# Characterization of waste — Sampling of waste materials —

Part 5: Guidance on the process of defining the sampling plan

ICS 13.030.10; 13.030.20



#### National foreword

This Published Document was published by BSI. It is the UK implementation of CEN/TR 15310-5:2006.

The UK participation in its preparation was entrusted by Technical Committee B/508, Waste management, to Subcommittee B/508/3, Characterization of

A list of organizations represented on B/508/3 can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 29 December 2006

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ISBN 0 580 49837 9

#### Amendments issued since publication

Amd. No.	Date	Comments

## TECHNICAL REPORT RAPPORT TECHNIQUE TECHNISCHER BERICHT

**CEN/TR 15310-5** 

November 2006

ICS 13.030.10; 13.030.20

#### **English Version**

#### Characterization of waste - Sampling of waste materials - Part 5: Guidance on the process of defining the sampling plan

Caractérisation des déchets - Prélèvement des déchets -Partie 5 : Guide relatif au processus d'élaboration d'un plan d'échantillonnage Charakterisierung von Abfall - Probenahme - Teil 5: Verfahren zur Aufstellung eines Probenahmeplans

This Technical Report was approved by CEN on 21 February 2006. It has been drawn up by the Technical Committee CEN/TC 292.

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#### **Foreword**

This Technical Report (CEN/TR 15310-5:2006) has been prepared by Technical Committee CEN/TC 292 "Characterization of waste", the secretariat of which is held by NEN.

This Technical Report has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This Technical Report is one of a series of five Technical Reports dealing with sampling techniques and procedures, and provides essential information and instructions for the application of the EN-standard:

EN 14899 Characterisation of waste - Sampling of waste materials - Framework for the preparation and application of a Sampling Plan.

The principal component of the EN Standard is the mandatory requirement to prepare a Sampling Plan. This EN 14899 standard can be used to:

- produce standardised sampling plans for use in regular or routine circumstances (i.e. the elaboration of daughter/derived standards dedicated to well defined sampling scenarios);
- incorporate specific sampling requirements into national legislation;
- design and develop a Sampling Plan on a case by case basis.

The Technical Reports display a range of potential approaches and tools to enable the project manager to tailor his sampling plan to a specific testing scenario (i.e. a 'shop shelf' approach to sampling plan development for waste testing). This approach allows flexibility in the selection of the sampling approach, sampling point, method of sampling and equipment used.

In practice, confusion can arise when translating the objective of the testing programme, which is often couched at a relative abstract level (e.g. 'the waste needs to be assessed to fulfil the demands of waste regulation') into an unambiguous technical instruction in the Sampling Plan, that will provide data to meet that objective (e.g. 'the mean concentration of each truck load should comply with a specified concentration level'). This Technical Report attempts to clarify the 'grey area' between the definition of an overall testing objectives and the definition of the practical Sampling Plan. It specifically provides guidance on the policy aspects that may be relevant for defining the objective of the testing programme, and how this will define the technical methods that can be used to prepare the Sampling Plan.

#### Introduction

Wastes are materials, which the holder discards, or intends or is required to discard, and which may be sent for final disposal, reuse or recovery. Such materials are generally heterogeneous and it will be necessary therefore to specify in the testing programme the amount of material for which the characteristics of interest need to be defined. The testing of wastes allows informed decisions to be made on how they should be treated (or not), recovered or disposed. In order to undertake valid tests, some sampling of the waste is required.

The principal component of the standard EN 14899 is the mandatory requirement to prepare a Sampling Plan, within the framework of an overall testing programme as illustrated in Figure 1 of EN 14899:2005. This standard can be used to:

- produce standardised sampling plans for use in regular or routine circumstances (i.e. the elaboration of daughter/derived standards dedicated to well defined sampling scenarios);
- incorporate specific sampling requirements into national legislation;
- design and develop a Sampling Plan on a case by case basis.

The development of a Sampling Plan within this framework involves the progression through three steps or activities.

- 1) Define the Sampling Plan;
- 2) Take a field sample in accordance with the Sampling Plan;
- 3) Transport the laboratory sample to the laboratory.

This Technical Report provides information to support Key Step 1 of the Sampling Plan process map and describes the selection of sampling approach that can be used in the recovery of a sample for a wide variety of waste types and arisings. Specifically CEN/TR 15310-1 provides information to support 4.2.7 (select sampling approach) of the Framework Standard. Due consideration and selection of statistical criteria is of key importance in the production of a Sampling Plan as it provides the sole means of ensuring that, wherever possible, the type and number of samples taken will address a clearly identified objective and will provide results that achieve a tolerable level of reliability.

In the process of defining the Sampling Plan (step 1 in Figure 1 of EN 14899:2005), the objective of the testing programme is translated into specific and concrete technical instructions for the sampler. Using these instructions the sampler will take the type and number of samples that are adequate to meet the objective of the testing programme, ultimately providing the decision maker with the required information on the waste material under investigation.

The process of defining the Sampling Plan, which takes into consideration both policy and technical requirements to produce technical instructions to the sampler, is therefore a fundamental step in sampling of a waste material.

In practice, problems arise when translating the objective of the testing programme, which is couched at a relative abstract level (e.g. 'the waste needs to be assessed to fulfil the demands of waste regulation') into a technical instruction that corresponds with that same objective (e.g. 'the mean concentration of each truck load should comply with a specified concentration level'). There is a 'gap' between the definition of the need to evaluate the waste material and the technical methods that should be applied in order to make an adequate evaluation possible.

This Technical Report aims to 'bridge the gap' between the chosen objective of the testing programme in policy terms, and that same objective defined in technical terms for sampling. It provides information and guidance on the <u>process</u> of defining a Sampling Plan. It deals specifically with the policy aspects relevant for defining the objective of the testing programme, and provides guidance for the definition of the Sampling Plan.

In addition to the main body of the Technical Report, an annex provides worked examples of Sampling Plans for a number of frequently occurring waste materials and situations in which these waste materials arise. Thereby the examples provide background to the main body of this Technical Report.

These examples clarify the process of defining the Sampling Plan. A number of assumptions have been made to produce each individual example, and therefore – although the examples represent actual daily practice – they are case specific and are not necessarily directly applicable to other similar generic situations.

This Technical Report is written for two distinctive groups of users:

- policy makers involved in sampling. For example, people working for the central, regional or local authority, government or administration, the management of a company involved in the production or disposal of waste, etc. Essentially, these people are, directly or indirectly, involved in making policy decisions that are based on the technical information gathered through sampling. Their interest lies in the requirement for testing a waste material to gain (general) knowledge about the waste material or to comply to national, regional or local legislation. They have in general no technical knowledge of sampling, but are responsible for making the right choices. They therefore need help to understand the definition of the testing programme in technical terms, in order to be able to judge if the suggested testing programme is adequate for the purpose.
- sampling specialists (specifically the project manager). These are the people who have to translate the objective of the testing programme, as defined by the policy maker, into a technically unambiguous Sampling Plan that will instruct the sampler on what to do in the field. The project manager is therefore confronted with the problem that not only technical information is necessary for the definition of the Sampling Plan, but also policy information.

Incorporated within the text of this Technical Report is an example. Each individual step of the process of defining the Sampling Plan made in this Technical Report is illustrated by the same step in this example. The example is meant to clarify the text of the individual paragraphs in more practical terms.

#### Example of a waste to be tested

Due to the incineration of residuals from paper production, filter dust is collected. The dust is trapped in an air filter unit and put into stockpiles before transport to the landfill. In order to allow land filling, the concentrations of a number of key constituents should comply with the acceptance criteria of that landfill. Therefore the waste must be sampled.

This Technical Report should be read in conjunction with the Framework Standard for the preparation and application of a Sampling Plan as well as the other Technical Reports that contain essential information to support the Framework Standard. The full series comprises:

- EN 14899 Characterization of waste Sampling of waste materials Framework for the preparation and application of a Sampling Plan;
- CEN/TR 15310-1, Characterization of waste Sampling of waste materials Part 1: Guidance on selection and application of criteria for sampling under various conditions;
- CEN/TR 15310-2, Characterization of waste Sampling of waste materials Part 2: Guidance on sampling techniques;
- CEN/TR 15310-3, Characterization of waste Sampling of waste materials Part 3: Guidance on procedures for sub-sampling in the field;

#### CEN/TR 15310-5:2006

 CEN/TR 15310-4, Characterization of waste – Sampling of waste materials – Part 4: Guidance on procedures for sample packaging, storage, preservation, transport and delivery;

CEN/TR 15310-5, Characterization of waste – Sampling of waste materials – Part 5: Guidance on the process of defining the Sampling Plan.

The Technical Reports contain procedural options (as detailed in Figure 2 of EN 14899:2005) that can be selected to match the sampling requirements of any testing programme.

#### 1 Scope

This Technical Report provides guidance on process of defining of a Sampling Plan based on the objective of the testing programme. It specifically deals with the strategic decisions that are needed, based on the sampling objective.

NOTE 1 Given the great variety of waste types, sampling situations and objectives, this Technical Report cannot provide definitive instructions that cover all scenarios. Instead, it discusses the basic statistical approach to be followed, and provides statistical tools that can be applied to determine the amount and type of sampling (e.g. number of samples and sample size) in any given situation to achieve results of adequate reliability (i.e. precision and confidence).

NOTE 2 The document provides considerable detail on current best practice, but is not exhaustive.

NOTE 3 To clarify the text, the document provides a number of worked examples.

#### 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 13965-1:2004, Characterization of waste - Terminology - Part 1: Material related terms and definitions

EN 13965-2:2004, Characterization of waste - Terminology - Part 2: Management related terms and definitions

#### 3 Terms and definitions

For the purposes of this Technical Report, the following terms and definitions given in EN 13965-1:2004 and EN 13965-2:2004 and the following apply.

#### 3.1

#### background information

information that is essential to understanding the setting of sampling

NOTE Among others, it consists of information on the production process of the waste, the nature of the waste, policy aspects and compliance levels set in legislation.

#### 3.2

#### basic characterisation

sampling that has the goal to describe the character or quality of a population of waste

#### 3.3

#### compliance testing

process of testing whether sample values meet a pre-defined set of criteria

#### 3.4

#### composite sample

two or more increments mixed together in appropriate portions either discretely or continuously (blended composite sample), from which the average value of a discrete characteristic may be obtained [ISO 11074:2005]

#### 3.5

#### confidence interval

interval within which the value of a particular population parameter may be stated to lie at a specific confidence level. The bounds of the confidence interval are termed the upper and lower confidence limits

#### 3 6

#### confidence level

value 100(1-α) of the percentage probability associated with a confidence interval (after ISO 3534-1)

#### 3.7

#### constituent

an essential part (component, element) of the waste

#### 3.8

#### decision maker

party that makes a decision based on the results of the testing programme

NOTE In most cases the regulator is the decision maker, but it can also be the waste producer or waste manager.

#### 3.9

#### field sample

quantity (mass or volume) of material obtained through sampling without any sub-sampling

#### 3.10

#### increment

individual portion of material collected by a single operation of a sampling device which will not be analysed / investigated as a single entity, but will be mixed with other increments in a composite sample prior to analysis

#### 3.11

#### involved parties

individuals who have an interest in the results of the sampling and who should therefore be involved in the (iterative) process relating to the exchange of information regarding the testing programme

#### 3.12

#### laboratory analyst

person conducting the analysis of the laboratory sample

#### 3.13

#### laboratory sample

sample sent to or received by the laboratory (IUPAC)

#### 3.14

#### legislator

body responsible for the definition of the rules that should be obeyed

#### 3.15

#### objective

underlying motivation for investigating a (potential) waste material

#### 3.16

#### on-site verification

normally simple test to evaluate if the involved waste material is indeed the type of material expected

#### 3.17

#### overall population

totality of items

#### 3.18

#### population

totality of items under consideration [ISO 3534-1]

#### 3.19

#### policy maker

person working for the central, regional or local authority, government or administration, the management of a company

#### 3.20

#### project manager

person who is responsible for deriving and / or fulfilling the testing programme

#### 3.21

#### regulator

body responsible for controlling if the rules of the legislator are met

#### 3.22

#### reliability

extent to which a test measures consistently

NOTE For scaled scores, a reliability coefficient of 1.00 indicates a test that is perfectly reliable.

#### 3.23

#### sample

portion of material selected from a larger quantity of material [ISO 11074:2005]

NOTE The use of the term 'sample' should be avoided as far as possible as it does not imply to what step of the total sampling procedure it is related.

#### 3.24

#### sampler

person carrying out the sampling procedures at the sampling locality [ISO 11074:2005]

NOTE Tools and devices to obtain samples are sometimes also referred to as 'samplers'. In this case it is recommended to write 'sampling devices' or 'sampling equipment'.

#### 3.25

#### sampling plan

predetermined procedure for the selection, withdrawal, preservation, transportation and preparation of the portions to be removed from a population as a sample [ISO 11074:2005]

#### 3.26

#### scale

quantity (mass or volume), defined in space and / or time, of material represented by the sample and considered relevant for the assessment of the material

#### 3.27

#### sub-population

defined part of a population [ISO 3534-1]

#### 3.28

#### technical goals

objective translated into specific, measurable, action oriented, realistic, timely (SMART) goals

#### 3.29

#### testing programme

total sampling operation, from the first step in which the objectives of sampling are defined to the last step in which data is analysed against the objectives

#### 3.30

#### waste

material, which the holder discards, or intends or is required to discard, and which may be sent for final disposal, reuse or recovery

#### 3.31

#### waste manager

company or organisation that accepts the waste

#### 3.32

#### waste producer

company or organisation that produces the waste

#### 4 The process of defining the Sampling Plan

#### 4.1 General description of the process

The project manager is responsible for the process, which defines the Sampling Plan. The first step is to identify the parties that have an interest in the results of the sampling and to ensure their full participation.

The involved parties come from various backgrounds and may have conflicting interests. Supported by the project manager, they must reach agreement on the objective of the testing programme, the translation of this objective into realistic technical goals and the translation of these technical goals into unambiguous instructions for the sampler. The project manager then records these instructions in the Sampling Plan.

The objective of the testing programme determines, directly or indirectly, the desired level of information (e.g. basic characterization, compliance testing or on-site verification) and the desired reliability of the sampling results.

Technical goals include statistical terms like the characteristic to be determined (e.g. a percentile value), the population, the scale, the confidence level and confidence interval to be reached and technical terms like the constituents of the waste that are to be determined; the moment when; or location where; the waste will be sampled. Therefore part of these technical goals provide direct input for the Sampling Plan, while others (e.g. the scale, the confidence level) still have to be translated into practical terms like the type of sampling, the sampling pattern and location, the number of increments and samples and the sizes of increments and samples.

Commonly, the reliability of the results improves when the number of samples is increased. This invariably leads to higher sampling and analysis costs. In short, the heterogeneous character of waste invokes the necessity to balance the desired reliability with the financial input. In fact, balancing the reliability and costs, may well be the most important decision the involved parties have to make in the process of defining a Sampling Plan.

A draft of the Sampling Plan should be discussed with all involved parties. By doing so, the practical implications of the choices that were made in the process of defining and translating the objectives become clear. For practical reasons, unrealistic objectives may be subject to change.

The process of defining the Sampling Plan may well be an iterative process that is repeated several times before it results in an accepted final version of the Sampling Plan. The project manager should actively manage this process.

The process of defining the Sampling Plan is provided in Figure 1.

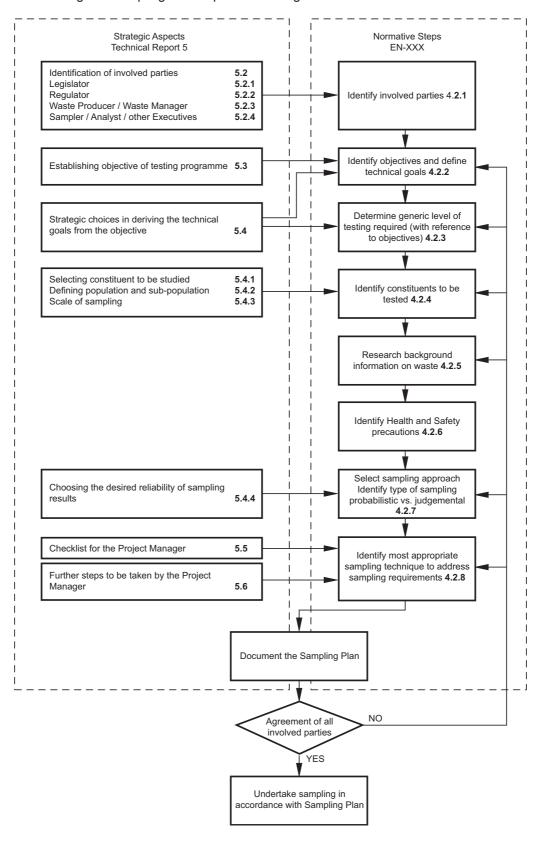


Figure 1 – The process of defining the Sampling Plan, providing information on the elements that are specified in the European Standard and this Technical Report

#### 4.2 Identification of involved parties

#### 4.2.1 General

It is the responsibility of the project manager to identify parties with an interest in the results of sampling and actually involve them in the decision process that is required to define the Sampling Plan. The involved parties are at a minimum the owners, users or buyers of waste and, directly or indirectly, the legislation to which the testing programme is related. Additionally, the project manager himself is also an involved party. The involved parties come from differing backgrounds and may have conflicting interests. Not all involved parties are easy to access. However, not all parties need to be personally represented.

The legislator<sup>1</sup> in particular will not be involved directly. The requirements of the legislator are usually defined in policy documents or directives. The regulator<sup>2</sup> may be involved, but the level of involvement will be decided by the regulator on a case-by-case basis, for example based on an evaluation of the risks that legislation is breached.

Identification of involved parties is not always easy. The same person that has more than one role may represent some parties (for example, in simple sampling programmes the client and sampler or regulator and legislator may be the same person. Furthermore, parties may not be aware that sampling is about to commence or may not understand the effects that sampling results could have on their situation. Failing to get input from all parties in the definition of the Sampling Plan may lead to resistance and loss of time in later phases of the testing programme.

The following roles can be distinguished in almost every testing programme:

- the legislator;
- the regulator;
- the company or organisation that produces the waste (waste producer);
- the company or organisation that accepts the waste (waste manager):
- the project manager and related personnel and organisations (like the sampler and the laboratory analyst).

#### **Example: Identification of the involved parties**

In the example, the waste originating from the incineration of residuals of paper production, might only be brought to the landfill if it meets the compliance levels defined for that landfill and specific waste.

In the example it is assumed that the regional government has defined compliance levels and can be seen as the legislator. In the involved country a generic Sampling Plan developed for sampling of heaps of homogeneous wastes is available and accepted for this type of waste material. So the national government may also be seen as an involved legislator. The responsibility for complying with the legislation is in this example however a responsibility of the regional government. The regional government therefore can be seen as the regulator. Other involved parties are the waste producer, the owner of the landfill and an independent consulting firm that is responsible for the sampling. The project manager is an employee of the consulting firm.

Before sampling the project manager directly consults the waste producer and the owner of the landfill. Directions on sampling are provided in the generic Sampling Plan, whilst more specific conditions are defined in both regional policy documents as well as in the contract for dumping this waste in the specified landfill. Therefore the legislator is involved indirectly.

<sup>1</sup> Legislator: the body responsible for the definition of the rules that should be obeyed.

Regulator: the body responsible for controlling if the rules of the legislator are met.

The regional government is aware of this waste stream to the landfill. The regulator can check the process when desired. The regulator is therefore not directly involved but decides if and when the land filling process will be checked.

#### 4.2.2 Legislator

In most cases, the legislator will be the European Commission, the national, regional or local government. However, company management can also have the role of legislator. A combination of legislators is therefore possible.

In case of international buying and selling of waste, the objectives of national legislation, European legislation and company management may all have to be included in the definition of the objective of the testing programme.

#### 4.2.3 Regulator

Most legislation authorises a regulator to base a decision on either the sampling results provided by the waste producer and / or buyer of the waste, or allow independent sampling by the regulator.

In complex situations, there may be several regulators (as many as there are legislators).

#### Example: multiple parties involved as regulator

The waste producer and the results of do the actual testing of the paper waste incineration dust that testing are delivered at the landfill together with the waste by the waste producer.

In addition to the testing results delivered by the waste producer, the owner of the landfill checks the waste periodically in order to check if the waste still complies with the specifications provided by the waste producer (not in a legislative definition of regulator, but self regulation).

In addition the regional government is the formal regulator from the perspective of the national and regional legislation. Thus, in this example, two of the involved parties act as regulator.

The regulator (but also other parties) could make demands on the quality, involvement and responsibilities of other parties like sampler and laboratory. Also, the legislator can prescribe procedures to safeguard the quality of sampling. These type of demands normally result in demands on certification or accreditation of the companies and / or personnel involved in the testing programme; see also 4.2.5.

#### 4.2.4 Waste producer and waste manager

The companies or organisations that produce and accept the waste have important interests in the outcome of the testing programme and should therefore be involved in the definition the objective of the testing programme and the translation of this objective into the Sampling Plan.

The waste producer and waste manager may well have conflicting interest but early discussion may resolve these or at least allow a negotiated compromise, well before committing time and resources to the testing programme.

Often the project manager is employed by one of these parties but in either case this does not imply that the waste producer or waste manager is the final decision maker.

#### 4.2.5 Sampler, laboratory analyst and other executives

The involved parties may also make demands concerning the quality of the parties that conduct the sampling and subsequent analysis of the samples. For example, by a system of certification or accreditation of sampler and analyst.

#### **Example: Certification or accreditation**

Member state A has determined on a national level that all waste sampling shall be done by an organisation that complies with a defined accreditation programme for the sampling of waste.

Recognised best practice is for the project manager to select the sampler, laboratory and other executives at an early stage of Sampling Plan development. In many cases, these involved parties have practical comments that improve the quality of the testing programme or positively influence the way the sampling should be conducted.

#### 4.3 Establishing the objective of the testing programme

In order to make sure that the testing programme is adequate, the underlying motivation for investigating a (potential) waste material must be clearly defined: what is the objective of the testing programme?

The objective of the testing programme can partly or fully consist of pre-conditions by international, national, regional or local legislation or regulation, however the waste producer or waste manager may also contribute to the objective setting process.

Examples of possible objectives of a testing programme are:

- to check the quality because of a change in ownership of the waste material (is this the type of waste we expect it to be?);
- to determine the (re) usability of the (waste) material;
- to assess the human health and / or environmental risks posed by the material;
- to determine the precautions that should be taken when the waste is disposed of in a landfill;
- etc.

The examples above provide objectives that are defined in very general terms. Basically, this type of objective provides no specific direction on how to evaluate the waste through sampling and analysis, as these objectives are couched to answer general policy issues.

The objective might also be defined in somewhat more technical terms like:

- the necessity to compare the quality of the waste material with quality levels defined in national and international legislation (does the waste meet the compliance levels?);
- to determine the leaching of substances from the material;
- etc.

Although the latter objectives are defined in technical terms and as such have a closer link to the actual sampling, there is still no technical specification available from these objectives that will allow the definition of an unambiguous Sampling Plan. The deduction of the Sampling Plan from the objective is discussed in 4.4.

It is important that all involved parties reach agreement about the objective.

#### **Example situation**

Due to the incineration of residuals from paper production, filter dust is collected. The dust is trapped in an air filter unit and put into stockpiles before transport to the landfill. In order to allow land filling, the concentrations of a number of key constituents should comply with the acceptance criteria of that landfill. Transport to the landfill happens when 30 t of dust is gathered. The stockpile is readily accessible from a hard standing.

The objective of the testing programme is to define whether the composition of the filter dust fulfils the criteria that are set for the acceptance of waste on the landfill. These acceptance criteria are formulated in light of the protective measures taken in the construction of the landfill and as such are aimed at protecting the environment – specifically groundwater quality – around the landfill.

#### 4.4 Strategic choices in deriving the technical goals from the objective

#### 4.4.1 General

As stated in 4.3, the objective of the testing programme defines what the involved parties want to achieve by investigating the waste material. In order to investigate the waste, samples should be gathered. For appropriate sampling the analytical results obtained from samples have to be adequate in light of the objective. Therefore, the sampling operation for obtaining these samples should be planned in detail. The detailed planning of the sampling operation and technical specifications for the sampling are formalised in the Sampling Plan.

In deriving the Sampling Plan, the original objective has to be translated in one or more technical goals. The relation between the testing programme, the objective, the technical goals and the Sampling Plan are depicted in Figure 2.

#### Example: Specifying instructions in the Sampling Plan by defining the technical goals of the objective

The objective 'compare the quality of the waste with quality levels as defined in legislation' has to be translated into technical goals like 'measure the pH and cadmium content of the waste'. In the Sampling Plan, technical goals (e.g. measurement of the pH) are translated into concrete instructions to the sampler. For instance by specifying the amount of sample to be taken and the necessary conservation measures for determining the pH.

The technical goals in this example are to take an adequate amount of waste material and conserve its characteristics by using an adequate sample container. In the Sampling Plan the term 'adequate' as used in these technical goals are replaced by actually stating the necessary amount of waste material (e.g. 1 kg) and prescription of the type of sample container (e.g. dark glass and air tight).

Note that one testing programme may often have more than one objective. In principle, each individual objective will result in a different Sampling Plan because the technical specifications for the necessary samples and the quality level to be achieved will vary between the different objectives. As a result it might be necessary to define more than one Sampling Plan to fulfil all objectives of the testing programme.

#### Example: Situation where the testing programme has more than one objective

Due to a fire in a nearby factory, on a specific moment the stockpile of filter dust might contain asbestos. Apart from regular sampling to determine the constituents relevant for land filling the filter dust waste, the testing programme now has a second objective that is to determine if there is indeed asbestos present in the waste. The latter obviously requires an approach, which considers stratified or hot spot sampling and therefore two different Sampling Plans will have to be defined.

The fact that there are two different Sampling Plans does not imply that the necessary sampling cannot take place at the same time.

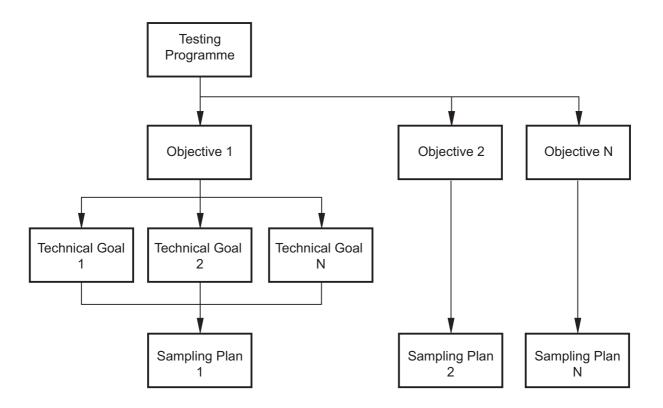


Figure 2 - Translation of objectives into technical goals and instructions in the Sampling Plan

- the technical goals are related to the following elements of the Sampling Plan<sup>3</sup>,
- constituents to be studied (4.4.2);
- the population that is represented by the sampling results (4.4.3);
- the desired reliability of the results (4.4.5);
- statistical parameter to be determined;
- choice of sampling methodology (probabilistic or judgemental);
- adequate sampling technique;
- sample pre-treatment;
- etc.

#### 4.4.2 Selecting constituents to be studied

The selection of constituents starts with an inventory of constituents that are raised in relevant legislation. The constituents identified by legislation are often a reflection of their potential to cause human, environmental and economic risks. Background data on the waste may also identify further relevant constituents.

<sup>&</sup>lt;sup>3</sup> Not all technical goals need to be discussed with (all) the involved parties. Choosing the constituents to be studied, defining the population and scale of sampling and choosing the desired reliability of the sampling results are most important because these choices influence to a large extent the efficiency and effectivity of the testing programme.

#### **Example: Selection of constituents**

In the example it is assumed that specific quality criteria are set for waste acceptance at landfill, based on a LS 10 (liquid to solid ratio 1: 10) leaching test. It defines compliance levels for 13 components and characteristics: Ag, As, Ba, Cd, Co, Cu, Cr, Hg, Ni, Zn, PAH (sum) and TOC.

Based on prior information TOC, Pb and Cu are to be considered as 'critical', since the 95-percentile value of earlier analyses (mean concentration for a stockpile) show these parameters exceed the compliance levels. Thus in the example the statistical definition of 'critical' is that there is more than 5 % probability that the mean concentration of a constituent in a stockpile will exceed the compliance level.

Background information about the composition and production process that leads to the production of the waste can be crucial in selecting the constituents to be studied.

#### **Example: Background information**

Different types of background information are available for the waste. There is technical information on the production process and input materials. There is also numerical information obtained from a previous basic characterisation as well as previous compliance testing.

Non-numerical information, in addition to the description of the example situation as given in 4.3, is that the annual capacity of the combustion operator amounts to 287.000 t of paper waste. This results in approximately 13.000 t of filter dust per year (600 waste movements a year). Around 60 % of the filter dust is destined for land filling; the rest is reused in cement industry.

Compositional data from periodic basic characterisation provides information that the components that are to be considered as 'critical' are TOC, Pb and Cu. From earlier basic characterisation and compliance testing the content of TOC in the last 4 years ranges from 2.775 up to 34.470 mg/kg. The mean of the TOC was 12.568 mg/kg. For Pb the mean concentration is 118 mg/kg with a standard deviation of 63 mg/kg. For Cu the mean concentration is 400 mg/kg with a standard deviation of 44 mg/kg. These types of data are required as a basis for determining requirements for any future sampling programme.

#### 4.4.3 Defining population and sub-populations

#### 4.4.3.1 General

The population is the total amount of material that we want to obtain information on by sampling.

In its most simple form, the population is a container, stockpile or lorry of waste. In this case identifying the population in terms of space and time is simple. But, where a production process results in a continuous stream of waste identifying the population is less straightforward. For example, the population might be the amount of waste that is produced in a continuous production process. To define the population in this case, the involved parties must specify the time period of production. The population might thus be defined as the amount of waste produced at a certain place in a year, month, week or other period. Additionally, this implies that the part of the waste produced outside the specified period also needs to be defined. For this purpose the term 'overall population' is used; describing the total quantity of material produced.

Depending on the objective of the testing programme and the available resources, the involved parties will have to make a choice between various options for defining a population. Furthermore, as will be made clear in the following paragraphs, it may sometimes be necessary to divide a population in sub-populations. From the perspective of sampling, a sub-population can be seen as the unity that is sampled separately and for which sampling results provide information (see also 4.4.4).

The production process of waste determines to great extent the definition of the population and the necessity to divide the population in sub-populations. For the purpose of sampling, the following production processes can be identified:

- one off production of waste (for example: container, stockpile, lorry);
  - continuous production (waste stream) of a homogeneous waste;
  - continuous production (waste stream) of a <u>heterogeneous</u> waste.

#### 4.4.3.2 One off production of waste

The simplest form of waste production is a one-off production of waste stored in a container, stockpile, lorry or other unit. In this case the population can easily be defined in terms of space (the amount of waste on a certain location).

In the simplest situation the one-off waste is stored in one container, stockpile, lorry or other unit, clearly identifying the full population. In this case there is no necessity to use any other term than 'population'.

In a more complex situation, the one off waste is stored in more than one container, stockpile, lorry or other unit, but still the set of 'units' is limited<sup>4</sup> and can be defined in terms of space. Potentially it might be possible and desirable to identify different sub-populations in relation to, for example, the method of storage. Then the population consist of different sub-populations.

Identification of sub-populations is, from the perspective of sampling, only necessary when these sub-populations are sampled and assessed on an individual basis. For example, when it is expected that sub-populations differ in quality or have different destinations with different acceptance criteria. However, this does not imply that each individual sub-population needs to be sampled.

#### 4.4.3.3 Continuous production of a homogeneous stream of waste

Contrary to a one off production of waste, a continuous production process generates a continuous stream of waste. The population will now be defined in time rather than in space (the amount of waste that is produced by a certain production process in a certain time span). Definition of the population in space is however also possible, depending on the location where the samples will be taken.

In some cases, a continuous production process produces a homogeneous stream of waste (Figure 3). The quality of this homogeneous waste can be established relatively easy by a limited amount of samples of appropriate size (4.4.4). It is not necessary to divide the population into sub-populations as these will not be sampled and assessed on an individual basis.

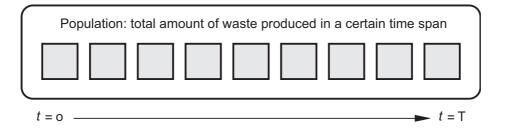


Figure 3 - Continuous production of a homogeneous stream of waste

There is a certain overlap between production of a 'limited' number of units and continuous production of waste. This overlap depends on the actual number of units and the time span they are produced in. See 4.4.3.3 and 4.4.3.4.

#### 4.4.3.4 Continuous production of a heterogeneous stream of waste

A continuous production process can also result in a stream of heterogeneous (of variable quality) waste. For example because the quality of primary products may change with time or because of variations in the production process.

The heterogeneity of the resulting waste might lead to a situation where it can be reasonably expected that part of the population is not suitable for the planned destination or use. If it might be expected that specific parts of the waste stream exceed the relevant specifications (e.g. compliance levels), the sampling should be organised in such a manner that these parts of the waste stream can be identified.

Thus the results of the testing programme should give insight in the heterogeneity of the waste. To accommodate sampling and to get insight in the heterogeneity within the population, the population has to be divided in several sub-populations. Preferably, sub-populations are physically separated until the results of the testing programme are available, allowing separate actions as a consequence of the potentially variable quality.

Any change in the production process that is expected to have an influence on the quality of the waste, like a new stock of primary product or the introduction of a new machine in the production process, can result in a new sub-population (Figure 4). Identification of sub-populations from a production perspective provides information on the (potential) heterogeneity of the population.

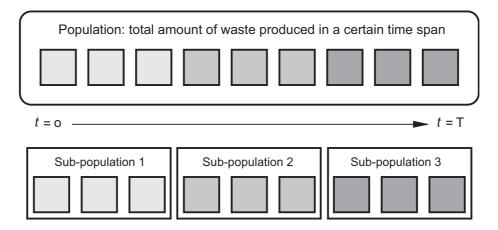


Figure 4 – Continuous production of a heterogeneous waste: identification of sub-populations from a production perspective

Sub-populations can also be identified from the perspective of transport and destination. For example, if a heterogeneous waste stream is collected on trucks to be transported, it may be wise and practical to identify individual truckloads as sub-populations because this allows the involved parties to gather information on the quality of waste that is to be transported (Figure 5).

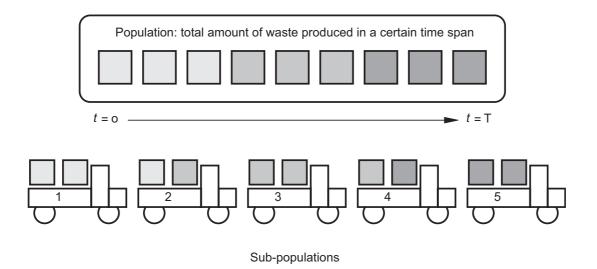


Figure 5 – Continuous production of a heterogeneous waste: identification of sub-populations from a transport perspective

One may also state that the truckload of waste is the result of a one off production and therefore is the population of sampling (4.4.3.2). However, this approach yields no information on the total amount of waste that was produced, because only part of the total amount of waste is included in the population.

For efficiency reasons, several truckloads that are transported to the same destination can be joined together in one sub-population (Figure 6). In this case the sub-populations is defined from a destination perspective.

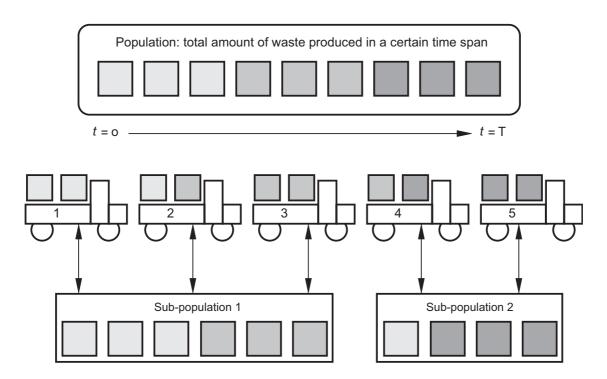


Figure 6 – Continuous production of a heterogeneous waste: identification of sub-populations from a destination perspective

Table 1 summarises the advantages and disadvantages of the various approaches to define sub-populations.

Table 1 – Advantages and disadvantages of various approaches to define sub-populations in sampling a continuous production of a heterogeneous waste

Perspective	Advantage	Disadvantage
Production perspective	Potentially a clear relation between the sub-population and the production process results in relatively lower costs for the testing programme.	Production process must be known and samples must be taken during or directly after production.
Transport perspective	Practical from the perspective of sampling.	Might result in high costs when there are a lot of sub-populations.
Destination perspective	Potentially a direct link can be defined between quantities of material that are considered relevant, for example from a toxicological perspective.	Variations caused by production, transport and/or mixing of quantities can no longer be identified.

#### Example: Identification of population and sub-populations

The population is defined as the amount of filter dust produced by incineration of paper waste in one year (approximately 13.000 t). However, because it is neither practical nor feasible to assess the production for a whole year, sub-populations have to be identified.

When 30 t of filter dust is gathered, it will be transported to the landfill. Considering the total amount of filter dust in a year, this equals approximately 430 truckloads, or based on a five days production week, approximately two truckloads per day.

Sub-populations might be identified based on these truckloads. However, that would result in 430 sub-populations to be sampled and assessed. To be more cost effective, the sub-population is defined as the production of a week. This implies that each sub-population consists of (approximately) 10 truckloads or 300 t.

In light of this definition of sub-populations, the landfill registers the exact location of each week's production. So, when sampling results indicate that a sub-population exceeds the compliance level for a constituent, the sub-population can be easily identified and appropriate measures taken.

#### 4.4.4 Scale

Scale is one of the essential issues of sampling. The scale defines the volume or mass of waste material that a sample directly represents. This implies that when the assessment of the waste is needed for example on one cubic metre, the sampling results should provide information on a cubic metre scale. Thus the analytical results should be representative for a cubic metre of waste.

Depending on the objective of the testing programme, the scale of sampling may be equal to the size of individual particles of the waste (for particulate waste materials), the size of the sub-population or even the whole population.

Scale can also be defined in terms of time: if the population is the total amount of waste produced in one year, the scale may be one year (the whole population) but also one month, week or day, depending in the objective of the testing programme.

Defining the scale is important, as heterogeneity is a scale dependent characteristic. Let's assume a particulate waste material that consists of small particles that only vary in colour. The particles in the waste are fully mixed.

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In a series of samples, each with the size of an individual particle, each sample will have a different colour. Therefore the observed heterogeneity in colour between these samples will be high.

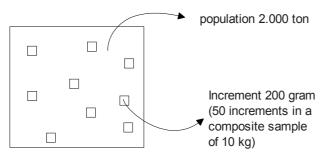
However, the degree of heterogeneity on a scale of for example 1 kg, consisting of several thousands of particles, will be low. Each of these samples will have approximately the same mix of colours, and – looking from some distance (thus really on the scale of 1 kg) – the samples will have the same mixed colour. Thus the observed heterogeneity will now be low.

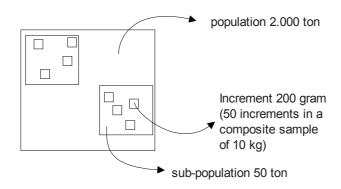
As a consequence of the direct relation between scale and heterogeneity, sampling results are only valid for the scale that is equal to the scale of sampling or higher scales. In general, the degree of heterogeneity will be higher for a smaller scale of sampling and will be lower for a larger scale of sampling.

#### **Example: Defining the scale of sampling**

Three specific examples for which the scale is defined are as follows:

<u>Situation 1</u> describes a population of 2.000 t from which randomly 50 increments are taken. The resulting composite sample is 10 kg.





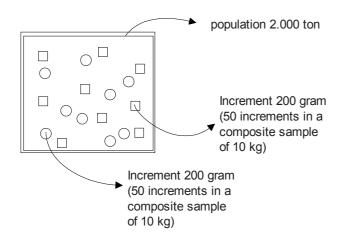
Assuming that the composite sample resulting from these 50 increments represents a good estimate of the mean concentration (but not of the variability) of the whole population, the scale for the composite sample in this example is 2.000 t.

Note that the variability of the population (on the scale of the increments) is fully incorporated in the composite sample; the sampling method will however provide no information on the variability.

<u>Situation 2</u> describes a population of 2.000 t. Within this population – perhaps only for the purpose of sampling – sub-populations are defined of 50 t each. From each sub-population 50 increments are taken. The resulting composite samples are 10 kg, each representing a sub-population.

The mass represented by each composite sample is now the mass of the individual sub-populations; thus 50 t. The scale for each composite sample in this example is 50 t. The mean value of all composite samples yields an estimate of the mean concentration of the whole population of 2.000 t

and the estimated variability within the whole population is estimated on a scale of 50 t.



<u>Situation 3</u> describes a population of 2.000 t. More than one composite sample is taken. However, each composite sample (existing of 50 increments) is obtained by taking random increments throughout the whole population. The mass represented by each composite sample is now equal to the mass of the whole population; thus 2.000 t.

The scale for each composite sample in this example is 2.000 t. The mean value of all composite samples yields an estimate of the mean concentration and the variability of the whole population of 2.000 t is estimated on a scale of 200 grams (the mass of the increments).

The following example illustrates the effects of different definitions of the scale of sampling. Depending on the objective of the testing programme, the involved parties must make a choice.

#### Example: Effects of different definitions of the scale of sampling

Consider the three sub-populations as shown in *Figure* 7. Each sub-population consists of thirteen individual parts that have a 'quality' that is symbolised by a number between 0 and 99. Heterogeneity is quantified by the coefficient of variation: a high coefficient of variation indicates a high heterogeneity.

When the scale of sampling is equal to the size of the sub-population, the sampling result will only be an estimate of the mean concentration for each sub-population. Comparing the sub-populations in *Figure 7* sub-population 1 and 2 are comparable while sub-population 3 has a higher mean.

When the scale of sampling is equal to the individual parts within each sub-population, we obtain not only an estimate for the mean concentration of the sub-population, but also an estimate for the heterogeneity within that sub-population. Comparing the sub-populations in *Figure 7* now still gives the same result for the mean of the whole sub-population, but additionally we discover that sub-population 2 has a higher degree of variability than sub-populations 1 and 3.

	Sub-pop. 1	Sub-pop. 2	Sub-pop. 3	3
	20	15	32	
	30	14	36	
	20	22	3	
	30	72	37	
	40	9	38	
	20	23	36	
	30	64	37	
	30	46	30	
	40	5	40	
	20	16	41	
	10	2	17	
	20	17	39	
	30	35	36	
				Population
Mean	26,2	26,2	32,5	28,3
Coefficient of variation	33,3%	84,2%	33,2%	

Figure 7 – Example of three different sub-populations, characterised on the individual samples, the mean and coefficient of variation (CV). A high CV indicates a heterogeneous sample.

Finally, when the scale of sampling is equal to the total population we obtain only an estimate of the mean for the whole population.

Different choices can now be made on the scale of sampling:

The scale of sampling is equal to the scale of the individual parts. It is not possible to define a smaller scale of sampling. The result of this definition of the scale is that information on the heterogeneity within the sub-populations can be obtained by calculating (for example) the coefficient of variation. Additionally, the heterogeneity between the sub-populations and within the population can be calculated. In this approach, the presumptions that led to identification of the sub-population as a relatively homogeneous part of the population can be verified. For example, it may be argued that sub-population 2 in Figure 7 is so heterogeneous that at least a part of sub-population 2 will not comply to certain quality standards, although the mean value is within the quality range. Many sub-populations of high heterogeneity may lead

to a re-evaluation of the Sampling Plan. Important disadvantage are the costs for measuring the individual parts, in this case thirteen per sub-population<sup>5</sup>.

- The scale of sampling is equal to the scale of the sub-populations. Therefore no information on individual parts within a sub-population is gathered. Characterisation of the sub-population is done by means of a composite sample per sub-population in which more than one of the individual items are put together prior to analysis. If this composite sample is taken and analysed correctly, the result of the composite sample will be a good estimate of the true mean of the sub-population. An important advantage of this approach is the low costs for measuring. Important disadvantage is the assumption that a composite sample can be obtained without a considerable sampling error. The analysis of a composite sample might pose problems as the amount of material in the sample will be (much) larger than the amount of material needed for the analysis and thus proper sample pre-treatment is necessary to obtain a representative analytical sample from a potentially highly heterogeneous composite sample. Additionally, there will be no information available on the heterogeneity within a sub-population.
- The scale of sampling is equal to the scale of the population. In the example (Figure 7) the population is defined as the combination of the three sub-populations. Individual parts are gathered from the involved sub-populations and put together in a composite sample. Now there will be no information available on a smaller scale than the scale of the population. An important advantage are the (very) low costs for measuring, while, as long as it is technically possible to mix a large number of these parts, the result of the composite sample will still be representative for the true mean of the total population. But the population has to be treated as one entity. In case of a heterogeneous population (for example sub-population 2 in Figure 7) sampling on the scale of sub-populations or individual parts would have given the involved parties information that may have led to different choices for the destination of sub-populations of different quality.

Given the relation between scale and the encountered degree of heterogeneity, the applied scale of sampling might determine if a waste is considered homogeneous (i.e. there is little variation between individual sample results) or heterogeneous (i.e. high variation between sample results).

The type of information that is desired, the possible destination, the financial means available and the technical possibilities of working with composite samples determine the choice on the scale of sampling.

#### **Example: Scale of sampling**

For the example of filter dust resulting from the incineration of paper waste, the population is defined as the amount of filter dust produced in one year (approximately 13.000 t). It was decided to define the sub-population as the production in a week. This implies that each sub-population consists of (approximately) 250 t (13.000 t in 52 weeks) or 9 truckloads (250 / 30). The scale of sampling is in principal equal to this amount of material. As it is technically possible to take 10 samples from each truckload and mix them into one composite sample (total of 90 increment), the scale of sampling is indeed 250 t, the week production.

When it becomes impossible to mix this many increments without an unacceptable analytical error, an approach could be to mix the samples of each truckload into a composite sample per truck, resulting in 9 composite samples per week. In this case the scale of sampling would be a truckload, which would also give insight in the heterogeneity within the sub-population (on the scale of the individual truckloads). Obviously, the analytical costs in the latter scenario will be higher.

In addition to the more technical perspective from which the definition of scale was described in the previous text, the scale of sampling can also (or even should) be defined by policy considerations. In principle the scale of sampling should be equal to the amount of material, which is considered relevant from a policy perspective.

<sup>&</sup>lt;sup>5</sup> It should be noted that it is not necessary (nor practical) to measure each individual item within a sub-population. A sample survey within each sub-population might be sufficient.

#### **Example: Policy defined scale of sampling**

Based on the radius of action of small animals living in soil, the mean concentration of a soil volume of 25 m³ is considered as relevant for assessing the seriousness of soil contamination. It is assumed that these animals throughout their whole life span are exposed to the mean concentration of the pollutants in this soil volume. Thus, when assessing the seriousness of polluted soil, we are interested in the mean concentration within this volume of 25 m³. When acute exposure to (very) high concentrations is considered not to be relevant, there is no need to gather information on a smaller scale than 25 m³. The scale of sampling is therefore 25 m³ and is achieved by taking a number of increments within this volume, an estimate of the true mean concentration on the scale of 25 m³ can thus be obtained.

#### 4.4.5 Choosing the desired reliability of sampling results

#### 4.4.5.1 General

Wastes are heterogeneous materials for several reasons:

- variability in the process the waste derives from;
- variability in the raw materials that are the input of the process the waste derives from;
- waste can be a mixture of materials requiring disposal;
- etc.

The fact that most wastes are heterogeneous has serious consequences for the Sampling Plan. In principle, it is impossible to know the exact composition of heterogeneous materials. Knowledge of whether the waste is consistent or erratic in composition will need to be considered in the design of the testing programme. The results of the sampling are always an estimate of the true composition of the waste that is studied. Two types of sampling error will influence the representability of the sampling results: the systematic error<sup>6</sup> and the random error.

Due to the fact that one or both of these errors occur, there is always a chance that the estimated characteristic of the waste material leads to an incorrect assessment of the waste.

If an incorrect assessment can have serious social, economical or environmental consequences, the reliability of the sampling results often needs to be high, although this does not imply that all sampling in these circumstances should be such. The necessary reliability also depends on the 'distance' between the measured characteristic and the relevant quality levels (compliance level). The nearer the specific waste characteristic is to the compliance level, the better the reliability should be in order to prevent an incorrect assessment. Thus, when the (expected) characteristic is much lower than the compliance level, the reliability of the measurement might still be poor as the chance that the compliance level is exceeded is still small. However, when the composition of the waste material is close to the compliance level, reliable estimates of the quality of the waste must be made.

Statistics enable us to specify the reliability of the estimate and the chances of an incorrect assessment based on the sampling results. For any random sample, confidence limits can be calculated. Confidence limits specify with a certain confidence that the true value of the waste will fall within a certain range (confidence

All activities that are necessary to obtain the analytical results are sources of variability. As long as the measurement in itself is correct, but the results vary due to coincidence, these errors should be considered as random error. However, when, for example due to the applied measurement technique, the results are per definition too high or too low, these errors are known as systematic errors. Although limiting the systematic error is essential for a correct assessment of the waste material, the prevention of systematic errors falls outside the scope of this Technical Report. It is a responsibility of the project manager.

interval) around the estimate. The narrower this confidence interval (distance between upper and lower confidence limit) the better the sampling mean estimates the true mean value of the population.

The size of the confidence interval is related to CEN/TR 15310-1:

- the heterogeneity of the population;
- the number of samples;
- the desired confidence level.

Given a specific sampling effort, a heterogeneous population has a wider confidence interval than a homogeneous population. Confidence limits decrease when more samples are taken and for a narrow confidence interval more samples are needed than for a wider confidence interval (Figure 8).

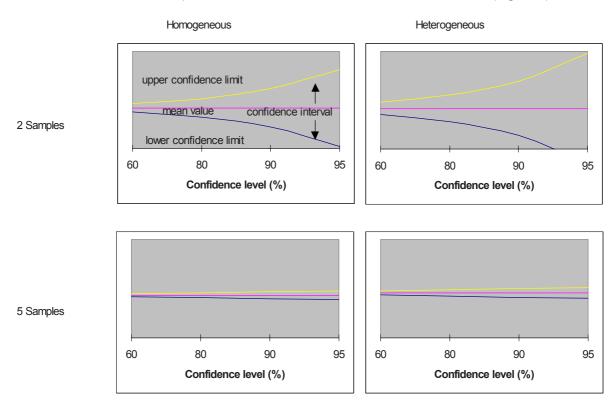


Figure 8 – The confidence interval increases with the desired confidence level and the heterogeneity of the population. The confidence interval decreases when more samples are taken. The narrower the confidence interval, the better the estimate of the mean represents the true mean of the population.

The involved parties influence the costs of the testing programme because the amount of samples is directly determined by the desired reliability that they define. The advice of statistical experts and additional research may be necessary to quantify the relationship between heterogeneity of the waste, the reliability of the estimate and the amount of samples.

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.

In most cases, the reliability of sampling results improves when the number of samples is increased (Figure 8). This leads invariably to higher costs for sampling and analysing. There are two important approaches possible to balance reliability and financial input:

- many field samples versus many increments joined in a composite sample (4.4.5.3);
- increasing the scale of sampling (4.4.4).

These two approaches can also be combined.

#### 4.4.5.2 Probabilistic versus judgemental sampling

It should be noted that whenever the reliability of the sampling is considered important, the type of sampling should comply with that need. Two principally different types of sampling are distinguished: probabilistic sampling and judgemental sampling (see also CEN/TR 15310-1).

The essential difference between probabilistic sampling and judgemental sampling is that in probabilistic sampling each individual part of the waste to be sampled has an equal chance of being sampled. While in judgemental sampling part of the population will not be considered while sampling. As a consequence the samples obtained by judgemental sampling can never be seen as (fully) representative for the whole population.

When the objective of the testing programme is to determine the type of material in a waste, which obviously differs from the rest of the waste, spot sampling is often the most appropriate sampling method. This is a specific type of judgemental sampling: only the parts that appear to be different are considered for sampling. In most other cases probabilistic sampling should be considered first and should only be replaced by judgemental sampling when there are good arguments for judgemental sampling. Still, the type of judgmental sampling should be as close to probabilistic sampling as possible in order to assure some degree of representativity of the samples.

#### 4.4.5.3 Many Field Samples versus many increments joined in a composite sample

When taking a large number<sup>7</sup> of field samples, the cost of analysis for all these samples will be high in relation to the costs of sampling. On the other hand, when these increments are joined in a composite sample, the variability of the sampled waste will effectively be summed within the composite sample. In this case, the amount of samples and the resulting costs of analysis are relatively low, but more effort (and thus costs) have to be made in sample pre-treatment to ascertain complete mixing of the increments. At the same time information on the range of concentrations that might be expected from the waste is lost. Whether that information is important depends on the objective of sampling.

The results of both options are different. Through analysing many field samples, information on the variability of the waste is obtained, but analysing a composite sample yields only a good estimate of the mean characteristic. Of course, intermediate solutions are also possible where a limited number of increments is joined in a limited number of composite samples.

#### Example: Defining the desired confidence and the estimating the resulting number of samples

The involved parties did some preliminary research on the amount of samples that is necessary to reach 90 % and 95 % confidence levels for an estimate with a confidence interval of  $\pm$  5 % for the constituents (TOC, Pb and Cu). The results of the experiment are shown in the following table.

Not only the number but also the size of samples or increments influences the reliability. The minimum sample size can be calculated (see @@@reference to CEN/TR 15310-1).

Confidence level	Number of composite samples (10 increments each)		
	TOC	Pb	Cu
90 %	15	6	1
95 %	21	9	1

The involved parties agree that the number of samples that is necessary for a 95 % confidence would lead to unacceptable costs for the testing programme. They decide that the estimate should be determined with 90 % confidence. Which means that 15, 6 and 1 composite samples will be taken for TOC, Pb and Cu respectively.

#### 4.5 Checklist for the project manager

In the interactive process of deriving technical goals from the objective, the involved parties must formulate answers to the questions that were raised in 4.4. However, not all Sampling Plans consist of the same elements, depending on the complexity of the objective. This paragraph contains a list of questions that helps the project manager (and the involved parties) through the process of defining the Sampling Plan<sup>8</sup>.

**Question**: Are all parties involved that should be involved?

See 4.2

Question: Is the objective clear and do all the involved parties agree on the objective?

See 4.3

**Question**: Are the constituents defined?

See 4.4.2

Question: Is the population defined?

See 4.4.3

**Question**: Is it necessary to identify sub-populations?

Identifying sub-populations is advisable when:

- parts of the population are going to be treated differently. For example in case of a
  continuous production process where part of the year production is transported every week
  to a waste manager and the quality of this week production (=sub-population) must be
  known;
- identifiable parts of the population are expected to be significantly different from other parts of the population.

Question: Is the scale of sampling defined?

In some cases, the scale of sampling is stated explicitly in legislation (see example: Policy defined scale of sampling). However, in most cases scale is not defined explicitly by legislation.

<sup>&</sup>lt;sup>8</sup> The order in which these questions are addressed might vary. For example the scale of sampling can be defined at various moments during the process.

Sometimes the scale can be derived from the objective. For example when information is necessary on the heterogeneity within the population<sup>9</sup>, the scale of this information should be known. In other cases, the scale is not defined a priori but becomes clear after the Sampling Plan is derived. Also in these situations it is important to identify the scale to check if it is possible to reach the objective by the chosen Sampling Plan. See 4.4.4.

Question: Is the desired reliability defined?

See 4.4.5

- confidence interval;
- confidence level;
- probabilistic sampling or judgemental sampling.

Question: Field samples or composite samples?

See 4.4.5.3

- (many) field samples: Good estimate of heterogeneity within the population (or subpopulation). Mean value of the population can be calculated. Reliability depends on the number of field samples;
- composite sample of (many) increments: Good estimate of the mean of the whole population (or sub-population). Reliability of the estimate depends on the number of increments and the quality of the sample pre-treatment.

#### 4.6 Further steps to be taken by the project manager

After the identification of the involved parties, the identification of the objective and translation of objective into technical goals, the project manager can make the Sampling Plan. As the policy related decisions are now made, the remaining aspects are purely technical and procedural. They include:

- the statistical parameter to be determined;
- the sample size;
- what sampling technique is adequate;
- the type of sample pre-treatment necessary in the field in order to obtain a quantity of material that can be transferred to the laboratory.

Or sub-population or lower scales: for example primary particles.

#### Annex A

#### **Examples of sampling plans for specific situations**

## A.1 Characterization of predominant aluminium content objects from the sorting of household and related waste

#### A.1.1 Introduction

The framework of legislation for household and related waste promotes the sorting of waste for recycling. Standardised evaluation methods are needed to verify the quality of the sorting of household and related waste. A national standardisation committee developed a generic Sampling Plan to characterise piles of waste resulting from the sorting of household and related waste.

#### A.1.2 Description of the process

Step in process	Results	Description
Identify involved parties	- Standardisation committee	The standardisation committee identified local authorities (or their suppliers) as waste producers
Paragraph 4.2	- Local authorities	because they are collecting and sorting waste. The waste recyclers are the waste users. Experts
	- Recyclers	of both parties took part actively in the work of the standardisation committee that was responsible
	- Experts of both parties	for elaborating the standard.
Define objectives	- The aluminium content (%) of the pile.	For the waste user, it is important to have an estimate of the total aluminium content of the pile,
Paragraph 4.3	- The ratio aluminium containing objects / non-aluminium containing objects (kg/kg) in the pile.	the percentage aluminium containing objects in the pile and the type of aluminium containing objects within the pile.
	The weight distribution over     categories of aluminium     containing objects (kg/kg) in	The standardisation committee defined 7 categories of aluminium containing objects:
	the pile.	Empty drink cans, empty tins, empty aerosol tanks, aerosol tanks with residual liquid, food containers and semi-rigid containers.
Determine generic level of testing required	Compliance testing (level 2) (and to some extent also basic characterisation)	The recycler defined a minimum acceptance level for the aluminium content of the waste.
	,	The weight distribution of aluminium objects in the waste is important information for optimising the recycling process.

Step in process	- Results	Description
Identify constituents to be tested  Paragraph 4.4.2	- Proportion of predominant aluminium content objects in the sample (%).	
	Distribution of predominant aluminium content objects (%).	
	Aluminium content of sample (%).	
Research background information on waste	Average weighted values, lowest values and highest values of the aluminium content for each of the defined categories	The standardisation committee reviewed available background information when elaborating this standard. This background information made it possible to identify suitable categories of aluminium containing objects and an estimate of the aluminium content for each of these categories.
Identify health and safety precautions	General health and safety precautions	No specific health and safety precautions are needed. Consequently, the general health and safety precautions are to be taken.
Select sampling approach	- Population: the totality of objects in the pile of waste.	The population consist of the objects in the pile that is made ready for shipment to the aluminium recovery facilities.
Paragraph 4.4	<ul><li>Scale: the pile of waste.</li><li>Reliability: not specified.</li></ul>	Increments are combined into one composite sample that represents the whole pile. Thus, the scale of sampling is the pile of waste.
		Based on knowledge and experience, the standardisation committee selected judgemental sampling as sampling approach.
		The minimum recommended weight of the composite sample is 15 kg. This is a compromise between available resources (operation duration, equipment, etc.) and expected results.
		Composite samples consist of three successive increments of 5 kg, each consisting of a large number of objects.

#### Comments

The involved parties also defined sampling equipment:

- Personal Protective Equipment.
- Sampling resources, excluding manual shovel (use of a manual shovel may lead to object segregation), with a capacity to hold at least 40 l of objects at the same time (bucket of a mechanical shovel, etc).
- Weighing machines with a maximum resolution of 5 g.
- Containers to hold the sample.

After sampling the aluminium content of the sample is estimated as follows:

- The sampler separates objects low in aluminium content from predominantly aluminium objects (by using a magnet).
- 2. The aluminium objects are divided into 7 sub-categories (empty drink cans, empty tins, empty aerosols, etc.).
- 3. The total estimated amount of aluminium in all categories divided by the total weight of the sample is an estimate for the aluminium content of the whole sample and represents the aluminium content of the population.

## A.2 Basic characterisation and compliance testing of waste from a continuous production process

#### A.2.1 Introduction

A production process generates a continuous stream of one type of waste throughout the year. The annual amount of the waste produced is more than 10.000 t per year. Based on preliminary examinations and process information, the quality of the waste is expected to be constant with variations within certain boundaries during the year.

According to national legislation, the quality of the waste shall be characterised before land filling. It is not possible to store the waste produced within one year prior to sampling. To facilitate acceptance and land filling of the waste during the one-year period, basic characterisation follows specific rules.

Table A.1- Description of the process

Step in process	Results	Description
Identify involved parties	- Legislator	The national legislator defined the involved parties in the landfill ordinance
Paragraph 4.2	- Waste producer	
	- Waste owner	
	- Laboratory (also responsible for sampling)	
	- Local authority	
Define objectives	The objectives of the testing	In year one, the production period is divided in 4
Paragraph 4.3	program can be divided in objectives for year 1 and objectives for the following years.	quarters. The quality of the waste in the first and the third quarter is characterised by three weekly assessments. If the results of basic characterisation show a constant quality below compliance levels in the first year, basic characterisation is replaced by
	Year 1: basic characterisation to evaluate if:	compliance testing in the following years.
	The mean concentration of the waste produced within one year of production complies with the limit values.	In the first week, composite samples of the waste produced on three days distributed evenly over the production week (e.g. Monday, Wednesday, Friday) have to be analysed. The waste can be land filled until the results of the next weekly assessment
	The waste has a constant quality so that only compliance testing may be done in year 2.	become available if the average over the 3 days of every parameter meets the limit value and no single daily result exceeds certain tolerance values.
	Following years: compliance testing based on a systematic sampling pattern.	If the mean value over 3 days does not exceed 80 % of the limit value and if the variability between days is not high, in the second weekly assessment the three days may be combined to a composite sample representing the weekly production. If this condition is not met the procedure of week 1 is repeated.

		If the second weekly assessment does not conflict
		with the landfill criteria, the waste can be land filled up to the third weekly assessment. If the values of the third weekly assessment still comply, land filling may continue without sampling until the third quarter.
		In the third quarter this procedure is repeated. If all the six 'weekly assessments' of the first and the third quarter confirm that the waste may be land filled, the waste is considered to be basically suitable for land filling.
		This means that in the following years, only compliance tests have to be performed. However, if changes in waste characteristics are expected (e.g. due to process modifications or changes in input materials) basic characterization has to be done.
		It is not allowed to mix different types of waste or waste with different quality levels caused by changes of the input or the process.
of testing required	Basic characterisation in year 1 and compliance testing in the following years.	According to national legislation, the quality of the waste shall be characterised before land filling.
		If the results of basic characterisation show a constant quality below compliance levels in the first year, basic characterisation is replaced by compliance testing in the following years.
		NOTE One part of each daily sample should be stored for eventual later use. If the measured value of one or more parameter in a composite sample exceeds 90 % of the limit value, the three daily composite samples should be analysed.
be tested	As, Ba, Pb, Cd, Cr, Co, Cu, Ni, Hg, Ag, Zn, TOC, HC, PAH, TOC, Pb, Cu.	
Paragraph 4.4.2	, ,, -,-	
information on waste	Production process, causes of variability in the waste stream, waste type and dimensions.	
-	General health and safety precautions.	No particular health and safety precautions are needed. Consequently, the general health and safety precautions are to be taken.

Step in process	Results	Description
Select sampling approach  Paragraph 3.4	Population: one-year production of waste from a continuous production process.	The population is defined by the legislator as the amount of waste produced within one year because most other regulation concerning waste also has a year as time period.
	- Sub-population:  - Year 1: one week production  - Following years: amount of waste that is	In the first weekly assessment of year 1, one composite sample is analysed per day. In the following assessments one composite sample per week is analysed.
	produced in a quarter  - Scale:  - Year 1: First week of the first and third quarter: the amount of	This composite sample consists of composite samples from three days. Therefore the result of testing is a weekly mean value. Daily composite samples have to be stored for eventually later use.
	waste that is produced in one day, in other weeks the amount of waste produced in a week.	In the following years, the three weekly composite are combined into a quarterly composite sample that is analysed. As a consequence, information on the scale of a weeks production is not available. The lowest scale we have information on is the quarterly production. If the stored daily composite samples
	- Following years: amount of waste that is produced in a quarter.	are analysed, information is obtained on the scale of a day.
	- Required level of confidence = 90 %,	
	- Required precision = 20 %	

#### Comments

The sample size and number of samples are calculated in line with the EN 14899; for the first weekly assessment it is recommended to take approximately 50 increments.

### A.3 Contaminated soil to be possibly land filled and to be tested as granular waste for basic characterisation

#### A.3.1 Introduction

National legislation only allows land filling of contaminated soil when it is impossible to re-use or clean the soil. The National government initiated the development of a generic sampling plan to characterise soil in order to evaluate if land filling is the only feasible solution.

#### A.3.2 Description of the process

Step in process	Results	Description
Identify involved parties  Paragraph 4.2	- Project manager - Legislator: the National	The legislator has the objective to minimise the amount of contaminated soil that has to be land filled and to maximise the amount of contaminated
	ministry of the environment  - Regulator: (representative of) local and national authorities.  - Samplers: (representative of) soil research companies.  - Representative of	soil that is cleaned or re-used while minimising human and ecological risks.  The regulator has partly the same objective as the legislator. In addition, the regulator wants legislation that is easy to assert.
	<ul><li>laboratories.</li><li>Producers of untreatable soil</li></ul>	The samplers and the laboratory are interested in clear and unambiguous legislation that can be easily translated into daily practice.
		The waste producers often are construction builders or and project developers (this is a divers group, depending on the specific situation: it might be local government (municipality), contractor, soil cleaning facility, etc.). They finance the testing programme and have an interest in keeping costs low. Also, the testing programme should not delay the redevelopment of the site.
Define objectives Paragraph 4.3	To evaluate if contaminated soil has to be land filled, can be cleaned and / or re-used.	The legislator decides to make a generic sampling plan with detailed information that must be applied in all cases. By doing so, the legislator guarantees uniformity and a constant quality level in sampling. This contributes to quality assessment and quality control.
Determine generic level of testing required	Basic characterisation	

Step in process	Results	Description
Identify constituents to be tested  Paragraph 4.4.2	The choice of constituents depends on local situation. However heavy metals, PAH and oil are always determined.	Based on scientific knowledge, compliance levels are defined in legislation for a (large) number of constituents. However, only a number of constituents are analysed in practice. The choice of which is based on historic information about the use of the site.
Research background information on waste	2 composite samples of 50 increments yield the necessary results.	Computer simulation and field tests of several sampling methodologies showed that two composite samples of 50 increments each (total 100 increments) yielded results with a reliability of 20 % (sampling error) due to heterogeneity in the soil.
Identify health and safety precautions	General health and safety precautions	No particular health and safety precautions are needed. Consequently, the general health and safety precautions are to be taken.
Select sampling approach  Paragraph 4.4	<ul> <li>Population: a population of more than 2000 t has to be divided in sub-populations.</li> <li>Scale: maximum of 2000 t.</li> <li>Reliability: sampling error 20 %</li> </ul>	Legislation defined a maximum scale of 2000 t (see scale). This means that a population bigger than 2000 t has to be divided in sub-populations smaller than 2000 t that have to be sampled separately. For example, a population of 10.000 t will be tested in (at least) 5 sub-populations.
		The compliance levels apply to a maximum of 2000 t of contaminated soil. The choice of 2000 t as the maximum that can be evaluated as one entity is a compromise between sampling costs per ton (waste producers), economic risks of rejecting waste (waste producers) and environmental considerations (legislator: part of the population of 2000 t may be contaminated above the compliance levels). Therefore, 2000 t is also the maximum scale of sampling. In case of a population smaller than 2000 t, the scale of sampling is equal to the size of the population, with (theoretically) a minimum of 25 m <sup>3</sup> .
		The choice of the desired reliability was based on the philosophy that sampling errors should be comparable to errors in sample pre-treatment, subsampling and laboratory analysis (the resulting total error from these activities was estimated to be 20 %).
		Lower sampling errors would lead to higher costs (waste producers) but not to more reliable results because errors in sample pre-treatment, subsampling and laboratory analysis are higher. Higher sampling errors would lead to lower reliability, which was not acceptable for the legislator.

Step in process	Results	Description

#### Comments

In some cases, it may be advisable to identify sub-populations that are smaller than 2000 t. For example, when a population is very heterogeneous and sub-populations with different quality can be easily identified, or when parts of the population are physically separated from each other.

The sampling methodology is: 2 samples that consist of 50 increments per sample. Increments are taken using a systematic three-dimensional pattern throughout the whole soil lot. As a result each composite sample originates from the whole soil lot.

Both composite samples are analysed. For quality control of the laboratory work the results of the two samples are compared. If the results differ more than a given figure, errors may have been made in sample pre-treatment, subsampling or laboratory analysis as it is unlikely that a soil lot is so heterogeneous that this causes the difference between the two analytical results (for inorganic components this might occur in less than 3 % of the soil lots).

## A.4 Zinc concentrate to be tested as granular waste for basic characterisation and compliance testing

#### A.4.1 Introduction

Zinc concentrate was shipped for treatment in another country, but the shipment was stopped as it was expected that it exceed waste regulations and should be considered as dangerous waste. After preliminary investigation and analysis, the zinc concentrate, originally loaded in a ship, was temporarily stored in a silo. Based on the result of the preliminary investigation, the zinc concentrate was tested both for compliance testing on zinc content as well as basic characterisation for a large number of other constituents in order to assess potential treatment of the waste.

#### A.4.2 Description of the process

Step in process	Results	Description
Identify involved parties  Paragraph 4.2	Project manager (from consulting firm)  Legislator: the National	In order to prohibit exportation of dangerous waste the regulator checks this type of transport on a regular basis.
	ministry of the environment - Regulator: National health inspection	The consulting firm (project manager and sampler) involved in the actual sampling and more in general responsible for the investigation.
	- Sampler (from consulting company) - Producers of the waste	The waste producer is not directly involved but will be when sampling and analysis show that the zinc concentrate is indeed a dangerous waste. If so, they will be held responsible for costs of storage, transport and treatment.
	- Owner of the intermediate storage facility	The owner of the storage facility is involved in relation to granting permission for sampling and fulfilment of health and safety requirements during sampling.
Define objectives  Paragraph 4.3	To evaluate if the zinc concentrate is a dangerous waste and if it can be treated.	The zinc concentrate is considered to be a dangerous waste if the mean concentration in the stockpile exceeds the levels for zinc in national waste legislation. Based on the preliminary investigation of the material this is to be expected. As a result, simultaneously with this compliance testing, the waste is characterised for a large number of constituents to assess possibilities for potential treatment. Therefore the testing programme is a combination of compliance testing and basic characterisation.
Determine generic level of testing required	Compliance testing and basic characterisation	See objectives.

Step in process	Results	Description
Identify constituents to be tested  Paragraph 4.4.2	Zinc for assessment of the classification (dangerous waste).  Broad spectrum analysis (heavy metals, PAH's, XRF-analysis, etc.) for assessment of potential treatment.	The compliance level for zinc is defined in legislation. There is no predetermined treatment method, so a broad spectrum analysis should provide indications of the exact type of waste and potential treatment methods.
Research background information on waste		
Identify health and safety precautions	General health and safety precautions	For the sampling of the waste itself, apart from general health and safety precautions, no additional health and safety precautions are needed. However, due to the location of the storage facility, sitespecific health and safety requirements should be fulfilled.
Select sampling approach  Paragraph 4.4  Comments	<ul> <li>Population: the whole amount of zinc concentrate stored in the silo.</li> <li>Sub-population: based on visual inspection of the zinc concentrate within the silo (location within the silo and colour of the material), a distinction is made in two sub-populations.</li> <li>Scale: the individual sub-populations</li> </ul>	The zinc concentrate should be seen as a 'one off' lot. However, based on the visual inspection of the material, there might be differences present between the two sub-populations.  The compliance levels apply to the mean concentration of zinc in the sub-populations; without predefined limits to the size of these lots.  The involved parties agreed to sample the zinc concentrate in accordance with a national standard
	- Reliability: not specified	on waste sampling. The standard describes the number of samples to be taken based on the size of the lot to be sampled. There is no statistical or scientific basis for this relation.

#### Comments

For the two sub-populations a different number of increments is taken as the two sub-populations have a significant difference in size. All increments from each sub-population are mixed in a composite sample prior to analysis.

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