Technical Guidance Note (Monitoring)

M4

Guidelines for Ash Sampling and Analysis

Environment Agency
Version 7
June 2016

Record of amendments

Version number	Date	Amendment
Interim draft v5	April 2002	Interim update to version 4. Introduced the requirement for a sampling plan
6	March 2011	Major rewrite to incorporate published CEN standards
6	April 2013	Rebadged as Technical guidance note M4, no other changes made.
7	June 2016	Remove references to hazardous waste assessment and landfill waste acceptance criteria as this is now on GOV.UK Updated hyperlinks Updated legislative references to include the Industrial Emissions Directive and updated Animal By-products Regulations Appendix E: removal of dates from references to standards. The most up-to-date version should be used.

Feedback

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1 INTRODUCTION

Note: this guidance is sometimes referred to as the 'Agency ash sampling protocol'.

Follow this guidance if your environmental permit requires you to sample and analyse solid industrial residues. You can also use this guidance to demonstrate compliance with Waste Protocols (GOV.UK), including product specifications (for example for re-use in the construction industry).

Solid residues (i.e. ash) are formed by incineration, co-incineration and some combustion processes. They may also be captured in dust abatement controls. Examples of industrial residues include:

- IBA (Incinerator bottom ash)
- APC (Air pollution control ash)
- Cement kiln dust (CKD)
- Other bottom ash, such as MBM (meat and bone meal) and poultry litter ash
- Furnace bottom ash (FBA)
- Pulverised fuel ash (PFA)

Following this guidance will help you ensure that samples of solid residues taken for analysis:

- are taken as representatively as possible to minimise sampling bias (see Section 2)
- has consistent sample treatment to ensure analytical results can be compared (see Section 3)
- are collected in a traceable manner (see Section 4)

This guidance is based on BS EN 14899 Characterisation of Waste – Sampling of Wastes – Framework for the preparation and application of a sampling plan. It is structured to guide you through a methodical series of steps that will help you decide how much sample to take, how often to take a sample or how to analyse the sample. An overview of these steps is shown in Figure 1. Many of the answers will be dependent upon the overall objective of the sampling exercise and will be specific to individual sites.

Practical guidance on how to apply BS EN 14899 can be found in the five supporting Technical Reports. The Technical Reports are quoted in this guidance document, where applicable:

- CEN/TR 15310-1 - Guidance on selection and application of criteria for sampling under various conditions.
- CEN/TR 15310-2 – Guidance on sampling techniques.
- CEN/TR 15310-3 – Guidance on procedures for sub-sampling in the field.
- CEN/TR 15310-4 – Guidance on the procedures for sample packing, storage, preservation, transport and delivery.
- CEN/TR 15310-5 - Guidance on the process of defining the sampling plan.

The design of the ash sampling will depend on its purpose. If ash is being sample for waste classification or landfill waste acceptance, for example, you will also be referred other documents where additional or more detailed guidance can be found. You must ensure that any separate guidance is also adhered to:

- waste classification: How to classify different types of waste (GOV.UK);
- landfill waste acceptance: Waste testing and sampling for disposal to landfill (GOV.UK);

Residues that are produced from incineration and co-incineration plants must undergo a number of physical and chemical tests to demonstrate compliance with plant operating conditions, re-use or disposal criteria. These tests are requirements of Articles 50 and 53 of the Directive on Industrial Emissions (IED) (2010/75/EU).

Incineration sites that are permitted to incinerate animal remains under IED and/or the Animal By-Products Regulations may have additional waste residue testing requirements. However, incinerators that only burn animal carcasses may also require a permit (under the Environmental Permitting Regulations) to operate if they meet capacity criteria, although IED conditions will not be imposed. See the following sources for more information:

- Sector Guidance Note on the Incineration of Waste (EPR 5.01) (GOV.UK)
- Guidance on the incineration of animal carcasses (GOV.UK)

2 SAMPLING METHODOLOGY

The following sections and sub-sections of this guidance will take you through each of the seven steps that are described in BS EN 14899. The first three steps are key steps because they focus on ensuring how the sample is taken. The first step is the development of a sampling plan:

1. Define the sampling plan
2. Take the sample in accordance with the sampling plan
3. Transport the sample to the laboratory
4. Prepare the test portion
5. Extraction
6. Analyse the sample
7. Produce the overall report

Key steps

2.1 KEY STEP 1 – DEFINING THE SAMPLING PLAN

The sampling plan is a **written record** of why sampling is to be carried out. It describes the overall aims (the objective) and includes specific and practical instructions on what is going to be sampled, how it is going to be sampled, how the sample is going to be tested and by whom. An example sampling plan template is given in Appendix A.

The development of a sampling plan can be broken down into a further eight steps. Figure 1 illustrates the eight steps and the technical reports where additional guidance can be found. This is an iterative process: as you proceed through the eight steps in turn and more information is obtained, the sampling plan will probably require review and refinement. Whatever changes are made to a sampling plan, do not forget the objective of the exercise, or the objective may not be achieved. If there is more than one objective, there may be a need for a different sampling plan for each objective.

In practice, generic sampling plans[2,3] may be developed to serve a common objective. Depending on the objective, this may encourage consistency for sampling or analytical requirements. However, it is advisable to review generic sampling plans, adapting them in order to meet site-specific situations, such as on-site health and safety considerations.

The next eight sub-sections describe each of the steps to developing a sampling plan, and sources of more information, in more detail.

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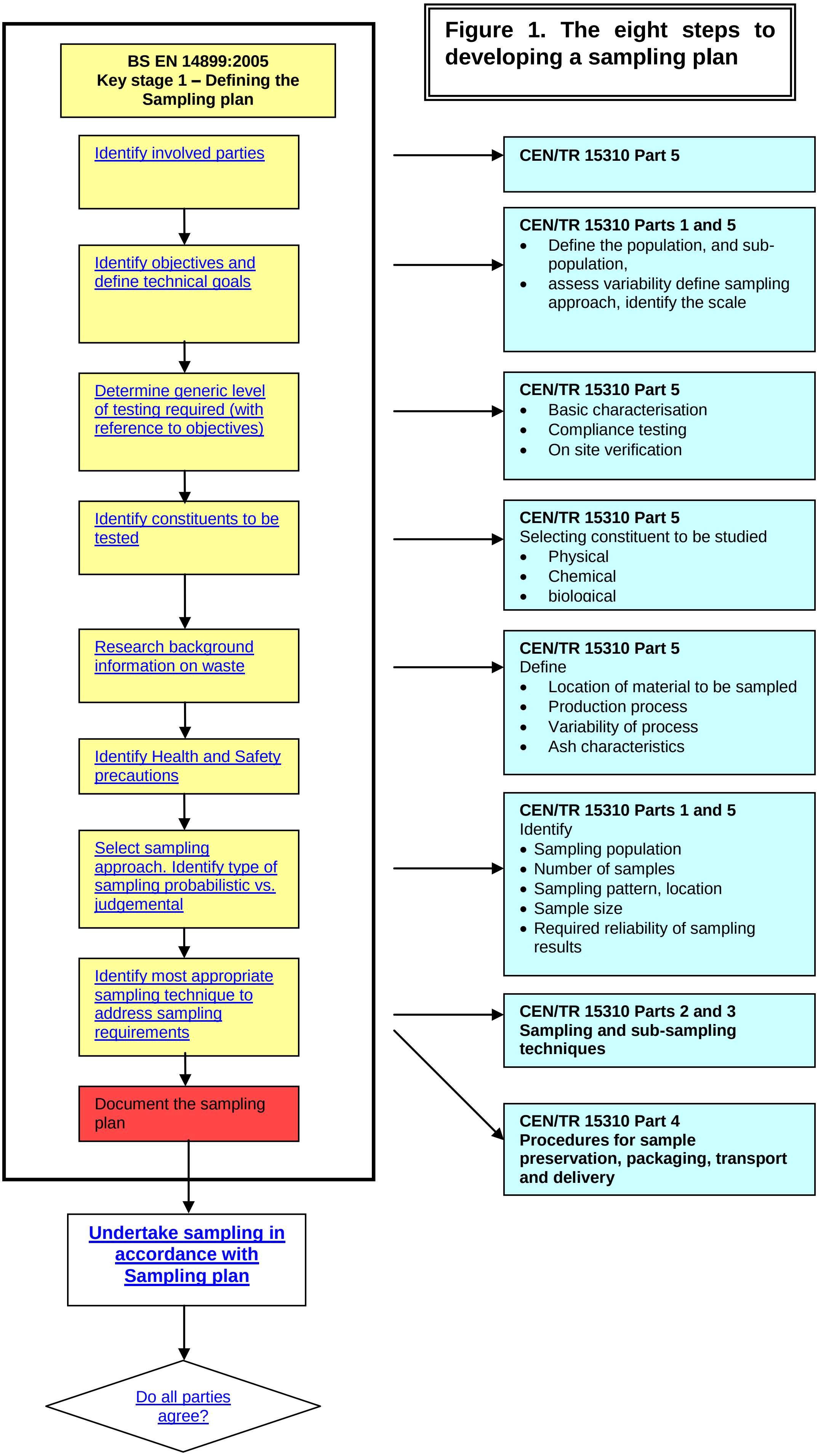
[2] Appendix F reproduces the ash sampling protocol developed by us for those incinerators permitted to burn suspected-BSE cattle. The protocol is highly prescriptive but may not be suitable for all incinerator types.

[3] Examples of sampling plans to meet different objectives can be found in The Environmental Service Association Research Trust (ESART)'s 'A practitioner's guide to testing waste' http://www.esauk.org/reports_press_releases/esa_reports/ESART_practitioners_guide_to_testing_waste.pdf and the IBA Sampling Protocol (see http://www.esauk.org/esa_reports/) for the latest version.

Figure 1. The eight steps to developing a sampling plan



2.1.1 Identify involved parties

Sampling plans are not developed in isolation. There may be a number of parties[4] who can contribute towards achieving the overall objective. Legislation places the responsibility on the waste producer and/or waste manager to characterise (often by sampling and analysis of) waste residues. It is therefore the waste producer who will lead the process of defining the sampling plan, in consultation with other interested parties (for example, the regulator, the sampler, the analyst, and the waste manager).

2.1.2 Identify objectives and define technical goals

The objectives define the purpose of the sampling exercise. Example objectives may include:

- Comply with the sampling and monitoring requirements of an environmental permit
- to determine the re-usability of the material;
- to carry out a risk assessment perhaps for human health or the environment;
- to demonstrate compliance with quality protocols;
- To classify a waste and determine if it is a hazardous waste (i.e. to assess and classify a ‘mirror’ entry in the List of Waste).

The waste producer, in consultation with involved parties, will translate the objective into unambiguous technical goals. The technical goals relate to other elements of the sampling plan: constituents to be tested; the population that is represented by the sampling results, the statistical parameter to be determined; sampling approach (probabilistic or judgemental); the sampling technique and any pre-treatment that may be required.

Example of an objective translated into a technical goal:

Objective: To demonstrate that the ash derived from incineration processes is in compliance for relevant determinands, as defined by permit.

Technical goal: a representative sample of IBA from storage bay 1 to be taken on the first day of each month and submitted to the laboratory for soluble metal analysis (BS EN xxxx).

Defining the population, assessing spatial and temporal variability and defining the scale are essential in determining how much material and how often to sample. This guidance document provides an overview but cannot reproduce the depth of guidance given elsewhere. You are recommended to refer to CEN/TR 15310:2006 part 1 (section 4.2, 4.3 and 4.4) and CEN/TR 15310:2006 part 5 (section 4.4.3 and 4.4.4) for additional guidance.

The **‘population’** is the statistical term used to define the total volume of material about which information is required through sampling.

Defining the population, an example:

Population:	All the ash produced by an industrial plant
Annual Sub-Population:	All the ash produced by an industrial plant in one year
Monthly Sub-population:	All the ash produced by an industrial plant in one month

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[4] CEN/TR 15310-5:2006 Paragraph 4.4.5.1 “It is very important that the involved parties are aware of the impact of their choices on both costs and reliability of the sampling and that they specify the desired reliability of the estimate before a Sampling plan is constructed.”

Using the example above, and depending on the rate of production, it may not be possible, let alone practical, to wait an entire year for all the ash to be sampled. Therefore the realistic population to sample may be all the ash produced in one month. This assumes that the one month's ash production is representative of the whole year's ash production and that there are no process variations.

Variability in the production process, for example using different waste derived fuels in an incinerator, may lead to a heterogeneous ash. To determine how heterogeneous the ash is, more samples will be required.

The **'scale'** is the minimum quantity (mass or volume) of material below which variations are judged to be unimportant. For example, the characteristics of incinerator bottom ash, from a municipal waste incinerator, may vary on a daily basis due to waste collections from different areas. However, these variations may be smoothed out over a week and therefore between weeks. If process variations on a weekly basis are unimportant then the scale is weekly. If variations on a daily basis are important, then the scale is daily.

2.1.3 Determine generic level of test required

The robustness of the sampling will be determined by the objective and the level of sampling required.

If the sampling objective is waste classification, you should refer to Appendix D of Technical guidance WM3 – Waste Classification – Guidance on the classification and assessment of waste. Most of the wastes subject to the 'Guidelines on Ash Sampling and Analysis' are classified under 'mirror entries' found in the following subchapters of the List of Waste (Appendix A):

- 19 01 Wastes from incineration or pyrolysis of waste, or
- 10 01 Wastes from power station and other combustion plants.

The 'mirror entries' from subchapter 19 01 are presented here.

'Mirror entries' that contain a general reference to 'hazardous substances' should be assessed to determine if any hazardous substances are present and whether the waste possesses **any** hazardous property. Persistent organic pollutants also have to be considered. Examples of hazardous properties associated with some of the hazardous substances that may occur in such wastes include Irritant (HP4), Specific Target Organ Toxicity/Aspiration Toxicity (HP5), Acute Toxicity (HP6), Corrosive (HP8), Carcinogenic (HP7), Toxic for Reproduction (HP10), Mutagenic (HP11), and Ecotoxic (HP14). However all hazardous properties should be considered.

'Mirror entries' from subchapter 19 01 Wastes from incineration or pyrolysis of waste

19 01 11*	bottom ash and slag containing hazardous substances	MH
19 01 12	bottom ash and slag other than those mentioned in 19 01 11	MN
19 01 13*	fly ash containing hazardous substances	MH
19 01 14	fly ash other than those mentioned in 19 01 13	MN
19 01 15*	boiler dust containing hazardous substances	MH
19 01 16	boiler dust other than those mentioned in 19 01 15	MN
19 01 17*	pyrolysis wastes containing hazardous substances	MH
19 01 18	pyrolysis wastes other than those mentioned in 19 01 17	MN

'*' Denotes the hazardous waste classification in each 'mirror entry' pair. The non-hazardous classification in each pair must only be used after assessment has determined that the waste possesses no hazardous properties and does not contain persistent organic pollutants.

The Landfill Directive and BS EN 14899 describe three levels of testing (but use slightly different terms for each level); each term is given in the examples below.

Examples of the three levels of testing:

Characterisation, basic or level 1: characterisation of ash, involving extensive sampling and analysis.

Compliance, periodic or level 2: sampling to show compliance of a particular determinand, involving robust sampling to give a composite sample with a lesser level of analysis.

Verification, quick check or level 3: equivalent of a quick on-site test to indicate the presence or absence of a particular determinand.

2.1.4 Identify constituents to be tested

The constituents to be tested will depend on the objective of the sampling plan. It may be necessary to identify physical (e.g. particle size distribution), chemical (pH, specific elements) and/or biological parameters depending upon the nature of the original material, the intended end use (i.e. disposal or recycling). National or European regulations may also determine what constituents are to be tested for.

It will be helpful to have some prior information or some research may be required on what constituents are of legislative or environmental concern[5].

For other compliance testing, constituents to be tested may be laid down in legislation through environmental permits. For example, the Industrial Emissions Directive is explicit in its monitoring requirements:

IED Article 50(1):

‘Waste incineration plants shall be operated in such a way as to achieve a level of incineration such that total organic carbon (TOC) content of the slag and bottom ashes is less that 3% or their loss on ignition is less than 5% of the dry weight of the material.’

IED Article 53(3):

’Prior to determining the routes for the disposal or recycling of the residues from, appropriate tests shall be carried out to establish the physical and chemical characteristics and the polluting potential of the residues. Those tests shall concern the total soluble fraction and heavy metals soluble fraction’

Table S3.10 ‘Residue quality’ lists the monitoring requirements in waste incineration EPR permits:

- **All incinerators routinely monitor bottom ash and APC residues** for metals (antimony, cadmium, thallium, mercury, lead, chromium, copper, manganese, nickel,

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[5] CEN/TR 15310-5:2006 Paragraph 4.4.2 “The selection of constituents starts with an inventory of constituents that are raised in relevant legislation. The constituents identified by legislation are often are reflection of their potential to cause harm to human, environmental and economic risks. Background data on the waste may also identify further relevant constituents”

arsenic, cobalt, vanadium, zinc) and their compounds, dioxins, furans and dioxin-like PCBs.

- **Before use of a new disposal or recycling route, incinerators monitor bottom ash and APC residues** for total soluble fraction and metals (antimony, cadmium, thallium, mercury, lead, chromium, copper, manganese, nickel, arsenic, cobalt, vanadium, zinc) soluble fraction
- For incineration sites that are permitted to burn animal remains (for example MBM, tallow, animal parts), analysis for protein in bottom ash may be required.

2.1.5 Research background information on waste

Knowledge of the process or activity that produced the waste, as well as the results from previous testing help to inform the decision making process. The information or data can be used to focus effort on where it is required, thus enabling best use of effort and financial resource in the sampling exercise.

Example 1: interested parties may agree that there is sufficient evidence to conclude that a certain determinand will not be present in the waste. Consequently, interested parties may agree that the specific determinand need not be tested for. The sampling plan will document the justification for removing the testing of the determinand from the analytical suite.

Example 2: results from previous tests demonstrate that the heterogeneous nature of the waste. Therefore the interested parties will know that more samples will be required.

Background information may also include: what inputs created the waste; what is the method of ash collection; how is the ash stored (is it stockpiled in heaps or in a container), how much ash is stored and where is it stored; is the solid waste a moving or static stream; are there any access issues that may affect sampling; is the ash heterogeneous, homogeneous, granular, or monolithic to help inform the sampling approach.

2.1.6 Identify Health and Safety precautions

Health and safety should have the highest priority.

For some industry-wide sampling objectives, details of levels of testing and components to be tested can be agreed at a generic level. However, generic sampling plans are unlikely to provide the detail of site-specific safety hazards and risk assessment. Of the interested parties involved in designing the sampling plan, the site operator has the duty of care for health and safety for their site.

Prior to undertaking site work, a risk assessment[6] shall be carried out. The risk assessment should be regularly reviewed; site safety rules must be adhered to and safety procedures followed.

2.1.7 Select sampling approach

The decisions that you make at this point in the process (and in section 2.1.8) will determine how statistically representative your final sample will be.

The sampling approach depends on the sampling objective. It is influenced by practical considerations, such as the physical characteristics of the residue and the risk assessment.

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[6] BS ISO 10381-3:2001 *Soil quality -- Sampling -- Part 3: Guidance on safety* for identifying potential hazards is a useful reference. Where the constituents of the materials are known, Material Safety Data Sheets are also useful sources of information.

2.1.7.1 Probabilistic versus judgemental sampling

The choice of whether to take a probabilistic or judgemental approach takes into account the variability within the sub-population and the acceptable (or agreed) degree of uncertainty. Probabilistic sampling provides greater confidence over judgemental sampling in that the obtained sample represents the whole population. Probabilistic sampling is therefore the preferred approach.

There may be practical, including safety, and financial constraints that preclude this approach. For example, IBA from clinical waste may contain a mixture of free flowing ash and fused lumps of material. In this situation it will not be possible to take a sample that is representative of the whole population; a judgemental approach will be the only practical approach. Judgemental sampling can also be useful to address a specific question (for example – to identity a specific piece of material present in the waste)

If judgemental sampling is the chosen approach, then sampling of the sub-population should be as representative as possible.

Figure 2 illustrates the two approaches. Sub-population 1 has the same composition as the whole population. A probabilistic approach gives each part of the sub-population an equal chance to be sampled. In sub-population 2, it is not feasible to take samples from the whole population. It is hoped that the sub-population is representative of the overall population but it is not possible to estimate the uncertainties. Sub-population 3 illustrates an appropriate use of judgemental sampling. Part of the waste clearly differs from the rest and information is required on the different part of the waste. This is referred to as ‘spot sampling’ or ‘targeted worse-case’ sampling.

The choice of approach taken will be site specific but, whichever approach is taken, it must be justified and the justification recorded in the sampling plan.

The sampling approach will also decide how much sample to take, whether individual or composite samples are taken and where to take the sample from. This is discussed next.

2.1.7.2 Determine the increment and sample size (CEN/TR 15310-1)

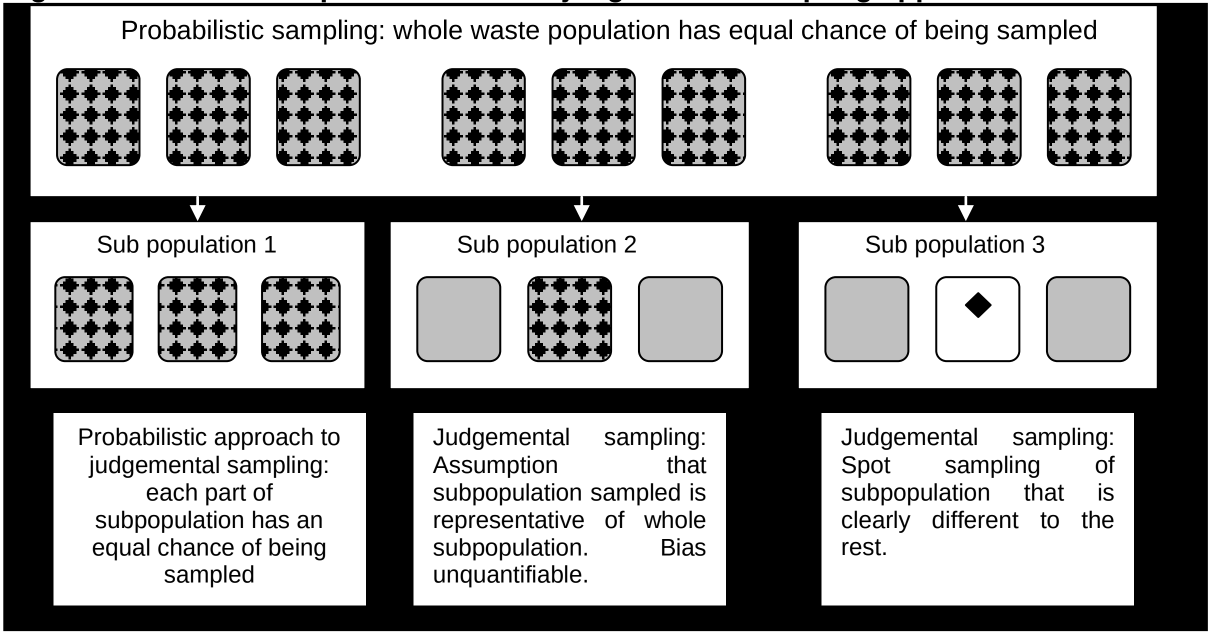
The actual size of the increments and samples will depend on the quantity of material required by the laboratory for analysis. It is advisable to consult with the analytical laboratory to ensure that sufficient but not excessive quantity of material is provided to them. Sending too small an amount will mean that there is insufficient quantity to complete the analysis; sending too much may have storage and/or cost implications. Depending upon the objective, you may also wish to consider if you need duplicate samples.

An **increment** is the amount of material (mass or volume) that is obtained through one single sampling action. It may be combined with other increments to form a **composite sample**. If individual increments are analysed separately, each increment will be an individual sample.

The **minimum increment size** is governed by the need for the sampling device to accommodate all particle sizes. The device opening should be at least three times the diameter of the largest particles.

The **minimum sample size** must take into account the homogeneity or heterogeneity of the individual particles, such that a representative sample is taken. Where residues are large, fused lumps of material (such as clinker-like lumps of un-incinerated metal items, glass and ash from clinical waste incinerators), a larger sample size will be needed. This will ensure that, after particle size reduction (see section 2.1.8), there is sufficient sample for analysis of the ash.

Figure 2 Illustration of probabilistic and judgemental sampling approaches



Probabilistic approach to judgemental sampling: each part of subpopulation has an equal chance of being sampled

Judgemental sampling: Assumption that subpopulation sampled is representative of whole subpopulation. Bias unquantifiable.

Judgemental sampling: Spot sampling of subpopulation that is clearly different to the rest.

2.1.7.3 The use of composite versus individual samples (CEN/TR 15310-1)

The choice of whether to obtain composite or individual samples depends on the objective:

Characterisation, basic or level 1 testing: to obtain a reliable estimate on the variability of individual components within the ash, a substantial number of samples are taken.

Compliance, periodic or level 2 testing: to obtain a reliable estimate on the mean quality of the ash over a period of time, a substantial number of increments should be taken over time to form one or a number of composite samples.

For this level of testing it may be useful to have a diagram showing the areas where the increments of ash should be taken. By defining a methodological approach to sampling, future samples can be taken in a similar manner so that results can be compared.

Verification, quick-check or level 3 testing: one sample or small number of samples to give an approximate indication of the quality of the material.

2.1.7.4 Determine the number of increments and samples (CEN/TR 15310-1)

The number of increments that will form the composite sample is dependent on the variability of the ash, as well as the overall sampling confidence and precision required. In other words, taking a 1 kilogram spot sample from one location from a 1 tonne ash stockpile, would not give a high degree of confidence in the resultant analytical result. CEN/TR 15310-1:2006 Annex C and Annex D of technical guidance WM3 describe detailed statistical approaches to determine the number and the size of samples and increments. A simpler calculation is given in section 5 of BS EN 932-1:1997 *Tests for general properties of aggregates Part 1. Methods for sampling:*

$$M = 6 \text{ x } \sqrt{D} \text{ x } \rho b$$

Where,

M is the mass of the sample (in kilograms)
D is the maximum particle size (in millimetres)
ρb is the loose bulk density (megagram per cubic metre, determined as specified in BS EN 1097-3:1998)

Annex B of BS EN 932-1:1997 can be used to check if the number of sampling increments is adequate.

2.1.8 Identify most appropriate sampling technique to address sampling requirements

You should choose the sampling technique and equipment that is appropriate for your site and that will meet the sampling objective. Document your choice in the sampling plan. Figures 13 and 14 in CEN/TR 15310-2:2006 provide guidance in the selection of appropriate sampling techniques and equipment that is most relevant to solid waste residues sampling;

The choice of sampling technique is dependent on the type of material (for example, solid material), the situation from which a sample is to be taken (for example, a stockpile or conveyor belt), the degree of heterogeneity (how well mixed the component of interest is) and the level of testing.

The sampling equipment to be used is dependent upon a number of factors, such as the nature of the waste, how much sample is required and where the sample is to be taken from. The equipment must be safe, practical and fit-for-purpose. For ash sampling, the most likely types of equipment are scoops, trowels, or a thief/trier. Appendix A of CEN/TR 15310-2:2006 describes each of the sampling equipment types.

Tables B1 and B2 in Appendix B are extracts of sampling methodologies from CEN/TR 15310-2:2006 most relevant to sampling ash from hoppers, heaps, silos, bags, kegs or drums. In all cases, sample preparation starts with gaining safe access to the material. After the material has been transferred to the sample container, the container is wiped and labelled as per the requirements stated in the sampling plan.

The composite sample may be of considerable mass therefore sample size reduction may be necessary to aid transportation of a sample to the laboratory. Sub-sampling, if required, should be carried out so that the integrity of the sample is protected; so that final sample is representative of the field sample (and representative of the stockpile); and carried out in and on a clean protected area, using clean equipment to avoid contamination of the sample.

Sub-sampling should only be done in the field if a homogeneous sample can be produced.

Before the sub-sample is taken, the sample must be mixed. For ash, this is done by forming a conical heap. The material is then shovelled or scooped from the bottom of the heap to the top. This action is repeated at least twenty times in order to transfer all the material.

CEN/TR TR-15310-3 describes the techniques for sample size reduction, which are dependent on the sample mass: riffling is practical for producing sub-samples of less than 100kg and or quartering is suitable for producing sub-samples down to 1 kg.

Particle size reduction

Depending on the nature of the analysis that is required, the particle size of the sample may need further reduction. In these circumstances all items that would interfere with particle size reduction should be removed from the sample i.e. metal, cans, bricks etc. The weight of the items removed should be recorded so that the analytical result can be determined for the total mass of sample taken and not just the fraction analysed. If, after manual particle size reduction there is insufficient sample available for analysis, more sample may be required and added to the first sample. These could be treated as two bulk increments (CEN/TR 15310-3:2006 paragraph 12.3.4.2)

2.2 KEY STEP 2 TAKING THE SAMPLE IN ACCORDANCE WITH THE SAMPLING PLAN

Samples should be taken in accordance with the sampling plan. Any deviations from the sampling plan should be documented on the Sampling Record. An example of a sampling Record template is given in Appendix C.

Observations made during sampling should also be recorded. These can be useful when interpreting the results. Examples of the type of observations that should be made include:

- Details of incinerator loading/process type
- Type of waste relative to the ash sample
- Odours

2.3 KEY STEP 3 – TRANSPORTING THE SAMPLE TO THE LABORATORY

Duty of Care requirements apply to everyone involved in handling waste, including waste samples taken for analysis. You may be able to register for a D5 waste exemption if you meet the specified criteria for transporting waste samples for testing or analysis.

Samples should be transported in a manner that does not cause deterioration. It is advisable to check with the chosen analytical laboratory that the packaging, transportation and storage procedures are appropriate to protect the integrity of the sample. CEN/TR 14310-4 is a useful source of guidance on sample packaging, storage, preservation, transport and delivery. Requirements for these should be documented in the sampling plan.

2.3.1 Packaging and labels

The sample container opening should be of the appropriate size for the material to be packaged. The samples must be packed such that they are protected from potential reactions with the packaging or light, deterioration (perhaps through moisture loss or gain) or contamination.

The packaging should be of suitable size for transportation and reception by the analytical laboratory. Consideration should be given to health and safety restrictions that could influence the size of the packaging.

Analytical laboratories should be able to provide advice on requirements recommended for designated tests. For example: Annex A of BS EN 1519:2007 gives a list of recommendations for the packaging, transport and storage requirements for samples destined for Loss on Ignition (LOI) tests. Key recommendations are:

- Air tight container of amber glass or plastic with no migration of constituents e.g. polypropylene or polyethylene.
- Transport in dark and cool conditions

All sample containers should be marked with a unique identifier that is recognisable to the sampler and the laboratory. This should be done in the manner identified in the sampling plan. A chain of custody form (see example in Appendix D) should be completed for each sample and sent with the sample to the analytical laboratory.

2.3.2 Preservation

Depending on the nature of the material, the time between sampling and analysis should be minimised to avoid deterioration or contamination of the sample. It is advisable to discuss and agree the requirements with the analytical laboratory prior to sampling.

3 OVERVIEW OF ANALYTICAL METHODS

The analytical methods and the level of uncertainty for the determinands required depend on the objectives of the sampling plan. This section provides an overview of determinands for various purposes.

When the objective of sampling is for characterisation or compliance purposes, our preference is that the analytical laboratory (whether in-house or external provision) should be accredited by the United Kingdom Accreditation Service (UKAS) (or equivalent accreditation) to BS EN ISO/IEC 17025 *General requirements for the competence of testing and calibration laboratories* for the scope of the work.

When the objective of sampling is to classify the waste under the List of Wastes, through assessment of whether or not it is a hazardous waste, then the approach must be consistent with that set out in Technical Guidance WM3.

3.1 PREPARATION OF LABORATORY SAMPLE

The sampling plan documented the details for taking a representative sample and transporting that sample to the laboratory. The laboratory now needs to prepare test portions that are representative of the field sample, for analysis.

BS EN 15002 Preparation of test portions from the laboratory sample should be followed to obtain suitable test portions for most analytical procedures. *BS EN 14735 Preparation of waste samples for ecotoxicity tests* describes the necessary preparatory steps, specific to carrying out ecotoxicity tests on wastes.

Analytical procedures may define the number and size of the test portion and how they must be preserved. Analytical procedures may also define requirements for drying or particle size reduction. Example: leaching behaviour test BS EN 12457-3. The test is carried out on material with a particle size below 4mm, achieved initially by sieving and then, if necessary, by crushing; compositional analysis using BS EN 15309 is carried out on a dry sample.

Guidance on the choice of sample treatment techniques (such as drying, phase/fraction separations and particle size reduction) is given in Annex A of BS EN 15002. Usefully, guidance is given on when not to use as a particular technique, as well as when to use it. Further information on requirements for test portions from aqueous and solid laboratory samples are given in Annex D of this standard.

3.2 ANALYTICAL METHODS

If the test and analytical standard is not dictated by mandatory requirements then the standards should be used in the following order of priority as given in the European Commission's IPPC BAT Reference Document on the General Principles of Monitoring:

- Comité Européen de Normalisation (CEN)
- International Standardisation Organisation (ISO)
- National standards

For the assessment of hazardous properties, guidance on how to assess a waste and analytical methods is provided by technical guidance WM3.

A competent laboratory will be able to give advice on which analytical and test methods should be chosen to meet the sampling objective.

Appendix E lists the main determinands that are required by the Industrial Emissions Directive. Leaching behaviour tests are included in this list for assessments of the potential to cause pollution when determining disposal routes. The analytical methods given are taken from those published by CEN /TC 292 ‘Characterisation of Waste’ (except total dioxins and protein). The selected standard must be appropriate for the intended application.

Tables C3, C4 and C5 of the ‘Guidance on sampling and testing of wastes to meet landfill waste acceptance procedures’ lists test methods and their principles for general properties and digestion of waste, and eluate analysis. Table C3, C4 and C5 also include references to UK equivalent ‘Blue book’ methods.

For tests carried out on eluates, the choice of analytical technique will depend on the achievable limit of detection.

4 SAMPLE RECORDS

To have traceability there must be records and documentation. All documentation must be traceable to the Sampling plan.

BS EN 14899 lists the following documents, examples of which are given in Annexes A and B of the Standard:

- **Sampling plan** – the instructions on how the take the sample. Completed by the waste producer in consultant with interested parties.
- **Sampling Record** – a record of changes to the agreed Sampling plan. Completed by the sampler.
- **Chain of Custody form**. Completed by the sampler.
- **Sample analysis request form**. Completed by the sampler.

Reporting requirements are given in each of the BS EN standards listed in Table 3. Test reports must contain details of sample preparation as well as the reference to the Sampling plan.

In addition to test results, the test report shall include at least the following information:
- Description and identification of the laboratory sample
- Which processes, procedures and apparatus were used
- Results of the determination expressed in the appropriate units
- Any details not specified in the standard or which are optional, and any other factors which may have affected the results
- Date of receipt of laboratory sample and dates(s) when the test was carried out
- Reference to the standard or procedure followed

Note: Samples of waste are waste, and are subject to Duty of Care and, where relevant, hazardous waste controls when transferred, moved, or received.

APPENDIX A: EXAMPLE SAMPLING PLAN

SAMPLING PLAN REFERENCE:

GENERAL INFORMATION

Sampling plan completed by:	
On behalf of:	
Involved parties:	Project manager & waste producer - industry
	End user -
	Regulator -
	Analyst -
Sampling to be carried out by:	
Name of sampler (company):	

IDENTIFY SAMPLING OBJECTIVES AND DEFINE TECHNICAL GOALS

Objectives

Technical goal

LOCATION

Sampling location (address):	
Source and origin of material (e.g. form and nature of arising):	**Form and nature of arising:**

GENERIC LEVEL OF TESTING REQUIRED

SAMPLING APPROACH (WITH JUSTIFICATION) – PROBABILISTIC OR JUDGEMENTAL

Sampling approach

Justification

MATERIAL

Process/activity producing the material:	
Causes of variability (spatial and temporal) in the waste stream (assists judgement of population and sub-set to be sampled):	

ANALYTICAL LABORATORY

Company details:	
Contact:	

ANALYSIS REQUIRED

Identify target constituents to be tested and LOD:

Determinand:	Relevant legislation:	LOD (specify units):

SPECIFIC MATERIAL	
Type of material:	•
Relevant characteristics:	•
Relevant tests (and duplicates)	•
ACCESS, HEALTH AND SAFETY	
Identify access problems or restrictions that may affect sampling programme:	•
Identify health and safety precautions (ref ISO 10381-3:2001 *Soil quality. Sampling. Guidance on safety*):	
SAMPLING METHODOLOGY	
Specify sampling pattern:	
Define place and point of sampling:	
Specify detailed sampling location (i.e. specific stockpile including description of site location; stockpile reference number; product type etc).	
Sample 1	
Sample 2	
Sample 3	
Sample 4	
Specify time period of production (waste produced over specified period at this location) to define population (i.e. dates stockpile was started and completed):	
Sample 1	
Sample 2	
Sample 3	
Sample 4	
Define population/sub-population (volume) to be sampled (i.e. stockpile size sampled):	
Sample 1	
Sample 2	
Sample 3	
Sample 4	
Specify persons (witness) to be present (name/address): Details also to be recorded on Chain of Custody documentation	
Specify use of individual or composite samples:	
Identify equipment:	
Specify no. increments/samples to be collected (CEN/TR 15310-1):	

Specify increment size/sample size (CEN/TR 15310-1):	
Specify any provision to be made for replicate samples (required for 5 samples as specified):	
Specify any requirements for sample reduction:	
Detail requirements for on-site determinations:	
Identify sample coding methodology:	
Identify safety precautions:	
Identify anticipated restrictions or limitations that may impact on reliability of data:	
SUB-SAMPLING	
Detail procedure (CEN/TR 15310-3):	
PACKAGING, PRESERVATION, STORAGE, TRANSPORT REQUIREMENTS (CEN/TR 15310-4)	
Packaging (size; shape; material considering risk of adsorption/reaction; cleaning):	
Preservation (samples shall be packed and transported in such a way that their condition at the time of sampling is preserved)	
Storage:	
Transport (A chain of custody form to be completed and sent with each sample):	
Delivery date:	

APPENDIX B: FIELD SAMPLING TECHNIQUES

TABLE B1 SAMPLING POWDERS, GRANULES AND SMALL GRANULES

Scenario	**Sampling small static volumes from hoppers, heaps and silos**		**Sampling large static volumes from hoppers, heaps and silos**	
Statistical approach	Probabilistic sampling	Judgemental sampling	Probabilistic sampling	Judgemental sampling
Apparatus	Core sampler	scoop	Vacuum probe or sampling auger/scoop	
Preparation for sampling	Obtain safe access to the material			
Sampling methodology	1. Using a core sampler to cut out the required shape and amount of material as per the SP[7]. 2. Place the sample in a sample container.	1. Dip the scoop in the material at the area defined in the SP. 2. Withdraw the scoop and level off the material so there is none above the sides of the scoop. 3. Transfer the sample into a sample container.	1. Push the vacuum probe or sampling auger/scoop through the material in the identified direction taking a series of individual samples until the traverse is complete. 2. Combine the individual samples to give a directional sample. 3. Transfer the sample into a sample container.	1. Push the vacuum probe or sampling auger/scoop through the material to the identified point and extract the sample as per the SP. 2. Transfer the sample into a sample container.

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[7] SP in Table B1 and B2 means sampling plan

TABLE B2 SAMPLING COARSE OR LUMPY SOLID MATERIALS

Scenario	**Sampling small volumes from a bag, keg or drum**		**Sampling large stockpiles**	
Statistical approach	Probabilistic sampling	Judgemental sampling	Probabilistic sampling	Judgemental sampling
Sampling methodology	1. Empty the material onto a clean surface 2. Using a straight edge, make a cut into the material at the spot identified in the SP. 3. Move the material to one side away from the pile. 4. Make a parallel cut into the remaining material as identified in the SP. 5. Take the material between the parallel cuts to form the directional sample. 6. Transfer the sample to a sample container.	1. Dip the scoop in the material at the spot identified in the SP. 2. Take the required sample size, as identified in the SP. 3. Transfer the sample into a sample container.	1. Flatten out the stockpile and collect samples across the length and breadth of the material as specified in the SP. 2. Place individual increments into a new pile, mix with the digger and repeat until the sample volume can be handled manually. 3. Transfer the sample into a sample container.	1. Collect individual samples from the specific part of the stockpile as detailed in the SP. 2. Transfer the sample into a sample container.

APPENDIX C: EXAMPLE SAMPLING RECORD (ADOPTED FROM ANNEX A BS EN 14899:2005)

SAMPLING RECORD	
Sampling plan reference:	
Sample Code: (Reflect site location, material type and date of collection)	
Date and time of sampling:	
Signature of Sampler:	
Other persons present:	
GENERAL INFORMATION	
Waste Producer: Contact:	Client (company): Contact:
Location of sampling:	Carried out by (company): Sampler:
MATERIAL	
Type of Material:	Estimate of moisture content:
Description:(colour, odour, consistency/ homogeneity/ grain size – uniform or diverse)	
SAMPLING METHODOLOGY	
Describe/ define batch or consignment sampled:	
Place and point of sampling:	
Access problems that effected areas or volumes of material sampled:	
Safety measures taken:	
Procedure (describe procedure adopted):	
Equipment used:	
Number of increments/samples collected:*	
Increment size/sample size:*	
Observations during sampling:	
Details of on-site determinations: (if undertaken complete field record sheet and append to Sampling Record)	
SUB-SAMPLING & PRE-TREATMENT	
Identify location: e.g. on-site or fixed laboratory facility (describe whether open air or enclosed)	
Procedure:	
PACKAGING, PRESERVATION, STORAGE AND TRANSPORT DETAILS	
Packaging:	
Preservation:	
Storage:	
Transport:	
DEVIATIONS FROM SAMPLING PLAN	
Detail:	
DELIVERY TO ANALYTICAL LABORATORY	
Company:	Delivery Date:
Received by:	Signature:

APPENDIX D: EXAMPLE CHAIN OF CUSTODY FORM

A copy of this sheet is to accompany the sample at all stages

LIST ANALYSIS REQUIRED

SECTION 1 (to be completed by Sender)

SAMPLE IDENTIFICATION NUMBER -----------------

SITE NAME --- PERMIT No --------------

SAMPLE DATE ------------------------ SAMPLE TIME -------------------------

SAMPLE DESCRIPTION ---

SAMPLER NAME ------------------------- SIGNATURE ----------------------------

SECTION 2 (to be completed by the Sender)

DATE SENT ---------------------------

RECORDED DELIVERY NUMBER ------------------------------

SECTION 3 (to be completed by Laboratory)

DATE RECEIVED ----------------------

IS SAMPLE PACKAGING DAMAGED? YES / NO

RECEIVERS NAME ------------------------------ SIGNATURE ---------------------------------

APPENDIX E: ANALYTICAL AND LEACHING BEHAVIOUR TESTS

Determinand	Analytical method	Comments
Dry Matter Content	• EN 14346 Calculation of dry matter by determination of dry residue or water content.	• This is based on a method for drying at 105 C to constant mass.
Loss on Ignition (LOI)	• BS EN 15169 Determination of loss on ignition in waste, sludge and sediments.	• Largely used for assessing the organic matter content. Note that: • Elementary carbon or any volatilisation or chemical reaction of inorganic compounds will also be included in the loss on ignition value. • Whether the determination is carried out on a dry matter or not will depend on whether there are significant amount of volatile matter or not. • For some materials it may be convenient to carry out the determination of both dry residue and LOI in successive operations in the same crucible.
Total Organic Carbon	• BS EN 13137 Determination of total organic carbon (TOC) in waste, sludges and sediments.	
Metals	• BS EN 13657 Digestion for subsequent determination of aqua regia soluble portion of elements. • BS EN 13656 Microwave assisted digestion with hydrofluoric (HF), nitric (HNO3) and hydrochloric (HCl) acid mixture for subsequent determination of elements.	Solutions produced by this method are suitable for analysis by atomic absorption spectrometry (AAS), inductively coupled plasma emission spectrometry (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS).

Total Dioxins	• Method based on EPA 1613	• This method gives a number of scenarios for extraction, analysis etc. The laboratory should use a method based on EPA 1613 that has been accredited by an external body such as UKAS
Dioxin like PCBs	• BS EN 15308 Characterization of waste. Determination of selected polychlorinated biphenyls (PCB) in solid waste by using capillary gas chromatography with electron capture or mass spectrometric detection	
Elemental composition	• BS EN 15309 Determination of elemental composition by X-ray fluorescence.	• Describes two procedures: a quantitative and semi-quantitative determination of major and trace element concentrations
Total Protein	• Aqueous extraction followed by PITC derivitisation and subsequent analysis by HPLC	
Leaching behaviour tests	• BS EN 12457-1 One stage batch test at a liquid to solid ratio of 2 litres/kg for materials with particle size below 4 mm • BS EN 12457-2 One stage batch test at a liquid to solid ratio of 10 litres/ kg for materials with particle size below 4 mm • BS EN 12457-3 (this employs a two stage batch test at 2litres and 8 litres / kg) for materials with particle size below 4 mm • BS EN 12457-4 One stage batch test a liquid solid ratio of 10 litres/kg for materials with particle size below 10 mm • CEN/TS 14405 Upflow percolation test (under specified conditions) • BS EN 14997 Influence of pH on leaching with	• This test is mandatory for assessing compliance with the waste acceptance criteria

	continuous pH-control • CEN/TS 15364 Acid and base neutralisation capacity test	

APPENDIX F: ASH AND PARTICULATE TESTING METHODS AND FREQUENCY FOR ANIMAL REMAINS INCINERATION, INLCUDING MBM (FOR ANALYSIS OF CARBON AND TOTAL PROTEIN CONTENT)

F1 INTRODUCTION

Appendix F is specific to the objective of testing residues following incineration of suspected BSE cattle. If your incinerator is not permitted to incinerate suspected BSE cattle, this appendix does not apply to you.

This appendix preserves the guidance issued in the draft v5 version of this document for the sampling of ash following incineration of suspected BSE infected cattle. Earlier versions of this document provided sampling guidance; including sampling frequencies, from all new plant and existing installations that were authorised to incinerate BSE suspects or cattle from the Over Thirty-Month rule (OTM). This rule was closed in November 2005. However, the sampling methodology may be appropriate for any future testing of residues from incinerators burning category 1 material (Article 8a(i)(ii) (Regulation 1069/2009/EU Animal By-Products Regulation).

The purpose of analysis is to confirm that the assumptions made in the risk assessment concerning releases to air are correct: that the risks from burning animal remains in incinerators are negligible if they are operated under the sector guidance notes on incineration. Therefore the sampling of IBA is a surrogate for the releases to air and is only carried out if sampling of the post APC is not possible (e.g. because of there being no abatement).

- ✓ Sampling is from processes permitted to incinerate animal remains
- ✓ Residues to be sampled are IBA and APC
- ✓ Analysis to be carried out for total organic carbon and protein
- ✓ Analysis to be carried out by UKAS accredited (or equivalent) laboratories
- ✓ Stack particulate samples to be taken by MCERTs certified personnel

The Operator will prepare the Sampling plan in accordance with these guidelines and submit the Sampling plan to Regulatory Officer for agreement. Sections 3, 4 and 5 of this guidance document support the preparation of a sampling plan.

F2 SAMPLING OBJECTIVE AND TECHNICAL GOALS

The sampling plan and accompanying risk assessment will propose how sampling is to take place to meet the following objectives:

(i) samples of ash are collected in a reasonably consistent manner
(ii) representative samples of ash are obtained as far as is reasonably possible by minimising sampling bias
(iii) sample preparation requirements minimise the sampling handling/preparation required by analytical laboratory staff

The technical goal is to take a representative sample from a 24 hour burn so that a Level 2 or ‘compliance’ sampling regime is achieved.

F3 SAMPLING FREQUENCY AND ANALYTICAL REQUIREMENTS FOR IBA AND APC

There are four Stages of sampling:

STAGE 1 **One sample per day** over a one week period from each incinerator (i.e 7 consecutive samples)

Sampling at Stage 1 should continue in subsequent weeks until results of the analyses are available and an assessment of the data can be made. Assessments should not be made on anything less than a complete week of data which has been collected in accordance with Stage 1 of this Protocol.

STAGE 2 **One sample per week over a one month period** from each incinerator (i.e. 4 consecutive samples)

STAGE 3 **One sample per month for three months** from each incinerator (i.e. 3 consecutive samples)

STAGE 4 **One sample per quarter for one year** from each incinerator (i.e. 4 consecutive samples)

If the criteria for total protein are met for **all** samples at each Stage, sampling frequency may be reduced to the next Stage. However, if at any time, samples exceed the action levels (Section F4) for total protein during Stages 2, 3 or 4, or the Regulatory Officer considers it necessary, for example unusual combustion conditions, sampling frequency should revert back to the previous Stage. If the failure is by more than twice that of the action level then a thorough investigation should be carried out.

TABLE F1 SAMPLING FREQUENCIES AND ANALYTICAL REQUIREMENTS FOR IBA AND APC

	No. of samples to be taken	Mass of sample (max)	Analysis required	**IBA**	**APC**
STAGE 1	One sample per day	10 g	Total Organic Carbon (TOC)	✓	✓
		10 g	Protein		✓
STAGE 2	One sample per week	10 g	Total Organic Carbon	✓	✓
		10 g	Protein		✓
STAGE 3	One sample per month	10 g	Total Organic Carbon	✓	✓
		10 g	Protein		✓
STAGE 4	One sample per quarter	10 g	Total Organic Carbon	✓	✓
		10 g	Protein		✓

APC samples are only required if a process has an abatement plant – generally a requirement of Part A processes only. However, if the process does not have an abatement system that can be easily sampled, then IBA will require to be sampled for protein at the frequency given in the APC section of Table F1

- All analysis is to be done in duplicate

F3.1 Plant loading

Care must be taken to ensure that the ash sample taken is representative of the specified loading rate. The Regulatory Officer should ensure that there is not more than one change of loading rate in any one day of the commissioning period.

- ⇒ At least one of the sets of samples from Stage 1 should be collected under "normal" plant throughput
- ⇒ One of the remaining sets of samples should be collected during maximum plant throughput.
- ⇒ All samples should be collected at the burnout rate initially proposed by the Operator. If these fail the action levels detailed below, then burnout time will need to be amended and sampling repeated.

F4 SAMPLING FREQUENCY AND ANALYTICAL REQUIREMENTS FOR PARTICULATE FROM THE STACK

All samples are to be taken by the operator, using MCERTS accredited personnel. However, the sampling does not need to meet the requirements of BS EN 13284.

The sample should be a minimum mass of 50mg. However, if insufficient material has been collected after 10 hours of sampling it will have to be assumed that the results of the bag filter samples are representative of the particulate samples.

- All analysis is to be done in duplicate

TABLE F2 SAMPLING FREQUENCIES AND ANALYTICAL REQUIREMENTS FOR PARTICULATES FROM THE STACK

Sampling frequency	Mass of sample	Analytical requirements
One sample taken during stage 1	50 g	total protein
One sample taken three months after first sample	50 g	total protein
Annual sampling thereafter	50 g	total protein

F5 LIMIT AND ACTION VALUES

Substance	Limit/action value	Preferred analytical methods
Total organic carbon	3 %	Remove inorganic Carbon and subsequent analysis by elemental analyser
Total protein	5 mg/100g sample	PITC derivitisation and subsequent analysis by HPLC

The following section F6 separately discusses the sampling requirements for IBA, APC and particulates from the stack

F6 SAMPLING REQUIREMENTS

- Health and safety is not covered in this guidance as any requirements must be identified in a site risk assessment
- Gloves should be worn to minimise the risk of sample contamination
- A clean dedicated working area for sample preparation and packaging should be available which is free from sources of contamination.
- An electrical or mechanical grinder, capable of milling ash to a fine powder will be required.
- Sampling equipment should be washed and dried after each use and stored in a clean, dedicated area.

F6.1 SAMPLING APPROACH – IBA

It is suggested that a number of samples of ash should be taken from either the de-ashing process or from the ash receptacle. The details of the sampling should be contained in the site sampling plan. These samples should be discrete and representative, from a burn out period of not more than 24 hours (or other time period as agreed by the Regulatory Officer) to give approximately 1 kilogram of sample.

This sample is further divided by coning and quartering, as per method given below.

Coning and Quartering of sample

F6.1.1 Place sample in a deep, flat-bottomed tray and mix.

F6.1.2 Form into a cone then flatten until an equal shallow depth of ash is obtained.

F6.1.3 Divide this into 4 equal portions using a fine edged rod.

F6.1.4 Remove two diagonally opposite portions for further coning and quartering.

F6.1.5 Discard the other two portions

F6.1.6 Repeat 5.2.1 to 5.2.3

F6.1.7 Remove two diagonally opposite, portions (A) and remove for particle size reduction

F6.1.8 Mill portion A to a fine powder

F6.1.9 Divide portion A in half.

F6.1.10 Place one half in storage (this sample to be retained until Regulatory Officer agrees it may be discarded).

F6.1.11 The remaining half is to be divided for analysis to satisfy the requirements of Table F1 and / or F2 in the Protocol and sent in accordance with Section 3.3.

If the IBA obtained from the incineration process is already of a finely divided nature, then on agreement with the Regulatory Officer, steps F6.1.1 to F6.1.8 above may be omitted.

F6.2 SAMPLING APPROACH – APC

F6.2.1 A sample taken at this stage is assumed to be of a finely divided nature, homogeneous and representative. There is no need for this to be ground further. A final sample can be obtained by following the procedure in 6.1 above from 6.1.9 to 6.1.11.

F6.2.2 If the sample is not of a finely divided nature, a primary sample should be taken in accordance with the site sampling plan. The process described in F5 should then be followed.

F6.3 SAMPLING APPROACH - PARTICULATE FROM THE STACK

F6.3.1 The analysis for protein is extremely sensitive so contamination of the sample can easily occur. It is important that at all stages the filter and unused filter are handled to ensure that contamination does not occur therefore, gloves must be worn and filters handled using forceps.

F6.3.2 If the stack operates at between 0 - 30 mg/m^3 particulate, the sampling time needs to be adjusted so that a mass of around 50 mg particulate is collected or until the filter blinds. In the case of very low particulate levels, sampling may take up to 10 or 12 hours.

F6.3.3 In addition to the filter from the stack an unused filter should also be submitted for analysis. This will act as a control.

F6.3.4 If practical, the sample is to be collected isokinetically at suitable points. In the case of a very low level or particulate in the stack, it is suggested that single point sampling take place. The average velocity should be set to achieve near to isokinetic conditions and the sampling time extended up to 12 hours.

F6.3.5 Filters should be of glass fibre and handled only whilst wearing gloves and with forceps.

F6.3.6 The stack filter and control filter should be placed in separate petri dishes and sent in accordance with Section 3.3.