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BSI Standards Publication

Construction products — Assessment of release of dangerous substances — Determination of emissions into indoor air

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Construction products - Assessment of release of dangerous substances - Determination of emissions into indoor air

Produits de construction - Détermination des émissions de substances dangereuses - Détermination des émissions dans l'air intérieur

Bauprodukte - Bewertung der Freisetzung von gefährlichen Stoffen - Bestimmung von Emissionen in die Innenraumluft

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Foreword

This document (CEN/TS 16516:2013) 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.

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Introduction

This Technical Specification was developed in the frame of the Mandate M/366 "Development of horizontal standardized assessment methods for harmonised approaches relating to dangerous substances under the Construction Products Regulation (CPR)" addressing the preparation of horizontal measurement/test methods for the determination of emission of regulated dangerous substances from construction products into indoor air. This mandate is a complement to the product mandates granted by the European Commission to CEN under the Construction Products Regulation. The harmonised products standards (hEN) developed in CEN under mandates from the European Commission specify construction product(s) as put on the market and address their intended conditions of use.

This Technical Specification has gone through a robustness validation for identifying how small changes in specific testing parameters can influence the test result. This study also delivered data on repeatability within one testing laboratory (see Annex A). It is planned to convert the TS into an EN standard immediately after publication of this TS taking into account any relevant information provided during that process. This may include data from further round robin tests.

It is vital that such information is clearly linked to a specified product in a product standard. The responsibility of product specification is with the product TCs, as described in CEN/TR 16496. This determination of emission into indoor air is to be carried out on products under their intended conditions of use. The intended use of a construction product is generally specified in the corresponding harmonised product standard. The specific emission rates determined using this Technical Specification are associated with application of the product in a defined European Reference Room under specified climate (temperature and humidity) and ventilation conditions. A reference room is needed since it is not possible to evaluate emissions by testing in all possible use scenarios.

The reference room dimensions, resulting product loading factors, as well as climate and ventilation conditions are selected to represent the general indoor environment (see Clause 4). Based on the huge amount of available European experience, it was possible to identify one emission scenario and one reference room and associated set of product loading factors to be used.

This Technical Specification specifies the horizontal reference method for testing the emission (release) of dangerous substances from construction products into indoor air. This method uses a test chamber in which emissions are generated under conditions which are kept constant during the test. These conditions are selected so that the test results can be expressed in terms of chemical concentrations in the air of the reference room (see Clause 7 and Clause 9). It is to be noted that the test chamber is defined in terms of performance requirements. This responds to the requirement of Mandate M/366 for a horizontal approach but still maintains sufficient flexibility on chamber dimensions to ensure representative samples of different materials can be accommodated (see Clause 5). Clause 8 of this Technical Specification specifies how emitted regulated dangerous substances should be analysed.

This Technical Specification also addresses separately (see Clause 11 and Annex B) indirect methods that provide, within their specific field of application, a result that is comparable or that correlates with the result of the reference method. Such methods may be easier to apply and/or be cheaper. They are in accordance with mandate M/366 provided that their comparability or correlation to the reference test method has been demonstrated in their specific field of application. They are especially suitable for Factory Production Control testing (FPC).

The selection of one emission scenario and one reference room for evaluating emissions to indoor air is in general accordance with the approach taken in existing European national regulations and voluntary schemes relating to emissions from construction products into indoor air. It also accords with the horizontal requirements of mandate M/366. The aim of this Technical Specification is not to develop a new testing method but to combine by normative references the use of existing standards complemented, when necessary, with additional and/or modified requirements so that – according to the horizontal concept specified in mandate M/366 – construction products can be evaluated under comparable conditions with regard to emissions into indoor air.

In summary, the horizontal test method specified in this Technical Specification determines the specific emission rate of volatile organic compounds from a construction product into indoor air. This can be converted into a concentration in the air of the reference room by calculation.

This Technical Specification has not been evaluated for the determination of 'steady state' concentration of formaldehyde.

NOTE A European Standard (EN 717-1) exists for the determination of formaldehyde emissions from wood-based panels, in terms of 'steady state' concentration.

1 Scope

This Technical Specification specifies a horizontal reference method for the determination of emissions of regulated dangerous substances from construction products into indoor air. This method is applicable to volatile organic compounds, semi-volatile organic compounds, and volatile aldehydes. It is based on the use of a test chamber and subsequent analysis of the organic compounds by GC-MS or HPLC.

NOTE 1 Supplemental information is given on indirect test methods (Annex B) and on measuring very volatile organic compounds (see informative Annex C).

NOTE 2 This Technical Specification describes the overall procedure and makes use of existing standards mainly by normative reference, complemented when necessary with additional or modified normative requirements.

2 Normative references

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

CEN/TR 16220:2011, *Construction products — Assessment of release of dangerous substances — Complement to sampling*

CEN/TR 16496:2013, *Construction Products — Assessment of release of dangerous substances — Use of harmonised horizontal assessment methods*

EN ISO 13137, *Workplace atmospheres — Pumps for personal sampling of chemical and biological agents — Requirements and test methods*

EN ISO 16000-9:2006, *Indoor air — Part 9: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test chamber method (ISO 16000-9:2006)*

EN 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 (ISO 16000-11:2006)*

EN ISO 16017-1, *Indoor, ambient and workplace air — Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography — Part 1: Pumped sampling (ISO 16017-1)*

ISO 554, *Standard atmospheres for conditioning and/or testing — Specifications*

ISO 16000-3:2011, *Indoor air — Part 3: Determination of formaldehyde and other carbonyl compounds in indoor air and test chamber air — Active sampling method*

ISO 16000-6:2011, *Indoor air — Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA sorbent, thermal desorption and gas chromatography using MS or MS-FID*

3 Terms, definitions and abbreviations

For the purposes of this document, the following terms, definitions and abbreviations apply.

NOTE Several of the defined terms on product sampling are closely related, which is also depicted in Figure 1. This figure and the relevant definitions are taken from CEN/TR 16220:2011.

3.1 Terms relating to sampling and products

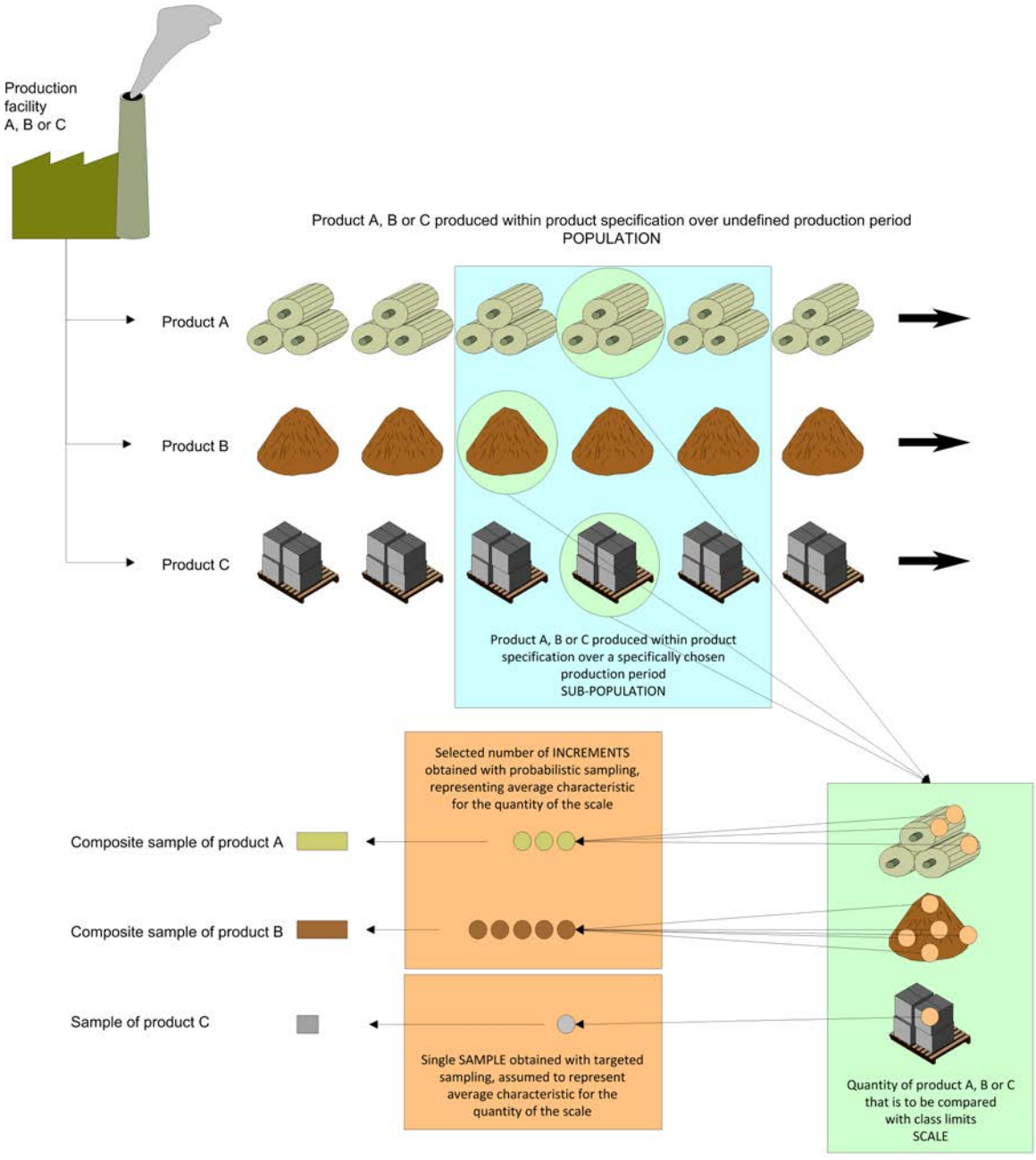


Figure 1 — Relation between the key terms of product sampling

3.1.1
composite sample
average sample, aggregated 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

[SOURCE: adapted from ISO 11074:2005, 4.3.3]

3.1.2
curing
hardening of freshly prepared mixtures under well-defined conditions (time, temperature, humidity, etc.) specified in harmonised product standards

3.1.3
curing time
minimal time defined necessary for curing before an emission test can be executed to perform relevant test results

3.1.4
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 1 to entry: 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 should fulfil both the criteria for the size of an increment as well as for a sample.

[SOURCE: adapted from ISO 11074:2005, 4.1.8 as in CEN/TR 16220:2011, 2.4.5]

3.1.5
laboratory sample
sample or sub-sample(s) sent to or received by the laboratory

Note 1 to entry: When the laboratory sample is further prepared by mixing, drying, grinding or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test/analysis or for the preparation of a test specimen.

Note 2 to entry: The laboratory sample is the final sample from the point of view of sample collection but it is the initial sample from the point of view of the laboratory.

[SOURCE: IUPAC, 2.5.5]

3.1.6
population
totality of items under consideration

Note 1 to entry: See also the term sub-population.

[SOURCE: adapted from ISO 11074:2005, 4.1.11 as in CEN/TR 16220:2011, 2.4.3]

3.1.7
sample
portion of material selected from a larger quantity of material

Note 1 to entry: The manner of selection of the sample should be described in a sampling plan.

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

[SOURCE: IUPAC, 2.1.1]

3.1.8
sampling plan
predetermined procedure for the selection, withdrawal, preservation and transportation of product samples

[SOURCE: CEN/TR 16220:2011, 2.3]

3.1.9
scale
minimum quantity (mass or volume) of the product for which test results are obtained

Note 1 to entry: Information on characteristics of the product, including emission and variations therein, for a quantity of product smaller than the defined scale, is judged to be not relevant for description of product properties, e.g. for evaluation of emissions into indoor air.

Note 2 to entry: Sometimes this quantity is called lot or batch.

[SOURCE: CEN/TR 16220:2011, 2.4.4]

3.1.10
sub-population
defined part of the population that is targeted for the purposes of testing

Note 1 to entry: See also the term population.

EXAMPLE Consider a continuous 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.

[SOURCE: adapted from ISO 11074:2005, 4.1.29 as in CEN/TR 16220:2011, 2.4.3]

3.2 Terms relating to emissions into indoor air and associated laboratory testing

3.2.1
air change rate
ratio of the volume of air brought into the test chamber per hour and the free test chamber volume measured in identical units

[SOURCE: EN ISO 16000-9:2006, 3.1]

3.2.2
air flow rate
ventilation rate
air volume entering into the emission test chamber per unit of time

Note 1 to entry: Air flow rate is expressed in litres per second or in cubic metres per hour (l/s, m^3/h).

[SOURCE: adapted from EN ISO 16000-9:2006, 3.2]

3.2.3
chamber blank value
test result obtained by carrying out the test procedure in the absence of a test portion/specimen

Note 1 to entry: Blank value is expressed in micrograms per cubic meter ($\mu g/m^3$).

3.2.4
compound recovery
measured mass concentration of a target volatile organic compound in the air leaving the emission test chamber during a given time period divided by the mass concentration of the same target volatile organic compound added to the emission test chamber air in the same time period, expressed in percent

Note 1 to entry: The recovery provides information about the performance of the entire method.

[SOURCE: EN ISO 16000-9:2006, 3.9]

3.2.5
emission
liberation of chemical substances from a construction product into air

Note 1 to entry: Emission may be expressed as an emitted quantity in terms of concentrations in a defined volume of air or in terms of emission rate per hour and per unit quantity of the construction product (i.e. per area, length, mass, volume, unit or component).

Note 2 to entry: The terms "emission" and "release" have fundamentally the same meaning. However, by tradition, the term "emission" is used when describing liberation of chemical substances or radiation into air and the term "release" is used when describing the liberation of chemical substances into soil or water.

3.2.6
emission test chamber
enclosure with controlled operational parameters for the determination of volatile organic compounds emitted from construction products

[SOURCE: adapted from EN ISO 16000-9:2006, 3.6]

3.2.7
emission test chamber concentration
mass concentration of a specific volatile organic compound, VOC, (or group of volatile organic compounds) in test chamber air measured in the emission test chamber outlet

[SOURCE: EN ISO 16000-9:2006, 3.7]

3.2.8
intended conditions of use
conditions that a product may experience during service life and that influence its release/emission behaviour

3.2.9
mass concentration of the compound in the reference room air
mass concentration of a specific volatile organic compound, VOCs, (or group of volatile organic compounds) in a reference room

3.2.10
product loading factor
ratio of exposed dimension of the test specimen to the free test chamber volume

Note 1 to entry: The product loading factor is often expressed as the ratio of the exposed area of the test specimen and the volume of the test facility (L_A expressed in m^2/m^3). The product loading factor can also be expressed as ratio of the exposed length, volume or unit(s) of the test specimen and the volume of the emission test facility (L_L expressed in m/m^3, L_V expressed in m^3/m^3 or L_U expressed in u/m^3).

[SOURCE: adapted from EN ISO 16000-9:2006, 3.8]

3.2.11
reference room
room with conventional dimensions, climate and ventilation used as reference for any specification of emission testing and any calculation of VOC concentration in indoor air

Note 1 to entry: In this Technical Specification, a reference room is specified in 4.2.

3.2.12
specific air flow rate
q
ratio of air change rate and product loading factor

Note 1 to entry: Specific air flow rate can be expressed as the area specific air flow rate q_A , equivalent to ratio of the air flow rate and the surface area of the test specimen in [m^3/m^2*h], which is equivalent to the expression [m/h].

Note 2 to entry: This definition includes other specific air flow rates than only the area specific air flow rate. Specific air flow rates can also be volume specific (q_V expressed in $m^3/(m^3*h)$), length specific (q_L expressed in $m^3/(m*h)$), mass specific (q_m expressed in $m^3/(kg*h)$), or unit specific (q_u expressed in $m^3/(unit*h)$).

[SOURCE: adapted from EN ISO 16000-9:2006, 3.4]

3.2.13
specific emission rate SER (emission factor)
product specific rate describing the mass of a volatile organic compound emitted per unit of product per unit of time at a given time from the start of the test

Note 1 to entry: This definition is intended to avoid confusion between the terms *q* (in 3.2.12) and *q* (used for specific air flow rate in EN ISO 16000-9). The specific emission rate can be related to area, length, volume, mass or unit, expressed as SER_A in µg/(m^2*h), SER_L in µg/(m*h), SER_V in µg/(m^3*h), SER_m in µg/(kg*h), or SER_U expressed in µg/(u*h).

[SOURCE: adapted from EN ISO 16000-9:2006, 3.11]

3.2.14
test portion
quantity or volume removed from the test sample for analysis purposes, generally of known weight or volume

[SOURCE: IUPAC, 2.5.7]

3.2.15
test sample
sample, prepared from the laboratory sample from which test portions are removed for testing or for analysis

[SOURCE: IUPAC, 2.5.6]

3.2.16
test specimen
test portion specially prepared for emission testing in an emission test chamber in order to simulate the emission behaviour of the product under intended conditions of use

EXAMPLE In case of floorings the test portion is a defined area of the flooring. The test specimen is prepared from this by covering the edges and the back of the flooring, because these surfaces do not have contact to the indoor air under service life conditions.

[SOURCE: adapted from EN ISO 16000-9:2006, 3.13]

3.3 Terms relating to determination of emitted substances

3.3.1
LCI value
Lowest Concentration of Interest
substance-specific value for health-related evaluation of the emission from construction products

3.3.2
limit value
numerical limit derived from national, European or contractual provisions

Note 1 to entry: Limit values for dangerous substances may be defined for environmental media or for product performance like emission or content of the product.

3.3.3
non-target compound
compound for which the test result is not compared with a compound specific limit value

3.3.4
***R* value**
sum of all R_i values

3.3.5
***R_i* value**
Ratio C_i / LCI_i, where

— C_i is the mass concentration in the air of the reference room

— LCI_i is the LCI value of compound *i*

3.3.6
semi-volatile organic compounds
SVOC
all organic compounds which, in a gas chromatographic column as specified in 8.2.2, are eluting with a retention range between n-hexadecane (excluded) and n-docosane (included)

Note 1 to entry: The measurement is carried out using a capillary column coated with 5 % phenyl/95 % methyl-poly-siloxane.

Note 2 to entry: This definition corresponds to volatile organic compounds with a boiling point approximately higher than 287 °C.

Note 3 to entry: Other definitions are given by the World Health Organization (WHO) 1987, ISO 16000-6.

3.3.7
target compound
compound for which the test result is compared with a compound specific limit value

[SOURCE: adapted from EN ISO 16000-9:2006, 3.12]

3.3.8
total semi-volatile organic compounds
TSVOC
sum of the concentrations of the identified and unidentified volatile organic compounds eluting with a retention range between n-hexadecane (excluded) and n-docosane (included) on a gas chromatographic column as specified in 8.2.2

Note 1 to entry: The measurement is carried out using a capillary column coated with 5 % phenyl/95 % methyl-poly-siloxane.

[SOURCE: adapted from EN ISO 16000-9:2006, 3.14]

3.3.9
total volatile organic compounds
TVOC
sum of the concentrations of the identified and unidentified volatile organic compounds eluting between and including n-hexane and n-hexadecane on a gas chromatographic column as specified in 8.2.2

Note 1 to entry: The measurement is carried out using a capillary column coated with 5 % phenyl/95 % methyl-poly-siloxane.

[SOURCE: adapted from EN ISO 16000-9:2006, 3.14]

3.3.10
very volatile organic compounds
VVOC
all volatile organic compounds eluting before n-hexane on a gas chromatographic column as specified in 8.2.2

Note 1 to entry: This definition corresponds to volatile organic compounds with a boiling point lower than approximately 68 °C.

Note 2 to entry: Other definitions are given by the World Health Organization (WHO) 1987, ISO 16000-6.

3.3.11
volatile organic compounds
VOC
all volatile organic compounds eluting between and including n-hexane and n-hexadecane on a gas chromatographic column as specified in 8.2.2

Note 1 to entry: The measurement is carried out using a capillary column coated with 5 % phenyl/95 % methyl-poly-siloxane.

Note 2 to entry: This definition corresponds to volatile organic compounds with a boiling point in the range of approximately 68 °C to 287 °C.

Note 3 to entry: Other definitions are given by the World Health Organization (WHO) 1987, ISO 16000-6, Decopaint Directive 2004/42/EC.

[SOURCE: adapted from EN ISO 16000-9:2006, 3.15]

3.4 Abbreviations

GC Gas Chromatography

HPLC High performance liquid chromatography

LCI Lowest Concentration of Interest

MS Mass Spectrometry

q	Specific Air Flow Rate
R	Ratio of concentration and limit value
SER	Specific Emission Rate
SVOC	Semi-Volatile Organic Compound
TSVOC	Total Semi-Volatile Organic Compound
TVOC	Total Volatile Organic Compound
VOC	Volatile Organic Compound
VVOC	Very Volatile Organic Compound

4 Intended conditions of use, emission scenarios and European reference room

4.1 Intended conditions of use and emission scenario

The intended conditions of use describe the purpose, place and circumstances of typical application(s) of a construction product as defined in a product standard. This includes the intended use, (e.g. for what purpose, how the product typically is installed, etc.), and an emission scenario.

Regarding the determination of emission into indoor air, the emission scenario specifies the climate and ventilation conditions of the air surrounding the product in a reference room. The actual condition(s) of use in reality may be different. It is also not possible to evaluate emissions by testing under all possible use scenarios. Therefore, this Technical Specification specifies a set of conditions that are generally agreed to be representative of use of the product in "normal" indoor environments, such that all construction products can be evaluated under comparable conditions.

These defined conditions assume a standardised installation of the product in a reference room with standardised dimensions, climate and ventilation.

4.2 Reference room and emission scenario

4.2.1 General

In this horizontal Technical Specification, only one reference room and one emission scenario are specified and used as conventional references for any specification of emission testing and any calculation of concentration of emitting compounds in indoor air.

If any piece of legislation or a particular application of the test requires a different reference room then the results may be transformed by calculations within the ranges where such calculations are valid (see Clause 7 and Clause 9).

4.2.2 Dimensions and loading factors in the reference room

The dimensions of the reference room are specified as listed below:

— The walls are 2,5 m high.

— Floor and ceiling both measure 3 m x 4 m resulting in surface of 12 m^2 each.

— There is one door of 0,8 m (width) x 2 m (height) (1,6 m^2).

— There is one window of 2 m^2.

— Then the total wall area (minus door and window) is 31,4 m^2.

Then the total air volume is 30 m^3. Using these reference room dimensions, the product standards shall specify one of the following loading factors L, depending on the product type:

— 1,0 m^2/m^3 – walls;

— 0,4 m^2/m^3 – floor, ceiling;

— 0,05 m^2/m^3 – small surfaces, e.g. a door;

— 0,007 m^2/m^3 – very small surfaces, e.g. sealants.

If the above surfaces and resulting loading factors do not represent the intended conditions of use of a specific product, the product TC shall specify that the nearest surface and resulting loading factor shall be applied.

If the intended conditions of use imply the possibility that a product is used on more than one of the above surfaces, the appropriate surfaces and loading factors either shall be summed, or the largest applicable surface and loading factor shall be applied.

NOTE In this Technical Specification, the reference room dimensions and associated loading factors are normative and correspond to a small, normal living room. However, the corresponding loading factors remain similar to those given in the informative annex of EN ISO 16000-9:2006.

4.2.3 Ventilation in the reference room

The rate of ventilation with fresh air is specified at 0,5 air changes per hour (15 m^3h^{-1}) for the reference room to represent, by convention, normal indoor air conditions. In conjunction with the climate and dimensions conditions this ventilation rate is the reference representing by convention the "general" indoor air conditions.

NOTE 1 Real air change rates have been observed as less than 0,3 air changes per hour in naturally ventilated energy efficient, air tight buildings to more than 1 air change per hour with continuous mechanical ventilation.

NOTE 2 In Clause 9, a calculation formula is given for recalculation of a test result (when expressed as concentration in air) to another air change rate than the specified one, within a valid range.

4.2.4 Climate conditions in the reference room

The climate conditions specified in ISO 554 and in EN ISO 16000-9, i.e. 23 °C and 50 % relative humidity, shall be applied to the reference room to represent, by convention, general indoor air conditions.

4.3 Time schedule of emission(s) determination

The time schedule specified in EN ISO 16000-9 shall be used for emissions evaluation, i.e. short-term emissions shall be tested at three days and long-term emissions shall be tested at 28 days after installation of the product.

At 28 days after installation of the product, either steady-state emissions have been reached, or the decay of emissions has at least slowed significantly. Such a situation may be reached much earlier for some products. In such cases, an optional shorter testing duration shall be clearly defined in the relevant product standards to evaluate the emissions at the above reference time schedule, if the comparability with the 28 days testing is shown in line with the requirements specified for applicability of indirect methods in Clause 11.

NOTE Emissions of some SVOCs can increase after day 3.

5 Product sampling and transport to the laboratory

5.1 Introduction

This clause summarises basic elements of sampling as a complement to specifications on sampling in the relevant product standards. Product specific details need to be specified in those product standards (see Annex D).

5.2 Objective of sampling

The objective of sampling from a construction product shall be to obtain a laboratory sample that is:

— sufficiently representative of the quantity of product being assessed, see D.1, D.2 and D.3;

— fulfilling the prerequisites for the test(s) to be performed (e.g. possibility to make a test specimen from the sample, fitness for the determination of the emission of regulated dangerous substances into indoor air).

5.3 Preparation of a sampling plan and sampling strategy

5.3.1 General

The relevant product standards shall require development of a sampling plan (see D.6) prior to actual product sampling for assessing the emission of regulated dangerous substances into indoor air.

Existing sampling plans for testing other product properties may not always cover sufficiently the needs of testing emissions of regulated dangerous substances from construction products into indoor air.

5.3.2 Sampling approach

The most appropriate sampling approach (see D.7.2) shall be selected in the sampling plan, either probabilistic sampling or judgemental (or informed) sampling.

NOTE This includes balancing the pros and cons of each sampling approach, such as the necessary effort and costs of sampling, and the practical possibilities for sampling against the (statistical) representativity of the test result for the sampled sub-population, weighed against the objective of sampling. Such a decision requires detailed knowledge of the product to be sampled and the process that product originates from.

5.3.3 Population and sub-population

The relevant product standards shall specify how to select the population and sub-population (see D.7.3) for which emission of regulated dangerous substances shall be determined.

For the determination of the emission of regulated dangerous substances, the same approach for selection of the sub-population may be used as for sampling of products for testing other properties. It should be noted however that an approach that works well for these other properties does not necessarily work for emissions of regulated dangerous substances.

5.3.4 Scale

The relevant product standards shall specify how to select the scale that shall be represented by the laboratory sample.

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 of the construction product. When applying that scale to the determination of the emission of regulated dangerous substances, it should be evaluated whether the variability between the results obtained from different laboratory samples within the same scale is limited to an acceptable level. If not, the scale may be too small for obtaining a sample for determination of the emission of regulated dangerous substances into indoor air sufficiently with an acceptable uncertainty (see D.7.4).

5.3.5 Size of samples, of increments when relevant, and sampling techniques

The relevant product standards shall specify the size of samples taken, and the sampling techniques, taking into account:

— specification and characteristics of the product to be sampled;

— minimum size of the sample(s) necessary for testing (and of increments, where relevant); the sample size should allow the production of at least three test specimens for allowing repetition of a test in case of doubt or dispute on the test results;

— any necessary measures to maintain the integrity of a sample.

Sampling activities shall have no, or as little as possible, impact on the integrity of the sampled product. The relevant product standards shall specify how to minimise:

— evaporation of volatile substances;

— creation of new surface, e.g. by cutting, if this cannot be avoided for obtaining a sample;

— deterioration of the product due to heat production during sampling or sub-sampling, e.g. sawing;

— contamination of the sample(s) by the sampling devices and/or by other sample(s).

The relevant product standards can specify that and how the actual test specimen shall be prepared from the sampled material in the testing laboratory.

NOTE This applies in particular to products that are placed on the market in another form then needed for application under intended use. As an example, tile adhesives are sampled as powder (as distributed). For making a test specimen, they need to be mixed with water before intended use.

5.3.6 Sampling of complex, composite and large products

Complex, composite and large products shall be evaluated as a whole unit. For practical purposes, the relevant product standards may foresee testing of:

— a small-scale model of the product,

— specified sections of the product,

— significant components of the product

if these represent the whole product, or if the individual test results of sections or components can be combined to represent the whole product.

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

5.3.7 Sampling location and moment

As specified in EN ISO 16000-11:2006 (there in 4.1), product samples shall be collected at the point of manufacture after the normal manufacturing. This may include curing time, drying time, etc., as specified in the relevant product standards.

Manufacturing may include drying and curing periods if necessary before placement on the market.

In defining the sampling location a practical first approach can be to use the same location and time as used for determination of other properties (e.g. mechanical properties). But it should be taken into consideration that the sampling requirements for determining the emission of regulated dangerous substances can be different from the sampling requirements for the other properties.

5.4 Information from the testing laboratory needed to complement the product sampling plan

The following information shall be provided by the analytical laboratory to the sampling body for complementing the sampling plan:

a) number of product laboratory sample(s) needed for the testing programme (e.g. two product samples for testing and one for possible later review);

b) minimum and/or maximum size of product laboratory sample required for testing;

c) instructions on packaging and transport;

d) sampling report to be filled in;

e) chain of custody report to be filled in.

5.5 Packaging and transport of laboratory sample

As specified in EN ISO 16000-11 with the following additional statements:

a) Product samples shall be placed in airtight, emission-free and absorption free packaging or containers to avoid contamination.

   Solid samples can be packaged by wrapping airtight in aluminium foil and then packaged in an unprinted airtight polyethylene bag and sealed. As an alternative, aluminium-coated packaging material may be used. To prevent external contamination, the package should be sealed maximally airtight with a film welding device, or with a low emission adhesive tape, or by mechanical tightening.

b) Product samples that shall be examined separately shall be packed separately to avoid cross-contamination.

c) Samples of different products shall be packed separately.

d) If several solid product samples are packed together in one bag then it is essential to consider that the upper side and backing may show very different emission levels. If then only one side shall be tested for emissions (e.g. floorings) then it is important that product samples are packed in such a manner that there is no direct contact between back and front.

e) Product laboratory samples that are taken in a permeable commercial packing shall be wrapped additionally to ensure airtight packaging to minimise risk of contamination. This does not apply to packing known to be impermeable such as metal cans, or laboratory bottles. Use of commercial packaging without further airtight wrapping is possible, especially for large products, if this is considered by the product TC as being sufficient for the testing.

f) Liquids and powders/granulates shall be dispatched in a commercial can, cartridge or bag, or in clean laboratory bottles made of glass or of polyethylene. For transport and storage of samples the air volume in cans and bottles shall be minimised.

g) Excessive heat, extreme pressure and other physical challenges shall be avoided during storage and transport.

h) Contamination by adsorption of volatile chemicals from, e.g. fuel cans, car exhaust gases, cleaners, during transport shall be avoided by proper packaging and by avoiding transport close to contamination sources such as gasoline canisters.

If there is uncertainty about the quality of packaging then installation (and later analysis) of passive air samplers inside packaging is recommended for monitoring any contamination.

5.6 Sample description, marking of laboratory sample and sampling report

As specified in EN ISO 16000-11:2006 (there in 4.3), with the following additional statements:

a) Marking of the sample shall not have any impact on emission testing.

b) Only solvent-free writing utensils may be used inside the packaging.

NOTE Examples are barcodes, ballpoint pens, pencils, self-adhesive labels.

c) The sample and the wrapping shall be marked identically.

d) Details of the client's identification of the sample shall be included in the test report.

e) A sampling report shall follow the sample. An example of a sampling report is given in Annex E.

5.7 Chain of custody report

The chain of custody report shall ensure that the product sample received in the laboratory is the same as the product sample taken in the field (traceability).

NOTE 1 It is good practice that the sampling plan specifies the completion of a chain of custody form for each sampling exercise, at the time of sampling and during transport to the testing laboratory.

NOTE 2 An example form for a chain of custody report is given in Annex F.

5.8 Dispatch of product samples, time schedule

As specified in EN ISO 16000-11:2006 (there in 4.3) and EN ISO 16000-9:2006 (there in clause 14) with the following additional statements:

a) Product samples shall be taken from the factory at the earliest point of time when the product is ready for dispatch or application. This point of time depends on the product types and information shall be specified in the respective product standard.

NOTE Emission level changes over time, especially with open surfaces and when in contact with surrounding air.

b) The product sample shall be packaged, as specified above, as soon as possible after collection and, in any event, within the same working day.

c) The product sample shall arrive in the laboratory not later than 14 days after the date of sampling.

d) The maximum time between date of sampling and beginning of a test in the laboratory (incl. storage at manufacturer, transport and storage at testing laboratory) shall not exceed eight weeks provided that the laboratory sample is stored in the specified packaging. On-site wet-applied products coming in a closed container (e.g. can, cartridge) shall be tested not later than four months after sampling.

The product standards specifying the considered product may specify different requirements depending on the behaviour of the involved products.

6 Handling of product samples in the laboratory

6.1 Storage of sample in the testing laboratory

Follow the procedures specified in EN ISO 16000-11, but with the following additional statements:

a) Samples shall be protected from intensive light, exposure to chemicals and mechanical damage.

b) Cleaners, solvents or other volatile chemicals shall not be stored close to the sample if there is a risk of contamination that could interfere with emission testing.

6.2 Preparation of the test specimen

The test specimen may be prepared in the laboratory or at the sampling site depending on the nature of the product and of its specification in a product standard.

Follow the procedures specified in EN ISO 16000-11 but with the following additional statements:

a) The preparation of the test specimen shall take place in a clean environment, and any tools used shall be clean.

b) When possible and necessary, adjust the size of the test specimen in accordance with 4.2.2 and Clause 7 of this Technical Specification. Representativeness with regard to intended use shall be maintained when performing any such adjustment.

   It may not be possible to adjust the size of the test specimen of complex products such as windows. However, in these cases, product TCs may specify how to perform testing of a small-scale model of the product, or specified sections of the product, or significant components of the product, for achieving a test result representative for the full-size product.

c) If a product requires an assembly specified in the corresponding product standard under normal conditions of use, the product shall be assembled accordingly prior to testing.

d) If only one surface of a product is exposed to the indoor environment under intended conditions of use, as specified in a product standard, cover the rear surface and edges of the test specimen immediately after specimen preparation, especially the cut edges, using aluminium foil or low emitting adhesive tape or by installing the test specimen into a frame or sealed box made of inert, non-porous and non-emitting material. Typical sample supports are made of glass, metal plates or metal foil.

NOTE 1 Efficient sealing techniques are:

— back to back storage of plates, with edges covered with low emission aluminium tape,

— tight coverage of edges and back with aluminium foil,

— seal box, as specified in JIS A 1901, or equivalent.

NOTE 2 When using aluminium tape then the emissions of every new batch of tape can be tested by fixing the tape on an inert surface and determining the emissions using the same test procedure as applied to normal samples. Any determined emissions from the tape will be taken into consideration when evaluating emissions of a test specimen prepared using such tape.

e) For liquid products check and weigh the sample support before and immediately after application of the product. Then report the applied amount as the resulting difference.

f) For application of liquid products, the application method and amount shall be defined in the product standard.

g) If specified in the relevant product standard, a conditioning period shall be applied to laboratory samples or test specimens before starting the test to enable the product to acquire properties representing in use conditions. This conditioning time shall be fixed in a unique manner for all similar products, and it has to represent typical use patterns. In this case the conditioning parameters (climate, ventilation, loading factor, closed and separate conditioning container for each test specimen or laboratory sample) shall be defined precisely and be within the ranges specified in this Technical Specification for test chamber parameters. Transfer of a pre-conditioned test specimen into the actual test chamber is regarded as the starting time of the test (T_0).

NOTE 3 Conditioning periods are typically representing hardening or curing of reactive systems, or drying of wet-applied products, as these periods are not relevant to in use conditions.

7 Test chamber conditions

7.1 Principles

A ventilated test chamber shall be used for generating emissions from the test specimen under constant, controlled conditions. Most test chambers are smaller than the reference room and can be thought of as a scaled down model of the reference room. In general, the test chamber parameters are kept as close as possible to those specified in 4.2.2 for the reference room, but deviations are accepted within a narrow range as specified below. Robustness validation has shown that flexibility within the stated ranges has negligible impact on product emission rates.

Generation of emissions from test specimen in a test chamber shall be performed as specified in EN ISO 16000-9 with the additional requirements as specified below.

7.2 Dimensions of test specimen

The dimensions of the test specimen shall be appropriate to the size of the considered test chamber and the product loading factor L as specified for the reference room in 4.2.2. The test specimen shall be large enough to be at least as representative as the laboratory sample (see 7.9 and 7.10 below).

7.3 Loading factor

The loading factor in the test chamber may deviate from the values in 4.2.2 within a narrow range. It shall not be below 50 % or above 200 % of the specified loading factor and shall not exceed 2,0 m^2/m^3. Within this interval the specific emission rate remains constant and test results given in terms of specific emission rate do not need any conversion. However, any test result reported in terms of concentration in the air of the reference room shall be re-calculated using the formulas given in Clause 9.

7.4 Ventilation

The ventilation (air change rate) in the test chamber may deviate from the specified value for the reference room (see 4.2.3). It shall not be lower than 0,25 air changes per hour and not higher than 1,5 air changes per hour. Within this interval, the specific emission rate is considered as remaining constant and test results presented in terms of specific emission rate do not need any conversion. However, any test result reported in terms of air concentration in the reference room shall be re-calculated using the formulas given in Clause 9. Variation of the air change rate during the testing period shall not exceed ± 5 %, as specified in EN ISO 16000-9.

NOTE While emissions from dry products are typically controlled by internal diffusion processes and are largely unaffected by ventilation (surface air velocity), the same cannot be said for "liquid" products (being wet when applied) during the drying/curing stage, i.e. while evaporation dominates the emission process. Given that building occupants are not usually present during the drying/curing phase of wet-applied products in construction works (although common for "do-it yourself" activities and redecoration), emission testing is normally only started after the drying/curing phase. For comparing emissions data, all testing and pre-conditioning parameters, including ventilation, should be within the ranges specified in this clause.

7.5 Air velocity

The air velocity above test specimen shall be in the range 0,1 m/s to 0,3 m/s.

NOTE High air velocity and high ventilation rate may increase emission rate of less volatile substances.

7.6 Cleanliness of test chamber

The test chamber and the supply air shall be clean, as specified in Clause 8 in terms of maximum acceptable background concentration for the different substances analysed.

7.7 Testing climate (temperature, relative humidity of supply air)

Temperature and relative humidity shall be registered as the average of 15 min period. The average temperature during the entire testing period shall not deviate from the target value of 23 °C by more than ± 1 °C. Moreover, not more than 10 % of the individual 15 min average values shall deviate from the target value of 23 °C by more than ± 1 °C. The average relative humidity of the air supplied to the chamber throughout the entire testing period shall not deviate from the target value by more than ± 5 % RH. Moreover, not more than 10 % of the individual 15 min average values shall deviate from the target value of 23 °C by more than ± 5 % RH.

NOTE 1 Test specimens emitting high amounts of water initially can lead to increased relative humidity in the air inside the test chamber and in the test chamber exhaust.

NOTE 2 As temperature and humidity have a significant impact on emissions, these parameters cannot be varied for any type of product or test equipment without compromising the comparability of emission data.

7.8 Storage of test specimen

The test specimen shall remain in the test chamber for the whole duration of the test, i.e. until the last air sample has been collected.

Moving a test specimen from an intermediate storage chamber to the test chamber and vice versa may significantly interfere with the emission testing in terms of contamination of the sample and of the chamber and shall be avoided.

NOTE 1 For VOC of lower volatility and for SVOC, the reliability of test results depends on effective control of ventilation, climate, and wall adsorption / desorption effects, which can only be guaranteed if samples are kept in the test chamber the whole time.

NOTE 2 During indirect tests according to Clause 11, sample storage outside the chamber between air sampling periods can be accepted.

7.9 Large bulk products

When testing large bulk products the required free air volume, product volume and chamber size shall be specified precisely. Large bulky products can impair the velocity in the chamber above the surface of the test specimen. If this occurs, the air flow rate shall be adjusted to address this (see 7.2 and 7.10).

7.10 Volume of test chamber

The volume of the test chamber selected shall be large enough to accommodate a test specimen that is representative of the product in normal use (see 7.2 and 7.9). The size of a test chamber shall be larger than 20 l.

The smaller the test chamber, the smaller the test specimen appropriate for such a small chamber. However, such small test specimens may not be representative for the entire laboratory sample or for the product in normal use unless it is homogeneous. Therefore, small test chambers may not be large enough especially for testing complex or inhomogeneous products.

If a representative laboratory sample consists of several similar increments, each increment may be tested separately in an appropriate test chamber (i.e. of such dimension so as to be representative of the product in normal use) and the individual results are combined by weighted averaging. The produced result corresponds to the laboratory sample which is a combined sample of the increments.

NOTE The maximum size of test chambers is not limited. Experiences with existing chambers range up to 50 m^3.

7.11 Placement of test specimen in test chamber

The test specimen shall be placed in the centre of the test chamber as specified in EN ISO 16000-9. If the efficiency of air mixing is proven to be in compliance with specifications of EN ISO 16000-9 in all compartments of the test chamber in the presence of the test specimen, then the test specimen can be placed in the test chamber on the bottom or at the wall or in another manner, as long as the main surface of the test specimen is parallel to the air flow direction.

8 Determination of volatile organic compounds in test chamber air

8.1 Common requirements

A measured volume of air from the outlet of the emission test chamber is drawn at a controlled flow rate through the samplers at specified times during the emission test. Volatile organic chemicals present in the chamber air are selectively trapped on the samplers as the air passes through. The type of air sampler differs depending on the compounds described in the following sections.

The sampling flow rate shall be calibrated with the sampler assembly in line, using an appropriate external calibrated meter as described in EN ISO 16017-1 and EN ISO 13137. The total sampling flow shall not exceed 80 % of the air flow rate of the chamber.

If air sampling is to be carried out early in an emission test (within the first 72 h), the sampling duration shall show the same time before and after the target sampling time, e.g. from 71,5 h to 72,5 h.

NOTE 1 Experience showed that, with adequate mixing of test chamber air, the concentration of emitted compounds in the air across the test chamber usually has balanced out after a time corresponding to air exchange of five times test chamber volume.

In the following analysis, retained compounds or their derivatives are desorbed or extracted from the sampler and transferred into an analytical system where they are identified and quantified.

Duplicate samples shall be collected and the results compared.

Any test results shall be reported with not more than two significant figures.

NOTE 2 This rule reflects the degree of precision that can be achieved by this testing method. Examples:

— 52.5 and 53.4 both are expressed as 53,

— 255.0 and 264.9 both are expressed as 260,

— 2 550 and 2 649 both are expressed as 2 600.

8.2 Determination of VOCs and SVOCs in test chamber air

8.2.1 Introduction

This section specifies procedures for sampling and analysing volatile organic compounds (as specified in 3.3.11) and semi-volatile organic compounds (as specified in 3.3.6) in test chamber air. ISO 16000-6 shall be applied except for the deviations and additional requirements specified in the clauses below. The method

involves pumped air sampling onto tubes packed with Tenax TA[1)] sorbent and subsequent analysis by thermal desorption with gas chromatography and mass spectrometer detection (TD-GC/MS).

NOTE Annex C provides general information regarding sampling and analysis of VVOCs (as specified in 3.3.10) in conjunction with VOCs.

8.2.2 Capillary GC column

A 5 % phenyl / 95 % methyl poly siloxane capillary column shall be selected with suitable dimensions (length, diameter, film thickness) to optimise separation of the compounds of interest in the sample, i.e. VOCs with a boiling point between 69 ºC and 287 ºC.

Summation of specific VOCs within a particular chromatographic retention time window may be required by the applied material emission testing. Examples of VOC summation include: total VOC, the sum of notified target VOCs, the R value, the sum of VOCs that are not on a list of notified target compounds, and/or the sum of non-identified VOCs. By convention, in these cases, the retention time window of interest typically relates to components eluting from (and including) n-hexane to (and including) n-hexadecane on a non-polar, 100 % polydimethylsiloxane (PDMS) stationary phase capillary column. The column specified in this Technical Specification gives better (sharper) peak shapes for the more polar compounds of interest and reduces analytical uncertainty. Any change in component elution order from that seen on a PDMS column is minimal and can be ignored.

8.2.3 Tube conditioning and laboratory blank tubes

This section refers to blank levels on conditioned sorbent tubes that have not been used for air monitoring.

Before sampling, a representative number of conditioned sorbent tubes shall be analysed using routine analytical parameters to ensure that the thermal desorption blank is sufficiently small. The tube blank is acceptable if no individual interfering artefact peaks exceed 2 ng and if TVOC levels do not exceed 20 ng after subtraction of non-interfering VOC artefacts. If the blank is unacceptable, recondition the tubes by desorbing them for 10 min under a 50 ml/min to 100 ml/min flow rate of inert carrier gas at a temperature ~20°C higher than that used for analysis and then repeat the analysis. If after repeated conditioning, the blank is still unacceptable, the tubes shall be re-packed.

One or two of the conditioned tubes from the batch selected for each sampling exercise shall be retained as laboratory blank tubes. These are noted. They shall be analysed with the sampled tubes and shown to meet the performance specification detailed above (i.e. < 2 ng of any individual interfering VOC and < 20 ng total VOC after subtraction of non-interfering VOC artefacts).

Do not exceed the maximum temperature of the sorbent during tube conditioning or analysis.

8.2.4 Checking the test chamber blank

The test chamber apparatus, including supply air, shall be checked for background contamination prior to carrying out a new materials emission test as described in EN ISO 16000-9. The TVOC background concentration shall be lower than 20 µg/m^3. The background concentration of any single target VOC shall be lower than 2 µg/m^3.

Tenax TA tubes can generate low levels of benzene artefact, even if the tube was clean of benzene before air sampling. False positive results may impair determination of low µg/m^3 range of benzene. It is recommended to verify any such low benzene value with an independent second test method (see Annex C and Annex G).

1) Tenax TA is a trade name of Buchem BV (NL). This information is given for the convenience of users of this standard and does not constitute an endorsement. Equivalent products may be used if they can be shown to lead to the same results.

8.2.5 Sampling test chamber air

Ensure that the air sampling tube is at approximately the same temperature as the chamber air to prevent risk of water condensation inside the sample tube when sampling emissions.

An appropriate air sampling flow rate is in the range of 20 ml/min to 200 ml/min.

NOTE VOCs and SVOCs ranging in volatility from n-hexane to n-docosane are normally quantitatively retained by a sorbent tube containing 200 mg (or 50 mm to 60 mm bed length) of Tenax TA provided air sample volumes are kept to 5 l or below.

Air samples shall be collected in duplicate, but using different pump flows such that two different volumes of air (such as 5 l and 2 l or 5 l and 1 l) are collected simultaneously (or immediately sequentially).

For determining background concentrations in the chamber or when a sample is known to be low-emitting, two duplicate 5 l sampling volumes may be sampled to optimise sensitivity.

8.2.6 Calibration and analysis

8.2.6.1 Reporting limit, quantification limit

The quantification limit of any VOC and SVOC shall be 1 µg/m^3 as far as feasible. All quantified substances shall be reported. Substances with 5 µg/m^3 or more shall be reported with their concentrations (as calculated for the reference room in 4.2.2).

Traces of VOCs and SVOCs include a significantly increased uncertainty of quantification. It is recommended to report test results below 5 µg/m^3 as "< 5 µg/m^3" or "less than 5 µg/m^3".

NOTE Quantitative reporting limit of carcinogenic substances is 1 µg/m^3, as far as feasible.

8.2.6.2 Identified compounds (target and non-target compounds)

During initial system set-up, the analytical system shall be calibrated using a minimum five-point multi-level calibration and be shown to be linear (or exhibit a predictable mathematical relationship) for each target compound over a range of at least 20 (i.e. the factor between the lowest and highest mass in the range shall be at least 20). Data from this initial calibration phase shall be used to determine a relative response factor (RRF) for each compound of interest, relative to toluene.

At least one appropriate (e.g. mid-level) standard, containing toluene and the expected target compounds (or at least a set of compounds that is representative of the volatility and polarity range of the relevant target compounds), shall be run at the start of each batch of samples and be interspersed with the sample tubes – e.g. every 10th sample – as a check on RRF stability. At least three single-level standards shall be run with each sample batch. The multi-level calibration for one or more specific compounds of interest shall be repeated whenever the single level calibration for that type of compound shows unacceptable drift in actual or relative response factor since the previous multi-level calibration. See also 8.2.8.

NOTE 1 Use of an internal standard (e.g. toluene D8 or cyclodecane) can also be helpful as a further check on analytical system stability

Identified target compounds shall be quantified using the actual or relative response factor for each compound. Identified non-target compounds shall be quantified using the response factor for toluene (i.e. in toluene equivalents).

NOTE 2 For target compounds see Annex H.

NOTE 3 The test result calculated and quantified with the respective calibration factor can be useful supplemental information for identified non-target compounds.

NOTE 4 Determining VOCs using toluene equivalents is semi-quantitative, since individual compounds in the mixture may have response factors which differ widely from the toluene response factor.

Details of the response factor used to quantify identified non-target compounds shall be included in the test report (response factor for toluene or response factor for the individual compounds).

The results from duplicate samples shall be compared (see 8.2.9) taking into account the different sampled volumes.

If a regulation or a test protocol requires the data to be analysed for a specific list of target compounds then the chromatographic data shall be reviewed for identifying and quantifying any components on that list that are present above the reporting limits as specified in 8.2.6.1.

8.2.6.3 Unidentified compounds

Unidentified compounds shall be quantified using the response factor for toluene and reported as toluene equivalent. The results from duplicate samples shall be compared (see 8.2.9) taking into account the different sampled volumes.

8.2.7 Total volatile organic compounds (TVOC) and total semi-volatile organic compounds (TSVOC)

The sum of VOCs (TVOC, see 3.3.9 and Annex H) shall be determined by summing the individual concentrations of every identified and unidentified compound eluting from a gas chromatographic column as specified in 8.2.2, between n-hexane and n-hexadecane inclusively, at a concentration above 5 µg/m^3, all calculated as toluene equivalent.

The sum of SVOCs (TSVOC, see 3.3.8) shall be determined by summing the individual concentrations of every identified and unidentified compound eluting from a gas chromatographic column as specified in 8.2.2 after n-hexadecane and not later than n-docodecane at a concentration above 5 µg/m^3, all calculated as toluene equivalent.

8.2.8 Quality control – Additional method performance checks required

As detailed above and in the EN ISO 16000 series standards, an appropriate level of quality control shall be employed and documented. Method performance checks shall include:

a) Single level standards interspersed with a batch of samples shall be compared with each other and with the most recent multi-level calibration. Single level standard results shall agree within 10 % and be within 10 % of the values given by the most recent multi-level calibration. If unacceptable, a new multi-level calibration shall be undertaken.

b) Results of duplicate chamber air sample shall be compared for individual compounds and for TVOC before reporting a test result. The compound concentration determined from duplicate air sample shall agree within ± 15 % of the average, after taking air sample volume into account. If there is any greater disparity, only the higher concentration shall be reported.

8.3 Determination of formaldehyde and some other volatile carbonyl compounds in test chamber air

When a determination of formaldehyde in the test chamber air is required, this shall be undertaken according to ISO 16000-3. The same applies to a number of other volatile carbonyl compounds: acetaldehyde, butyraldehyde, propionaldehyde, acetone, crotonaldehyde.

NOTE ISO 16000-3 specifies simultaneous determination of formaldehyde, acetone and a range of other volatile carbonyls using pumped air sampling with a cartridge or tube used to trap aldehydes by chemical reaction for subsequent analysis by high performance liquid chromatography.

8.4 Quality control – External references

Notified and accredited laboratories shall verify performance of the whole method by comparing against external references and by following the quality control requirements of ISO 16000-3, ISO 16000-6, ISO 16000-9 and ISO 16000-11.

NOTE 1 Use of external reference materials spiked with VOCs with known emission rate, and with known emission decay profiles, are a useful tool for evaluating the performance of the whole procedure against primary standards, provided the quality of the reference materials is known. Determination of test chamber sink effects by recovery tests using target compound sources can be applied if suitable reference materials are not available, as described in EN ISO 16000-9.

NOTE 2 Participation in round robin tests and relevant independent analytical proficiency testing schemes are useful for comparing performance against a group of laboratories.

NOTE 3 Routine laboratory checks of analytical system performance can be conveniently carried out by spiking samplers (e.g. Tenax tubes) with a mix of compounds that is representative (in terms of analyte mass, polarity, volatility range, etc.) of the compounds of interests.

9 Calculation of specific emission rates and expression of results at the reference room

Calculate the impact of product emissions on reference room air concentration as specified in EN ISO 16000-9, taking into account any variation in parameters (e.g. air change rate or loading factor) as allowed in Clause 7 of this Technical Specification.

a) The peak areas of a single compound in the chromatogram are proportional to the mass of compound injected. For each target compound, the relationship between the mass of analyte injected and the corresponding peak area is determined. The slope of the calibration curve over the linear range is the response factor of the compound analysed:

$$A_{St} = b_{St} m_{St} + i_{St} \quad (1)$$

where

A_{St} is the compound peak area in the chromatogram of the analytical standard St, in the area units;

b_{St} is the slope of the calibration curve (response factor);

m_{St} is the mass of compound in the analytical standard St, in nanograms;

i_{St} is the intersect of ordinates and calibration curve. If the calibration curve crosses the origin, i_{St} is considered zero.

b) The mass of compound present in the air sample is calculated from the detector peak area using the response factor of the compound:

$$m_a = \frac{A_a - i_{St}}{b_{St}} \quad (2)$$

where

m_a is the mass of compound *a* in the air sample, in nanograms;

A_a is the peak area of compound *a* in the chromatogram of the sample, in area units;

i_{St} is the intersect of ordinates and the calibration curve. If the calibration curve crosses the origin, i_{St} is considered zero;

b_{St} is the slope of the calibration curve.

c) The mass concentration of the compound in the sampled air is calculated by means of the following equation:

$$c_a = \frac{m_a - m_{ab}}{V} \quad (3)$$

where

c_a is the mass concentration of compound *a* in the sampled air, in micrograms per cubic metre;

m_a is the mass of compound *a* present in the chamber air sample, in nanograms;

m_{ab} is the mass of compound *a* present in the chamber blank, in nanograms;

V is the sampling volume, in litres.

d) The emission rate of the compound is calculated from the mass concentration in the sampled air by means of the following equation:

$$ER_a = c_a VR_t \quad (4)$$

where

ER_a is the emission rate of compound *a* into air, in micrograms per hour;

c_a is the mass concentration of compound *a* in the sampled air, in micrograms per cubic metre;

VR_t is the hourly ventilation rate of the test chamber, in cubic metres per hour test chamber.

e) The area specific emission rate of the compound is calculated from the emission rate in the sampled air by means of the following equation:

$$SER_A = \frac{c_a AC_t}{L_{At}} \quad (5a)$$

where

SER_A is the area specific emission rate, in micrograms per square metre and hour (also called emission factor);

c_a is the mass concentration of compound *a* in the sampled air, in micrograms per cubic metre;

AC_t is the hourly air change rate of the test chamber, in air changes per hour;

L_{At} is the loading factor in the test chamber, in square metres sample per cubic metres.

Specific emission rate can also be calculated as volume specific emission rate, length specific emission rate or unit specific emission rate if this reflects better the essential properties of a product:

$$SER_V = \frac{c_a AC_t}{L_{Vt}} \quad (5b)$$

$$SER_L = \frac{c_a AC_t}{L_{Lt}} \quad (5c)$$

$$SER_u = \frac{c_a AC_t}{L_{ut}} \quad (5d)$$

where

SER_V is the volume specific emission rate, in micrograms per cubic metre and hour;

SER_L is the length specific emission rate, in micrograms per metre and hour;

SER_u is the unit specific emission rate, in micrograms per unit and hour;

c_a is the mass concentration of compound *a* in the sampled air, in micrograms per cubic metre;

AC_t is the hourly air change rate of the test chamber, in air changes per hour;

L_{Vt} is the loading factor in the test chamber, in cubic metre sample per cubic metre test chamber;

L_{Lt} is the loading factor in the test chamber, in metre sample per cubic metre test chamber;

L_{ut} is the loading factor in the test chamber, in sample units per cubic metre test chamber.

f) The mass concentration of the compound in the reference room air is calculated by means of the following equation:

$$c_R = \frac{SER_A L_{AR}}{AC_R} \quad (6a)$$

where

c_R is the mass concentration of compound *a* in the air of the reference room, in micrograms per cubic metre;

SER_A is the area specific emission rate in micrograms per square metre and hour (also called emission factor);

L_{AR} is the loading factor in the reference room, in square metre sample per cubic metre reference room;

AC_R is the hourly air change rate in the reference room, in air changes per hour.

NOTE This calculation is based on a linear relation between the test chamber results and the reference room concentration based on reaching an equilibrium between the emission source and the indoor environment.

If the specific emission rate is expressed not as area specific emission rate but as volume, length or unit specific emission rate, one of the following formulas applies:

$$c_R = \frac{SER_V L_{VR}}{AC_R} \quad (6b)$$

$$c_R = \frac{SER_L L_{LR}}{AC_R} \quad (6c)$$

$$c_R = \frac{SER_u L_{uR}}{AC_R} \quad (6d)$$

where

c_R is the mass concentration of compound *a* in the air of the reference room, in micrograms per cubic metre;

SER_V is the volume specific emission rate, in micrograms per cubic metre and hour;

SER_L is the length specific emission rate, in micrograms per metre and hour;

SER_u is the unit specific emission rate, in micrograms per unit and hour;

L_{VR} is the loading factor in the reference room, in cubic metre sample per cubic metre reference room;

L_{LR} is the loading factor in the reference room, in metre sample per cubic metre reference room;

L_{uR} is the loading factor in the reference room, in sample units per cubic metre reference room;

AC_R is the hourly air change rate in the reference room, in air changes per hour.

10 Reporting for the horizontal reference method

10.1 General

To conform to this Technical Specification, the test report shall include the following information:

a) general reference to the relevant product samples and emission test procedure used;

b) reference to this Technical Specification;

c) if relevant, reference to the applied product standard of the specific construction product.

10.2 Sampling

As far as available, the following shall be reported:

a) time and date of the sampling, duration of transport, batch reference;

b) key elements of the sampling plan, population, sub-population, scale, size and number of sample(s), location for taking the sample(s) and when applicable number of increments in composite sample;

c) sampling conditions.

10.3 Handling of samples in the laboratory, preparation of test specimen

The test report shall include the following information:

a) description of the preparation of the test specimen with photos (taken either after preparation or after placement in the test chamber).

10.4 Test chamber conditions

The test report shall include the following information, as far as relevant:

a) description of the test chamber and test chamber conditions.

10.5 Determination of vapour-phase organic compounds in test chamber air

To conform to this Technical Specification, the test report shall include the following information, as far as relevant:

a) details of which analytes or ranges of analytes were measured;

b) description of the analytical procedure;

c) detection limit and quantification limit of the analytical method;

d) performed elements of the quality control (e.g. calibration, breakthrough, laboratory blanks, desorption efficiency test, duplicates). Results of the quality control shall be reported if relevant for interpretation of test results.

10.6 Calculation and reporting of test results

The test report shall include the following information, as far as required (see Annex H):

a) specific emission rates and respective calculated air concentration in the reference room air of:

   1) identified target compounds, provided with CAS number;

   2) identified non-target compounds, provided with CAS number;

   3) non identified compounds;

   4) volatile carcinogens of categories CARC 1A and CARC 1B (according to Regulation (EC) No 1272/2008);

   5) sum of identified non-target compounds and non-identified compounds;

   6) TVOC;

   7) TSVOC;

b) *R* value;

c) details of the uncertainty of the reported results.

11 Indirect methods

This Technical Specification specifies the horizontal reference method in Clause 1 to Clause 10 and addresses indirect methods in this clause and informative Annex B. The term indirect method is variously applied to mean any simplified, screening, secondary, derived or alternative method.

An indirect method can be applied if it provides within their specific field of application a result comparable or correlated to the result of the reference method in accordance with the purpose of the emission determination. Guidance is given in CEN/TR 16496.

The requirements, especially the sampling requirements, specified for the reference method apply also for the indirect method unless this indirect method specifies different requirements, especially sampling requirements, adapted to its specific field of application.

Such indirect methods may be easier and/or cheaper to apply for a specific application. An indirect method is generally not horizontal, but in most cases dedicated to a specific product or range of products (as specified in a product standard).

NOTE 1 The term "indirect method" has been selected to underline that, instead of a direct determination by the horizontal reference method, an indirect method provides a result indirectly through the mandatory comparability and/or correlation to the reference method (in accordance with the purpose of the emission determination).

NOTE 2 These indirect methods are in accordance with mandate M/366 (see point IV-10) provided that their comparability, correlation have been demonstrated.

The specific and limited field of application of any indirect method may extend to raw materials, product formulation and operating parameters. Anyhow, the validity of the above correlation remains strictly in the field of application for which it has been established.

Annex A
(informative)

Repeatability

A study on robustness of this horizontal standard delivered data on repeatability of VOC emissions testing within one laboratory.

All test results were obtained by testing two or three test specimens from the same sample under identical conditions (in terms of temperature, relative humidity, loading factor, and ventilation rate) in the same laboratory during that study. Each result was analysed for main individual VOCs. The deviation of individual emission chamber test results from their mean value was calculated as % of their mean value for each main individual VOC.

This resulted in:

a) Number of chamber tests: 36.

b) Number of involved products: 6.

c) Number of involved testing laboratories: 9.

d) Number of repeatability data (= number of individual VOCs analyses in these tests): 171.

   1) 56 data for individual VOCs from duplicate determination.

   2) 115 data for individual VOCs from tests with triplicate determination.

Repeatability was found as follows:

— 50 % of all test data showed a deviation of individual test results from their mean value below 13 % (the median of all findings).

— 75 % of all test data showed a deviation of individual test results from their mean value below 26 % (75-percentile of all findings).

— 95 % of all test data showed a deviation of individual test results from their mean value below 54 % (95-percentile of all findings).

— Standard deviation (1 σ) of all test results was 18 %.

— The expanded uncertainty (2 σ) of all test results, representing the 95 % confidence interval, was 35 %.

Repeatability can be very different for VOCs of different chemical characteristics, for products with different emissions mechanisms, and for test results close to the detection limit. These values include also material inhomogeneity and may show higher deviation with small test specimens of products being very inhomogeneous in terms of VOC emissions.

Annex B
(informative)

Examples of indirect methods (also called simplified, screening, secondary, derived or alternative methods)

B.1 General

This annex is informative and is not aimed to be exhaustive. It provides only examples in broad terms as background of Clause 11 of this Technical Specification where the basic requirements are specified that are to be fulfilled by any indirect method for the determination of emissions from construction products of regulated dangerous substances in indoor air.

If the indirect method includes a determination of organic compounds in the air of a specific test chamber, the correlation – comparability with the reference method is easier and closer when this determination is in accordance with Clause 8.

For details of the below cited standards, see Bibliography.

B.2 Indirect methods relative to testing devices for generating emissions

B.2.1 Emission cell

The emission cell testing method is specified in EN ISO 16000-10 and may be applied for most volatile compounds, such as VOC, SVOC, volatile aldehydes. An example of a commercially available emission cell is the FLEC (Field and Laboratory Emissions Cell).

B.2.2 Micro-chamber

Micro-chamber methods are applicable to volatile and semi-volatile organic compounds and aldehydes.

Examples of methods specifying micro-chambers for fast emissions measurement include:

— GUT test method;

— ISO 12219-3.

B.2.3 Thermal extraction

An example of a thermal desorption testing method is specified in VDA 278; this method is applicable to most volatile compounds, such as VOC, SVOC, volatile aldehydes and cetones. Thermal extraction is another way of generating emissions. In this case organic compounds thermally desorbed from the sample are swept away in a stream of inert gas and either analysed using on-line gas chromatography, or by adsorption and thermal desorption as specified in Clause 8 of this Technical Specification.

B.3 Indirect methods relative to analysis

B.3.1 Headspace and in-can VOC determination

Headspace / gas chromatography and in-can testing methods are specified in EN ISO 17895 and in VDA 277 and are applicable to many volatile substances, such as VOC.

B.3.2 VOC content determination

Content determination methods are specified in many standards for many different substances, e.g. for VOC in EN ISO 11890-1 and EN ISO 11890-2.

B.3.3 Formaldehyde specific methods

Formaldehyde-specific testing methods are:

— Chamber test: EN 717-1, ISO 12460-1;

— Gas analysis: EN 717-2, ISO 12460-3;

— Flask test: EN 717-3;

— Perforator test: EN 120, ISO 12460-5;

— Dessicator test: ISO 12460-4, ASTM D5582.

For details of the standards, see Bibliography.

B.4 Indirect methods relative to the evaluation of the test result

B.4.1 Method integrated in production monitoring

When it is required only to verify that the emission of a substance is below a certain level it could be demonstrated by an indirect test method provided that certain manufacturing, raw material or operating parameters remain within certain domains, and the product formulation is unchanged.

B.4.2 Prediction of results at 28 days

When it is required to provide a value of the emission at the reference room, a correlation may be established between an indirect measurement (e.g. the emission at 10 days) and the reference measurement (e.g. the emission at 28 days), based either on accumulated knowledge or to new experiments. The uncertainty attached to the value obtained by correlation is higher than the uncertainty of the reference value obtained by direct testing. But it can be fit for purpose in many cases. Anyhow, as specified in Clause 11, the validity of such correlation remains strictly in the field of application for which it has been established (raw materials, product formulation and production parameters).

Annex C
(informative)

Information on very volatile organic compound (VVOC) testing

The purpose of this annex is to provide information and guidance. Its purpose is not to specify specific requirements since VVOCs determination would require different specific methods under the present stage of practice.

This informative annex addresses the test chamber air sampling and analysis for determining emissions of VVOC in conjunction with VOC and SVOC emissions from construction products in a test chamber.

Air sampling tubes or filters, air sampling velocity and air volume, and analysis, need to be adapted to the respective VVOC in test. In the framework of the present Technical Specification, relevant information and guidance can be found in:

— the informative Annex D of ISO 16000-6:2011;

— EN ISO 16017-1.

Annex D
(informative)

Key concepts for product sampling

NOTE This annex is taken from CEN/TR 16220:2011 and provides information on product sampling with regard to testing emission of regulated dangerous substances from construction products into indoor air.

D.1 Representativeness

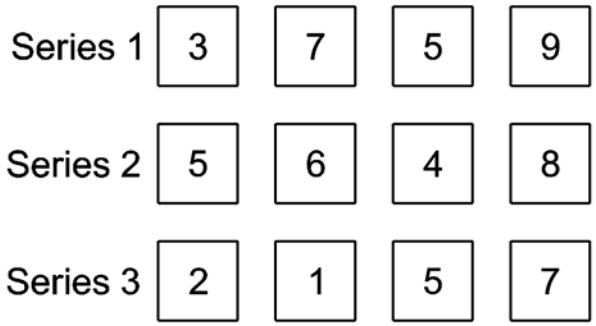
The ultimate goal of product sampling is to obtain a representative portion of the sampled construction product; maintaining the representativeness is essential in all steps where a (partial) sample of the product is involved. Whenever there is variability in the product, measures are to be taken in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

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

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.

Figure D.1 — Numerical example to illustrate the standard deviation

In order to obtain comparable test results, it is important that in a product standard a choice is made with respect to the quantity of product (the scale) on which that product is tested. See also D.7.4.

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.

Sampling might well result in a laboratory sample of 10 kg, while the size of the 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 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.

The size of the 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 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.

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. 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 create fresh surfaces which potentially may disturb the emission test result.

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 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.

D.2 Uncertainty

The associated uncertainty of the final test result is of major importance when assessing the 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 is still 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 by:

— 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 text 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.

D.3 Aspects of repetitive sampling

CEN/TR 16220 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. CEN/TR 16220 provides guidance on how to obtain a sample, either by a single sampling operation or by incremental sampling in which increments are joined into a composite sample. It provides guidance to ensure that the obtained (possibly composite) sample is sufficiently representative for the quantity of product it represents. As such, it provides a basis for the CE marking.

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

NOTE 2 The user expects that a product under CE marking fulfils the specified requirements on the level of the individual product. Testing of each individual product as one entity is often not realistic for environmental characteristics of construction products.

NOTE 3 Repetitive sampling and subsequent assessment of a series of test results against limit values set by national authorities are highly related.

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 D.1). 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.

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 t. 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.

D.4 Series of steps in sampling

A full test procedure can be described as a series of steps, i.e. definition of a sampling plan, taking of a sample, on site sample pre-treatment, packaging, preservation, storage and transportation, delivery, storage

and preservation, preparation of the test specimen, test to determine the emission, analysis/quantification, data management and reporting. These steps should be closely interlinked. CEN/TR 16220 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 D.2 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.

When for example the emission of dangerous substances from a construction product is strongly dependent on the presence of new surface, the sampling and subsequent preparation of the 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 specimen might by itself consist of a number of steps.

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

D.5 Objective of sampling

The objective of sampling a construction product is to obtain a sample in accordance with the specification of the test specimen representing the product given in the product standard and that is:

— sufficiently representative of the quantity of product being assessed, D.1, D.2 and D.3;

— fulfilling the prerequisites for the test(s) to be performed.

D.6 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 annex can be used to produce a sampling plan for:

— the development of a full horizontal test procedure as done in the present document;

— the development of sampling instructions in a product standard by product TCs;

— the production of standardised sampling plans for use under routine circumstances.

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 D.3) are to be addressed. The definition process should:

a) identify those individuals and organisations 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.

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

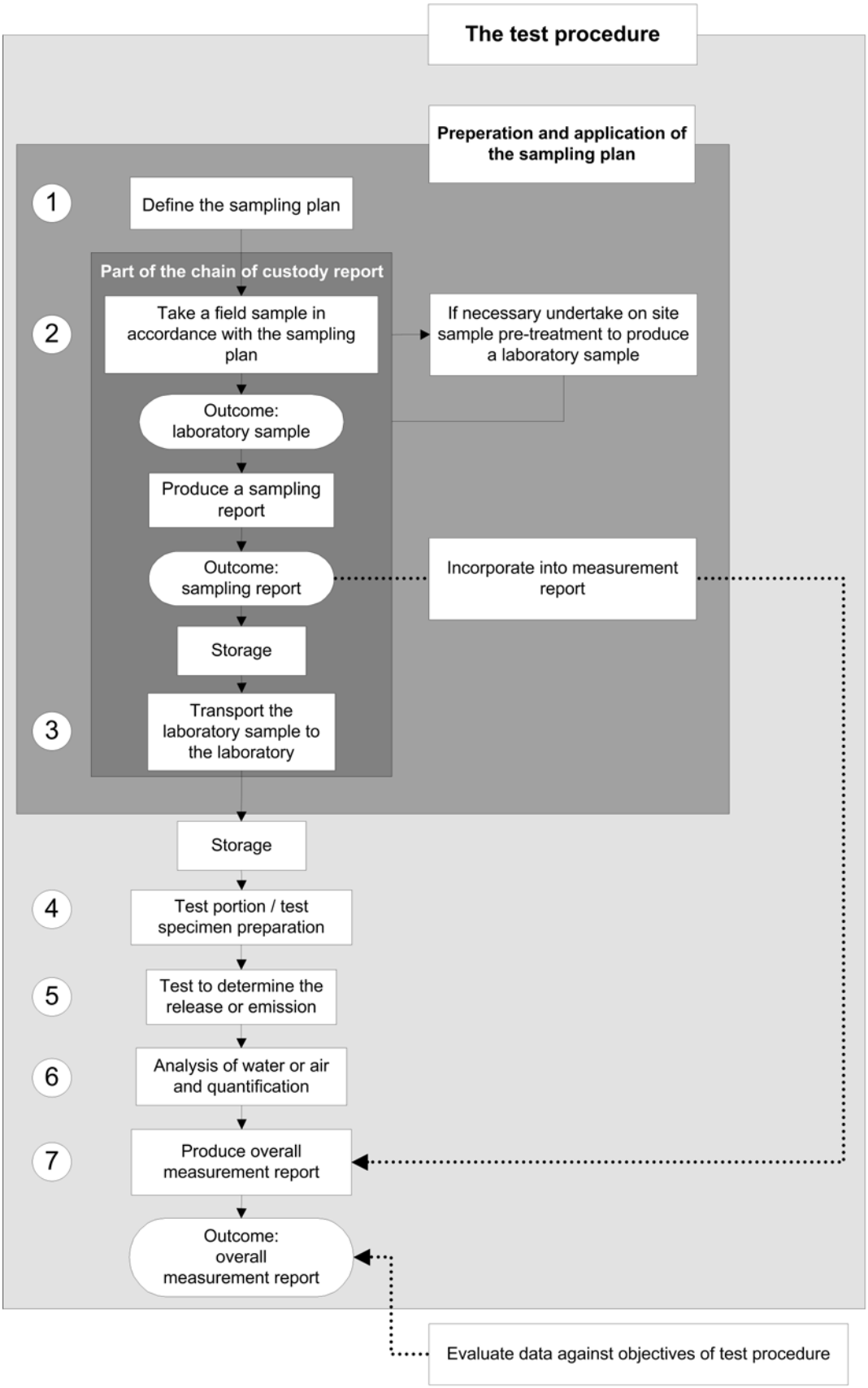


Figure D.2 — Links between the essential elements of a test procedure wherein the main steps are numbered (1 to 7)

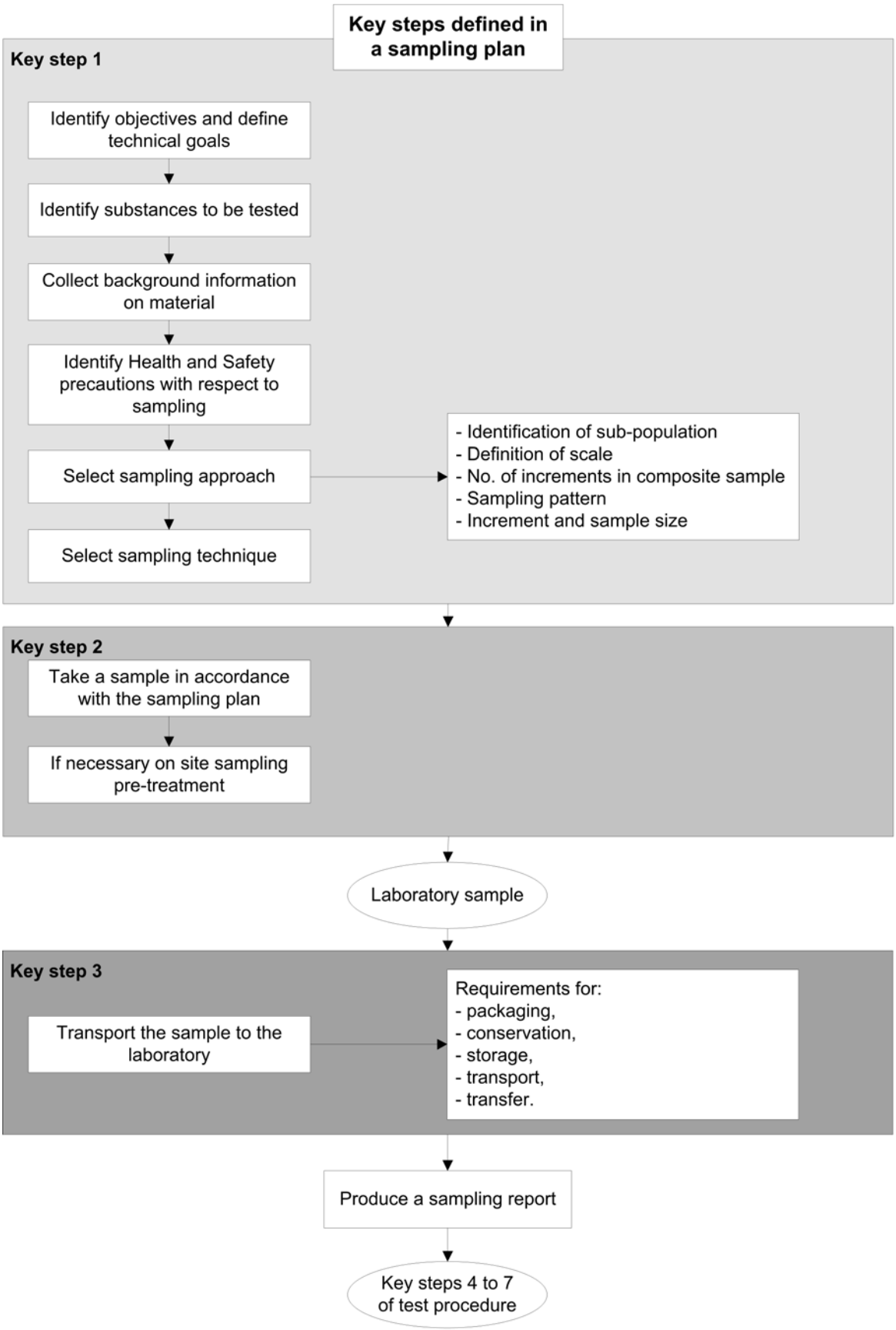


Figure D.3 — Details on the key steps of product sampling

D.7 Sampling strategy

D.7.1 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. Here this term relates to the aspects as referred to in the following subclauses: sampling approach, population and sub-population, scale, increment and sample size, and the sampling location and moment. In general terms, the sampling strategy takes into account 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 subclauses, see also the definitions in Clause 3.

D.7.2 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 and a random mechanism determines which items are part of the sample. This should be preferred if knowledge on product and process is insufficient. Sound statistical conclusions can be applied on the obtained test results only under the conditions of probabilistic sampling. However, when sufficient information is available, the quality of the results obtained with judgemental sampling can be the same as for probabilistic sampling, or even more reliable.

— 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. This term 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, one specifically chosen item might by itself provide an excellent estimate of the characteristic of interest, while probabilistic sampling would require 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:

a) full understanding of the concepts of probabilistic and judgemental sampling;

b) the circumstances under which product sampling is to be performed;

c) the availability of detailed knowledge on the product to be sampled and the process it originates from;

d) the test(s) to be performed.

D.7.3 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" (see Clause 3).

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

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.

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 2 Referring to the example in D.1, 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 emission of dangerous substances is tested on the product that has the highest 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 t. 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 emission of dangerous substances is basically independent of the dimensions of the individual products. To a minor degree, variations in the 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 emission of dangerous substances. A sub-population therefore is defined as all products produced with a specific process using raw materials of specified sources.

D.7.4 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 emission and variations therein, for a quantity of product smaller than the defined scale, is judged to be not relevant for the test results.

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. When obtaining information about a product at the specified scale, then 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 possibility to obtain a smaller, but still representative, laboratory sample by an informed decision on the place or moment of product sampling;

— the possibility to obtain a smaller, but still representative, laboratory sample by on site sample pre-treatment;

— the possibility to obtain a smaller, but still representative test specimen;

— 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. IT 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.

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. 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 often is beneficial (see Example 2 in this clause). As a consequence, the size (mass or volume) of the laboratory sample is larger than the test specimen necessary. On site sample pre-treatment of the obtained large sample or composite sample can be necessary to obtain a representative laboratory sample. The on-site sample pre-treatment, as well as the production of the test specimen in the laboratory, has to ensure that the test specimen is representative for the mean value of the large sample or composite sample. Selecting a larger scale then results in a much better estimate of the mean characteristic of the sub-population, which can result in a lower sampling frequency.

NOTE 2 Choosing a too small scale leads to test results that do not sufficiently represent the product sampled. The variability then is higher than what is expected.

NOTE 3 Fixing the scale in the product standard establishes a level playing field in testing costs between different producers of the same product.

NOTE 4 If the scale would be fixed differently between IT and FPC, there is a considerable chance that the results of IT would no longer be representative for the results obtained through FPC and vice versa.

EXAMPLE 1 Referring to the example in D.1, 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 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 for the test result.

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 D.1 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.

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 for the test result, 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.

If a test specimen for testing a product is 1 kg, when the scale is not considered when defining the sampling plan, a sample of 1 kg is obtained. Variability between samples of 1 kg is substantially higher than variability on a scale of 1 000 kg. At the same time, it is not possible to take a 1 000 kg sample. Therefore, the scale of

1 000 kg may be sampled by incremental sampling. The number of increments taken from the selected 1 000 kg needs to ensure that the resulting composite sample still provides a good estimate of the mean value for 1 000 kg. The number of increments necessary depends on the variability on the scale of the individual increments, for example 250 g, and the accepted uncertainty in the estimated mean value. Assuming this is fulfilled with 20 increments, the resulting composite sample is 5 kg. Preparation of the test specimen in the laboratory then has to ensure the representativeness of a test specimen of 1 kg from the composite sample. As the variability on the scale of 1 kg is substantially higher than the variability on the scale of 1 000 kg, the chance of non-compliance is substantially higher when using a (implicit) scale of 1 kg instead of a scale of 1 000 kg.

EXAMPLE 2 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 (possibly composite) sample is obtained, and the quantity of product that is usually applied by the consumer. If a product TC decided to set the scale for a specific type of floor covering at the median daily production of the involved producers, e.g. 25 000 m^2. Consequently, the test result represents the average emissions of a quantity of 25 000 m^2 with a test specimen size of 1 m^2. This type of floor covering may be used mainly by private consumers, where the median quantity applied is 50 m^2. The consumer expects that the emissions declared are valid for the quantity that he used.

In case of small variability of emissions within the production of one day, a single randomly sampled 1 m^2 shows emissions comparable to the emissions of any randomly chosen 1 m^2 sample during that day. In such a situation, the producer does not need to worry (too much) about the difference between the chosen scale (25 000 m^2), and the quantity of the product that is normally used (50 m^2).

In case of large variability of emissions within the production of one day, there might be an unacceptable large chance that the product does not comply for a certain quantity of 50 m^2, even though the mean of the whole day does comply. Consequently, the producer might expect consumer complaints. Apart from making changes to the process to enable a more stable quality of the product with respect to the emission of regulated 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 complaints is far less.

When comparing test results of a relatively heterogeneous construction product with a given set of limit values, a well-chosen scale can have a significant effect on the conclusions with respect to (non-)compliance. In Figure D.4, the test results of a product over time are depicted. Tests have been performed with test specimens obtained from composite samples of 500 g. These composite samples were obtained from a product sampled over time on a scale of 100 kg, 1 000 kg and 2 000 kg. Non-conformity is assumed at a value 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 1 000 kg, the product always complies. Consequently, the test frequency can be much lower. Finally, when looking at the product quality on a scale of 2 000 kg, there is little variability in the product, it lies well below the limit value and consequently a low test frequency is possible. It should be noted that regulations may not accept values above the limit value at any time.

EXAMPLE 3 The manufacturer of autoclaved aerated concrete in Example 2 in D.7.3 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 t each.

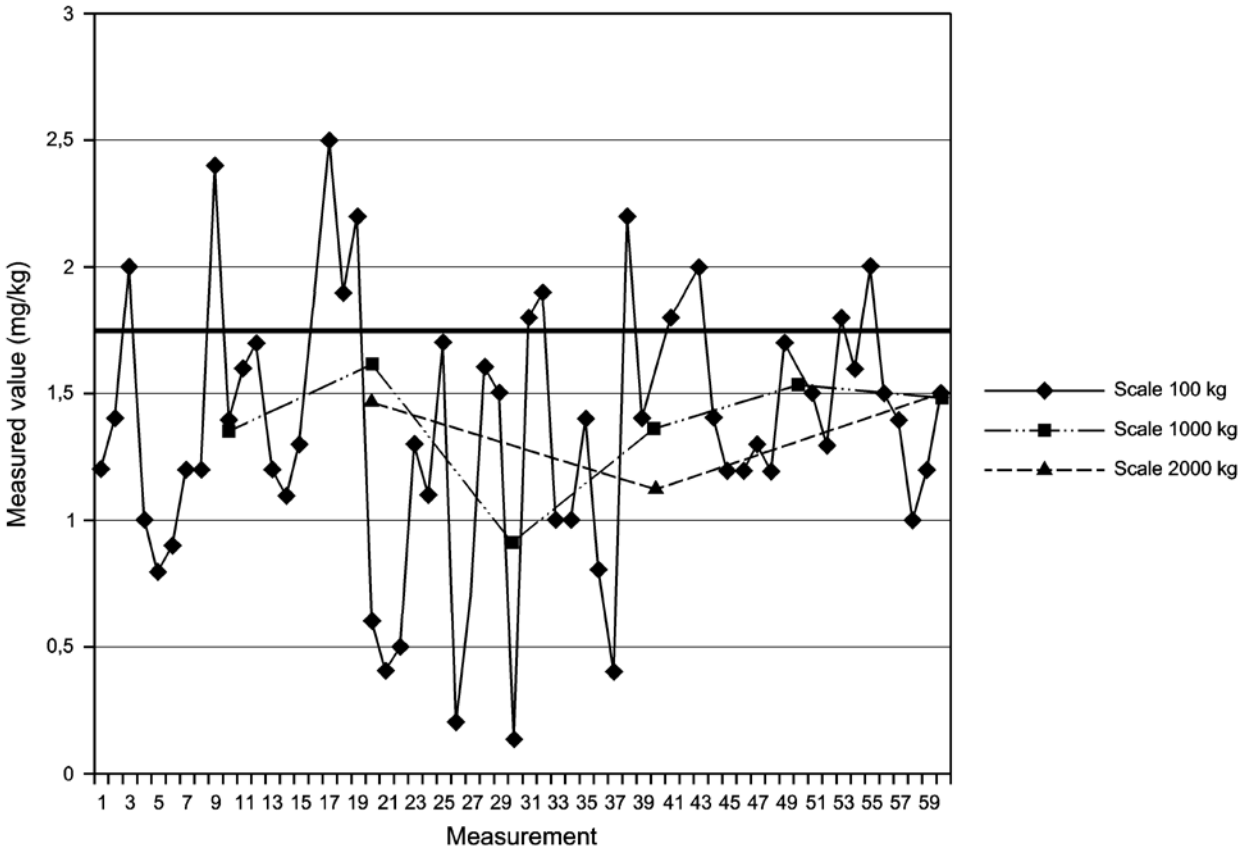


Figure D.4 — Results over time for a product tested at three different scales, 100 kg, 1 000 kg and 2 000 kg, where the horizontal bold line is the cut off for non-conformity

D.7.5 Size of increments and samples

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.

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. This has to be weighed against the fact that new cutting edges may impact emissions test result in an unacceptable manner.

NOTE 1 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.

Incremental sampling does not per definition imply mixing of the product. Given the example of D.1, 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 might become necessary when the 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 specimen.

NOTE 2 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.

D.7.6 Sampling of complex, composite and large products

Samples of complex, composite and large products are taken and test specimens are prepared for emission testing in accordance with the specifications given in the product standard specifying the considered product.

Complex, composite and large products are evaluated as whole unit. For practical purposes, the product standard specifying the considered product may foresee testing a model representing the whole product.

CEN/TR 16220 provides guidance on obtaining samples and increments (joint in a composite sample) which are sufficiently representative for the sub-population sampled.

The declaration of performance is targeted at the product in its intended use. As some products per definition are applied together with other products, this should be taken into account, specifically as the emission of the product might be highly dependent on the combination of products.

D.7.7 Sampling location and moment

CEN/TR 16220 provides guidance on the location where and moment when a sample or individual increment is taken.

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 for which the performance is declared. 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. Another good strategy is worst-case sampling, where the sample taken represents products with the highest expected emissions.

NOTE 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 available, it would be highly beneficial to obtain that information as this allows for informed decision on the cost effective testing of the product.

Annex E
(informative)

Example of a form for the sampling report

<table>
<tr><td colspan="2">Testing laboratory / certification body:</td><td colspan="2">Sampler (name, company, telephone):</td></tr>
<tr><td colspan="2">Name of the manufacturer at the place of sampling (address/stamp):</td><td colspan="2">Manufacturer (if deviating from company's name at the place of sampling):</td></tr>
<tr><td colspan="2">Name of the product:</td><td colspan="2">Type of product (e.g. laminate, textile flooring, PVC-flooring):</td></tr>
<tr><td colspan="2">Model/program/series:</td><td colspan="2">Batch No:</td></tr>
<tr><td colspan="2">Article No:
Misc.:</td><td colspan="2">Date of batch production:</td></tr>
<tr><td rowspan="2">Sample is taken from</td><td>⌗ Production
⌗ Store
⌗ Miscellaneous</td><td rowspan="2">How had the product been stored prior to sampling?</td><td>⌗ open
⌗ in the stack
⌗ wrapped up</td></tr>
<tr><td>Place of storage:</td><td>Packing material:</td></tr>
<tr><td colspan="4">Specifics (possible negative influences by emission at the place of taking the sample, petrol emissions, solvent emissions from production, uncertainties, questions, etc.):</td></tr>
<tr><td colspan="4">Cut edges (identification of cut edges when present and identification of new surfaces and surface to be exposed in the emission test):</td></tr>
<tr><td colspan="4">Confirmation
The signer herewith confirms the correctness of the data given above. The sample was selected, drawn and packed personally in accordance with the instructions for the taking of samples.</td></tr>
<tr><td colspan="2">Date of sampling:</td><td colspan="2">Signature:
(Stamp)</td></tr>
</table>

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.

Annex F
(informative)

Example form for a chain of custody report

Chain of custody report							Reference no.:			
Site:					File name:					
Start:	Date		Time		File no.:					
End	Date		Time		Person in charge:					
Sampler, initials:					Other specifications:					
Sampling report reference no.:										
Sample no. start:			Total no. of samples:							
Sample no. end:			Laboratory:							
Sample ID:										

Handed over between:		Time	Justification	Conditions
Handed over by:	Initials			
	Signature			
Handed over to:	Initials			
	Signature			
Handed over by:	Initials			
	Signature			
Handed over to:	Initials			
	Signature			
Handed over by:	Initials			
	Signature			
Handed over to:	Initials			

Annex G
(informative)

Benzene artefact generation on Tenax

Benzene can be generated as an artefact on clean Tenax TA tubes. The level of benzene is thought to be primarily linked to tube desorption temperature and is minimised if tubes are stringently conditioned at temperatures up to 320 °C, then desorbed at temperatures of 280 °C or below. However, other factors may also contribute, for example interaction with ozone or certain VOCs in the sampled air. It has not yet been possible to identify rules or correlations explaining completely under which conditions this occurs or not. If significant benzene artefact levels are generated from Tenax TA during analysis, it can interfere with benzene measurements in the low µg/m^3 range giving falsely high readings.

In the course of a robustness study of this Technical Specification, investigations on the generation of benzene during air sampling and analysis were performed in different laboratories with different experimental settings.

These investigations showed that some laboratories reported unexpected increase of benzene levels on Tenax TA tubes after sampling from an atmosphere known to be free of benzene. In these cases, this benzene level was higher than blank level determined from the same Tenax TA tube before air sampling.

In most (but not all) of the cases, benzene increased during air sampling in the low nanogram range, resulting in false chamber air concentrations generally below 0,2 µg/m^3, but up to 2 µg/m^3 in other cases. This can impair accuracy of benzene determination in low levels in the µg/m^3 range.

One possible assumption is that benzene is generated on Tenax TA tubes during air sampling under certain conditions. The mechanism has not been identified. It cannot be excluded that certain VOC mixtures after adsorption undergo chemical reaction while sampling air passes over surface of Tenax TA.

As benzene test results in the low µg/m^3 range can be overestimated by artefact generation to a significant extent, it is recommended to verify low-level test results with an independent second testing method (see Annex B for additional information) before assessing a test result against any low limit value of e.g. 1 µg/m^3.

If the product manufacturer or the testing laboratory expects or experiences a significant risk of artefact generation of benzene, especially in cases of low test results, this should be communicated between the involved parties and considered when reporting and interpreting the test results.

Annex H
(informative)

Information on regulations concerning the emission into indoor air of dangerous substances from construction products

At the time when this CEN/TS was adopted several national regulations were requiring the determination of the emission into indoor air of dangerous substances from construction products. They are listed below in alphabetical order and include the references of these regulations.

It should be noted that these regulations may require different and/or additional calculation methods for assessing the test results.

— **BELGIUM**
Draft Royal Decree establishing threshold levels for the emissions to the indoor environment from construction products for certain intended uses and notified in October 2012 under the 98/34 Directive with number 2012/568/B.

— **FRANCE**
Décret n° 2011-321 du 23 mars 2011 relatif à l'étiquetage des produits de construction ou de revêtement de mur ou de sol et des peintures et vernis sur leurs émissions de polluants volatils, NOR: DEVL1101903D http://www.developpement-durable.gouv.fr/Chapitre-II-Industriels-comment.html

Arrêté du 19 avril 2011 relatif à l'étiquetage des produits de construction ou de revêtement de mur ou de sol et des peintures et vernis sur leurs émissions de polluants volatils, NOR: DEVL1104875A, http://www.developpement-durable.gouv.fr/Chapitre-II-Industriels-comment.html.

Arrêté du 30 avril 2009 relatif aux conditions de mise sur le marché des produits de construction et de décoration contenant des substances cancérigènes, mutagènes ou reprotoxiques de catégorie 1 ou 2, NOR: DEVP0908633A

Arrêté du 28 mai 2009 modifiant l'arrêté du 30 avril 2009 relatif aux conditions de mise sur le marché des produits de construction et de décoration contenant des substances cancérigènes, mutagènes ou reprotoxiques de catégorie 1 ou 2 NOR: DEVP0910046A, http://www.developpement-durable.gouv.fr/Chapitre-II-Industriels-comment.html

Arrêté modifiant l'arrêté du 19 avril 2011 relatif à l'étiquetage des produits de construction ou de revêtement de mur ou de sol et des peintures et vernis sur leurs émissions de polluants volatils, NOR: 1133129A, http://www.developpement-durable.gouv.fr/Chapitre-II-Industriels-comment.html

— **GERMANY**
Grundsätze zur gesundheitlichen Bewertung von Bauprodukten in Innenräumen (August 2011) http://www.dibt.de/en/Divisions/Referat_II4.html

The construction products for which the requirements are binding are listed in: http://www.dibt.de/en/Service/Dokumente-Listen-BRL.html

Volatile carcinogenic substances of categories CARC 1A and CARC 1B of Annex VI to Regulation (EC) No 1272/2008 (Table 3.1).

The current list of LCI compounds and values is published by the AgBB (Ausschuss zur gesundheitlichen Bewertung von Bauprodukten; http://www.umweltbundesamt.de/en/topics/health/committee-for-health-related-evaluation-of-building).

EXAMPLE of different and/or additional calculation methods that may be required by regulation for assessing the test results: Supplementary calculation of $TVOC_{SUM}$

If required, it may be necessary to supplement the TVOC test result (as specified in 3.3.9) with a $TVOC_{SUM}$ value calculated as follows:

a) The $TVOC_{SUM}$ is determined by summing the individual concentrations of every identified and unidentified VOC at a concentration of ≥ 5 µg/m^3, after subtracting non-interfering artefacts. In general, the highest possible degree of identification – beyond the information of ISO 16000-6 – should be pursued in order to enable an individual substance assessment.

b) $TVOC_{SUM}$ is defined as the sum of:

   1) all individual target VOCs (compounds with a LCI value) (see Ad c.1 below);

   2) all volatile carcinogenic substances of EU categories 1A and 1B (see Ad c.2 below);

   3) all individual non-target VOCs and non-identified VOCs (see Ad c.3 below).

Ad c.1) Every target VOC (compounds with a national LCI value) is identified via its mass spectrum and retention time, and quantified via its substance-specific response factors, provided the available chromatogram allows reliable quantification. For this, it is assumed that all of these compounds are available in the testing laboratory either as pure substances or in form of certified standard solutions.

Ad c.2) Every carcinogenic substance in EU categories 1A and 1B is defined via its mass spectrum and retention time, and quantified via its substance-specific response factors, provided the available chromatogram allows reliable quantification. For carcinogenic substances, a detection threshold of ≤ 1µg/m^3 should be ensured.

Ad c.3) Every non-target VOC and non-identified VOC is quantified as toluene equivalent (TE).

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