazardous to staff or cause damage to equipment, then consideration may be given to removing them from the sample⁽¹¹⁾. The removal of any material from the sample submitted to the laboratory prior to commencement of analysis should, therefore, be retained and documented. Such details should include the nature and quantity of the material removed, as this may affect the interpretation of the results reported, and all details should be reported to the client.

Whatever sample preparation and pre-treatment procedures are used, including drying, crushing, grinding and/or sieving etc, all details should be recorded and provided with the final report, as a minimum.

Sub-sampling

Sub-sampling for unstable and volatile determinands

As stated above, samples for volatile analysis should be sampled into separate containers on site – **samples received into the laboratory which are not in the correct containers will be classified and reported as deviating samples.**

However, if the client/supplier insists on the laboratory performing the subsampling, extreme care needs to be exercised to ensure losses of the volatile determinands are minimised.

For the analysis of unstable determinands, it is necessary to obtain a sub-sample that is as representative as possible of the as received sample submitted to the laboratory. This should 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 (either on site or in the laboratory) and care is required to ensure the portion taken for analysis is as representative (and homogeneous) as possible of the whole sample. Procedures for homogenising samples include stirring, cone and quartering, shredding, kneading or other mixing protocols, but for unstable determinands, these procedures should be undertaken as quickly as possible to minimise changes.

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.

It is not possible to analyse the < 2 mm fraction for unstable and volatile determinands, as the as received sample cannot be dried and sieved without significant loss of these target determinands.

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, where possible, and the weight of the whole of the sample submitted to the laboratory should be recorded. If any material is removed or rejected prior to drying then the corresponding amounts should also be weighed and details recorded (as in Section 1.3).

Procedures for homogenising samples include stirring, cone and quartering, shredding, kneading or other mixing protocols, using appropriate equipment. The sample, or subsample, can then be dried (see section 1.2), recording the loss in weight, and then crushed to provide a homogenous powder with a nominal particle size of 250 microns or less. A portion of this prepared sample should then be used for the analysis of stable and non-volatile determinands in the whole sample.

If only the < 2 mm fraction is required for analysis, then after drying, the sample is sieved through a 2 mm sieve, and only this fraction should be collected, crushed, and then used for analysis. The weight of the < 2 mm fraction should also be recorded.

Quantity of sample

Upon sample receipt, the laboratory staff should check there is sufficient sample to perform all the requested analyses (as per TPS 63) – see section 6 and Appendices 1 and 3 for further details. Any potential problems should be reported back to the sample provider.

There should be good communication between the laboratory and the sample provider to ensure all requirements are identified and addressed, prior to submission of samples – this is part of Contract Review as specified in ISO 17025.

If the weight of sample submitted to the laboratory is reduced, then difficulties may arise if the sample no longer remains representative of the location sampled. Further difficulties may be introduced in the production of suitable sub-samples and whether these remain truly representative of the original sample. This will be exacerbated, for example, where high levels of determinands need to be analysed and the amount of sub-sample taken for analysis becomes ever smaller.

See Appendix 1 for details of appropriate containers and quantities.

Reporting of samples

The report should comply with MCERTS requirements wherever possible, and therefore should contain details of, or make reference to, the procedures used to obtain sub-samples, and any unusual aspects of the sample. A brief sample description and all details of the sample preparation and pre-treatment procedures, should be recorded, as per section A1.9.

A1.1 Scope

This document provides a method that should be followed when preparing and pre-treating soil and associated materials from potentially contaminated sites, prior to chemical analysis.

Two procedures are detailed – when analysing the whole sample, and when analysing the < 2 mm fraction.

Appendix 2 shows a schematic flow chart of the main procedures to be considered.

A1.2 Hazards

Care should be taken when handling potentially contaminated samples and all samples should be considered as potentially toxic.

Skin contact with the sample should be avoided, and appropriate safe 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 (including, but not limited to, 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.

Odoriferous samples should alert staff to the presence of potentially toxic volatile substances and hence the potential risks arising from their presence. In all cases, 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. 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. All samples should, therefore, be viewed as potentially hazardous.

Staff must be trained in recognising potential hazards, including asbestos recognition awareness training.

A1.3 Reagents

- A1.3.1 Deionised water
- A1.3.2 Solvents, typically acetone or methanol, AR grade
- A1.3.3 High purity washed sand
- A1.3.4 Proprietary phosphate free surfactant such as Decon 90

Cleaning reagents should not contain concentrations of the determinands of interest which could interfere with the analysis.

These reagents may be used for quality control purposes to ensure significant cross-contamination does not arise between the processing of highly contaminated samples and samples which are not highly contaminated.

A1.4 Apparatus

- A1.4.1 Air Drying Facility

This may be a clean designated area open to the atmosphere at ambient temperatures. The temperature should not be allowed to exceed 30°C.

- A1.4.2 Assisted-Drying Facility

An oven, with thermostatic control, forced ventilation etc., capable of maintaining a specified constant temperature between 30 and 40°C. The temperature used should not have any adverse effect on the determinands being analysed.

- A1.4.3 An Additional Oven

Maintained at 105 ± 5 °C, may be required to enable results to be expressed on a dry weight basis according to BS 1377.

- A1.4.4 Jaw crusher

For size reduction of very large fragments - this may be a mechanical or manual device.

- A1.4.5 Grinder

Capable of grinding and reducing the particle size of the sample to less than 250µm.

A1.4.6 Calibrated Balance

Capable of weighing to an appropriate tolerance (More than one balance may be needed depending on the range required).

A1.4.7 Sieve

Capable of producing a particle size of < 250 µm is appropriate 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 particle size is produced.

A1.4.8 Sieve – 2 mm

For separation of < 2 mm fraction of the sample.

A1.4.9 Riffle box

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 samples). When used repeatedly, a homogeneous sample is produced.

A1.4.10 Pestle and mortar

Useful for crushing small samples.

A1.4.11 Cold storage facility, typically 5°C +/- 3°C

All systems listed above should be made of material that does not contaminate the samples with the determinands under investigation.

A1.5 Sample collection and storage

Upon sample receipt, laboratory staff should check sufficient sample is received for all requested analyses, and that appropriate containers have been used. Any problems should be communicated to the sample provider.

See Appendix 3 for guideline notes for site staff.

A1.6 Preparation and pre-treatment procedures

A schematic flow chart of these procedures is shown in Appendix 2.

Step	Procedure	Notes
A1.6.1	Sample Description The as received sample should be tipped into a clean tray and a brief description of the sample recorded - details may include the following: <ul style="list-style-type: none">• Quantity (record weight), if required• Soil type• Colour• Particle size - approximate• Inclusions such as vegetation, plastic, brick, etc• Any noticeable odour	(a) This description should be recorded before any preparative or pre-treatment processes are performed.
A1.6.2	Homogenisation & Sub Sampling The whole as received sample should be homogenised by whichever means is best suited to the sample type, using one or more of the following: <ul style="list-style-type: none">• Stirring• Cone and quartering*• Kneading• Paddle mixer• Other electric mixers	(b)*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 whole process repeated until the sample is thoroughly mixed. The sample is then divided into four segments and two opposing quarters should then be combined and thoroughly mixed together and this subsample will be used for analysis. The remaining two quarters may be returned to the container for storage.
A1.6.3	If there are any components present in the sample which will not be amenable to further stages of sample preparation (e.g. large pieces of metal), then these may be removed, weighed, retained and recorded on the final report.	(c) If the analysis required is for unstable determinands, then steps A1.5.2 to A1.5.4 should be performed as quickly as possible to minimise potential changes.

A1.6.4 Once the sample is well mixed, subsamples may be removed for subsequent analysis – this can be two quarters of the sample (if cone and quartering is used), or up to 10 smaller aliquots taken from different areas of the whole sample, and placed in a clean container, depending how many analyses are required on the as received sample. The residue of the sample should be returned to cold storage for possible future use.

(d) If the sample contains very large components, such as gravel, ballast, aggregate or hardcore, then some size reduction procedure may be required, such as a jaw crusher. This is not recommended for unstable determinands.

A1.7 Drying

Step	Procedure	Notes
A1.7.1	For the analysis of stable and non-volatile compounds, a portion (sufficient for the requested analyses) of the well mixed sample (see section 7.2) should be transferred to a clean, non-absorbent pre-weighed tray.	(a)The tray material should not adversely affect the sample. The weight of the tray plus sample should be recorded
A1.7.2	The sample should be spread into a thin layer to facilitate the drying process, and then placed in a suitable drying facility at a specified temperature in the 30 – 40°C range until the sample is dried (overnight), typically for approximately 16 hours.	(b)Depending upon individual sample matrices, this period of time may need to be increased. For example, clay soils or soils containing high levels of organic matter may require longer drying times.
A1.7.3	When the sample has dried, the weight of the dried material should be recorded in order to determine the loss in weight due to the drying procedure.	(c)This factor will be used to adjust results performed on as received samples so that all results are reported on a dried weight basis, when required.
A1.7.4	For results to be expressed on a dry weight basis as per BS 1377, a second sub-sample should be dried at 105 °C to enable a suitable factor to be calculated.	
A1.7.5	As an aid to the production of an homogeneous sample, a riffle box may be used on the dried sample, which 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.	(d)This technique is equivalent to that of the cone and quartering process. Again, this is not suitable for unstable determinands. (e) For all determinands performed on dried material, it is important for the laboratory to produce validation data proving there is no loss on drying

A1.8 Crushing, grinding, sieving etc. for analysis of the whole sample

Step	Procedure	Notes
A1.8.1	Using a suitable crushing system, all the dried material can now be crushed and/or ground to produce a finely powdered homogenised material of the requisite particle size (less than 250 µm). Sub-samples of the ground material should be taken for the analysis of specific determinands.	
A1.8.2	If the method has been adequately validated across a range of matrices, it should not be necessary to sieve every sample, although this can be done on a spot check basis for quality control purposes, if required.	(a) There may be some atypical samples where it may be necessary to sieve all the crushed material to ensure it will all pass through a 250 µm sieve
A 1.8.3	The crushing vessels should be thoroughly cleaned after each sample: methods include the use of surfactants, solvents and deionised water, as appropriate.	(b) Sand or gravel may be used as an abrasive cleaning agent, particularly if highly contaminated samples are known to be present.
A 1.8.4	It should then be possible for the cleanliness of the equipment to be randomly checked for quality control purposes 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.	(c) All cleaning protocols should be validated.
A 1.8.5	The sequence of samples crushed should be recorded, to facilitate traceability if a highly contaminated sample is subsequently found to be present.	

A1.9 Crushing, grinding, sieving etc. for analysis of the < 2 mm fraction

Step	Procedure	Notes
A1.9.1	<p>The dried, weighed, sample is placed onto a 2 mm sieve, and the fraction passing through is collected and weighed. There should be no crushing or size reduction of the larger non-friable components – the sample should be gently agitated or easily disaggregated by hand.</p> <p>The < 2 mm fraction can now be crushed as per the steps in A1.8</p>	<p>(a) The percentage of the 2 mm fraction in the whole sample should be reported</p> <p>(b) Some samples may contain only a small amount of < 2 mm material, and it may be necessary to dry and sieve the whole sample in order to obtain sufficient sample for analysis. If this is still not adequate, the sample provider should be informed.</p>

A1.10 Reporting

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 the following information ⁽¹²⁾:

- a brief description of the sample
- if the analysis is performed on a dried or as received basis
- if the analysis is performed on the whole sample or the < 2 mm fraction
- details (including amounts and descriptions) of any portion of the sample or subsample 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

A1.11 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 10175:2011 - Investigation of potentially contaminated sites – Code of practice
- 3 ISO/FDIS 2002:10381-1 – Soil Quality – Sampling - Part 1 – Guidance on the design of sampling programmes.
- 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.
- 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 2006:11464 – Soil quality - Pre-treatment of samples for physico-chemical analysis.
- 9 ISO FDIS 2003:14507 – Soil quality - Pre-treatment of samples for determination of organic contaminants.
- 10 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.
- 11 Initial sample preparation, M Allen, in Chemical Analysis of Contaminated Land, (Eds) K C Thompson and C P Nathanail Blackwell, 2003, ISBN 1841273341.
- 12 MCERTS Performance Standard for Laboratories Undertaking Chemical Testing of Soil, Environment Agency, Current Version.
- 13 ISO/IEC 17025:2005 General requirements for the competence of testing and calibration laboratories.
- 14 BS1377-1:1990 Methods of test for soils for civil engineering purposes. General requirements and sample preparation
- 15 BS ISO18512:2007 Soil quality. Guidance on long and short term storage of soil samples.

- 16 EPA 8260C:2006 Volatile Organic Compounds by Gas Chromatography /Mass Spectrometry (GC/MS)
- 17 EPA 9010C:2004 Total and Amenable Cyanide: Distillation
- 18 EPA 9012B: 2004 Total and Amenable Cyanide (Automated Colorimetric, with Off-line Distillation)
- 19 EPA 1664:2009 N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM; Non-polar Material) By Extraction and Gravimetry. This document is currently under revision.
- 20 EPA 9071B:1998 n-Hexane Extractable Material (HEM) for Sludge, Sediment and Solid Samples
- 21 EPA 8100:1986 Polynuclear Aromatic Hydrocarbons
- 22 EPA 8270D:2007 Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
- 23 EPA 8015C:2007 Nonhalogenated Organics by Gas Chromatography
- 24 BRE SD1:2005 Concrete in aggressive ground (Special Digest 1)
- 25 EPA 9251:1986 Chloride (Colorimetric, Automated Ferricyanide AAll)

Appendix 1 Relative stabilities, typical sample containers and holding times

This table provides a guide to containers and the relative stability (as received or dried), and holding times, with holding times, for some common determinands, although other holding times may be used where appropriate (see notes below table).

Soil

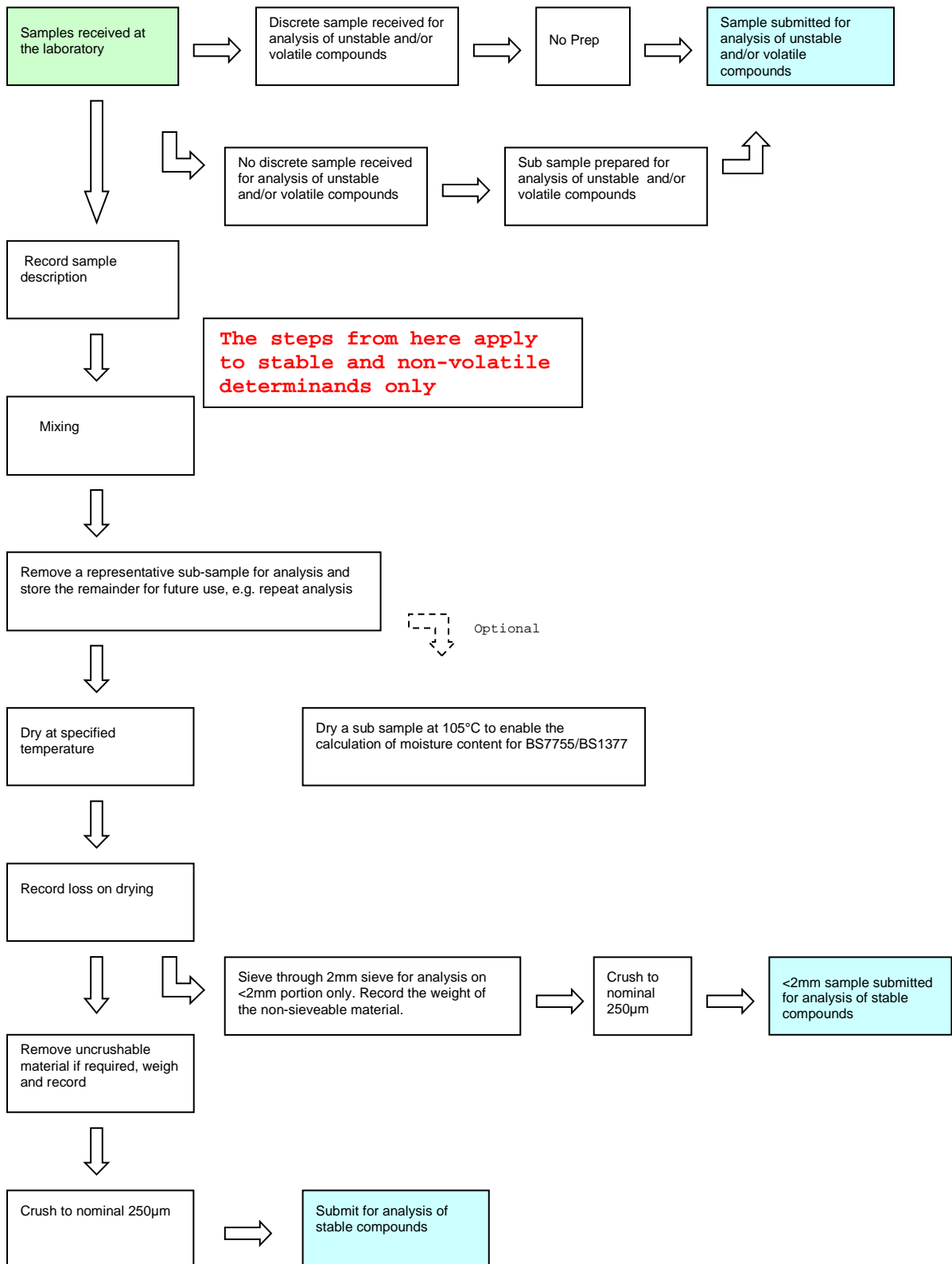
Analyte	Container type	Minimum sample required	Dry or As received ³	Maximum holding time from sampling ¹	Reference/ in house ²
Ammonia/Ammonium	Plastic tub	20g	As received	1 week	BS ISO18512:2007
Anions - sulphate, chloride	Plastic tub	20g	Dry	3 years	BS ISO18512:2007
Anions - nitrate, nitrite	Plastic tub	20g	As received	1 week	BS ISO18512:2007
BTEX	60ml coloured glass jar	Full container, no headspace	As received	1 week 4 days	EPA 8260 BS ISO 18512
Conductivity	Plastic tub	20g	As received	1 week	BS ISO18512:2007
Cyanide	Plastic tub	20g	As received	2 weeks	EPA 9010B/9012
Dioxins	250 ml Coloured glass jar	50g	Dry	1 year	EPA 1613
Heavy metals general suite	Plastic tub	10g	Dry	3 years	BS ISO18512:2007
Heavy metals - organo/elemental mercury, organo lead/tin	Plastic tub	10g	As received	4 days	BS ISO18512:2007
Hexavalent chromium	Plastic tub	20g	As received	30 days	BS ISO18512:2007
Loss on ignition	Plastic tub	10g	Dry	1 month	BS ISO18512:2007
Oil & grease, EPH	Coloured Glass	20g	As received	1 month 1 week	EPA 9070/1 BS ISO18512:2007
Pesticides	Coloured Glass	20g	As received/dry	1 month	BS ISO18512:2007
PAH	Coloured Glass	20g	As received/dry	2 weeks	EPA 8100/8270 BS ISO18512:2007
PCB	Coloured Glass	20g	As received/dry	2 weeks	BS ISO18512:2007
pH	Glass or plastic	20g	As received	1 week	BS ISO18512:2007
Phenols	Coloured Glass	20g	As received	2 weeks	EPA 8270
PRO	60ml coloured glass jar	Full container, no headspace	As received	1 week	EPA 8015
Sulphide	Glass or plastic	20g	As received	3 weeks	BRE SD1
SVOC	Coloured Glass	20g	As received/dry	2 weeks	EPA 8270

Thiocyanate	Glass or plastic	20g	As received	No special requirement	EPA 9251
Total Organic Matter	Glass or plastic	20g	Dry	1 month	
Total sulphur	Glass or plastic	20g	Dry	1 month	
Total Extractable Petroleum Hydrocarbons (C10-C40)	Coloured Glass	20g	As received	2 weeks	EPA 418.1
VOC	60ml coloured glass jar	Full container, no headspace	As received	1 week 4 days	EPA 8260 BS ISO 18512

Notes: Sample storage environment is 5 +/- 3°C

1. Holding times are measured from the sampling time until the point where the analytical process commences. For certain determinands, chemical preservation may also be an option to extend the holding times. Post extraction, some determinands will be stable for more extended periods, e.g. PAHs, PCBs, and SVOCs stored in solvent in sealed vials.
2. Where in house validation data extends the holding time, this is acceptable.
3. Some determinands can be performed on either dried or as received sample, but validation will always be required to prove no significant loss on drying. However, low molecular weight PCBs, some SVOCs, pesticides, and PAHs may be lost if dried at 40°C.

Appendix 2 Schematic flow chart for sample preparation and pre-treatment



Appendix 3 Summary guideline notes for on-site staff and samplers

1 Sample Collection and Storage

For the analysis of potentially contaminated samples, it is essential that the sample submitted to the laboratory is representative of the material or location under investigation⁽¹⁻⁷⁾. 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 sufficient sample should be provided.

If suppliers of samples are aware of any known hazard, they should inform the laboratory accordingly.

Suitable equipment and containers should be available for taking and storing samples. Specific guidance on details of sampling, sample containers and storage conditions is usually given in the individual methods describing the analysis of specific determinands.

The following are examples of suitable containers (see Appendix 1 for more details):

Metals, inorganics, Leaching tests	1 x Plastic tub (min. 500 g) 1 x Plastic tub (min. 500 g) – needed to provide sufficient volume of leachate
Asbestos	1 x double wrapped tub or plastic bag (1 kg) – this is essential for H & S, and representative sample size
Organics - PAHs, EPH, pesticides	1 x 250 g coloured glass jar as plastics can leach organics into the sample
VOCs and other volatile species	2 x 60 g coloured glass jars for each test – VOCs can be lost if the laboratory has to subsample

Certain types of sample, such as ballast or aggregate, may require more than this, possibly as much as 10 kg.

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. A cool environment, with a typical storage temperature of 5 +/- 3°C is recommended, and samples should be despatched to the laboratory as soon as practicable.

If samples are not stored correctly, they may be reported as deviating samples.

2 Documentation

The sample provider should complete a Chain of Custody form containing all relevant information such as:

- The date sampled
- Trial pit/borehole location or similar unique identifier
- Site location/identification code
- Name of sampler

- Contact details
- Analytical requirements
- Quotation reference
- Turnaround required
- Any known hazards

3 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 determinands) or separate individual containers are submitted for specific analytical determinands.

4 Samples for volatile organic analysis

The analysis of volatile organic determinands requires on-site sampling into small (typically) 60 g amber glass jars. Each sample should ideally be taken in duplicate, and care should be taken to pack the soil tightly into each jar, smoothing across the top to ensure a good seal with the lid, minimising headspace.

A duplicate sample is required, as once the jar is opened and a subsample removed, volatiles will migrate into the created headspace and be lost, so repeat or further analysis is not possible.

The sample will not receive any further mixing, homogenising, or drying in the laboratory, so it is important that the sample is as representative as possible.

Once taken, best endeavours should be made to chill the samples to below 8°C, both during storage and transportation to the laboratory.

Appendix 4 Glossary

Air-dried sample	A sample that has undergone an air-drying process at less than 30 °C
As-received sample	The sample submitted to the laboratory in a suitable container as removed from a specific location
Assisted-dried sample	A sample that has undergone a specified accelerated drying process. This may involve oven-assisted drying at a specified temperature between 30 – 40°C, or some other process
Determinand	The parameter or analyte being determined
Deviating samples	Samples where the results may be potentially compromised by incorrect sampling, storage or inappropriate containers (as per UKAS TPS 63)
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 (as per BS1377, see references)
EPH	Extractable petroleum hydrocarbons
Moisture content	Loss on oven drying at a specified temperature and used to correct data derived from as received samples, and expressed as % weight of sample as received
Natural moisture content	The loss on drying expressed as % of dry weight (for geotechnical purposes) – results can therefore be >100%. For waste classification, moisture content ratio and dry matter content ratio are used, as in BSEN 12457
Non-friable	Not easily disaggregated by hand
PAHs	Polycyclic aromatic hydrocarbons
PCBs	Polychlorinated biphenyls
Sample	A quantity of material submitted to the laboratory. This comprises the “as-received” sample
Soils	A generic term that includes contaminated land and associated materials, but within the context of this booklet, does not refer to agricultural soils
Sub-sample	A representative portion of the sample taken from the original sample

SVOCs	Semi volatile organic compounds
Test	An analysis which consists of several determinand results produced in one run, e.g. VOCs
Unstable	Determinands which are subject to change or degradation (apart from volatilisation) caused by drying, crushing or sieving
VOCs	Volatile organic compounds with boiling points between - 25°C and 200°C

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.

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Standing Committee of Analysts

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