PD CEN/TR 16220:2011



BSI Standards Publication

Construction products — Assessment of release of dangerous substances — Complement to sampling

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National foreword

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Construction products - Assessment of release of dangerous substances - Complement to sampling

Produits de construction - Evaluation de l'émission de substances dangereuses - Complément relatif à l'échantillonnage

Bauprodukte - Bewertung der Freisetzung von gefährlichen Substanzen - Ergänzung zur Probenahme

This Technical Report was approved by CEN on 24 April 2011. It has been drawn up by the Technical Committee CEN/TC 351.

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Foreword

This document (CEN/TR 16220:2011) has been prepared by Technical Committee CEN/TC 351 "Construction products: Assessment of release of dangerous substances", the secretariat of which is held by NEN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN (or CENELEC) by the European Commission and the European Free Trade Association.

0 Introduction

0.1 Objective

This CEN/TR provides a complement to the sampling of construction products. Sampling of construction products for other characteristics than the release or emission of regulated dangerous substances is described in product standards and ETAs¹⁾. This CEN/TR is based on mandate M366 of the European Commission²⁾. It provides requirements which are specific for the sampling of construction products for the determination of the release or emission of regulated dangerous substances. The mandate implies that existing sampling standards from product TCs, or sampling instruction in product standards from product TCs, are to be used as much as possible. Consequently this CEN/TR and the sampling parts of the standards prepared by WG 1 and WG 2 of CEN/TC 351 (see below 0.3) should be used as a *complement* to the sampling of construction products as described in existing standards and ETAs. It does not provide full guidance to sampling of construction products.

NOTE 1 As a consequence of the fact that this CEN/TR is a *complement* to existing standards of product TCs, some instructions that would be an integral part of a full sampling standard, are missing in this CEN/TR. An obvious example thereof is the fact that this CEN/TR contains no instructions for actually taking a sample.

Existing sampling standards and instructions³⁾ for the sampling of construction products are to be compared with this CEN/TR, in order to determine if the requirements recommendations for sampling as described in this CEN/TR can be met with the existing sampling standards and instructions. If not, product TCs may have to adapt their sampling standards and instructions following appropriate provisions included in the standards to be produced by WG 1 and WG 2. For this purpose this CEN/TR contains a checklist in Annex G.

NOTE 2 Product TCs should be aware of the fact that sampling for the determination of the emission and/or release of dangerous substances, might differ from their current sampling procedures which are used to determine product characteristics.

0.2 Terminology

It is essential that a number of key terms, as mentioned in Clause 2, are well understood when working with this CEN/TR. These key terms are defined in Annex A, which annex also contains Figure A.1 that depicts the relation between these key terms.

0.3 Relation with the deliverables of CEN/TC 351/WG 1 and WG 2

At the time that this CEN/TR is developed, CEN/TC 351 comprises two Working Groups (WGs): CEN/TC 351/WG 1: Release from construction products into soil, ground water and surface water and CEN/TC 351/WG 2: Emissions from construction products into indoor air. Both WGs have to, within their scope, deliver a complete test procedure of which sampling is just a part. The interface between these sampling parts, product standards and this TR have been defined in TC 351 resolution 81⁴⁾. The test results are to be used for CE-marking (and corresponding AoC) and are produced according to WG 1 and WG 2

¹⁾ ETA: European Technical Approval issued by the European Organisation for Technical Approvals (EOTA).

²⁾ Mandate M366 "Development of horizontal standardised assessment methods for harmonized approaches relating to dangerous substances under the Construction Products Directive"; European Commission, DG Enterprise, Brussels 16 March 2005.

³⁾ This document refers both to sampling standards as published by product TCs as well as to product standards that contain sampling instructions as part of an overall test procedure.

⁴⁾ Resolution 81 taken by CEN/TC 351 on 23-24 April 2008 reads: CEN/TC 351 confirms the recommendation 1 of TG 4 taken at its March 2008 meeting as given in document N 149, which is "It is the responsibility of product TCs to specify the detailed procedure for sampling. However, they have to follow the general requirements provided by WG 1 and WG 2 that are to be based on the technical report prepared by TG 4." The decision was taken by unanimity.

standards. Since it is not possible to test all possible conditions, WG 1 and WG 2 establish reference conditions under which the test results are expressed.

0.4 Users of this CEN/TR

This CEN/TR is intended to be used by two principal users:

- CEN/TC 351/WG 1 (Release from construction products into soil, ground water and surface water) and CEN/TC 351/WG 2 (Emissions from construction products into indoor air).
- CEN/TCs and EOTA committees responsible for the development and maintenance of standards for products under the Construction Products Directive (CPD). These CEN product TCs fall under the framework of mandate M366 on the "Emission of dangerous substances from construction products into indoor air, soil, surface water and ground water". This mandate is a "horizontal complement" to the construction product mandates.

Additionally, this CEN/TR might for instance be used as a reference document by individual producers when indirect test procedures are derived e.g. for Factory Production Control (FPC).

0.5 Two sampling domains

Two different sampling domains are relevant to regulated dangerous substances:

- sampling of the construction product to obtain a quantity of the product which is used in a test;
- sampling of the air (emission) or water (release) with which a quantity of the product has been in contact.

This CEN/TR is only of relevance to the first sampling domain, the sampling of the construction product. At the same time, restrains which result from the second sampling domain might impose boundary conditions on the first sampling domain.

NOTE To avoid confusion, this Technical Report often uses the term 'product sampling' for the first sampling domain.

0.6 Uncertainty and statistical testing

The number and type of samples to be taken relates directly to the accepted uncertainty of the test result(s). A number of individual sources of uncertainty can be identified, which can be clustered in three groups: the variability of the product, the variability introduced due to sampling activities and the variability introduced by the laboratory activities.

NOTE 1 In most situations the uncertainty caused by the variability of the product dominates the other sources of uncertainty.

NOTE 2 The variability of the release or emission of dangerous substances often differs from the other characteristics tested by product TCs.

Variability of the product results in uncertainty of the obtained test result(s). By taking account of the variability when sampling, a representative test result can be obtained. Representative within the context of this CEN/TR means the acceptance of a certain level of uncertainty. The level of uncertainty should at least be such that the chance that another sample would result in another assessment of conformity than the original sample is acceptably small.

NOTE 3 This means that the test result obtained from the sample can be used to assess the sampled product, while the uncertainty of that assessment is sufficiently small: the risk of false positive or false negative results is acceptable.

This CEN/TR focuses on obtaining an individual laboratory sample that is representative for a defined quantity of the construction product. Implementation of the guidance of this CEN/TR provides individual samples which are sufficiently representative. Whenever repetitive sampling is necessary, for example to quantify the risk of

exceeding a limit value, a second source of variability in the product is introduced. This is the variability of the relevant product properties over a period of (production) time. This CEN/TR does not provide the necessary guidance to deal with that level of uncertainty, nor does it provide the tools to define the statistical testing that does.

NOTE 4 Considering the fact that the uncertainty of the actual test and measurement often is much smaller than the uncertainty that is due to the heterogeneity of the sampled construction product, it is important to realise that the quantity of the product represented by the test portion / test specimen should be sufficiently large to incorporate that heterogeneity.

In Annex B, more information is provided with respect to the assessment of the uncertainty related to sampling activities as part of the overall test procedure.

0.7 Structure of this CEN/TR

This CEN/TR consists, apart from the scope in Clause 1, of two main parts:

- Clause 2 describes in general the principle requirements for sampling construction products for the determination of the release or emission of dangerous substances. It provides explanatory texts on the key issues that are to be covered in sampling standards and sampling instructions for construction products;
- Clause 3 provides a practical translation between the theoretical principles as described under Clause 2, and the test procedures as developed by CEN/TC 351/WG 1 and WG 2, as well as the product standards as developed and maintained by product TCs.

In addition to these two clauses a number of annexes provide background information and examples:

- Annex A provides definitions for the key terms on sampling as used in this CEN/TR;
- Annex B discusses the assessment of the uncertainty resulting from sampling as part of the overall test procedure;
- Annex C provides help for the estimation of the minimum increment and sample mass when applying probabilistic sampling;
- Annex D provides methods for the calculation of the required number of increments and samples when applying probabilistic sampling;
- Annex E provides some details on sample containers and storage conditions;
- Annex F provides example forms for the sampling plan, the field report and the chain of custody report;
- Annex G provides a checklist for the product TCs to assess their existing sampling standards or sampling paragraph against the essential elements as identified in this CEN/TR.

1 Scope

This Technical Report covers the specific requirements for sampling construction products to determine the release or emission of dangerous substances in their intended use. It is complementary to existing sampling standards and sampling instruction in product standards or test methods for construction products of CEN product TCs and EOTA committees which fall under the CPD.

The scope of this Technical Report covers all activities related to product sampling, starting with the initial planning of sampling until the delivery and formal transfer of the laboratory sample at the laboratory.

This Technical Report:

- does not deal with sub-sampling in the laboratory as a step towards the preparation of the test portion / test specimen⁵⁾;
- does not deal with the second sampling domain in which a sample is to be taken from the air (emission) or water (release) with which the test portion / test specimen has been in contact;
- does not deal with the statistical testing of a construction product against (legislative) limit values, nor
 does it deal with the definition of repetitive sampling, suitable for fulfilling requirements with respect to a
 minimum level of uncertainty in a series of test results.

This Technical Report focuses on obtaining a single sample. Repetitive sampling is outside the scope as the boundary conditions for routine testing against a limit are not yet defined (e.g. the necessary reliability). Despite the fact that repetitive sampling is not covered, the conditions provided in this Technical Report apply for an individual sample, as well as for a sample that is part of a series.

2 Key concepts

2.1 Introduction

2.1.1 Key terms

A number of key terms for product sampling are introduced in this clause, including: population, sub-population, scale, increment, composite sample, sample, laboratory sample and test portion / test specimen. The definition of these key terms is independent whether the release or emission of dangerous substances is to be assessed.

NOTE 2.1 gives a general description of some of the key terms and Annex A gives a formal definition together with a figure showing the relationship between some of these terms.

2.1.2 Representativeness

The ultimate goal of product sampling is obtaining a representative portion of the sampled construction product; maintaining the representativeness is essential in all steps where a (partial) sample of the product is involved. Whenever there is variability in the product, measures are to be taken in order to ensure the representativeness of the sample.

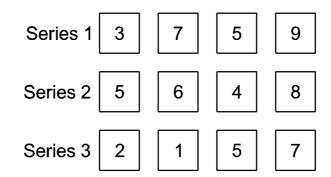
NOTE 1 When it comes to maintaining the representativeness of the sampled product, the full test procedure needs to be taken into account.

⁵⁾ This document regularly refers both to the term 'test portion' and the term 'test specimen' which are equivalent terms. However, as the term 'test portion' is used in the field of release to soil and water, and the term 'test specimen' is used in the field of emissions to indoor air, both are referred to.

NOTE 2 The same set of samples may show a different distribution of test results for different properties.

The degree of variability encountered, depends on the quantity of the product for which a sample is representative.

EXAMPLE A simple numerical example might be four tiles with a slightly different characteristic property, represented by single numbers. Observations are available for three series of four individual tiles:



The mean and standard deviation for these three series are:

Series 1: mean 6,0 standard deviation 2,6 Series 2: mean 5,8 standard deviation 1,7 Series 3: mean 3,8 standard deviation 2,8

The overall mean and standard deviation are 5,2 and 2,4 respectively.

When, instead of individual tiles, a group of four tiles is tested in a single test, the mean values for these three series would become the new measurements. The standard deviation between these three measurements is decreased to 1,2 (instead of 2,4 when measuring all individual tiles). Using a bigger quantity of product (four tiles) reduces observed variability from 2,4 to 1,2. The results are less variable when a larger quantity of the product is tested. Consequently the product might comply more easily.

In order to obtain comparable test results, it is important that in a harmonized product standard a choice is made with respect to the quantity of product (the scale) on which that product is tested. See also 2.4.4.

Representativeness of the test portion / test specimen is ensured differently for the release to soil and water and the emission to indoor air, reflecting the different nature of influencing factors (see below). For the determination of the release, incremental sampling and subsequent use of a composite sample is possible when sampling particulate products. Even for monolithic and shaped products this is still a potential, although less simple, option, when assessing the release to soil and water.

NOTE 3 Sampling might well result in a laboratory sample of 10 kg, while the size of the test portion / test specimen can only be 1 kg. This implies that maintaining the representativeness of the sample is essential, in order to ensure that the test result of the 1 kg test portion / test specimen indeed represents the original laboratory sample of 10 kg. As should the laboratory sample of 10 kg actually be a representative portion of the original product. Maintaining representativeness throughout the whole test procedure, from the first stage of sampling until the actual testing, is therefore essential.

NOTE 4 The size of the test portion / test specimen might put demands on the size of the laboratory sample, i.e. the laboratory sample should at least be sufficiently large to accommodate all test portions / test specimens necessary.

Especially when determining the emission into indoor air, probabilistic sampling may result in less effective sample selection at higher costs than educated or skilled selection of samples. Such sampling is to be based on knowledge of the key parameters influencing emissions properties of a certain piece of sample. The emission of dangerous substances across a certain amount of product often does not follow a statistically describable distribution, showing rather distinct changes depending on parameters such as actual composition, raw materials used, details of manufacturing process and storage conditions (e.g. temperature control, drying period), age of product and more.

NOTE 5 Some examples: Use of another source of tree may influence emissions of a wood based product. Purchase of nominally identical resin or dispersion from a different supplier may influence emissions of a water-based adhesive.

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Changing to cement from another mill may influence emissions of a cement-based product. Slightly elevated temperature due to sunshine on the roof of a manufacturing plant may influence remaining volatiles in the final product.

Additionally, incremental sampling shall be avoided when cutting is essential to obtain the individual increments, because the cutting edges creates fresh surfaces which potentially may disturb the release or emission test result.

Thirdly, products manufactured in a discontinuous manner are not always available as freshly manufactured products (although some products need aging before testing).

Therefore, the alternative approach comprises a targeted and informed selection of sampling date and sampling site, such that the sampled product represents either typical emission properties, or worst case elevated release or emission properties, taking into account the availability of the product at the selected sampling site. In this approach, specific technological knowledge is used to ensure representativeness instead of statistical observations.

The key terms as used in this CEN/TR are defined in Annex A.

2.1.3 Uncertainty

The associated uncertainty of the final test result is of major importance when assessing the release or emission of dangerous substances. The uncertainty is the result of variability in the obtained test result. Although often only one test result is obtained, that test result still is affected by the different sources of variability. When there is only one test result available, the variability is unknown; nevertheless the test result is partly determined by that variability.

Each activity necessary to obtain a test result has an effect on the variability and consequently on the uncertainty of that test result. Additionally, the variability of the product itself also contributes to the uncertainty. The sources of uncertainty of sampling are identified:

- Variability in the product (over time and / or space);
- Variability introduced by sampling activities and all subsequent activities until delivery of the sample to the laboratory;
- Variability introduced by laboratory activities up to the reporting of the results.

This CEN/TR deals with the first two sources of variability.

Sampling is at the very start of the assessment of a product. A series of subsequent steps is necessary to obtain the test result, based on which the actual assessment is performed. Starting at the end of that chain, the chemical analysis, and moving towards the first step of sampling, the quantification of the uncertainty associated with each of these individual steps become more and more difficult and costly. Consequently it is practically almost impossible to separate the uncertainty of sampling from the uncertainty of the subsequent steps.

There is no specific level of uncertainty that can be considered as being acceptable. The acceptability of a certain degree of uncertainty depends on the risk of non-compliance. If the risk of non-compliance becomes too big, the overall uncertainty associated with the test result(s) should be smaller in order to still be able to come to a decision. The risk of non-compliance is determined by the 'distance' of the obtained test result to the limit / limit value against which the product is assessed, and the variability of these test results (or potential test results if only one result is available). So, a relatively large degree of uncertainty is acceptable when the risk of non-compliance is low, while only a small amount of uncertainty is acceptable when there is a large risk of non-compliance.

With respect to the uncertainty see also Annex B.

2.1.4 Sampling under various stages for CE-marking

This CEN/TR provides guidance with respect to sampling for the product declaration under CE-marking although it does not cover the repetitive sampling which might be necessary.

NOTE 1 In the process of CE marking various stages can be identified, amongst which Initial Type Testing (ITT) and Factory Production Control (FPC), which result in a need for product sampling.

This CEN/TR provides guidance to obtain a sample, either by a single sampling operation or by incremental sampling in which increments are joined into a composite sample. This CEN/TR provides guidance to ensure that the obtained (composite) sample is sufficiently representative for the quantity of product it represents. As such it provides a basis for the CE-marking.

NOTE 2 The user expects that a product under CE-marking fulfils the specified requirements on the level of the individual product. While for consumer products, testing of each individual product might be an option, for environmental characteristics of construction products this often is not realistic.

NOTE 3 Repetitive sampling and subsequent assessment of a series of test result against limit values set by national authorities are highly related. This CEN/TR does not deal with the statistics necessary to determine whether a product complies with the limit values. As, at the time this CEN/TR was developed, no statistically defined objectives are set for determining if a test results complies, it is not possible to provide concrete guidance on the number of samples or test frequency necessary.

Whenever repetitive sampling is applied, not only the representativeness of a sample for a chosen quantity of product is of importance, but also the variations that occur over time. These are the variations that occur on the level of the quantity for which a sample is representative (see also 2.1.2). Consequently, the producer might be providing proof on the compliance of the product on a different quantity of product than the user expects. When making choices with respect to product sampling, this should be taken into consideration. The producer has to ensure that what is sampled is representative for the CE-marked product that is to be assessed.

NOTE 4 A consumer who buys a bag of cement of 25 kg assumes that this product fulfils the requirements. The consumer is unaware of the fact that the producer tests the product for quantities of for example 25 tons. Consequently, there is a certain, undefined risk that the quantity bought by the consumer does in fact not meet the requirements. The producer should be aware of this when defining the sampling (and testing) procedure.

2.1.5 Series of steps

A full test procedure can be described as a series of steps, i.e. definition of a sampling plan, taking of sample, on site sample pre-treatment, packaging, preservation, storage and transportation, delivery, storage and preservation, preparation of the test portion / test specimen, test to determine the release or emission, analysis / quantification, data management and reporting. These steps should be closely interlinked. This CEN/TR only provides guidance on the first few steps, from defining the sampling plan up to the delivery of the laboratory sample to the laboratory. This is depicted in Figure 1, wherein the main steps are numbered (1 to 7).

When defining part of this whole chain of activities, the implications of the whole chain should be taken into consideration in order to ensure that the test result is fit for purpose.

EXAMPLE When for example the release of dangerous substances from a construction product is strongly dependent on the presence of new surface, the sampling and subsequent preparation of the test portion / test specimen should be such that no new surface is created or measures are to be taken to isolate new surfaces prior to testing.

NOTE 1 The preparation of the test portion / test specimen might by itself consist of a number of steps.

NOTE 2 The preparation of the test portion / test specimen is often referred to as 'sample pre-treatment', but in this CEN/TR that term is exclusively related to on site sample pre-treatment, aimed to obtain a representative laboratory sample of acceptable size.

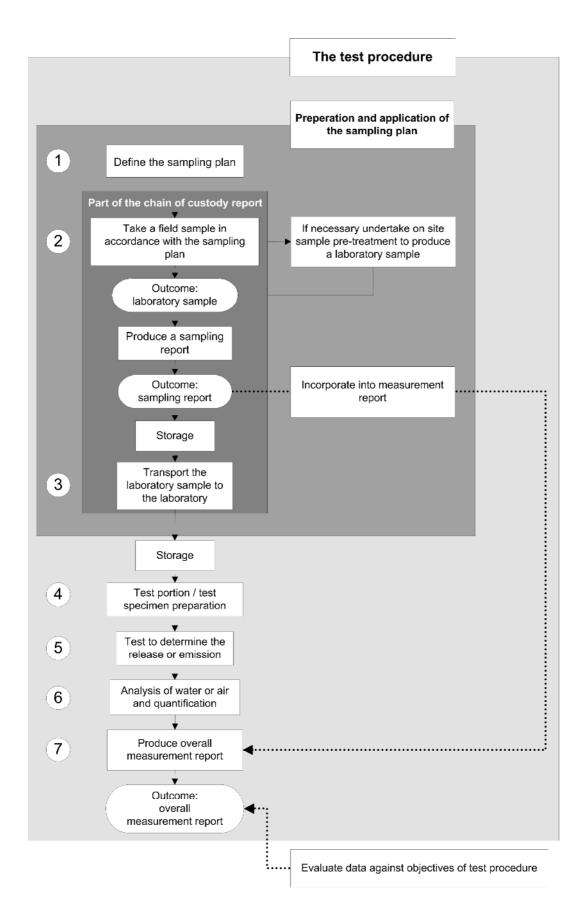


Figure 1 — Links between the essential elements of a test procedure wherein the main steps are numbered (1 to 7)

2.2 Objective of sampling

The objective of sampling the construction product is to obtain a sample that is:

- sufficiently representative of the quantity of product being assessed, see 2.1.4;
- fulfils the prerequisites for the test(s) to be performed.

See, with respect to being sufficiently representative, 2.1.2 and 2.1.3.

2.3 Preparation of a sampling plan

A sampling plan is to be completed prior to undertaking any product sampling.

By providing specific and practical instructions, the sampling plan defines the boundaries and logistics of product sampling as part of the test procedure.

The principles laid out in this CEN/TR can be used to produce a sampling plan for any test procedure within the framework of the CPD.

These principles can be used in:

- the development of a full test procedure by CEN/TC 351/WG 1 and WG 2 (see Clause 3);
- the development of a sampling standard or sampling instructions in a product standard by product TCs (see Clause 3);
- the production of standardized sampling plans for use under routine circumstances.

NOTE 1 The latter may be applied for example by an individual producer for application within the context of FPC.

In the process of defining a sampling plan, the key steps of the test procedure (as shown in Figure 2) are to be addressed. The definition process should:

- a) Identify those individuals and organizations with an interest and detail the proposed sampling design in agreement with the requirements as specified by those involved parties;
- b) Identify the requirements arising from other key steps in the test procedure;
- c) Establish specific instructions for when and where, and how many samples and / or increments should be taken;
- d) Identify all safety precautions that are to be taken.

NOTE 2 The specific details contained within any sampling plan differ according to the objectives of the test procedure, the product to be sampled and the sampling circumstances.

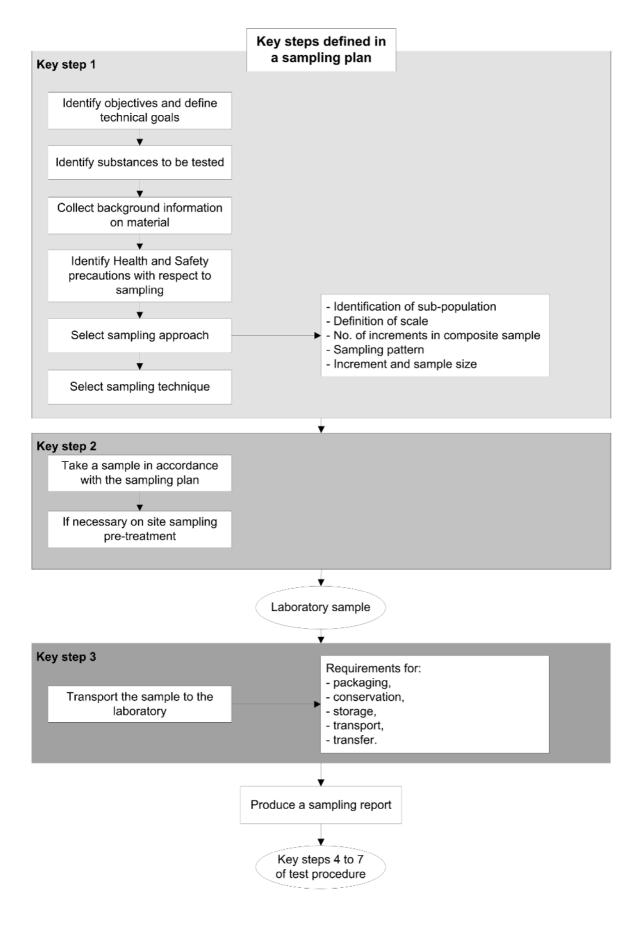


Figure 2 — Details on the key steps of product sampling

2.4 Considerations on sampling strategy

2.4.1 General

General considerations taken from statistical observation techniques are helpful for specifying a sampling strategy for achieving representative test results.

NOTE 1 The term 'sampling strategy' is used in other documents with different definitions. In this CEN/TR the term 'sampling strategy' relates to the aspects as referred to in the following sub-clauses: the sampling approach, population and sub-population, scale, increment and sample size and the sampling location and moment. In general terms, the sampling strategy takes account of the factors that determine what the sample is representative for, and how the representativeness of the sample is ensured.

NOTE 2 For good understanding of the concepts in the next sub-clauses, also see the definitions in Annex A.

2.4.2 Sampling approach

Two basically different approaches for sampling can be used:

- probabilistic sampling, in which approach every item within the scale has an equal chance of being (part of) the sample;
- judgemental sampling (or informed sampling), in which the selection of (an) item(s) within the scale that is (part of) the sample is based on knowledge about the product to be sampled and the process it originates from.

Probabilistic sampling, wherein a random mechanism determines which items are part of the sample, should be preferred if insufficient knowledge on product and process is available, as only under the conditions of probabilistic sampling sound statistical conclusions can be applied on the obtained test results. However, when sufficient information is available, the quality of the results obtained with judgemental sampling can be the same as for probabilistic sampling.

The term 'judgemental sampling' covers a wide range of sampling approaches, going from small deviations of a fully probabilistic approach, towards the judgemental selection of specifically identified items.

NOTE The use of judgemental sampling does not imply that the quality of sampling and specifically the representativeness of the obtained sample are insufficient. When for example the production process is well known, the specifically chosen item might by itself provide a good estimate of the characteristic of interest, while using probabilistic sampling would need a number of increments and/or samples to obtain a good estimate.

When developing the sampling plan, an informed decision should be made on the sampling approach to be used. This choice is to be based on:

- Full understanding of the concepts of probabilistic and judgemental sampling.
- The circumstances under which product sampling is to be performed.
- The availability of detailed knowledge on the product to be sampled and the process it originates from.
- The test(s) to be performed.

2.4.3 Population and sub-population

The term 'population' is defined as the "totality of items under consideration". A sub-population is defined as a "defined part of the population that is targeted for the purposes of sampling".

The quantity of products covered by the CE marking declaration is defined as the sub-population.

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The sub-population is to be defined, taking into account:

- The objective of testing;
- The resources available;
- The consequences of non-conformity;
- The production process and the raw materials used.

NOTE 1 As each sub-population is, from the perspective of sampling and testing, seen as an entity, testing is necessary for each sub-population. Defining small sub-populations consequently implies a high sampling and testing effort for the population, and consequently a big demand on available resources. Opposite, when defining large sub-populations the financial consequences of non-conformity are high. Ultimately, the definition of the sub-populations is based on a balanced decision between costs of sampling and testing and the financial consequences of non-conformity.

NOTE 2 Whenever there is a possibility to link the definition of sub-populations to the production process, this should be done, as in general this allows the definition of sub-populations that in themselves are less variable in comparison to randomly defined sub-populations of the same size.

NOTE 3 Referring to the example in 2.1.2, the three series from each of which four tiles are taken, would be three individual sub-populations.

In specific cases, the sub-population might be defined as a group of related products, for which the release or emission of dangerous substances is tested on the product that has the highest release or emission potential within that group. If this specific product conforms, it might be assumed that all other products in that group also conform. Obviously it is essential in such a situation that the assumptions on which the proof is based are sufficiently substantiated.

EXAMPLE A manufacturer produces autoclaved aerated concrete, with various product dimensions. The production is batch-wise, wherein the batch size is 20 tons. Raw materials used in the production process are sand, lime, cement and gypsum in a constant mixture. Assuming a constant density of the products, the release or emission of dangerous substances is basically independent of the dimensions of the individual products. To a minor degree, variations in the release or emission relate to the batch in which a product is produced, but the major determining factor are variations in the raw materials. In this (somewhat simplified) example, the population covers all products produced with that same process. In the definition of a sub-population, account is taken of the fact that changes in the origin of the raw materials might have an impact on the release or emission of dangerous substances. A sub-population therefore is defined as all products produces with a specific process using raw materials of specified sources.

2.4.4 Scale

The 'scale' is a crucially important element in defining a test procedure. It defines the minimum quantity (mass or volume) of the product for which test results are obtained. Information on characteristics of the product, including release or emission and variations therein, for a quantity of product smaller than the defined scale, is judged to be unimportant.

The amount of variability in the population cannot be quantified without defining the scale on which that variability occurs.

A given scale represents the quantity of product on which the measurement is based. Consequently, the mean value obtained for that scale is used to assess the construction product.

It follows that when obtaining information about a product at the specified scale, each numerical value is a mean for the volume or mass of product at that scale.

The scale should be fixed in the product standard. In establishing the scale, the following aspects should be taken into account:

the variability of the product;

- the possibilities to obtain a smaller, but still representative, laboratory sample through on site sample pretreatment:
- the possibility to obtain a smaller, but still representative test portion / test specimen;
- the possibility to obtain a smaller, but still representative, laboratory sample through an informed decision on the place or moment of product sampling;
- the costs of product sampling and when necessary subsequent on site sample pre-treatment;
- the risk of non-compliance and the acceptability of that risk for both producer and consumer;
- the practicalities of product sampling;
- the possibility to obtain a representative sample for a (much) larger quantity of product.

The scale should be fixed, independently of the level of testing (e.g. ITT or FPC) performed on the product.

NOTE 1 The final quantity of product used for a test still has a certain mass (or volume). Assuming some variability even within this quantity of product, implies that the test result is a mean value, influenced by both highs and lows within that quantity. Consequently, all test results represent a mean value of a certain mass or volume, independent of the size of that mass or volume and independent of the degree of variability within that mass or volume.

NOTE 2 Quite often, the choice of scale is not consciously made and is just based on the amount of product that is practical for sampling or necessary for the tests to be performed. Be aware of the fact that such an implicit decision on the scale might result in high variability between results and consequently a larger chance of non-compliance of the product. Assessing the product on a larger scale is often beneficial (see Example 4 in this clause). As a consequence, the size (mass or volume) of the laboratory sample is larger than the test portion / test specimen necessary. On site sample pretreatment of the obtained large sample or composite sample is necessary to obtain a representative laboratory sample. The on site sample pre-treatment, as well as the production of the test portion / test specimen in the laboratory, has to ensure that the test portion / test specimen is representative for the mean value of the large sample or composite sample. As such, it results in a much better estimate of the mean characteristic of the sub-population. A direct effect of this approach can be a lower sampling frequency.

- NOTE 3 Choosing a too small scale leads to test results that do not sufficiently represent the product sampled. The variability is higher than what is expected.
- NOTE 4 Fixing the scale in the product standard enables a level playing field in testing costs between different producers of the same product.
- NOTE 5 If the scale would be fixed differently between ITT and FPC, there is a considerable chance that the results of ITT would no longer be representative for the results obtained through FPC.
- EXAMPLE 1 Referring to the example in 2.1.2, two different scales are used. In the situation where four individual tiles are taken from each of the three sub-populations, the scale is a single tile. Information on the release or emission is obtained on the level of an individual tile. As the tile is the lowest quantity of product for which information is obtained, no information is available on the variability within an individual tile. In this example this is considered to be unimportant.

In the second part of the example, the four tiles from a sub-population are tested together. The scale has been enlarged to the quantity of four tiles. No information is obtained on a smaller quantity than four tiles and in this second part it is therefore assumed that this variation is unimportant. At the same time, as long as only one set of four tiles is obtained from a sub-population, there is no information available on the variability within the sub-population. The total number of tiles in the sub-population is far larger than four, so there is still a difference between the quantity of product to which the test result is directly related (four tiles) and the quantity of product that is assessed on the basis of that result (the sub-population).

Finally, in the example in 2.1.2 the results of the three sets of four tiles are compared to each other. In this situation the scale is still a quantity of four tiles, but now the definition of the sub-population has changed. The sub-population in the last part of the example is equal to the three sub-populations in the first part of the example.

EXAMPLE 2 The variability in a product from gram to gram is likely to be larger than the variability in that same product from kilogram to kilogram. If variations on such a fine scale as grams are believed to be important, then that is the scale on which the sampling should operate. If, conversely, variations within any one kilogram of product are irrelevant, the primary aim of the product sampling should be to quantify variability solely on the kilogram-to-kilogram scale. It is therefore of vital importance that the scale is stated explicitly.

EXAMPLE 3 Let's assume that the test portion / test specimen for testing a product is 1 kilogram. When the scale is not considered when defining the sampling plan, a sample is obtained of 1 kilogram. Variability between samples of 1 kilogram is substantially higher than variability on a scale of 1000 kilograms. At the same time, it is not possible to take a 1000 kilogram sample. Therefore the scale of 1000 kilograms may be sampled by incremental sampling. The number of increments taken from the selected 1000 kilograms needs to ensure that the resulting composite sample still provides a good estimate of the mean value for 1000 kilograms. The number of increments necessary depends on the variability on the scale of the individual increments, for example 250 grams, and the accepted uncertainty in the estimated mean value. Assuming this is fulfilled with 20 increments, the resulting composite sample is 5 kilograms. Preparation of the test portion / test specimen in the laboratory then has to ensure the representativeness of a test portion / test specimen of 1 kilogram from the composite sample. As the variability on the scale of 1 kilogram is substantially higher than the variability on the scale of 1000 kilograms, the chance of non-compliance is substantially higher when using a (implicit) scale of 1 kilogram instead of a scale of 1000 kilograms.

EXAMPLE 4 An evaluation of the risk on non-conformity is specifically important when there is a large difference between the quantity of product from which a (composite) sample is obtained, and the quantity of product that is usually applied by the consumer. Imagine a situation where there is a product TC for a specific type of floor covering. This product TC has chosen to set the scale at the median daily production of the involved producers, being 25.000 m². Consequently, the test result for the release represents the average release for a quantity of 25.000 m² with a test portion / test specimen size of 1 m².

This type of floor covering is mainly used by private consumers, where the median quantity applied is 50 m². Obviously, it is the expectation of the consumer that, in light of the CE-marking, the release of the product complies for the quantity that is used. Assuming little variability over the days production, a single randomly chosen part of 1 m² from that daily production has a release which is comparable to the release of any randomly chosen part of that days production. In such a situation, the producer does not need to worry (to much) about the difference between the chosen scale (25.000 m²), and the quantity of the product that is normally used (50 m²). However, if a large degree of variability is to be expected over a days production, there might be an unacceptable large chance that the product does not comply for a certain quantity of 50 m², despite the fact that the mean of the whole day does comply. Consequently, the producer might expect consumer complains. Apart from making changes to the process to enable a more stable quality of the product with respect to the release of dangerous substances, the product TC should also consider to define a smaller scale than which was originally chosen. By choosing a scale equal or comparable to the median quantity applied, the risk of consumer complains is far less. At the same time, test results clearly indicate the need to stabilize the production with respect to the release of dangerous substances.

EXAMPLE 5 When comparing test results of a relatively heterogeneous construction product with a given class limit, a well chosen scale can have a significant effect on the conclusions with respect to (non-)compliance. In Figure 3 the test results of a product over time are depicted. Tests have been performed with test portions / test specimens obtained from composite samples of 500 grams. These composite samples were obtained from a product sampled over time on a scale of 100 kilograms, 1000 kilograms and 2000 kilograms. The cut off level for non-conformity is set at 1,75 and is shown as a horizontal line. It is obvious that due to variations in the product on a small scale (100 kg), the product frequently exceeds the level of non-conformity. Assuring the quality of the product on the market based on test for the 100 kg scale, would, in light of the considerable risk of non-conformity, imply high frequent testing and would consequently be expensive. However, when testing the same product on a larger scale of 1000 kg, the product complies always. Consequently, the test frequency can be much lower. Finally, when looking at the product quality on a scale of 2000 kg, there is little variability in the product, it lies well below the cut off and consequently a low test frequency is possible.

EXAMPLE 6 The manufacturer of autoclaved aerated concrete in the example in 2.4.3 defines the scale on which information on the sub-population is obtained as the amount of products produced during a single autoclave step. Given the production facility, this is a quantity of four batches of 20 tons each.

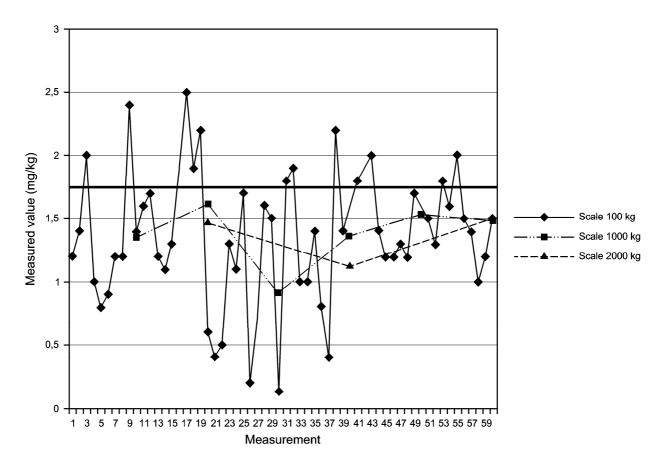


Figure 3 — Results over time for a product tested at three different scales, 100 kg, 1000 kg and 2000 kg, where the horizontal bold line is the cut off for non-conformity

2.4.5 Size of increments and samples

2.4.5.1 General

An increment is an individual portion of a product collected by a single operation of a sampling device which is not tested as a single entity, but is mixed with other increments in a composite sample.

NOTE 1 Whenever the portion of product collected by a single operation of a sampling device is analysed individually, the obtained product is called a sample. In such a situation the quantity of product has to fulfil both the criteria for the size of an increment as well as for a sample.

A composite sample consists of two or more increments, put together in appropriate portions, from which the mean value of a desired characteristic may be obtained.

Whenever the scale is larger than the quantity of product used in the test, measures should be taken to ensure the representativeness of the sample for the quantity of product at the chosen scale. When technically possible, incremental sampling is an effective strategy to obtain a good estimate of the mean characteristic for the scale.

NOTE 2 The representativeness of the composite sample obtained directly depends on the variability of the product on the scale of the individual increments. In principle variability on that small scale is considered to be unimportant as the chosen scale is (much) larger. However, when the variability on the scale of the increments is known, and the accepted level of uncertainty is defined, the number of increments that are to be combined in a composite sample can be calculated.

NOTE 3 Incremental sampling does not per definition imply mixing of the product. Given the example of 2.1.2, an increment is an individual tile, while the composite sample consists of four individual tiles. When the quantity of product obtained in this way is suitable for the test, the full composite sample can be tested. Sub-sampling from the individual tiles

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might become necessary when the test portion / test specimen has the size of an individual tile. Then a quarter of each of the tiles should be obtained through sub-sampling when producing the test portion / test specimen.

NOTE 4 Except for granular products and small shaped products, incremental sampling normally is no option when determining the emission into indoor air. Cutting small pieces out of a larger product and later recombination in a composite sample cannot be used because the cut edges disturb the emission test result.

More details on the increment and sample size can be found in Annex C as well as in CEN/TR 15310-1.

2.4.5.2 Particulate / granular products

Sampling of particulate / granular products for determining the emission to indoor air does, within the scope of the CPD, not often occur. However, if it might occur, the sampling strategy for product sampling might be the random selection of the smallest commercially available packaging. If the so obtained quantity of product is too large for transport to the laboratory, then sub-sampling may be performed, see 2.6.

NOTE 1 Typical products appearing as particulate or granular products are mortar, unbound aggregate, drainage aggregate, porous granular material and construction debris.

The degree to which account needs to be taken of the sample and increment size depends very much on the product sampled. For particulate / granular products, the minimum increment size is governed by the need for the sampling device to accommodate all particle sizes.

A sample should be sufficiently large in order to minimize or exclude errors caused by the fundamental variability of the particulate product that is determined by the differences between individual particles.

NOTE 2 The fundamental variability is the inherent variability which is shown by a particulate product at the smallest possible scale of measurement; for particulate products the scale of the individual particles. Whenever a product consists of a mixture of different raw materials, there are differences between the individual particles. The fundamental variability should not be confused with the variability that is due to the preferential presence of a specific type of material in a specific part of the sub-population. The latter type of variability (segregational variability) should be dealt with by the sampling pattern.

Powders are basically particulate products with a (very) small particle size. Provided the sample device allows the entry of all particles present in the product being sampled, there are no additional requirements for the minimum increment size. Similarly, given the small size of the particles in these types of products, the differences between individual particles do not have a major affect on the characteristics of a sample, as in practice, the sample is large enough to consist of a (very) high number of particles. Therefore no practical requirement applies for the minimum sample size due to sampling. Thus the minimum sample size is governed by the quantity of material required by the laboratory for analysis, whilst the dimensions of the sampling device determine the increment size.

NOTE 3 The particulate behaviour of powders with a very fine maximum particle size (e.g. $< 63 \mu m$) is, as long as the material is free flowing, highly comparable to the behaviour of liquids, for which also no specific requirements are set for sample and increments size.

NOTE 4 Although the distinction between a powder and a granular product is not always obvious, the consequences on the minimum increment and sample size are potentially large. Care should be taken, therefore, that the aperture of the sampling device is suitable for the particle size distribution in the product to be sampled.

When sampling particulate products with a very large particle size, these products are to be sampled as being small or large shaped products, which ever is the most appropriate. See 2.4.5.3 or 2.4.5.4.

Particulate / granular products are to be sampled with a sampling device of which the aperture is large enough to allow the entry of all particles present in the product. The aperture of the sampling device should also be large enough to allow the simultaneous entry of all different particle sizes within the product. In practice this means that the device opening should be at least three times the diameter of the largest particles. Further details can be found in Annex C.

For particulate or granular products the composition of individual particles could have a substantial influence on the composition of the sample, and the minimum sample size should be large enough to compensate for this. This is particularly important when the characteristic of interest constitutes only a small proportion or fraction of the product and consequently this implies that with minor constituents a larger sample size is required.

It is assumed that, in characterizing the product, the composition of the individual particles is not of interest. It is the average composition of the product (at the specified scale) that needs to be determined. To measure that average composition, the sample should contain a sufficient number of particles to ensure that the effect of any individual particle within the sample does not have a disproportionate effect on the total composition of the sample.

NOTE 5 In some situations the analysis of individual particles e.g. asbestos might be of interest. Still this does not imply that the material is to be sampled on a particulate level.

The actual size of the increments and samples does not depend only on the minimum increment and sample size but also on:

- the quantity of material required by the laboratory for analysis;
- the number of increments in a composite sample (when increments are taken); and
- the relation between the mass of the minimum increment size and the minimum sample size (in relation to the number of increments in a composite sample).

NOTE 6 In cases where a powder or granulate product is produced in, for example, bags or another package of acceptable size, an individual bag might be considered as the increment or sample, see 2.4.5.3.

Whenever the primary increment or sample is too large for transportation to the laboratory, on site sample pretreatment should be applied to obtain a representative laboratory sample, see 2.6.

2.4.5.3 Small shaped products

For shaped products with a product size that is sufficiently small to handle, an increment is of the same size as the product. Depending on the expected product variability with respect to the release or emission of dangerous substances, an individual product might also be a sample. If there is too much variability in the release or emission of dangerous substances between individual products, incremental sampling should be performed. The resulting composite sample in this situation comprises of two or more individual products which are separately obtained during sampling, but are tested together as being one test portion / test specimen.

NOTE When the quantity of product so obtained is too large to perform an emission test, a single product obtained by targeted sampling can be used as a sample assuming representativeness as explained in 2.1.2.

EXAMPLE The manufacturer of autoclaved aerated concrete of example 2 in 2.4.3 (population and sub-population) and example 5 in 2.4.4 (scale) delivers both small as well as large shaped products, depending on the market demands. In order to obtain a sample from the scale, three random choices are made:

- a) the selection of the moment when the sample is taken, i.e. which day, time and groups of batches;
- b) the exact batch from which the sample is obtained;
- c) the product that is the sample.

When this specific product is sufficiently small to be handled, the full product is the sample.

2.4.5.4 Large shaped products

For shaped products that are too large to handle in a reasonable manner, two options are available for sampling:

selection of an individual product and subsequent cutting of an increment or sample;

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 sampling of the raw mixture from which the product is produced and subsequent production of a small scale specimen, representative of the product for testing.

Which option is chosen depends on what is the best applicable choice in a specific situation. The choice has to be fixed in the product standard.

In the first option, product sampling is a two steps procedure, wherein the first step an individual product is selected. In the second step a part of that product is cut off which is the increment or sample. If it is the sample, it should comply with the specifications of the test portion / test specimen. If it is an increment, those specifications are to be met by the composite sample obtained of this increment together with other increments.

NOTE 1 Cutting of a part of the shaped product is necessary in light of practical restrictions to sampling. The cutting itself results in new surface on part of the sample which might result in enhanced release or emission of dangerous substances. Consequently the sample is to be obtained in such way that a minimum of new surface is created. Any newly created surfaced should be wrapped or sealed.

Alternatively, for shaped products that are too large to handle in a reasonable manner, it might also be an option not to sample the product as available for its intended use, but sample the raw mixture from which it is produced. A test portion / test specimen is then to be produced from the raw mixture of materials. The conditions for particulate products in 2.4.5.2 apply when sampling the raw mixture. The specifically produced test portion / test specimen has to comply with the specification set for the test portion / test specimen.

NOTE 2 The sampling of the raw material offers some major advantages; i.e. the easiness of sampling and the final product is not destroyed by sampling. A major disadvantage is however the fact that a new product is produced for testing which might have different properties with respect to the release or emission of dangerous substances, especially with respect to ageing of the final product.

EXAMPLE If in the example in 2.4.5.3 a product is selected which is too large to be handled, an additional step is to be performed in order to cut a piece, the size of the test portion / test specimen, of the selected product.

2.4.5.5 Composite products

Composite products are products in which two or more individual products are jointly applied.

NOTE 1 The product declaration under the CPD is targeted at the product 'in their intended use'. As some products per definition are applied together with other products, this should be taken into account, specifically as the release or emission of the product might be highly dependent on the combination of products.

NOTE 2 Examples of composite products are windows, heating appliances, coated roof products, coated bricks and tiles, wall systems, road equipment (e.g. a lighting column), etc.

For sampling purposes, composite products can either be sampled in their final joint status, or as individual products prior to their final application.

When sampling composite products in their final joint status, they are to be sampled in accordance with the procedures for sampling of small shaped products (see 2.4.5.3) or large shaped products (see 2.4.5.4), depending on the size of the composite product. If the size of the composite product implies cutting, this should be done in such way that all individual products are equally present in the resulting sample as they are in the original product.

When sampling the individual components of the composite products, sampling of the individual components has to be performed as for any product. However, specific demands might result from the need to produce a test portion / test specimen prior to testing in which the different products are joint together into a model of the original composite product.

Another option is to sample and to test the ingoing components separately. In that case, total emissions are re-calculated from the individual test results by weighted addition of emissions, taking into account the surfaces of each component relative to total surface of the composite product. A similar approach can also be used for the determination of the release.

2.4.5.6 Not finalized products

Products that are not brought onto the market in their final form (e.g. cement based adhesives) need to be brought into their final form (e.g. a typical layer of tile adhesive) before testing to determine the release or emission of dangerous substances. Sampling of these products follows the rules provided in 2.4.5.2 up to 2.4.5.5 depending on the nature of the product.

2.4.6 Sampling location and moment

Two different aspects are covered by 'sampling location and moment':

- a) The definition of the location where and moment when a sample or individual increment is taken;
- b) The definition of the locations where and moments when a series of samples is taken.

This CEN/TR provides direct guidance on aspect a), while for aspect b) only limited guidance is provided. Apart from the variability within the sub-population on the predefined scale, the necessary number of samples directly relates to the requirements for the overall uncertainty of the test procedure.

NOTE 1 As the requirements for the overall uncertainty are not set at the time this CEN/TR was prepared, the statistical testing is no part of this CEN/TR.

This CEN/TR provides guidance on obtaining samples and increments (joint in a composite sample) which are sufficiently representative for the sub-population sampled. Detailed guidance on the calculation of the required number of increments and samples is provided in Annex D as well as in CEN/TR 15310-1.

NOTE 2 Knowledge on the variability of a product, in light of defined choices about the sub-population to be tested and the scale on which these tests are to be performed, might be available for product TCs. If this information is not yet available, it would be highly beneficial to obtain that information as this allows for informed decision on the cost effective testing of the product.

Samples or increments should be taken from the factory as brought onto the market (ready for dispatch to distributors or users), as typical as possible for the total volume of production that is CE-marked (the sub-population). Sample location and moment should be selected such, that raw materials and production process are as typical as possible with respect to the tested properties. Within these boundary conditions, samples or increments are to be taken based on random or stratified random sampling.

2.5 Application of sampling techniques

A sampling technique should be used that takes account of:

- the size of the sample or increment to be taken;
- the product to be sampled;
- the measures necessary to maintain the integrity of the sample or increment.

For considerations with respect to the size of samples and increments see 2.4.5.

Samples may be selected in form of discrete units, e.g. tiles or a window, or in commercial packaging (e.g. bags or cans). In these types of situations, samples are obtained by hand picking. Samples may also be taken from bulk (e.g. during loading or unloading of silos or large containers).

Sampling activities should have no, or as little as possible, impact on the integrity of the sampled product. For the determination of the release or emission of dangerous substances, this relates to:

exclusion of the evaporation of volatile substances;

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- exclusion of the loss of material;
- exclusion of the creation of new surface (unless necessary for obtaining a sample, see 2.4.5.4);
- exclusion of the deterioration of the product due to heat production during sampling or sub-sampling;
- exclusion of contamination of the sample or increment, either by sampling equipment, by other samples, by the surrounding environment or by cosmetic products used by the sampler;
- exclusion of contact of the sample or increment with water (release) or air (emission) after sampling.

NOTE Consequently the use of cutting liquid or oil is prohibited when cutting a part of a shaped product.

Taking the necessary measures relates also to packaging, preservation and storage conditions, see 2.7.

2.6 On site sub-sampling / on site sample pre-treatment

Sub-sampling is the process of selecting one or more sub-samples from a sample of a sub-population, wherein the sub-sample is a quantity (mass or volume) of product obtained by procedures in which the characteristics of interest are randomly distributed.

The scope of this CEN/TR is limited to the on site sub-sampling, directly after sampling and prior to packaging of the sample. It is aimed at obtaining a laboratory sample that fulfils the requirements with respect to:

- representativeness (see 2.1.2);
- integrity;

NOTE 1 The integrity of the sample implies that no material is lost or obtained after sampling without the explicit purpose to reduce the amount of material, and that the properties of the sample with respect to the characteristic of interest – in this case specifically the release or emission of dangerous substances – are not changed.

- being a quantity that is practical for transport to the laboratory;
- being a quantity that is sufficient for the test(s) to be performed;
- fulfilling the demands posed by the minimum sample size (see 2.4.5).

On site sub-sampling of granular products is limited to division of the sample into smaller quantities, but always without reducing the size of the product.

Additional guidance on sub-sampling in the field can be found in CEN/TR 15310-3.

NOTE 2 Two potentially confusing terms are encountered here: the reduction of the sample size and the reduction of the particle size. Sample size reduction is allowed – within certain boundaries – and is the objective of on site subsampling.

With respect to particle size reduction, a distinction is to be made between granular / particulate products and shaped products. For granular / particulate products particle size reduction implies some kind of grinding technique. This is not allowed on site due to a considerable risk with respect to the integrity of the sample. Particle size reduction of granular products is only allowed under laboratory conditions. For shaped products reduction of the size of the product means taking / cutting smaller units than originally obtained through sampling. This is an activity that can be performed on site.

NOTE 3 Specific care should be taken when sub-sampling a composite sample obtained through incremental sampling. Depending on the variability of the product on the scale of the individual increments (the 'within scale' variability), a composite sample can be highly heterogeneous. In such cases the process of sub-sampling can easily result in a biased sub-sample / laboratory sample.

EXAMPLE Tiles are packed in quantities of 10 tiles. During the first stage of product sampling, a package of 10 tiles is selected as a sample. As only one tile is supposed to be sufficient for reliable testing in this example, the sub-sampling

process would be the random selection of one of these tiles from the original package. Note that from the perspective of sample integrity it might be a good idea to keep the original package closed and transport it as a unit to the laboratory, in which case sub-sampling is performed in the laboratory.

NOTE 4 Particle size or product size reduction is allowed under laboratory conditions under the provision that the integrity of the sample can be maintained, but falls outside the scope of this CEN/TR except for situations where a sample is to be obtained from a large shaped product, see 2.4.5.4.

2.7 Packaging, preservation, storage and transport

2.7.1 Packaging

The purpose of the sample container (packaging) is to protect the sample during transport and storage until it is further treated or analysed. A container should be compatible with the nature of the product sampled and the substances to be analysed. In general:

- pack samples for the determination of release in plastic containers (if only release of inorganic substances is of interest) or metal / glass containers (if release of organic substances is of interest);
- pack granular and liquid samples for the determination of emission in glass or PE containers;
- wrap samples of shaped products for the determination of the emission in aluminium foil and then in PE foil, or in combined aluminium / PE material.

Product packaged in commercial packaging of acceptable size per unit, which can be sent to the laboratory as an entity, might be sent to the laboratory without additional packaging.

NOTE 1 The risk of contamination and / or loss of substances might be unquantifiable when the original package is used as packaging material, in which case it should be considered to repack the product.

NOTE 2 Glass containers often are not available, specifically for granular products with larger particle size or shaped products. For these types of products plastic containers are to be used to ensure at least gas tight packaging. However, several plastic packaging materials are not appropriate for testing for a number of volatile substances which might be of interest. Consequently, the selection of adequate packaging material needs thorough consideration.

The following factors are most important with respect to the release or emission of dangerous substances:

- adsorption of substances at the walls of the container or packaging material;
- contamination of the container prior to sampling (e.g. by improper cleaning);
- contamination of the sample by the material of which the container or packaging material is made;
- reaction between substances in the sample and the container or packaging material;
- introduction and then adsorption of substances from surrounding air through the walls of the container or packaging material;
- the container or packaging material should fit tightly around the sample.

NOTE 3 Sample containers can be made from many different types of materials, some of which may react or contaminate a specific type of sample. To avoid any accidental contamination by the sample container or derogation of the sample, advice should be sought, usually from the receiving laboratory, regarding the type of container(s), appropriate preservation method(s) if applicable, maximum storage time prior to analysis, and the labelling system. The maximum storage time prior to analysis indicates the period of time available before the sample has to arrive at the laboratory. In general, this period has to be as short as possible.

Annex E provides details for the use of containers for different substances.

2.7.2 Preservation

The method of preservation relates to the stability of samples. Preservation methods are considered under the following broad headings:

- products for which the release or emission is stable no action needed;
- products for which the release of emission is unstable action needed.

NOTE 1 Examples of products which can be unstable with respect to the release to soil and water are alkaline cement based products or alkaline fly ash, for which the level of carbonation determines the leaching properties.

For products for which the release or emission is unstable, preservation measures should be taken in order to minimise loss of dangerous substances or changes to the product. Changes may occur due to various environmental factors, including:

- oxidation of substances by atmospheric oxygen;
- loss of dissolved volatile substances due to direct contact with surrounding air;
- photochemical reactions;
- modification of the pH, conductivity, solubility and carbon dioxide by absorption of CO₂ from the air;
- reaction with carbon dioxide or water;
- microbial activity.

The selection of a suitable preservation method depends on the product to be preserved and the dangerous substances for which the release or emission is to be determined.

Applied preservation methods are:

- air tight storage;
- dark storage;
- cooled storage (< 4 °C ± 2 °C)

NOTE 2 Cooled storage is not applicable to the determination of the emissions to indoor air because cooling and reconditioning may impair product properties by condensation of water on the laboratory sample.

NOTE 3 The time between product sampling and testing is particularly important for products for which the release or emission is unstable. This is specifically relevant in situations where the preservation measures result a reduction of the changes (e.g. cooling with respect to microbial degradation), instead of a total stop of changes. Despite the preservation measures taken, for those situations it is recommended to keep the time between product sampling and analysis as short as practically possible.

Annex E provides details for preservation for different substances.

2.7.3 Storage

Storage time should be kept to a minimum.

NOTE In general, the storage time covers the time the sample is available in the field after sampling and prior to delivery at the laboratory, as well as the time between arrival in the laboratory and start of the test(s). Within the scope of this CEN/TR storage time is limited to the period between sampling and delivery at the laboratory, but obviously account should be taken of the total storage time.

Storage conditions should be in line with the preservation conditions for the sampled product, see 2.7.2.

Annex E provides details for storage conditions for different substances.

2.7.4 Transport

Storage conditions should be maintained during transport.

NOTE Transport conditions during long transport in cars or during flights are to be considered if they occur. Specifically when products are transported by car excessive heat can cause problems with sample integrity. For transport by airplane, the partial loss of air pressure might, if samples are not packed airtight, be of influence specifically on the emission of volatile substances.

2.8 Sampling report and chain of custody report

The sampling report provides information on all field activities during product sampling and includes any important observation of the sampler. A sampling report is to be prepared for each sampling operation.

It is important that the sampling report is detailed and accurate, as the information is useful when interpreting the results and assessing the representativeness of the samples.

Two example forms of a sampling report are provided in Annex F.

NOTE In case a large number of samples are taken it can be helpful to use a specific sampling report containing information on actual sampling conditions and observations.

The chain of custody report provides information on who is responsible for the sample at any point in the test procedure. A chain of custody report is to be prepared for each test procedure.

The chain of custody report is the link between the product sampling and the preparation of the test portion / test specimen in the laboratory and serves to maintain representativeness and traceability of the test portion / test specimen back to sampled sub-population.

An example form for the chain of custody report is provided in Annex F.

3 Recommendations to CEN/TC 351/WG 1, CEN/TC 351/WG 2 and product TCs

3.1 Introduction

The essential elements for sampling of construction products to determine the release or emission of dangerous substances are covered in general terms in Clause 2. Clause 2 is therefore essential for a good understanding of what is to be incorporated with respect to sampling in the overall test procedures as developed by CEN/TC 351/WG 1 and WG 2, as well as for the incorporation in sampling standards or sampling instructions in the product standard of product TCs.

It should be noted that this CEN/TR is a complement to sampling. Consequently the guidance provided in this document is no sampling instruction on its own.

This Clause 3 provides some specific guidance to WG 1, WG 2 and product TCs, as well as providing examples to clarify what is stated under Clause 2. Consequently Clause 3 is to be read in conjunction with Clause 2.

NOTE 1 This Clause 3 and its sub-clauses do not contain any specific references to Clause 2 and its sub-clauses; for easy reference the same clause number in Clause 2 can be consulted.

NOTE 2 The determination of the release or emission of dangerous substances may imply the involvement of specialised laboratories. Probably these are other laboratories than those used for testing the construction properties of the product.

CEN/TC 351/WG 1 and WG 2 should pay attention to the overall uncertainty. See also Annex B.

3.2 Objective of sampling

The objective of sampling a construction product is to be specified within the scope of the product standards when adding ER3 properties to product standards for CE-marking.

Based on an evaluation of Clause 2, product TCs have to identify if the objective for the determination of the release and / or emission of dangerous substances can be integrated in the scope of their sampling standard or instructions, or that an additional sampling standard or instruction is to be developed.

3.3 Preparation of a sampling plan

For CEN/TC 351/WG 1 and WG 2 as well as product TCs, it is important that the development of a sampling plan prior to actual product sampling is included in the complete test procedures that they have to develop. In the sampling plan, the topics as addressed in this CEN/TR are to be concretized.

Existing sampling plans for other testing may not cover sufficiently the needs of release into soil and water or emissions into indoor air testing.

For repetitive sampling the same product at the same location, a single sampling plan can be developed which can be used each time when a sample is to be taken.

3.4 Considerations on sampling strategy

3.4.1 General

Good understanding of the concepts for the key terms population, sub-population, scale, increment, composite sample and sample is essential for the development of a test procedure. See the definitions in Annex A.

3.4.2 Sampling approach

CEN/TC 351/WG 1 and WG 2, as well as product TCs, have to make an informed decision on the sampling approach, either applying probabilistic sampling or judgmental (or informed) sampling.

NOTE 1 An informed decision in this context means that they have to balance the pros and cons of possible sampling approaches. This implies balancing the necessary effort and costs of sampling and the practical possibilities for sampling against the (statistical) representativeness of the test result for the sampled sub-population and the objective of sampling. Obviously such an informed decision is only possible when detailed knowledge is available on the product to be sampled and the process that product originates from.

EXAMPLE When sampling a moulded product for the emission to indoor air, it might be necessary to obtain a smaller part of the product due to the product size. Probabilistic sampling might be used for the random selection of the product from which a part is taken. However, in light of the influence new surfaces have on the emission to indoor air, at least the final selection of the part of the chosen product that is the sample, has to be an informed choice; taking a part with as small as possible new surface.

NOTE 2 The use of Judgemental sampling is much more common in sampling for the determination of the emission to indoor air than for the determination of release to soil and water. A strong motivation for judgemental sampling when determining the emission to indoor air is the practical impossibility to work with increments. Either due to the fact that small increments might have new surfaces, or that the quantity of a composite sample becomes too large for the emission test procedures.

3.4.3 Population and sub-population

Small changes in the product, like the organic raw material composition and / or process parameters (e.g. drying or curing temperature and duration) may influence the release or emission level significantly. This effect is especially strong for products that contain natural and/or recycled organic raw materials. In most cases, these variations are following neither a normal nor a lognormal distribution, because they are caused by distinct changes of raw material (e.g. different batches or different purchase sources). Consequently the identification of sub-populations with respect to the production process might be hard if detailed information on the production process is not available. Consequently, in order to be successful in diminishing the variability in the sub-population in comparison to a randomly defined sub-population, detailed knowledge of the production process is essential.

The variation of the release and / or emission between produced batches or over time may in some cases be very high. High variations in the release and / or emission could also be expected for complex manufacturing processes. It should be noted that these variations might only be related to the release of emission of dangerous substances, and are of no relevance for other characteristics. Obviously, these aspects are to be taken into account when defining the sub-population.

If a sample is taken from a product that comprises different modifications, and the whole group of products is to be evaluated for its release or emission for CE-marking, than the one with the highest release or emission potential is to be chosen as the product to be sampled. If this specific modification complies with all requirements, than also the other modifications are assumed to comply.

NOTE Choosing a specific product with the highest potential release or emission from the sub-population, implies that a worst case scenario is applied.

EXAMPLE If a specific type of carpet is manufactured day in day out, or, alternatively, that same type of carpet is produced in five batches per month, then the population can be defined as all carpets of that specific type ever produced. The sub-population consists of all batches or all volume produced during the validity of CE-marking (e.g. five years).

In product sampling for the determination of the release or emission of dangerous substances to soil and water, usually the same approach for selection of the sub-population can be used as for sampling of products for testing of other properties for CE-marking. It should be noted however that an approach that works well for these other properties not necessarily works for dangerous substances.

3.4.4 Scale

In defining the scale for sampling, a practical (first) approach can be to use the same scale as is already used for the determination of other properties in the construction product. When applying that scale for the determination of the release or emission of dangerous substances, it should be judged if the variability between the obtained results is limited to an acceptable level. If not, it might well be that the scale is set too small for obtaining a good representative result for the release or emission of dangerous substances.

When determining the scale to be used, account should be taken of the sampling strategy that can be applied. When probabilistic sampling is possible and incremental sampling can result in a composite sample, there is no direct need to take account of the variability of the sampled product when choosing the scale.

NOTE Obviously, when for example account can be taken of the production process in such way that product variability can be minimised, this is helpful to obtain a representative composite sample with only a small number of increments. So, although there is no technical need to define the scale in relation to the expected variability of the product, it is still interesting to do so as it can simplify the sampling strategy and consequently lower the involved costs.

When probabilistic sampling is not an option, or when incremental sampling is not an option, the need for homogeneity on the level of the scale is significantly larger. The remaining option for the sampling strategy is judgmental sampling of one, or at best only a few, individual sample(s). This single sample should than be sufficiently representative for the average characteristic or, when using a worst case approach, should represent that worst case scenario.

EXAMPLE 1 For textile flooring, typically a single production batch is selected as scale, assuming that raw materials and production process are sufficiently stable during manufacture of that batch.

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EXAMPLE 2 For a thermal insulation product made of glass wool, manufactured continuously, typically the production of one day is selected as scale, assuming that raw materials and production process are sufficiently stable during manufacture during that day.

Depending on the selected scale, the use of composite samples to obtain a good estimate of the mean for that quantity of product and the possibilities for sub-sampling, an intermediate solution between the mentioned options for probabilistic sampling and judgemental sampling might be to enlarge the test facilities in order to accommodate the testing of larger test portions / test specimens.

Ultimately, the scale is the quantity of product for which test results become available and based on which the product is assessed.

EXAMPLE 3 Let us assume that national regulations set a limit value of 1,0 mg/kg for the release of a substance from a construction product. It should be defined for which volume/mass of product this mean value is valid for. So for example the average release for 10 kg of product has to comply to the set limit value.

EXAMPLE 4 Considering the production of a specific type of carpet as given in the example in Clause 0, wherein the population covers all carpets of that specific type ever produced and the sub-population covers all carpets of that type for which the CE-marking applies, the scale for that carpet is the part (or quantity) of the production for which the (composite) sample is assumed to be representative. The scale could be a single batch or production day, but could also be a surface of for example 10 m² when a limit value is set for the average emission over a surface of 10 m².

3.4.5 Size of increments and samples

The harmonized Technical Specification has to enforce that the sampling plan for a specific product specifies the actual size of samples (and when relevant increments).

Whenever it is necessary to cut a test portion / test specimen from the original product, this should be done in such way that it minimizes the number of newly cut edges and the surface thereof.

In case of dispute or potential doubt on the test results, it might be desirable to be able to provide a comparable test portion / test specimen. Consequently, when the sampling strategy is to take account of this possibility, the minimum sample size should be such that it allows the production of at least two test portions / test specimen.

The size of the test portion / test specimen should be defined by the product specific test method as specified in the harmonized Technical Specification. Samples taken from a product known to be heterogeneous with respect to emissions should be as large as practically possible for levelling out heterogeneity.

If the minimum representative sample is too large for sampling – which may be the case for complex structures – then separate samples may be taken of the raw material. Instructions are to be supplied to the testing laboratory specifying how representative quantities of the individual parts should be fixed together. All surfaces which are not open in the final product, should be covered in such a way that these surfaces do not contribute to the emission or release of the product.

In testing of surface related release, it is important that the apparent geometrical surface of the product can be determined by simple calculations or simple measurements.

When testing shaped construction products, it is important to perform a visual inspection on the individual selected product. Whenever the release of the construction product is surface related, the surface should fulfil the requirements that apply to the specific product.

EXAMPLE 1 There should be no cracks or resent break lines in the surface.

EXAMPLE 2 A test portion / test specimen of a shaped product can be tested in different shapes (e.g. block, cube, cylinder, block with open spaces). Important for expressing the results and interpreting the release, is the need to be able to quantify the surface area as results are expressed per unit of surface area. Shaped products with irregular surfaces are therefore more difficult to handle. In that case the geometric surface area can either be estimated, or a specimen with regular surfaces is to be obtained by cutting or coring.

Different procedures of moulding, cutting or coring (e.g. wet or dry) may influence the leaching properties of the product differently. The choice of the procedure used should be justified and described in the test report. Oily fluid, water and emulsions as cooling fluid shall be excluded.

NOTE 1 When the construction product results from a stabilisation process, the product should be cured sufficiently long to avoid major variations in leaching due to ongoing changes in pore structure and in formation of release controlling mineral phases.

NOTE 2 The test portion / test specimen can be a laboratory prepared product, which is prepared under conditions similar to the normal manufacturing. It can also be a part of a construction product, unless cutting results in significant differences in surface properties influencing the release or emission of dangerous substances.

EXAMPLE 3 Considering the example of the carpet in Clause 0 and 3.4.4, wherein the scale is set to be the quantity of either a production day, a single batch or a certain surface for which a limit value is set for the average emission, a sample is a small part of that defined quantity for which it is assumed that the emission is sufficiently equal to the average emission of the full quantity (the scale). An increment is also a small part of that quantity of material (the scale), but per definition two or more increments are taken from that quantity to be put together in a composite sample.

3.4.6 Sampling location and moment

If possible, sampling is performed on the same location and moment as used for other properties.

NOTE It should be taken into consideration that the sampling requirements for determining the release or emission of dangerous substances can be quite different from the sampling requirements for the determination of other properties (e.g. mechanical properties).

The emission level may vary between the core and edge regions of a product when it leaves the manufacturing process. The sample should be taken from a spot that is rather in the core if that is representative for the product in use with respect to emission potential. An example is given for products in rolls in Annex A of ISO 16000-11:2006.

Emission level changes over time, especially with an open surface and in contact with surrounding air. The sample should be taken from the factory (from production or from stock, possibly including a necessary curing time) at the earliest point of time when the product is ready for dispatch, but absolutely not earlier because that might impair emission properties in a non-representative manner. This time depends on the product types and information should be given in the respective harmonized Technical Standard. The sample should be packed in accordance with given specifications directly after sampling.

Information on the sampling date and for example the minimum curing time prior to testing, should be given to the laboratory.

EXAMPLE 1 For products undergoing changes in physical properties (e.g. increase in compression strength, decrease in water permeability or porosity) a decrease as well as an increase of release can occur. Chemical incorporation into the matrix can result in a decreasing release, but might also result in degradation, resulting in an increased release of degradation products.

It is important to record information on the exposure of the sample to surrounding conditions, both when still part of the product at the sampling location as well as during and after sampling, in order to facilitate preparation of a representative test portion / test specimen of the construction product for testing.

EXAMPLE 2 The release of dangerous substances from coal fly ash depends on both variation on fuel (origin of fuel, composition (e.g. sulphur content)) and process conditions (e.g. combustion technique, loading degree, temperature, etc). The heterogeneity in ash quality may appear both randomly (e.g. variations in the uniformity of fuel feed) and non-randomly (e.g. due to variation of fuel origin with time). If a critical property is for example time dependent, this means that the sampling period needs to be long enough to cover the variations caused by the critical process conditions.

3.5 Application of sampling techniques

Liquid and powder-like samples can be taken out of larger units after homogenization, by pouring or digging into a smaller inert package. Alternatively, commercial packages (e.g. cans, bags) may be selected as the laboratory sample.

For the determination of the emission of dangerous substances to indoor air, all sampling procedures where humans are in skin contact with the sample to be taken require actions for avoiding contamination by e.g. skin creams. Typically this means that gloves are worn, best gloves made of polyethylene foil, but in any case not gloves made of vinyl for avoiding contamination of sample with plasticizers.

Application of a sampling technique may change the properties of the sampled construction product. New surfaces are exposed when samples are cut, sawed, crushed or drilled from a larger unit. Measures are to be taken into account to deal with these new surfaces prior to testing (e.g. sealing of new surfaces).

The cut edges should be clearly identified on the product as well as reported in the sampling report for later identification. The surface to be exposed in the release test also needs to be clearly identified. When cut edges are present, this should be mentioned in the chain of custody report.

Dust and small particles arising from the drill or cut from shaped products should not be included in the sample. Drill cores may be treated with pressure air (free from oil) to remove dust, if needed.

When selecting a sampling technique, preference should be given to the method which creates least possible deterioration or contamination of products (e.g. cross-contamination due to improper cleaning of the sample equipment in between sampling).

EXAMPLE 1 For sampling of granular products a sampling shovel or scoop made of suitable material such as plastic or stainless steel is generally suitable. If organic compounds are of interest, only stainless steel sampling equipment is allowed in order to minimize the risk for sorption. Sorption to the sampling equipment results in the loss of substances while it might be a source of contamination of subsequent samples.

EXAMPLE 2 It is important that the sample collection vessel and sample equipment (e.g. shovel) are cleaned and dried (if water used in cleaning) before and after the sampling operation. In case samples are drilled or cut from a product, the sample equipment needs to be cleaned carefully prior to sampling to avoid cross contamination (e.g. small particles may be attached to the sampling equipment and transferred to the next sample). Water, pressurized air, non-reactive material (e.g. towel, sand) may be used for cleaning sample equipment.

NOTE Cleaning of equipment is usually not necessary between taking successive increments from the same product which are intended to form the composite sample.

Typical sampling techniques are presented in CEN/TR 15310-2.

3.6 On site sub-sampling / on site sample pre-treatment

If a shaped product needs to be sub-sampled, a sample can be cut or sawed from the original product. The technique that creates least possible deterioration of the product is to be selected. Deterioration occurs by heat and by destructive separation techniques.

In practice it is of no use to try to homogenize granular products or samples of granular products, unless the production process has specifically designed apparatus for mixing and homogenization installed. In the latter situation, sampling should be performed after the homogenization step, which is anyway necessary as the sampling should be aimed at the finalized product ready for its intended use. Sub-sampling techniques are available to deal with heterogeneous products, see CEN/TR 15310-3.

If release or emission of volatile or semi-volatile substances is of interest, sub-sampling under field conditions should be avoided due to risk of loss or change of compounds.

NOTE Despite the reluctance to allow sub-sampling in the field prior to the determination of volatile or semi-volatile substances, the size of the primary sample might necessitate sub-sampling prior to packaging and transport.

Sub-sampling in the field should be done rapidly, especially if there is a risk that product properties are changed under field conditions (e.g. loss or gain of moisture).

3.7 Packaging, preservation, storage and transport

3.7.1 Packaging

Samples are to be thoroughly protected from chemical contamination or any physical exposure, e.g. heat, light and humidity. In order to avoid contamination, product samples are to be placed in air tight, emission and absorption free packaging or containers. Requirements for storage and conservation of the laboratory sample are related to the degree to which release or emission properties change when no precautions are taken to preserve the construction product.

For solid products, sampled for determining the emission of dangerous substances to indoor air, ISO 16000-11 suggests that emission and absorption free packaging can usually be achieved by wrapping each sample separately in aluminium foil and in a polyethylene bag or alternatively, in aluminized packaging lined with polyethylene or clear polyvinyl fluoride film.

Individual samples should be packed separately for avoiding cross-contamination. If a commercial package contains several shaped products, it is essential to consider that for some products the upper side and backing may show very different emission levels (e.g. floorings). If only one side is to be tested for emissions, it is important to avoid direct contact between back and front when packing the sample.

Suitable sealing options for foiled packaging have been found to include welding equipment, low emission adhesive tape or thorough mechanical tightening.

Product laboratory samples that are taken in a permeable commercial packaging should be wrapped additionally for ensuring airtight packaging to minimise risk of contamination. This does not apply to packing known to be impermeable such as metal cans, or laboratory bottles.

For liquid products as well as powders and granulates, ISO 16000-11 suggests that samples are to be shipped in unopened cans, tubes, etc. Liquids and powders / granulates should be dispatched in a commercial can or bag, or in clean laboratory bottles made of glass or polyethylene.

To prevent oxidation, volatilization and loss of moisture, the containers should be filled completely and closed tightly.

EXAMPLE Products that are subject to carbonation due to high material pH, are to be stored in airtight containers to avoid changes in pH and subsequently changing the release behaviour of the product.

The use of new packaging material is essential in order to avoid sample contamination.

Marking should be done in such a way that the identity of the sample is known at all times. If marking the sample itself, it should be done in such way that the marking is not of any influence on the release or emission of dangerous substances.

3.7.2 Preservation

Preservation should occur by air-tight and dry packaging that also prevents direct contact with sunlight and - if relevant - prevents exposure to elevated temperatures.

3.7.3 Storage

Samples should be stored under the preservation conditions (see 3.7.2).

3.7.4 Transport

Samples should be transported under storage conditions (see 3.7.3).

3.8 Sampling report and chain of custody report

The sampling report should contain all information necessary for unambiguous identification and characterisation of the laboratory sample.

The chain of custody report should contain information on all persons involved in, and responsible for, sampling.

Additionally, sampling report and chain of custody report should focus on any risk of contamination prior to delivery of the laboratory sample to the laboratory.

Any reports on product sampling and transport should be referenced in, or copied into, the final test report.

NOTE For repeated measurements under the same conditions, the same sampling plan can be used.

Annex A (informative)

Terminology

NOTE This annex on terminology contains the key terms on product sampling. The defined terms are closely related, which is also depicted in Figure A.1.

1

population

totality of items under consideration

NOTE See also the term sub-population.

2

sub-population

defined part of the population that is targeted for the purposes of sampling

NOTE Unfortunately, the term 'population' (ISO 3534-1: 2006) is open for various interpretations. Therefore the term 'sub-population' (CSS99033: 2007) is of much more used for the assessment of a product. For clarification of the terms population and sub-population an example is introduced.

EXAMPLE Consider a continue production process that results in a specific product. The population for that product is all the individual products produced between the moment the production process started (this may be years ago) and the moment the production process ends (this may be years ahead). From the perspective of testing, this definition does not provide a practical concept. Products produced in the past are no longer available for testing, while products that might be produced in the (far) future are neither available. The term sub-population provides a workable alternative, as the 'start' and 'end' of the sub-population can be defined in a practical way. For the same product, already in production for a number of years, the sub-population might be the production of a year, the production of a month, or what other definition is practical.

3

scale

minimum quantity (mass or volume) of the product for which test results are obtained

NOTE Information on characteristics of the product, including release or emission and variations therein, for a quantity of product smaller than the defined scale, is judged to be unimportant.

4

increment

individual portion of product collected by a single operation of a sampling device which is not tested as a single entity, but is mixed with other increments in a composite sample

NOTE Whenever the portion of product collected by a single operation of a sampling device is analysed individually, the obtained product is called a sample. In such a situation the quantity of product has to fulfil both the criteria for the size of an increment as well as for a sample.

5

composite sample

sample that consists of two or more increments, put together in appropriate portions, from which the mean value of a desired characteristic may be obtained

6

sample

portion of material selected from a larger quantity of material

NOTE 1 The manner of selection of the sample should be described in a sampling plan.

NOTE 2 The term 'sample' is often accompanied by a prefix (e.g. laboratory sample, test sample) specifying the type of sample and/or the specific step in the sampling process to which the obtained material relates.

NOTE 3 After ISO 11074:2005, definition 4.1.16

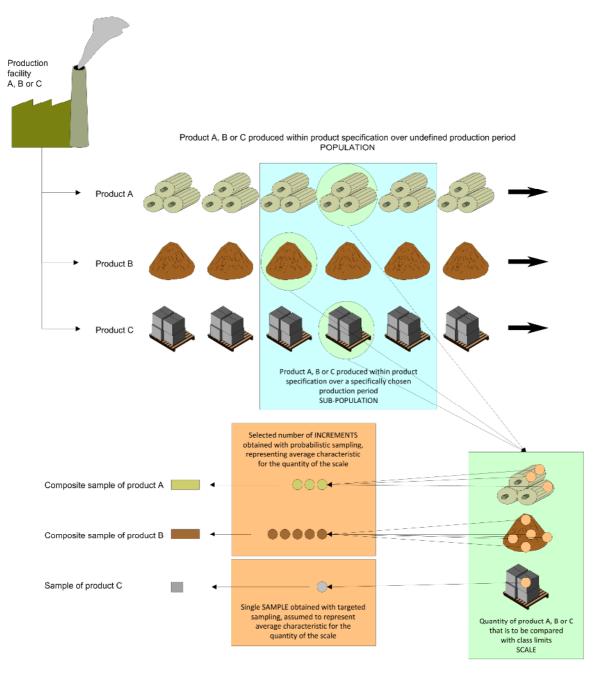


Figure A.1 — Illustration of the relation between the key terms of product sampling

7 test portion

amount or volume of the material taken for analysis

NOTE the term 'test portion' is commonly used in the field of soil and water testing, whereas the term 'test specimen' is commonly used in the field of air testing. These terms are synonyms.

8

test specimen

amount or volume of the material taken for analysis

NOTE the term 'test portion' is commonly used in the field of soil and water testing, whereas the term 'test specimen' is commonly used in the field of air testing. These terms are synonyms.

Annex B

(informative)

Assessment of uncertainty resulting of sampling activities as part of the overall test procedure

B.1 General

Quantifying the uncertainty of the overall test procedure is essential when assessing the release or emission of dangerous substances.

The uncertainty of an assessment can be considered from two perspectives:

- a) What is determining the uncertainty?
- b) What level of uncertainty is accepted?

More details on the expression of uncertainty in measurements can be found in ENV 13005.

B.2 Determining factors

Three sources of variability, and consequently of uncertainty, can be identified:

- Variability in the product (over time and / or space);
- Variability introduced due to sampling activities, including all activities until the delivery of the laboratory sample to the laboratory;
- Variability introduced due to laboratory activities, including preparation of the test portion / test specimen, testing and analysis of test media.
- NOTE 1 In general, the variability of the product sampled is often a dominant factor. At the same time, this variability can be accommodated through a well designed sampling strategy. The larger the variability is, the more samples (or increments in a composite sample) are necessary to get an estimate with a predefined reliability.
- NOTE 2 Some of the involved activities, like mixing and sub-sampling of particulate products, are actually aimed to minimise the encountered variability.
- NOTE 3 There is not one variability of a product. The variability of the product is different depending on the substance tested. The product can be considered as homogeneous for one substance, while at the same time it is heterogeneous for another substance. Additionally, the variability of the product for release of emission properties might be significantly different from the variability of that product for other e.g. mechanical properties.

Which source of variability has the biggest influence on the uncertainty depends on the specific situation considered, but in general the variability of the product dominates the total variability.

Variations caused by the sampling and laboratory activities can be estimated by replications of the various steps in the assessment process, or might already be known when methods have previously been validated. If the resulting variability of the different steps would be known, the overall variability can be calculated. However, the variability of all individual steps is hardly ever known, while the intrinsic variability with respect to the release of dangerous substances in construction products is also largely unknown.

B.3 Variability of the product

Ultimately, sampling of products within the context of the CPD is aimed at assessing whether a product complies with the requirements for CE-marking. The determined release or emission should not exceed the set limits, wherein the determined release or emission is a value for which is assumed that it is representative for the product. That value represents the mean for a predefined scale within a sub-population. Changes over time or in space within the product result in differences in test results, as such representing the variability of the product at the predefined scale. In general the variability of the product is larger when the samples represent a smaller scale.

As variability of the product influences the risk of non-compliance, it is desirable to use samples which are representative for a relatively large scale. At the same time, the larger the scale is, the more demanding is the efforts which are necessary to obtain a representative sample. Additionally, the consequences of non-compliance become larger with a larger scale.

If the risk of non-compliance due to variability of product properties is known, collecting and testing several samples over time can be an option.

B.4 Variability caused by sampling and testing

All activities that are performed have an influence on the test result due to random errors (or even systematic errors). Obtaining information on the effect of these random errors implies that more than one sample is to be tested. In fact quantification of the errors involved in each of the subsequent steps implies additional repetitive testing for those steps. Through statistical analysis, given a well designed experiment, the variability of each individual step can be estimated.

In practice it is (very) costly to determine the separate influence of sampling and testing on the registered variability.

Errors caused by sampling and testing do not per definition result in extremes as they can both strengthen as well as weaken each other. Additionally, some activities, like mixing and sub-sampling of particulate products, are specifically aimed at minimizing the variability between results.

B.5 Determination of the overall uncertainty

The most common approach for the determination of the uncertainty is to determine it for each of the individual steps of the test procedure. Validation of the final analysis of the water or air sample obtained with the test procedure, implies repetitive analysis of a series of samples and the determination of the variability between results. Adding the actual testing of the test portion / test specimen to the analysis implies not only repetitive analysis, but also repetitive testing. In this situation, both the variability of the analysis itself as well as the variability of the test is to be quantified. This implies a large additional test effort for each individual step of the procedure that is added. Consequently, the validation of all the steps of the test procedure, going from product sampling down to final analysis, is practically and financially impossible. Additionally, and perhaps even more important, this approach only provides an approximation of the overall uncertainty.

NOTE 1 Obtaining information for validation purposes is not up to the individual producers.

General information on validation can be found in CEN Guide 13.

For daily practice, the uncertainty of the overall testing procedure is of far more importance than the uncertainty of the individual steps. It is after all the uncertainty of the overall procedure with which the producer is confronted. Given the importance of the uncertainty of the overall test procedure, it could therefore be preferable to determine the overall uncertainty instead of the uncertainty of the individual steps.

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The uncertainty of the overall test procedure can be quantified when a system of parallel testing is adopted. The essential characteristic of this approach is that it can be applied in practice, thus in collaboration with characterizations which are already necessary for another reason (other than validation purposes).

NOTE 2 The determination of the overall uncertainty should be performed for all substances of interest (e.g. all substances for which limit values are defined) as there might be large differences in the associated uncertainty between substances in the same construction product.

As emissions into indoor air may deal with lists containing more than 200 compounds of interest, a pragmatic approach may be to determine analytical uncertainty only for a smaller number of substances, each representative for a group of those 200 compounds of interest, and especially leave out those that are known to appear very seldom or even never, even though they are included in such lists. Furthermore, the impact of other elements of the testing procedure than analytical methods showed to be similar for most volatile compounds and does not need to be addressed for each of these 200 substances separately. This is true especially for the sampling strategy and sampling technique.

NOTE 3 If parts of the steps of the overall test procedure already have been validated, the associated uncertainty can be estimated for the remaining steps that have not been validated.

NOTE 4 The determination of the overall uncertainty assumes that only random errors occur. Systematic errors are not covered by the approach introduced in this clause, despite the fact that in reality systematic errors also are a cause for uncertainty.

B.6 Accepted level of uncertainty

What level of uncertainty is acceptable depends on the assessment that is to be made. In general the accepted level of uncertainty depends on the distance between an observed value and the limit value against which that observed value is to be assessed. Apart from the value of the obtained measurement, also the uncertainty determines the risk of non-compliance. Due to cost constrains, in most environmental assessments there is only one observed value available. Knowledge of the potential variability of that single result is therefore in practice often not available.

NOTE Spending a significant amount of money to reduce the amount of uncertainty is not relevant for a product that always complies; a simple test procedure is sufficient in such a situation. Whether a test procedure is adequate should be judged based on the question whether it is fit for purpose, instead of just setting a simple limit to the acceptable uncertainty.

When more dangerous substances are to be assessed, the test procedure should be designed in such a way that the substance with the (expected) largest degree of uncertainty still meets the acceptable uncertainty.

Annex C

(informative)

Minimum increment and sample size mass (mass/volume) in case of probabilistic sampling

C.1 General

The following applies primarily to granular products for release of dangerous substances into soil and water testing.

The sampling plan should contain specific instructions on the type of samples to be taken, the size of increments and/or samples, the number of increments and/or samples to be taken and, when relevant, the number of increments that should be put together in a composite sample.

NOTE Emissions into indoor air testing normally requires strategic sampling (taking into account all relevant technical parameters influencing emissions into indoor air). Probabilistic sampling results in less representative samples because emissions properties are not related to particle size.

C.2 Estimation of increment and sample size

C.2.1 General

A key feature of probabilistic sampling is that all parts of the sub-population have the chance of being part of the sample. For the sampling of a granular product, this has an effect on the scale (volume or mass) of both increments and samples. This paragraph and subsequent sub-paragraphs show how the increment and sample size should be determined according to the following steps:

- a) Determination of the minimum increment size;
- b) Determination of the minimum sample size;
- c) Determination of the number of increments and/or samples;
- d) Calculation of the actual increment and/or sample size.

C.2.2 Determination of the minimum increment size

Particulate / granular products should be sampled with a sampling device of which the aperture is large enough to allow the entry of all particles present in the product. The aperture of the sampling device should also be large enough to allow the simultaneous entry of all different particle sizes within the product. In practice this means that the device opening should be at least three times the diameter of the largest particles.

In general, for a three dimensional sampling device, the volume of the increment should be equal to:

$$V_{\rm inc} = (3d)^3 = 27d^3$$
 (C.1)

where

 $V_{\rm inc}$ = the volume of increment

d = the diameter of the largest particles.

For practical reasons, the diameter of the largest particles can be substituted by the size of the (estimated) 95-percentile of the particle size distribution.

The minimum increment size should meet the following requirements:

- The actual width, height and length of the sampling equipment should be at least equal to three times the 'maximum' particle size (D_{95}) of the product to be sampled in the case of products with a maximum particle size (D_{95}) of at least 3 x 10⁻³ m (3 mm);
- The actual width, height and length of the sampling equipment should be at least equal to 1 x 10^{-2} m (10 mm) in the case of products with a maximum particle size (D_{95}) of less than 3 x 10^{-3} m (3 mm).

EXAMPLE 1 $D_{95} \ge 3 \times 10^{-3} \text{ m (3mm)}$

If the maximum particle size is at least 3×10^{-3} m (3 mm) and the width, height and length of the increment are chosen to be equal to three times the maximum particle size (D_{95}), the following formula applies to the mass of the minimum increment size:

$$M_{inc} = 10^{-9} \rho (3D_{95})^3 = 2.7 \times 10^{-8} \rho D_{95}^3$$
(C.2)

where

 $M_{\rm inc}$ is the mass of minimum increment size, in kg,

 D_{95} is the 95-percentile particle size, in m, and

 ρ is the bulk density of the product, in kg/m³.

Moreover, the mass of the maximum particle is:

$$\left(\frac{4}{3}\right)\pi 10^{-9} \left(\frac{D_{95}}{2}\right)^3 = 5.2 \times 10^{-10} \rho D_{95}^{3} \tag{C.3}$$

Thus the quantity (mass of increment)/(mass of maximum particle) = 27/0,52 = 51,6. In other words, the mass of the minimum increment should be about 50 times that of a maximum particle (95-percentile of the particle size distribution).

EXAMPLE 2 $D_{95} < 3 \times 10^{-3} \text{ m (3 mm)}$

In the case of products with a maximum particle size (D_{95}) of less than 3 x 10⁻³ m (3 mm), the following formula applies to the mass of the minimum increment size:

$$M_{inc} = 1 \times 10^{-6} \rho$$
 (C.4)

where

 $M_{\rm inc}$ is the mass of the minimum increment size, in kg, and

 ρ is the bulk density of the product, in kg/m³.

EXAMPLE 3 Assume a particulate construction product with a maximum grain size (D_{95}) of 5 x 10⁻³ m (5 mm) and a bulk density of 2 200 kg/m³. For this product the minimum increment size is:

$$2,7\times10^{-8}\times\rho\times D_{95}^{3} = 2,7\times10^{-8}\times2200\times5^{3} = 0,0074kg = 7,5gram$$

C.2.3 Determination of the minimum sample size

For particulate or granular products the composition of individual particles could have a substantial influence on the composition of the sample, and the minimum sample size should be large enough to compensate for this. This is particularly important when the characteristic of interest constitutes only a small proportion of the product.

The minimum sample size to be applied to the product in question is given by:

$$M_{sam} = \frac{1}{6}\pi (D_{95})^3 \times \rho \times g \times \frac{(1-p)}{CV^2 \times p}$$
 (C.5)

where

 M_{sam} is the mass of the sample in kg;

 D_{95} is the 'maximum' particle size (defined as the 95-percentile), in m;

 ρ is the specific mass of the particles in the product, in kg/m³;

g is the correction factor for the particle size distribution of the product to be sampled;

p is the fraction of the particles with a specific characteristic (m/m);

CV is the desired coefficient of variation caused by the fundamental error.

Note that this calculation results only in a rough estimate of the minimum sample size. The estimate however is precise enough to know the order of magnitude of the sample size. Two, partly related, aspects determine the correctness of the estimate: the quality of the assumptions made for the parameters in the formula (thus how correct are the estimates) and the correctness of the formula itself for non-spherical particles. As the aim is to obtain a (rough) estimate of the minimum sample size, the formula can also be used for non-spherical (e.g. irregularly shaped products) or even non-granular products.

NOTE 1 The variables in the formula for the estimation of the minimum sample size are expressed in CGS units for practical reasons.

NOTE 2 The minimum sample size is directly related to the desired coefficient of variation of the fundamental error (CV) and to the size of fraction of the particles with the characteristic to be determined (p). The result is derived from binomial sampling theory as follows. Suppose n samples are taken from the product. The standard error of the observed

proportion of particles with the characteristic of interest is $\sqrt{\frac{p(1-p)}{n}}$, and so the coefficient of variation CV is given

by:

$$CV^2 = \frac{\left(1 - p\right)}{pn}$$

Thus to achieve an adequately small value of CV, the value of n should be:

$$n = \frac{(1-p)}{(CV^2p)}$$
, which is the final term in the equation for M_{sam} .

NOTE 3 As the influence of the fundamental variability should be low, a well accepted value for the coefficient of variation due to the fundamental variability is 0,1.

NOTE 4 The actual value of p, the fraction of particles with a certain characteristic, depends on the product to be sampled and the substances in it to be determined. Knowledge of the product consistency is required in order to determine this value.

NOTE 5 The formula for estimating the minimum sample size is derived for spherical particles of diameter *d*, and so is only an approximation for non-spherical particles.

NOTE 6 The following applies for the correction factor for the particle size distribution (g):

Broad particle size distribution: $D_{95}/D_{05} > 4 \text{ g} = 0,25$ Medium particle size distribution: $2 < D_{95}/D_{05} \le 4 \text{ g} = 0,50$ Narrow particle size distribution: $1 < D_{95}/D_{05} \le 2 \text{ g} = 0,75$ Uniform particles: $D_{95}/D_{05} = 1 \text{ g} = 1,00$

Where D_{05} = the 'minimum' particle size (defined as the 5-percentile of the particle size distribution).

NOTE 7 For the sampling of fine granular products with a broad particle size distribution, the following default values can be used for the factors in the formula:

 $\rho = 2 600 \text{ kg/m}^3$ g = 0.25

p = 0.02

EXAMPLE Continuing the example as provided in C.2.2, and assuming that for this particulate construction product the default values of note 3 and note 7 apply, the minimum sample size for this product would be:

$$M_{sam} = \frac{1}{6}\pi (D_{95})^3 \times \rho \times g \times \frac{(1-p)}{CV^2 \times p} = \frac{1}{6} \times 3,14 \times 0,5^3 \times 2,6 \times 0,25 \times \frac{(1-0,02)}{(0,1^2 \times 0,02)} = 208$$

As the formula provides just an approximation, a practical minimum sample size for this product would be 200 grams.

C.2.4 Determination of the number of increments and/or samples

The number of increments and/or samples is directly related to the objective of the testing programme, the variability of the product to be sampled, and the accepted uncertainty of the result. Reliable information on variability is commonly unavailable – in which case it is not possible to fulfil the exact requirements for the uncertainty without carrying out a preliminary sampling investigation.

Guidance for the determination of the number of increments and samples can be found in Annex D.

C.3 Calculation of the actual increment and/or sample size

C.3.1 General

On the basis of the relationship between the minimum increment size (C.2.2), the minimum sample size (C.2.3) and the number of increments to be included per composite sample (C.2.4), the actual increment size and the actual sample size should be determined according to the following rules.

C.3.2 Taking individual samples

Where composite sampling is not being considered, the question of increment size is in most cases irrelevant as the mass of the minimum sample size (C.2.3) exceeds the mass of the minimum increment size (C.2.2). When the amount of product necessary for the analysis exceeds the mass of the minimum sample size, the actual sample size should of course be sufficient for the analysis.

C.3.3 Composite sampling

Where composite sampling is to be undertaken, there is a potential conflict between:

- a) The previously calculated minimum values for increment size (C.2.2) and sample size (C.2.3); and
- b) The planned number of increments, *m*.

Such conflict should be resolved as follows:

- If *m* increments of minimum size amount to less than the required minimum sample size, then the increment size needs to be increased accordingly, so that *m* actual increments produce an adequately large composite sample;
- Conversely, if *m* increments of minimum size amount to more than the required minimum sample size, then this larger quantity defines the actual sample size.

Annex D

(informative)

Calculation of the required number of increments and samples in case of probabilistic sampling

D.1 General

The following applies primarily to granular products for release of dangerous substances into soil and water testing.

NOTE Emissions into indoor air testing normally requires strategic sampling (taking into account all relevant technical parameters influencing emissions into indoor air). Probabilistic sampling results in less representative samples because emissions properties are not related to particle size.

D.2 Symbols

The following symbols are used:

n = total number of samples or observations;

m = number of increments per composite sample;

 μ = population mean;

 u_p = standard normal deviate corresponding to cumulative probability p;

 χ_0^2 = chi-squared deviate corresponding to cumulative probability p;

 χ_{P} = population P-percentile;

SE(z) = standard error of the statistic z;

 $\sigma_{\rm w}$ = standard deviation of local (i.e. within-composite) spatial variation;

 $\sigma_{\rm b}$ = standard deviation of between-composites spatial and/or temporal variation;

 $\sigma_{\rm s}$ = standard deviation of total spatial and/or temporal variation (= $\sqrt{\left(\sigma_{\rm w}^2 + \sigma_{\rm b}^2\right)}$).

NOTE In cases where composite sampling is not being considered, spot samples can be thought of as composite samples with just a single increment, and so the 'within-composite' standard deviation becomes zero, and the 'between composites' standard deviation becomes the 'between-spots' standard deviation.

 $\sigma_{\rm e}$ = standard deviation of analytical error;

C = desired confidence level (%);

a = cumulative probability related to the desired confidence level;

d = desired precision.

D.3 Estimating a mean concentration

D.3.1 Using composite samples

The standard error of the mean is given by:

SE(mean) =
$$\sqrt{\frac{\left(\frac{\sigma_w^2}{m} + \sigma_b^2 + \sigma_e^2\right)}{n}}$$
 (D.1)

Thus for a given value of m, and assuming normality, the number of composites required to achieve the desired precision (d) and confidence (C), as specified by the user, is given approximately by:

$$n = \left(\frac{u_a}{d}\right)^2 \times \left(\frac{\sigma_w^2}{m} + \sigma_b^2 + \sigma_e^2\right) \tag{D.2}$$

where a = 1 - (1 - C/100)/2. Alternatively, Equation (D.2) can be re-written to determine the number of increments (m) needed per composite sample if n, the total number of composite samples, has been set in advance. Thus:

$$m = \frac{\sigma_w^2}{\left(n\left(\frac{d}{u_a}\right)^2 - \sigma_b^2 - \sigma_e^2\right)}$$
(D.3)

NOTE It might be desirable to take only a single composite sample. Provided $(\sigma_b^2 + \sigma_e^2)$ is sufficiently small, this can be achieved by setting n equal to 1 in Equation (D.3).

In practice, the true standard deviations are unknown and so estimates have to be used. In some cases it might be appropriate to use the values obtained from the past analysis from similar investigations. Otherwise the estimates should, where possible, be obtained from a preliminary pilot study.

EXAMPLE 1 Suppose that:

- Estimates of σ_w , σ_b and σ_e are 4, 2 and 0,5 mg/kg;
- 10 increments are to be taken per composite (i.e. m = 10); and
- The mean is required to be estimated to a precision of d = 1 mg/kg with 90 % confidence.

For C = 90, a = 1 - (1 - 90/100)/2 = 0.95, and so $u_a = 1.65$.

From Equation (D.2), n = (1,65)2(16/10 + 4 + 0,25) = 15,9.

Thus about 16 composite samples would be needed to produce a mean to the required reliability.

To decide on the most appropriate value of m it is necessary to consider the relative costs of sampling and analysis. Suppose that the sampling cost per increment is A, and the analysis cost per sample is B. The total cost, TC, is accordingly given by:

$$TC = (Am + B)n \tag{D.4}$$

Thus, using Equation (D.2) with various trial values of m, it is possible to find the combination of m and n which minimizes TC.

EXAMPLE 2 Continuing with the earlier example, suppose that:

Values of m ranging from 1 to 20 are considered; and

B/A = 30 - that is, a sample analysis is 30 times more expensive than the cost of sampling an increment.

Figure D.1 shows the n value given by Equation (D.2) for each trial value of m. Figure D.2 shows the corresponding values of the total sampling cost TC (in arbitrary units). It is apparent that the optimum number of increments per composite sample is about 6.

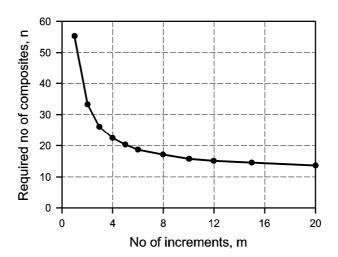


Figure D.1 — Illustration of the relationship between *m*, *n* and TC (see text for details) – Samples needed to achieve specified precision and confidence

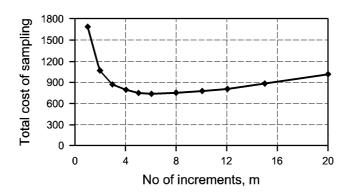


Figure D.2 — Illustration of the relationships between *m*, *n* and TC (see text for details) – Costs of sampling in relation to number of increments per composite sample

D.3.2 Using individual samples

The standard error of the mean is given by:

SE(mean) =
$$\sqrt{\frac{\left(\sigma_s^2 + \sigma_e^2\right)}{n}}$$
 (D.5)

Thus the number of samples required to achieve the desired precision (*d*) and confidence (*C*), as specified by the user, is given approximately by:

$$n = \left(\frac{u_a}{d}\right)^2 \times \left(\sigma_s^2 + \sigma_e^2\right) \tag{D.6}$$

Where

$$a = 1 - (1 - C/100)/2$$
.

NOTE Individual sampling can be thought of as composite sampling with just one increment per composite. Thus the results of the previous section apply to the case of spot sampling by substituting m = 1 and replacing $\sigma_w^2 + \sigma_b^2$ with σ_s^2 .

In practice, the true standard deviations are unknown and so estimates should be used. In some cases it may be appropriate to use the values obtained from the past analysis of sample data from similar investigations. Otherwise the estimates should, where possible, be obtained from a preliminary pilot study.

EXAMPLE Suppose that:

- Estimates of σ_s and σ_e are 4,5 and 0,5 mg/l; and
- The mean is required to be estimated to a precision of d = 2 mg/l with 90 % confidence.

For
$$C = 90$$
, $a = 1 - (1 - 90/100)/2 = 0.95$, and so $u_a = 1.65$.

From Equation (D.6), $n = (0.825)^2 (20.25 + 0.25) = 13.9$.

Thus about 14 individual samples would be needed to produce a mean to the required reliability.

D.4 Estimating a standard deviation

The following approach is applicable when the sub-population can be assumed to be normally distributed. Even for non-normal sub-populations, however, the method is useful as a rough approximation.

For normal populations, the confidence intervals for σ can be calculated using

$$s\sqrt{\frac{(n-1)}{\chi_{1-p}^2}} \text{ to } s\sqrt{\frac{(n-1)}{\chi_p^2}}$$
(D.7)

where

$$p = (1 - C/100)/2$$
.

For a given choice of confidence C, this can be evaluated for a range of trial n values, and this identifies the number of samples that provides the required precision.

EXAMPLE Suppose it is required to estimate the standard deviation to a precision of 20 % with 90 % confidence. For 90 % confidence, the lower and upper p values are = $(1 \pm C/100)/2 = 0.05$ and 0.95. With the help of statistical tables of the χ^2 distribution at the p = 0.05 and 0.95 points, the following table can be constructed:

Table D.1 — 90 % confidence limits for σ /s for various numbers of samples

No of samples	Lower 90 % confidence limit for σ/s	Upper 90 % confidence limit for σ/s
n	$\sqrt{\frac{(n-1)}{2}}$	$\sqrt{\frac{(n-1)}{2}}$
	$ \begin{array}{l} \sqrt{\chi^2} \\ (\rho = 0.05) \end{array} $	$ \begin{array}{c} V \chi^2 \\ (p = 0.95) \end{array} $
20	0,79	1,37
30	0,83	1,28
40	0,85	1,23
50	0,86	1,20
60	0,87	1,18
70	0,88	1,16
80	0,89	1,15
90	0,89	1,14
100	0,90	1,13
120	0,90	1,12
150	0,91	1,11
200	0,92	1,09

By inspection it can be seen that with 50 samples, the lower and upper confidence limits are 0,86 and 1,20. That is, the sub-population standard deviation σ may be 14 % below or 20 % above s, the observed standard deviation. Note that the interval is not symmetrical. Thus, at the 90 % confidence level, a precision of 20 % or better is achieved by a standard deviation calculated from 50 random samples.

Annex E

(informative)

Sample containers, preservation and storage conditions for different parameters for the determination of the release to soil and water

Table E.1 — Sample containers, preservation and storage conditions for different parameters for the determination of the release to soil and water

Parameter to be tested	Container / Packaging material	Remark
Heavy metals	Plastic, Glass	 For alkaline product (e.g. cement) uptake of carbon dioxide minimised For metals (e.g. chromium) subjected to oxidation air-contact to be avoided.
Mercury	Glass, PTFE	Air-tight container/package, head- space to be minimised (completely filled)
Volatile organic substances or groups	Glass, aluminium foil, stainless steel	Air-tight container/package, head- space to be minimised (completely filled)
Cyanides	Plastic	Air-tight container/package, head- space to be minimised (completely filled)
Non-volatile organic substances or groups	Glass, aluminium foil, stainless steel	See product specific conditions
Inorganic substances	Emission and adsorption free packaging material, e.g. plastic, glass, cartoon	See product specific conditions

Table E.2 — Sample containers, preservation and storage conditions for different parameters for the determination of the emission to indoor air

Parameter to be tested	Container / Packaging material	Preservation / storage
Any	Aluminium foil + PE foil or combined material for solid samples. Glass or PE container for powder-like and liquid samples.	 Storage in transport package at room temperature without excessive heat or direct sunlight.

Annex F

(informative)

Example forms for the sampling plan, the field report and the chain of custody report

F.1 Example of a form for the sampling plan

SAMPLING PLAN					
Sample Code: (Reflect site location, product type and date of collection)					
Date of sampling:					
Signature of project manager:					
GENERAL INFORMATION					
Producer:	Client (company):				
Contact:	Contact:				
Location of sampling:	Sampler:				
PRODUCT TO BE SAMPLED					
Type of product:	Estimate of moisture content:				
Description:(shape, colour, odour, consistency/ homo	ogeneity/ grain size – uniform or diverse)				
SAMPLING METHODOLOGY					
Definition of sub-population (mass, volume, time peri	,				
Definition of scale (mass, volume) a (composite) same	pple represents:				
Place and point of sampling:					
Sampling procedure:					
Sampling equipment:					
Number of samples / increments:					
Sample size / increment size:					
Safety measures:					
SUB-SAMPLING / ON SITE SAMPLE PRE-TREATI	MENT				
Identify location:					
Procedure:					
PACKAGING, PRESERVATION, STORAGE AND T	RANSPORT DETAILS				
Packaging:					
Preservation:					
Storage:					
Transport:					

F.2 Example of a form for the sampling report

For sampling construction products to determine the release to soil and water

Sampling report	for construct	tion product	(name of pro	duct):					
Sampling objecti	ve (ITT/FPC	5):							
Type of products	e.g. bulk, p	oiece, packa	ge):			_			
Producer of prod	luct:								
Sampler (organis	sation):								
Sampling plan (r	eference do	cument)							
Sampling point:									
Sampling equipn	nent:								
Sample identification code (e.g. individual sample)	Date /time	Sample amount	Sampler	Sample handling (removal of fractions not belonging to sample)	Sample preservation or storage conditions	Visual description of sample (e.g. colour)	Identification of new surfaces (cutting edges)	Delivery to testing laboratory (date)	Other observations or remarks
/cont.									

For sampling construction products to determine the emission to indoor air

Testing laboratory/ Inspection body:		Sampler (Name, company, telephone):					
Name of the manufacturer at (address / stamp):	the place of sampling	Manufacturer (if deviating from company's name at the place of sampling):					
Name of the product:		Type of product * (e.g. lami	inate, textile flooring, PVC-flooring):				
Model / Program / Series:		Batch N°.:					
Article N°.: Misc.		Date of batch production:					
Sample is taken from	☐ the ongoing productio☐ stocks	on .	Date of sampling:				
			Time:				
Where had the product been stored prior to sampling?	□ Production□ Store□ Miscellaneous	How had the product been stored prior to sampling?	□ open□ in the stack□ wrapped up				
	Place of storage:		Packing material:				
Specifics (possible negative influences by emission at the place of taking the sample, petrol emissions, solvent emissions from production, uncertainties, questions, etc).							
Cut edges (identification of cut emission test)	edges when present and	identification of new surfaces	and surface to be exposed in the				
Confirmation							
The signer herewith confirms th personally in accordance with the			selected, drawn and packed				
Date: Signature: (Stamp)							

NOTE This form is based on the form as used by German DIBT authority (German Institute for Construction Technology) within notified German regulation of emissions from construction products into indoor air.

F.3 Example form for a chain of custody report

Chain of custody report								R	Reference	no.:		
Site:						File na	File name:					
Start:	Date	Time			File no	File no.:						
End	Date	e Time				Persor	n in ch	arge	:			
Sampler	, initials:						Other	specifi	catio	ns:		
Samplin	g report	referen	ce r	no.:								
Sample	no. start	:				Total r	o. of sam	ples:				
Sample	no. end:					Labora	atory:					
Sample	ID:											
Handed	over bet	tween:			•	Time Justif		tifica	ation Conditions		ns	
Handed	over	Initials										
by:		Signature										
Handed	over	Initials										
to:		Signature										
Handed	over	Initials										
by:		Signature										
Handed	over	Initials										
to:		Signature										
Handed	over	Initials	8									
by:		Signa	ture									
Handed over Initials to:												

Annex G (informative)

Checklist for product TCs to be used for the evaluation of their sampling standard or sampling paragraph

This document refers both to sampling standards as published by product TCs as well as to product standards that contain sampling instructions as part of an overall test procedure.

Checklist

Topic	for consideration	§	Decision	Motivation
	Identification of the objective	2.2		
	Definition of a sampling plan	2.3		
	Sampling approach	2.4.2		
	Definition of the sub- population	2.4.3		
	Identification of the scale	2.4.4		
	Incremental sampling or single samples	2.4.5		
	Size of increment and/or sample	2.4.5		
	Number of increments in a composite sample	Annex C		
	Sampling technique to be applied	2.5		
	Need for on-site sub- sampling	2.6		
	Sub-sampling technique to be applied	2.6		
	Packaging of samples	2.7.1		
	Preservation of samples	2.7.2		
	Storage of samples	2.7.3		
	Transport of samples	2.7.4		
	Report of sampling	2.8		
	Chain of custody report	2.8		

Bibliography

This CEN/TR refers to the following standards and documents:

- [1] CEN/TR 15310-1:2006 Characterization of waste Sampling of waste materials Part 1: Guidance on selection and application of criteria for sampling under various conditions
- [2] CEN/TR 15310-2:2006 Characterization of waste Sampling of waste materials Part 2: Guidance on sampling techniques
- [3] CEN/TR 15310-3:2006 Characterization of waste Sampling of waste materials Part 3: Guidance on procedures for sub-sampling in the field
- [4] ISO 3534-1:2006 Statistics Vocabulary and symbols Part 1: General statistical terms and terms used in probability
- [5] CSS99033:2007 Sludge, treated biowaste, and soils in the landscape Sampling Vocabulary, CEN/BT/TF 151
- [6] ISO 16000-11:2006 Indoor air Part 11: Determination of the emission of volatile organic compounds from building products and furnishing Sampling, storage of samples and preparation of test specimens
- [7] ENV 13005:1999 Guide to the expression of uncertainty in measurement





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