New European VOC emissions testing method CEN/TS 16516 and CE marking of construction products

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Abstract CE marking of construction products used indoors soon will require a documentation of product emissions of Volatile Organic Compounds (VOC) into indoor air for several product types. A new horizontal testing method for VOC emissions was published in October 2013 as CEN/TS 16516. It is based on ISO 16000 standard series, but it contains additional refinements for improving reliability and has gone through extensive validation. This article summarizes the role of the new testing standard for CE marking, the main characteristics of that standard, major differences to other relevant testing standards (such as EN 717-1 for formaldehyde emissions), and key findings of the validation studies.

Die neue europäische Prüfnorm CEN/TS 16516 für VOC-Emissionen und die CE-Kennzeichnung von Bauprodukten

Zusammenfassung Für das CE-Zeichen benötigt man demnächst Angaben zur Emission flüchtiger organischer Verbindungen (VOC) für eine Reihe von im Innenraum verwendeten Bauprodukten. Eine neue horizontale Prüfnorm wurde im Oktober 2013 als CEN/TS 16516 veröffentlicht. Diese beruht auf den Normen der ISO-16000-Serie, jedoch mit einigen Ergänzungen zur Erhöhung der Zuverlässigkeit, und wurde einer gründlichen Validierung unterzogen. Der Beitrag skizziert die Rolle der neuen Prüfnorm im Rahmen der CE-Kennzeichnung, fasst die wesentlichen Elemente der Prüfnorm sowie die Unterschiede zu anderen Prüfnormen (z. B. EN 717-1 für Formaldehydemissionen) zusammen und stellt die wesentlichen Ergebnisse der Validierung vor.

1 Introduction

CE marking of construction products used indoors soon will require a documentation of product emissions of Volatile Organic Compounds (VOC) into indoor air for several product types. This requires a series of actions: a decision of the European Commission, an amendment of a harmonized product standard by CEN, national legislation in the targeted EU Member States, and publication of a horizontal testing standard for the determination of VOC emissions. The new horizontal technical specification CEN/TS 16516 [1] is based on ISO 16000 standard series, but with additional refinements for improving reliability. It has gone through extensive validation and it was published in October 2013.

2 Background

2.1 CE marking

A CE mark is required for several construction products as a prerequisite for being sold in any Member State of the European Union (EU). The objective of CE marking within the frame of the Construction Products Regulation (EU/305/2011) is not to define the safety of construction products, but to ensure that reliable information on product performance is presented in a harmonized manner across Europe. The intention is to substitute national approval systems by CE marking and to facilitate cross-border trade.

This is achieved by providing a common technical language for use in the Declaration of Performance that has to follow the CE marked product. Details are specified mainly in harmonized European product standards that shall be used by manufacturers, and by public authorities when these set requirements regarding the performance of construction products. The actual requirements (limit values, performance classes) still are specified by each EU Member State separately. The Construction Products Regulation does not entitle the European Commission to harmonize the requirements; this can happen only on a voluntary basis by agreement between the involved national governments.

By affixing the CE mark on a product, the manufacturer declares and is responsible for that the product is in conformity with the so-called essential requirements of the regulation that apply to it, and that this conformity was assessed as specified in the relevant product standards. Products bearing the CE mark then benefit from free circulation in the European Market.

Soon the Declaration of Performance following CE marking will also include information on VOC emissions for several product types. Once established, no concerned construction product without CE mark – and thus without a statement on VOC emissions level – must be sold in countries where a national regulation of the emissions into indoor air exists.

But this does not cover all construction products. It only concerns products for which

- the European Commission decided to issue a mandate to the European Commitee for Standardization (CEN) that includes VOC emissions;
- CEN produced or amended a harmonized standard covering VOC emissions as one of the product performance categories to be declared;
- national regulations exist.

Even when a harmonized standard covers VOC emissions, these can be assigned a „No Performance Declared“ (NPD) declaration without any testing if a product is distributed only in EU Member States without national legislation on VOC emissions. But in countries with relevant national legislation (such as Germany, France and soon Belgium) the reader shall be able to read from the declaration of performance whether it is legal to sell that product in his country – and which VOC emissions class label has to be attached in France.

2.2 Existing testing standards

Regulatory requirements on VOC emissions in Member States of the EU refer to ISO 16000 testing standards (parts 3, 6, 9, 11 and sometimes also to part 10).

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The German DIBt agency [2] added to those standards a number of supplemental specifications on details of testing, and some modifications of the original testing details. This helped achieving an improved reproducibility as the ISO 16000 standards do not specify all testing parameters in sufficient detail. But some of the testing details specified by DIBt were not made public, or they were made available only in German language. This can make it difficult to follow the testing requirements for non-German industries and testing laboratories and can be seen as a barrier to trade.
The French VOC regulation [3] just refers to ISO 16000 testing standards and added some additional specifications on how to test paints, coatings, doors and windows – available only in French language, again making it difficult to follow the testing requirements for non-French industries and testing laboratories.
The draft Belgian VOC regulation [4] refers to ISO 16000 testing standards, but it declared the intention to refer to the new European technical specification (CEN/TS 16516) in the final version of that regulation. Several language versions are available.
Several voluntary low-VOC labels are referring to either ISO 16000 standards, or to the German DIBt testing method, or they use in-house testing methods. But most of these standards are compatible with the new technical specification CEN/TS 16516.
Another testing standard, EN 717-1, was established for determination of formaldehyde emissions from wood-based panels. EN 717-1 does not determine any emissions other than formaldehyde. The testing method is targeted at monitoring a very special release mechanism of formaldehyde. Most wood-based panels are glued with a polymer producing small amounts of formaldehyde continuously by hydrolysis (decomposition by reaction with water) in contact with normal air humidity. This reaction can produce a stable "steady-state" concentration.
EN 717-1 requires many formaldehyde determinations during the testing period. The test can be stopped after 4 days if no formaldehyde is detected. Otherwise, steady-state concentration is reached when the results do not change with more than 5% during 4 days. If this is not reached until 10th day, the test is prolonged until up to 28th day. If a steady-state concentration then still is not reached, the extrapolated 28th day test result is taken as end result. EN 717-1 test results are used mainly for assigning formaldehyde E1 and E2 classes to wood-based products in the frame of CE marking, e.g. for wood-based panels (EN 13986), wooden floorings (EN 14342), etc.
Some programs such as BREEAM in the UK required EN 717-1 testing also for other products (e.g. carpets) not showing this equilibrium reaction with a steady-state concentration. This is based on a misunderstanding of the scope of application of EN 717-1. The much simpler ISO 16000 test after 28 days would deliver the same information on long-term formaldehyde emissions with fewer efforts. While this requirement was revised in the most recent version of BREEAM International, some local BREEAM versions did not yet realize that change.

3 Harmonization

While it is not the task of the European Commission to harmonize testing methods of any private labels, it can harmonize the testing methods that are required for showing compliance with legal requirements across Europe. CEN received the mandate „M/366" [5] to develop a horizontal testing method for determining VOC emissions. CEN established the Technical Committee (TC) 351 "Construction products: Assessment of release of dangerous substances". Working Group (WG) 2 of TC 351 developed a testing standard that brings together the different existing standards that are relevant for national regulations of VOC emissions, making use of present state-of-the-art of VOC emissions testing. The new horizontal standard for determination of VOC emissions was published as CEN Technical Specification CEN/TS 16516 [1] in October 2013. Within CEN, a Technical Specification is a testing standard that has not yet gone through complete validation. Transferral into a full EN standard is possible after the validation was completed.
CEN/TS 16516 specifies the operation of ventilated test chambers, the analysis of emitted compounds from test chamber air, calculation and reporting of test results. The standard also covers the principles of taking representative samples and of preparation of test specimens, but it is expected that the details are further developed in product specific TCs when editing the product related harmonized European Standards (hEN). This process started in 2013. The product standards must not re-define the testing method. Instead, it is the intention of the EU Commission that the product standards will refer to the horizontal VOC emissions standard CEN/TS 16516 – this is why that standard is called horizontal, meaning that it will be used by a variety of product specific standards.
Another testing standard interpreting CEN/TS 16516 for the specific aspects of paints and coatings was published as EN 16402 [6] by end of 2013. It will specifically determine application amount and technique when making a test specimen for emissions testing, and pre-conditioning periods for simulation of drying before (re-)occupancy of the building.
CEN/TC 351/WG 2 started the process of transforming the CEN/TS 16516 into a European Standard until 2016, based on the below summarized validation studies.

3.1 The new testing standard

In the following the basic principles of CEN/TS 16516 are outlined, and differences to existing testing standards are highlighted.

3.1.1 European Reference Room

CEN/TS 16516 [1] defines a European Reference Room (see **Table 1**). This is not a test room. The design figures of the reference room serve as guidance for operation of test chambers, and as exposure scenario to which all test results shall be calculated back.
The European Reference Room is already in use in regulations on VOC emissions in France and in Germany. The planned Belgian regulation also refers to that reference. The European Reference Room defines an equal framework for all products in all types of buildings, intending to allow fair competition. This common reference for all emissions tests across Europe is one of the major achievements of the new testing standard CEN/TS 16516. It allows an unambiguous comparison of VOC emissions between different products and avoids „green-washing" of non-complying products that would occur if applying another reference room with

Table 1. European Reference Room, compared to reference scenarios in ISO 16000-9 [7] and EN 717-1 [8].

	CEN/TS 16516	ISO 16000-9	EN 717-1
Temperature in °C	23	23	23
Relative humidity in %	50	50	45
Ventilation rate in ach*	0.5	0.5	1.0
Dimensions	3 m x 4 m x 2.5 m 1 door, 1 window	Not specified	Not specified
Volume in m³	30	17.4	Not specified
Loading factor in m²/m³			1.0
Walls	1.0	1.4	
Floor or ceiling	0.4	0.4	
Small surfaces, e.g. door or window	0.05	not specified	
Very small surfaces	0.007	0.011	

* ach = air change per hour

higher ventilation and/or lower material loading, resulting in larger dilution of the emissions and lower test results.

3.1.2 Taking samples for testing

It is essential to take samples for testing in a representative manner. CEN/TS 16516 describes two principles of sampling. Statistical sampling would collect a significant number of random samples (e.g. from different batches) and then either all of these are tested, or these samples are combined into a mixed sample for testing – which is not possible for solid samples without impairing the test result by emissions from cutting edges.

The second option is judgemental sampling and requires knowledge of the production and the product parameters that influence VOC emissions of the product after installation, such as temperature during manufacture, drying conditions, raw materials, material thickness, coatings applied, etc. Then one sample representing the worst case with the highest expected emissions can substitute many random samples and still deliver information of higher significance and reliability.

In any case the person selecting a sample is requested to make a clear decision on the sampling approach, and to use sampling report sheets and a chain-of-custody form for allowing traceability of the tested sample. Neither ISO 16000-11 nor EN 717-1 includes specifications of how to select samples for testing from production or storage in such detail.

Guidance is given on the maximum age of the sample when starting the test. Product samples shall be taken from the factory at the earliest point of time when the product is ready for dispatch or application. Unopened canned products are given maximum 4 months before start of test; in other cases 8 weeks is the maximum accepted sample age. Product specific deviations may be specified by the responsible product specific Technical Committees.

3.1.3 Test specimens

CEN/TS 16516 contains a number of principles for making the test specimen from the sample, leaving many details open for later specification in product specific standards. It also contains guidance on appropriate techniques for sealing back and edges where appropriate, based on most recent experimental findings.

A flooring test specimen with sealed back and edges is shown in **Figure 1**. Liquid samples can be applied to Petri dishes (see **Figure 2**).

Figure 1. A flooring test specimen with sealed back and edges.

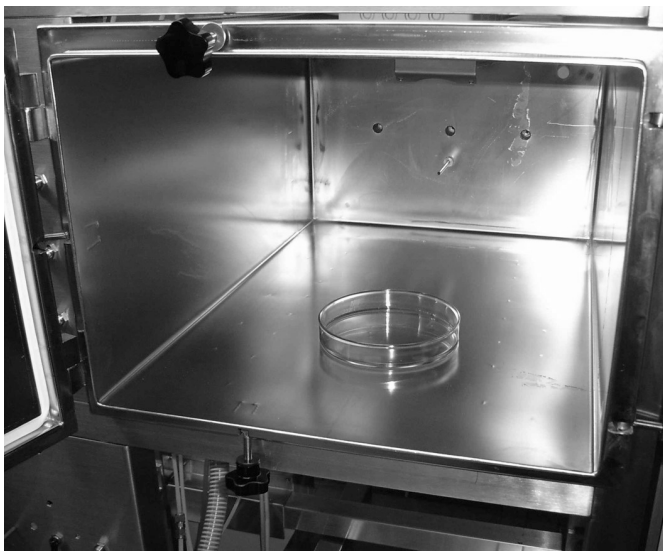

Figure 2. Liquid sample in a Petri dish.

Table 2. Test chamber parameters.

	CEN/TS 16516	ISO 16000-9	EN 717-1
Temperature in °C	23 ± 1	23 ± 2	23 ± 0.5
Relative humidity in %	50 ± 5	50 ± 5	45 ± 3
Air change rate in ach	0.25 to 1.5	Variable *	1.0 ± 0.05
Volume in m³	Minimum 20 l	Not specified	12 m³, 1 m³, or 225 l
Chamber material	Stainless steel or glass	Stainless steel or glass	Stainless steel, aluminium, glass, PVC, PMMA, …
Loading factor in m²/m³	< 50 to 200% of ref. room – max. 2.0	Variable *	1.0 ± 0.02

* Loading factor and ventilation can be changed simultaneously for ISO 16000-9 as long as the target area specific air flow rate is achieved.

Figure 3. VOC emissions are tested in ventilated stainless steel climate chambers.

3.1.4 Test chamber operation

CEN/TS 16516 specifies the operation of the ventilated test chambers in a partly different manner than the other existing standards (see **Table 2**).

CEN/TS 16516 specifies the accepted variation of air change, loading factor and relative humidity in a broader interval for allowing to combine testing for CEN/TS 16516, EN 717-1 and US American testing standards into one single test setup. The robustness validation study [9] showed that testing within these narrow intervals will not deteriorate the test result.
CEN/TS 16516 also specifies how precisely the once selected chamber parameters shall be maintained during the whole testing period in a more clear and pragmatic manner than the other standards do. It is expected that this leads to better reproducibility of test results, compared to ISO 16000 testing.
Figure 3 shows an arrangement of ventilated stainless steel climate chambers.

3.1.5 Chamber air sampling and analysis

CEN/TS 16516 specifies test chamber air sampling and analysis (see **Tables 3** and **4**) and gives more specific and precise guidance than ISO 16000 does on

- timing of air sampling,
- air sampling volume,
- sample storage (no storage outside the test chamber during the test and before the last air sampling was performed),
- calibration of individual VOCs,
- reporting limits, and more details,
- specification of the gas chromatographic column type in a compulsory manner, while ISO 16000 only gives a non-binding recommendation.

It is expected that all this leads to better reproducibility of test results, compared to ISO 16000 testing.

Table 3. Test chamber air sampling.

	CEN/TS 16516	ISO 16000-9	EN 717-1
Air sampling dates	Either 28 days, or 3 plus 28 days, depending on legislation	72 ± 2 h and 28 ± 2 days	Minimum 3h difference between two sampling dates
VOC air sampling duration	Same time before and after the target sampling date	Not specified	Not applicable
VOC air sampling volume in l	Max. 5; two different volumes in parallel air sampling	Max. 5	Not applicable
VOC air sampling velocity in ml/min	20 to 200	50 to 200	Not applicable
Storage between two air sampling dates	In test chamber	In or outside test chamber, free choice	In test chamber

Table 4. Test chamber air analysis. *DNPH: Dinitrophenylhydrazone*

	CEN/TS 16516	ISO 16000-9	EN 717-1
Aldehydes	DNPH (ISO 16000-3)	DNPH (ISO 16000-3)	Acetylacetone; DNPH (ISO 16000-3) also is accepted
VOC – sampling	Tenax TA	Tenax TA	Not applicable
VOC – desorption	Thermal desorption	Thermal desorption	Not applicable
VOC – analysis	GC/MS only	GC/MS or GC/FID	Not applicable
Gas chromatographic column	Compulsory: slightly polar, 5% phenyl/95% methyl poly siloxane	Recommended: non-polar, 100% dimethyl poly siloxane	Not applicable
Reporting limit in µg/m³	5; 1 for carcinogens if technically feasible	2	Not specified
Calibration	All target VOCs: with their response factor. All others: as toluene equivalent. *	Best possible	Formaldehyde specific

* Target VOCs: VOCs with a limit value

Table 5. Expression of results.

	CEN/TS 16516	ISO 16000-9	EN 717-1
TVOC calculation	Sum of all VOCs above 5 µg/m³, all calculated as toluene equivalent	Total area of chromatogram, calculated as toluene equivalent	Not applicable
Expression of result	Concentrations of individual VOCs and SVOCs, TVOC, TSVOC, some other parameters and R value, (depending on included legislation) – all calculated for the European Reference Room	Specific emission rates of individual VOCs and TVOC	Extrapolated formaldehyde steady-state concentration in test chamber

3.1.6 Expression of results, reporting

CEN/TS 16516 specifies how to express test results (see **Table 5**).

The specific emission rate (e.g. per surface of the tested product) is the primary result of any VOC emission test. But all limit values are expressed as mass concentration in air, not as specific emission rate. Therefore the specific emission rate has to be calculated into a contribution of the emissions to the mass concentration in the air of the European Reference Room.

The exclusion of small traces of emitted VOC below 5 µg/m³ aims at excluding results with high uncertainty. A harmonized cut-off limit for VOC and TVOC calculation will increase comparability between testing labs significantly.

There was some dispute on TVOC calculation. The compromise in CEN/TS 16516 is to calculate TVOC as the sum of all individual VOCs, all of them calculated as toluene equivalent, if they show emissions higher than 5 µg/m³ when calculated for the European Reference Room. This is an unambiguous definition compared to the TVOC definition given in ISO 16000-6. In contrast to both, German regulation foresees that all target VOCs (those with a limit value) are calculated with their respective response factors, and all VOCs without a limit value are calculated as toluene equivalent. German TVOC then is the sum of all such calculated VOCs. As there are three national regulations across Europe, all with different lists of limit values, an adoption of this principle on European level would have resulted in different TVOC values per country – with the risk to confuse the market. CEN/TC 351/WG 2 decided that only one uniform TVOC calculation across all countries was specified instead, as described above.

3.2 Validation of the standard

The CEN/TS 16516 testing standard had gone through validation of robustness [9], repeatability [9; 10], and reproducibility [10]. Robustness validation means an investigation on how a modification of individual testing parameters (e.g. testing temperature) will influence the test result. The robustness validation study compared area specific emission rates, as these are independent of specific testing parameters, in contrast to test chamber air concentrations. Such information helps setting acceptable tolerance values of the relevant testing parameters.

Repeatability validation means how similar test results of different test specimens from the same test sample will be within one testing laboratory. Reproducibility validation means how similar test results of different test samples taken from the same product will be between different testing laboratories.

3.2.1 Chamber operation

Main findings of the robustness validation of chamber operation parameters are [9]:

- Temperature had some impact on specific emission rate, but less than expected.
- Relative humidity of supply air had some impact on specific emission rate, but less significant than temperature.

● Test chambers with volume between 0.02 and 3 m³ gave equivalent specific emission rates (within the normal variation of test results).
● Higher loading factor had some impact on specific emission rate for some products.
● Higher ventilation rate had some impact on specific emission rate for some products.
The present narrow tolerance intervals in CEN/TS 16516 were confirmed by the study for all above parameters. No significant impact on test result was seen when changing between testing parameters of CEN/TS 16516 and of EN 717-1 – even not for products where a larger effect was expected (wood-based panel, wooden flooring, and glass wool).
● Using a solid reference material for toluene gave recovery of 80 to 120% in most involved test chambers.
This was considered as benchmark.

3.2.2 Interfering factors

Findings of the robustness validation on other interfering factors are [9]:
● On-site wet-applied products can give falsified increased level of emissions because some portion of high initial emissions may be adsorbed and then remain on the test chamber walls. These will be re-desorbed from the test chamber walls later during the test duration and then increase the test result at later points of time.
– This is different from reality where typical indoor walls will not show that strong re-desorption rate.
– This can be solved by applying some days of pre-conditioning the fresh test specimen in separate ventilated chambers, before transferring the test specimen into the actual test chamber and starting the test.
– For similar reasons the test specimen shall remain in test chamber all the time during testing. Otherwise the described adsorption/desorption equilibrium will be disturbed during temporary storage outside the test chamber, leading to non-comparable test results between laboratories if they handle this issue in a different manner.
● Sample age at start of test showed not to be important for most evaluated products.
● If only emissions from the top surface are relevant then edges and back shall be sealed.
– Most efficient sealing techniques were
-- back to back storage of plates, with edges covered with aluminium tape,
-- tight coverage of edges and back with aluminium foil,
-- seal box as specified in the Japanese standard JIS A 1901 [11].
– If aluminium tape is used then a regular monitoring of its blank value is essential for avoiding false findings.
● Homogeneity of emissions across the surface of the laboratory samples was between < 10 and 20% relative standard deviation for most of the investigated products.
● Low levels of benzene artefacts can be generated on clean Tenax TA air sampling tubes during air sampling.
– Any low-level benzene test results should be verified with an independent second testing method before comparing a test result with any low limit value of e.g. 1 µg/m³.

3.2.3 Repeatability within one testing laboratory

Some robustness tests were repeated within the same testing laboratory two or three times under the same conditions. These data were analysed for repeatability within one testing laboratory. Repeatability of VOC emissions testing within one testing laboratory showed to be influenced strongly by:
● Inhomogeneity of the tested product.
● Chemical characteristics of the identified VOCs; bad response in the detector device (MS) gives low signal and poor reproducibility.
● Height of emissions when testing; both small traces of VOC emissions and very high emissions are difficult to analyze.
● Emissions mechanisms of the tested product and emissions decay over time.
The deviation of individual emission chamber test results from their mean value was calculated as percent deviation from their mean value, and a frequency distribution was calculated. Repeatability within the same testing laboratory resulted as follows [9]:
1. 50% of all test data showed a deviation of individual test results from their mean value below 13% (the median of all findings).
2. 75% of all test data showed a deviation of individual test results from their mean value below 26% (75 percentile of all findings).
3. 95% of all test data showed a deviation of individual test results from their mean value below 54% (95 percentile of all findings).
4. Standard deviation (1 σ) of all test results was 18%.
5. The expanded uncertainty (2 σ) of all test results, representing the 95% confidence interval, was 35%.
As described above, these are average figures. The height of the emissions, the chemical characteristics of the emitted VOCs, and the inhomogeneity of the tested products can cause both better and worse performance in specific cases.

3.2.3 Reproducibility between different testing laboratories

Existing data of several round robin tests were analysed [10] that had been performed in the recent years. These test data were differentiated such that in one approach only test results were evaluated that had been obtained exactly under the conditions as specified in CEN/TS 16516, and in another approach all test results were evaluated – including those where the testing conditions were slightly different from CEN/TS 16516.
The relative standard deviations of reproducibility between different laboratories testing the same product were between 15 and 79%, where the high deviations occurred only with difficult to analyse VOCs (acetic acid, ethanediol). There were no significant differences whether all values were included in the evaluation, or only those obtained exactly under the testing conditions of CEN/TS 16516.

4 Conclusions

It is intended by the European Commission that CEN/TS 16516 will be referenced in updated harmonized product performance standards (hEN) that are used for CE marking. Furthermore, it is expected that the new testing standard will become the key benchmark also for voluntary low VOC emissions specifications, such as ecolabels and programs for sustainable buildings, at least across Europe.
CEN/TS 16516 includes refinements of existing testing standards for improving reliability. It is expected that this leads to significantly better repeatability and reproducibility of test results, compared to ISO 16000 testing. The key testing

parameters were confirmed by a robustness validation study. Data on repeatability within a testing laboratory and reproducibility between different testing laboratories were evaluated.

CEN/TC 351/WG 2 started the process of transforming the CEN/TS 16516 into a European Standard until 2016, based on the validation studies.

CEN/TS 16516 will allow using one test for all European countries and for several purposes in one single test set-up, even including tests for US low VOC specifications. This can reduce the testing costs for manufacturers and thus contribute to improved competitive strength of European industry.

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