BS EN 16105:2011

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Paints and varnishes — Laboratory method for determination of release of substances from coatings in intermittent contact with water

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The UK participation in its preparation was entrusted to Technical Committee STI/10, Test methods for paints.

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

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Paints and varnishes - Laboratory method for determination of release of substances from coatings in intermittent contact with water

Peintures et vernis - Méthode de laboratoire pour la détermination de la libération de substances provenant de revêtements en contact avec l'eau par intermittence

Beschichtungsstoffe - Laborverfahren zur Bestimmung der Freisetzung von Substanzen aus Beschichtungen in intermittierenden Kontakt mit Wasser

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Foreword

This document (EN 16105:2011) has been prepared by Technical Committee CEN/TC 139 “Paints and varnishes”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2012, and conflicting national standards shall be withdrawn at the latest by March 2012.

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Introduction

Leaching of substances from coatings into water needs to be quantified to enable an environmental risk assessment for the use of substances in coating materials. Substances can be leached from coatings, particularly by driving rain, and transferred into the environment.

1 Scope

This European Standard specifies a laboratory method to determine the leaching behaviour of substances from coatings into water over defined time intervals.

The release of substances from coatings under natural conditions cannot be determined with this method.

2 Normative references

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

EN 23270, *Paints and varnishes and their raw materials — Temperatures and humidities for conditioning and testing (ISO 3270:1984)*

EN ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling (ISO 15528:2000)*

3 Terms and definitions

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

3.1
substance
single chemical element or compound, or a complex structure of compounds, that is contained in the coating and can potentially be extracted from the coating via water contact

3.2
target substance
substances to be tested according to 6.3.2

NOTE One or more target substances may be defined. For example, biocides can be the target substances (see Annex B).

3.3
biocide
additive added to a coating material to prevent organisms responsible for microbiological degradation from attacking a coating material or a film thereof

[EN ISO 4618:2006]

NOTE A list of biocides is given in Annex I and IA of the Biocidal Products Directive 98/8/EC (Directive 98/8/EC of the European Parliament and of the Council of 16 February 1998 concerning the placing of biocidal products on the market – BPD).

3.4
coating
continuous layer formed from a single or multiple application of a coating material to a substrate

[EN ISO 4618:2006]

3.5
coating material
product, in liquid, paste or powder form, that, when applied to a substrate, forms a film possessing protective, decorative and/or other specific properties

[EN ISO 4618:2006]

3.6
test specimen
body to be tested consisting of substrate with coating

3.7
emission
release of substances from a coating, which pass through the external surface of the coating under specific conditions into the environment

NOTE The emission is expressed in units of released mass per surface area, i.e. milligrams per square metre.

3.8
leaching
release of substances from a coating, which pass through the external surface of the coating under specific conditions into water

NOTE The leaching is expressed in units of released mass per surface area, i.e. milligrams per square metre.

3.9
immersion
exposure of test specimen to the leachant

3.10
immersion cycle
i
sequence consisting of 1 h immersion, 4 h drying and 1 h immersion

NOTE i is the running number of immersion cycles.

3.11
eluate
solution obtained by one immersion

3.12
merged eluate
solution obtained at a specific immersion cycle, consisting of the eluates of the two immersions

3.13
specific emission
E
released mass of a target substance from a coating through the surface during a specific immersion cycle

NOTE The unit is mass per surface area, i.e. milligrams per square metre.

3.14
leachant
liquid that is brought into contact with the test specimen in the leaching procedure

NOTE Standard leachant as specified in 4.1.

4 Reagents

4.1 Standard leachant.

Deionised water with a pH-value of (6 ± 1) and a water temperature of (23 ± 2) °C shall be used.

5 Apparatus

5.1 General

Check the materials and equipment specified in 5.2.1 to 5.2.6 before use for proper operation and absence of interfering elements that might affect the results of the test.

The equipment specified in 5.2.2 to 5.2.5 shall also be calibrated.

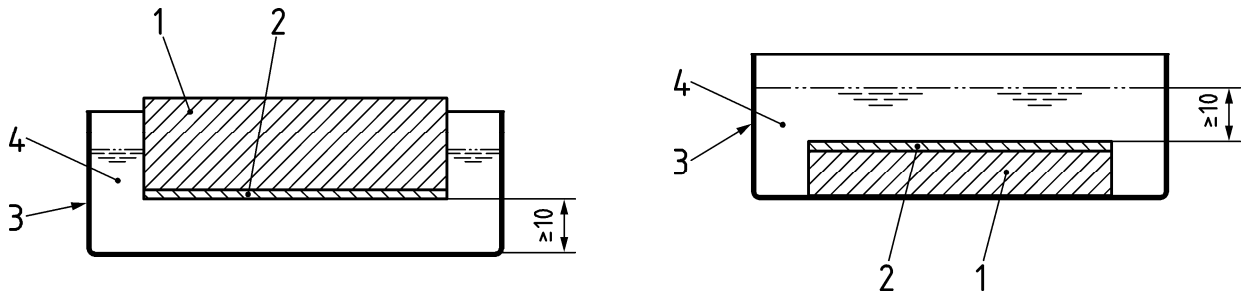
5.2 Equipment

5.2.1 Immersion container

The container for immersion shall be made of a material inert to the target substances in the eluates (e.g. glass, PTFE coated). The immersion container shall be large enough to allow the test specimens to have the coated face completely exposed to water and contain 25 l water per square metre exposed face. The water column below (see Figure 1 a)) or above (see Figure 1 b)) the test surface shall be ≥ 10 mm.

NOTE For example, a test specimen of 100 cm^2 requires 250 ml water.

Dimensions in millimetres



a) water column below **b) water column above**

Key
1 substrate
2 coating
3 immersion container
4 leachant

Figure 1 — Possible orientations of the test specimen in the immersion container during the immersion process

5.2.2 Analytical balance

Analytical balance, with an accuracy of ± 0,1 g.

5.2.3 Device for measuring

Device for measuring sample dimensions, with an accuracy of ± 1 mm.

5.2.4 Measuring cylinders for volume determination

Measuring cylinders for volume determination, with 1 % accuracy.

5.2.5 pH meter

pH meter, with an accuracy of ± 0,05.

5.2.6 Glass or plastic bottles

Glass or plastic bottles, e.g. glass, HDPE, PMMA, PTFE, PE, PET, PP, PVC.

Use bottles with an appropriate volume, and with screw cap, for eluate collection and preservation of merged eluates.

6 Test procedure

6.1 General considerations

Coatings are exposed under natural weather conditions to intermittent cycles of wetting (rainfall, thaw, condensate) and drying. To assess the leaching of substances from coatings, a wetting and drying method is described in this European Standard. For this purpose, samples with coatings are immersed into water and dried in time intervals.

The water from each immersion cycle i is analysed to determine the concentration c_i of leached target substances.

6.2 Sample preparation

6.2.1 Substrate

The substrate used to carry the coating shall have a homogeneous planar surface, shall be inert and coatings shall adhere well to it during the immersion in water. As the substrate can influence the results, it shall be stated in the test report.

NOTE Examples of inert substrates are XPS and EPS. Other substrates might influence the leaching (e.g. concrete, mineral renders and fibre cement board and wood substrates).

6.2.2 Sampling

Take a representative sample of the coating material, as described in EN ISO 15528.

The sampling and conditioning procedure should be recorded in a sampling report

6.2.3 Number and size of test specimens

The tests shall be performed with at least two replicate test specimens containing target substances and with one control test specimen with the same coating, but without target substances. If the coating without target substances is not available only the substrate can be used as control test specimen.

The test with the control test specimen may be stopped if no background signal has been determined after the second immersion cycle.

Test specimens shall have a minimum surface area of 100 cm^2. Each test specimen shall be marked to identify it throughout the test.

6.2.4 Preparation of the test specimens

The substrate shall be clean and dry.

The substrate shall be coated with the coating material in the specified quantity in accordance with the recommendations of the manufacturer. Determine and record the total mass applied.

Absorptive substrates shall be sealed on the reverse side and the edges against penetration of water. For example, two coats of a two-component coating material based on epoxy resin, overlapping the test surface by ± 5 mm but not more than 10 mm may be applied. A thixotropic coating system for sealing is recommended.

6.2.5 Conditioning

Condition the test specimens at minimum for 7 days with freely circulating air at (23 ± 2) °C and (50 ± 5) % relative humidity according to EN 23270 without UV influence or according to the requirements of the manufacturer.

6.3 Immersion method

6.3.1 Test procedure

Perform nine immersion cycles i, e.g. on day 1, 3, 5, 8, 10, 12, 15, 17 and 19.

Each immersion cycle i consists of:

— 1 h immersion,

— 4 h drying at (23 ± 2) °C and (50 ± 5) % relative humidity and

— 1 h immersion.

Carry out the immersion at a water temperature of (23 ± 2) °C.

The immersion container shall be washed and dried prior to the first test.

The required volume of water (e.g. 250 ml per 100 cm^2) shall be added. Fresh deionised water shall be used for each immersion. Determine and record the pH-value.

Determine and record the mass of each specimen before each immersion cycle.

The test specimens shall be positioned in the containers (each test specimen in a separate container) in a way that free contact of water to the coated surface is allowed.

The immersion container shall be covered to avoid evaporation of water during the immersion.

Test specimens shall be removed from the immersion containers for drying.

Merge the eluates of the two immersions of one immersion cycle i for the analysis of the target substances.

Determine and record the pH-values of the merged eluates.

If the analysis cannot be achieved on the same day, store the merged eluates in a refrigerator (+4 °C) in the dark to reduce microbial activity, but not longer than 7 days. Alternatively, the merged eluates may be stored in a freezer at ≤ -10 °C, but not longer than 30 days.

NOTE The analysis of the merged eluates should be done depending on the stability of the individual substance and depending on the storage conditions which can be applied.

Store the test specimens between the immersion cycles for a minimum of 42 h according to 6.2.5.

6.3.2 Analysis

The target substances in the merged eluates shall be analysed separately for each replicate with an appropriate method that is validated for this purpose. It might be necessary to concentrate the target substances in the merged eluates depending on the concentration and the sensitivity of the analytical method.

7 Results

7.1 Results in concentrations

Express the analytical results as concentration c of each target substance analysed in the merged eluate for each immersion cycle in milligram per litre.

7.2 Results in terms of specific emissions

If the analysis of the merged eluates of the control samples shows detectable levels of the target substances, implying a background signal in the water, it shall be subtracted from the analytical results for the test specimens with target substances. Convert the analytical results c_i expressed in milligram per litre to specific emissions E_i in milligram per square metre for each immersion cycle i with Equation (1):

$$E_i = \frac{c_i \times V_i}{A} \quad (1)$$

where

i is the running number of immersion cycles;

E_i is the specific emission at the immersion cycle i, in milligram per square metre;

A is the surface of the test specimen, in square metre;

c_i is the measured concentration of the target substance in the merged eluate at the immersion cycle i, in milligram per litre;

V_i is the real volume of the merged eluate after the immersion cycle i, in litre.

Express the result in E_i as a mean value of the two replicate measurements for each immersion cycle i. Plot the specific emissions E_i against immersion cycles.

NOTE Table A.1 shows an example of a recording form for one set of test specimens. Table A.2 summarises the mean specific emissions for all immersion cycles. An example graph is given in Annex B.

Further calculations might have to be done.

8 Test report

The test report shall contain at least the following information:

a) reference to this European Standard, i.e. EN 16105;

b) all details necessary to identify the coating material tested (e.g. binder type determined by IR-spectroscopy, binder content, liquid water absorption, ash content);

c) specific and unique name or code of the target substances determined;

d) number of test specimens;

e) method of application of coating material, applied mass, number of coats, duration and conditions of drying;

f) substrate and dimensions of the test specimens;

g) amount of target substances in the coating of the test specimens in milligrams per square metre;

h) total surface area of the test specimens exposed to water in square metres;

i) time and date of each immersion and the nominal volume of water used for each immersion in litre;

j) concentration of the target substance c_i for each immersion cycle i;

k) specific emission E_i for each immersion cycle i;

l) plot of specific emissions E_i versus immersion cycles i;

m) any variation from the method described in this standard and any factors that might have influenced the results;

n) quality (e.g. deionised or distilled) and pH-value of the merged eluates;

o) particular observations;

p) analytical methods used to determine target substances in the merged eluates.

Annex A
(informative)

Forms for recording

Table A.1 — Form for recording of masses of test specimens and pH-values

Cycle	Immersions		Test specimen mass before immersion cycle			pH-values of			
i	Date	Time	Test specimen 1 g	Test specimen 2 g	Control test specimen g	Standard leachant pH	Merged eluate of test specimen 1 pH	Merged eluate of test specimen 2 pH	Merged eluate of control test specimen pH
1									
2									
3									
4									
5									
6									
7									
8									
9									

Table A.2 — Analytical results

Cycle	Immersions		Control test specimens		Test specimens					
i	Date	Time	c_i	E_i	c_i			E_i		
					Test specimen 1	Test specimen 2	Mean	Test specimen 1	Test specimen 2	Mean
			mg/l	mg/m^2	mg/l	mg/l	mg/l	mg/m^2	mg/m^2	mg/m^2
1										
2										
3										
4										
5										
6										
7										
8										
9										

A separate table should be prepared for each target substance that has been analysed.

Annex B
(informative)

Examples of data: Leaching of biocides

B.1 General

The feasibility of the test procedure described in this European Standard has been evaluated by a round robin test for a paint and a render frame formulation for six target substances each. Details of the test parameters, the analytical method and the results are outlined in the following paragraphs as an example.

B.2 Sample preparation

Frame formulations of a paint and a render have been synthesised. Part of the material was used to produce control test specimens without biocides. The rest of the material was doped with six biocides, namely Diuron, (CAS 330-54-1), Terbutryn (CAS 886-50-0), 2-octyl-4-isothiazolin-3-one (OIT, CAS 2653-20-1), 4,5-dichloro-2-n-octyl-4-isothiazolin-3-one (DCOIT, CAS 64359-81-5), Carbendazim (CAS 10605-21-7) and 3-iodoprop-2-ynyl N-butylcarbamate (IPBC, CAS 85045-09-6).

Test specimens were produced for each material by coating XPS-panels of 40 mm thickness and 1 m × 0,5 m size with paint or render according to the technical specifications. Subsequently, the coated XPS-panels were cut into test specimens of 100 mm × 100 mm size (100 cm^2 surface area) using a dry cutting process.

B.3 Test procedure

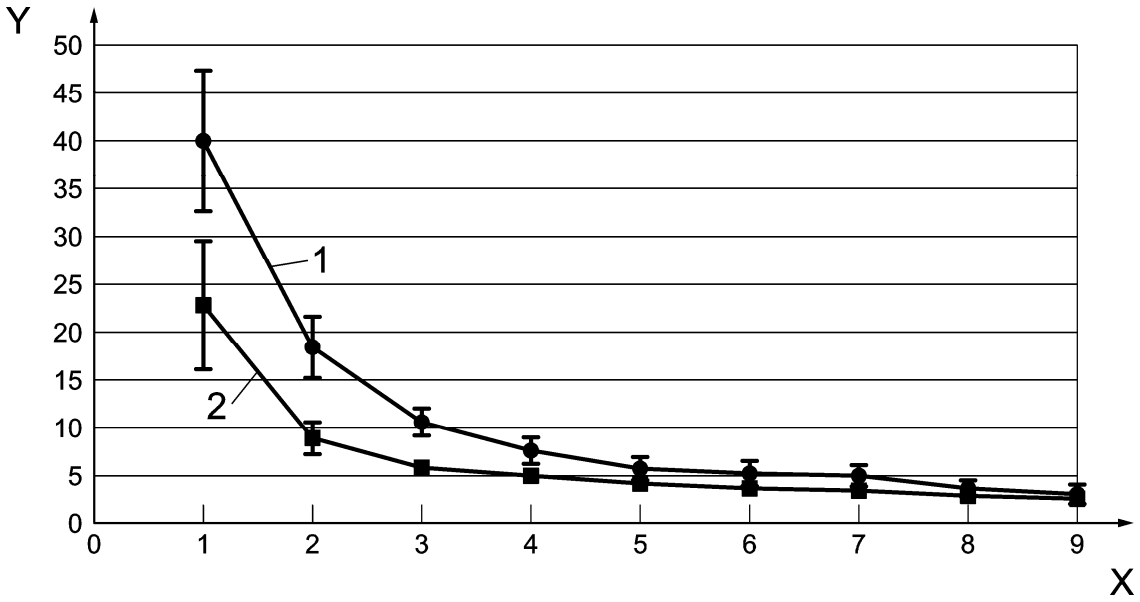
The test specimens were sent to eight different laboratories and the test procedure was performed according to this European Standard.

B.4 Analytical Methods

High pressure liquid chromatography coupled with diode array detection (DAD) or mass spectrometry was used to quantify the amount of biocides in the eluates. Some laboratories used solid phase extraction (SPE) to enhance the concentration of the biocides in solution.

B.5 Test results

The results were listed and calculated according to 7.1 and 7.2. According to 7.2, the specific emission (E_i) was plotted against immersion cycles i. Figure B.1 shows an example plot for Diuron emitted from paint and render. The standard deviation obtained in the round robin test with eight laboratories is indicated as error bars in the graph.



Key

1 plot for a render
2 plot for a paint
X immersion cycle
Y specific emission, in milligram per square metre

NOTE Error bars represent the standard deviation obtained in the round robin test.

Figure B.1 — Example plots for the specific emission of a target substance (here Diuron) vs. immersion cycle

B.6 Assessment of the uncertainty

a) General

There are given examples of the variability of the product and the overall standard deviation received in the round robin test.

b) Variability of the product

The coating thickness was not homogenous, but shows a certain variability. This leads to a difference in the applied mass of the coating material on the test specimens of 100 cm^2 size, which is reflected in a difference in the applied amount of biocides in milligrams per square metre. The mass distribution of the test specimens used exhibited a standard deviation of 7 % for paint and 11 % for render.

c) Overall uncertainty

The standard deviation of the interlaboratory test results varies for the different target substances. The relative standard variation between the laboratories is the lowest for Terbutryn and Diuron (7 % to 12 %), followed by IPBC, OIT and Carbendazim (22 % to 36 %) and is highest for DCOIT (70 % to 85 %)[1)] in the tests for paint and render. The standard deviation can be used as a measure of the overall uncertainty under the assumption that only statistical errors and non-systematic errors are relevant.

1) The amount of DCOIT was close to the detection limit.

Bibliography

[1] EN 998-1, *Specification for mortar for masonry — Part 1: Rendering and plastering mortar*

[2] EN 1062-1:2004, *Paints and varnishes — Coating materials and coating systems for exterior masonry and concrete — Part 1: Classification*

[3] EN 15824, *Specifications for external renders and internal plasters based on organic binders*

[4] EN ISO 4618:2006, *Paints and varnishes — Terms and definitions (ISO 4618:2006)*

[5] EN ISO 10993-9:2009, *Biological evaluation of medical devices — Part 9: Framework for identification and quantification of potential degradation products (ISO 10993-9:2009-12)*

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