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Construction products: Assessment of release of dangerous substances — Determination of biocide residues using liquid chromatography with mass spectrometric detection (LC-MS/MS)

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The UK participation in its preparation was entrusted to Technical Committee B/557, Construction products - Assessment of dangerous substances.

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

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Construction products: Assessment of release of dangerous substances - Determination of biocide residues using liquid chromatography with mass spectrometric detection (LC-MS/MS)

Produits de construction : Évaluation de l'émission de substances dangereuses - Détermination de la teneur en biocides par chromatographie en phase liquide et détection par spectrométrie de masse en tandem (LC-MS/MS)

Bauprodukte: Bewertung der Freisetzung von gefährlichen Stoffen - Bestimmung von Biozid-Rückständen mittels LC-MS/MS

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European foreword

This document (EN 17845:2023) 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.

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of an identical text or by endorsement, at the latest by May 2024, and conflicting national standards shall be withdrawn at the latest by May 2024.

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Introduction

This document deals with the determination of the content of biocides in construction products and eluates using liquid chromatography and tandem mass spectrometric detection (LC-MS/MS).

Following an extended evaluation of available methods for content and eluate analysis in construction products (CEN/TR 16045) and subsequent method evaluation in the robustness validation [Van De Weghe et al., 2018] it was concluded that eluate analysis and content analysis for biocides can be based on EN 15637 after some modifications.

This document is part of a modular horizontal approach and belongs to the analytical step. An overview of all modules which belong to a chain of measurement and the manner how modules are selected is given in CEN/TR 16220.

In the growing amount of product and sector-oriented test methods it was recognized that many steps in test procedures are or could be used in test procedures for many products, materials and sectors. It was supposed that, by careful determination of these steps and selection of specific questions within these steps, elements of the test procedure could be described in a way that can be used for all materials and products or for all materials and products with certain specifications.

In this context a horizontal modular approach is adopted in CEN/TC 351. “Horizontal” means that the methods can be used for a wide range of materials and products with certain properties. “Modular” means that a test standard developed in this approach concerns a specific step in assessing a property and not the whole “chain of measurement” (from sampling to analyses). A beneficial feature of this approach is that “modules” can be replaced by better ones without jeopardizing the standard “chain”.

The use of modular horizontal standards implies the drawing of test schemes as well. Before executing a test on a certain material or product to determine certain characteristics, it is necessary to draw up a protocol in which the adequate modules are selected and together form the basis for the entire test procedure.

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1 Scope

This document describes a method for the determination of the content of biocides in construction products, (either finished (dried) or in a ready-to-use state) and in eluates thereof, using liquid chromatography and tandem mass spectrometric detection (LC-MS/MS).

For content analysis liquid chromatography with UV-detection can also be used, if sufficient sensitivity and selectivity is ensured (see Annex A (normative)).

The method in this document is validated for the product types listed in Annex D (informative). For eluate analysis quantification limits of 0,1 µg/l can be achieved.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 16637-2, *Construction products: Assessment of release of dangerous substances — Part 2: Horizontal dynamic surface leaching test*

EN 16637-3, *Construction products: Assessment of release of dangerous substances — Part 3: Horizontal up-flow percolation test*

EN 16687:2023, *Construction products: Assessment of release of dangerous substances — Terminology*

EN 17087, *Construction products: Assessment of release of dangerous substances — Preparation of test portions from the laboratory sample for testing of release and analysis of content*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 16687:2023 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
blank value
test result obtained by carrying out the test procedure in the absence of a test portion

[SOURCE: EN 16687:2023, 3.3.1.10; modified – Note 1 to entry removed]

3.2
clean-up
purification of a crude extract to remove interfering compounds

[SOURCE: EN 16687:2023, 3.2.2.27; modified – Note 1 to entry removed]

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3.3
external standard
known quantity of the target analytes that is measured in the same series as the solution to be measured and is used for identification and quantification

[SOURCE: EN 16687:2023, 3.2.2.20]

3.4
extract
solution resulting from extraction of a sample with a solvent

[SOURCE: EN 16687:2023, 3.2.2.13]

3.5
extraction
dissolution of substances in a solvent for subsequent chemical analysis

[SOURCE: EN 16687:2023, 3.2.2.14; modified – Note 1 to entry removed]

3.6
final extract
solution that is obtained after clean-up of the Soxhlet extract through a purification stage

[SOURCE: EN 16687:2023, 3.2.2.18]

3.7
internal standard
known quantity of a substance (where applicable deuterated) not present in the sample, which is added to the analysis sample in order to determine the recovery

[SOURCE: EN 16687:2023, 3.3.2.10]

3.8
laboratory sample
sample or subsample(s) sent to or received by the laboratory

[SOURCE: EN 16687:2023, 3.2.2.1; modified – Notes to entry removed]

3.9
method detection limit
MDL
lowest analyte concentration that can be detected with a specified analytical method including sample preparation with a defined statistical probability

[SOURCE: EN 16687:2023, 3.3.1.12; modified – Note 1 to entry removed]

3.10
product matrix
main composition of the product dictating the manner of sample preparation and the type of digestion or extraction for later chemical analysis

[SOURCE: EN 16687:2023, 3.1.1.2; modified – Note 1 to entry removed]

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3.11
sample
portion of material selected from a larger quantity of material

[SOURCE: EN 16687:2023, 3.2.1.5; modified – Notes to entry removed]

3.12
Soxhlet extract
solution that is obtained after extraction of a solid subsample by the Soxhlet technique for determining organic compounds

[SOURCE: EN 16687:2023, 3.2.2.17]

3.13
Soxhlet extraction
chemical pre-treatment of a solid subsample, where the organic compounds to be determined are dissolved by the Soxhlet technique

[SOURCE: EN 16687:2023, 3.2.2.16]

3.14
test portion
analytical portion
amount of the test sample taken for testing/analysis purposes, usually of known dimension, mass or volume

[SOURCE: EN 16687:2023, 3.2.2.3; modified – Examples removed]

3.15
test sample
analytical sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis

[SOURCE: EN 16687:2023, 3.2.2.2]

4 Abbreviations

For the purposes of this document, the following abbreviations apply.

a.u.	Arbitrary units
DTL	Detection limit
HPLC	High performance liquid chromatography NOTE High pressure liquid chromatography is an (outdated) synonym.
ISTD	Internal standard solution
LC	Liquid chromatography
LOD	Limit of detection
MQL	Method quantification limit
MRL	Maximum residue limit

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MRM	Multiple reaction monitoring
MS	Mass spectrometry; Mass selective detection
PTFE	Polytetrafluoroethylene
SPE	Solid phase extraction

5 Sample preparation

To obtain test samples for extraction (and analysis) guidance on sample preparation as specified in EN 17087 shall be applied.

Precautions shall be taken before and during transport of the laboratory sample as well as during the time in which the samples are preserved in the laboratory before being analysed, to avoid alteration of the sample (see CEN/TR 16220).

Extracts are susceptible to change due to physical or chemical reactions which can take place between the time of extraction and the analysis.

It is therefore essential to take the necessary precautions to minimize these reactions and in the case of many parameters to analyse the extract with a minimum of delay. The maximum delay is given in the respective analytical standards.

6 Principle

Quantification of biocides is performed by liquid chromatography with tandem mass spectrometric detection (LC-MS/MS), using electrospray ionization. Eluates are injected directly into the LC-MS/MS system.

Solid and pasty samples are extracted with methanol or a methanol/water mixture. Clean-up may be applied when necessary by solid phase supported liquid-liquid extraction.

To achieve the required selectivity the mass spectrometer is operated in the MRM mode.

7 Reagents

Use reagents of recognized analytical grade, unless otherwise specified. Take every precaution to avoid possible contamination of water, solvents, inorganic salts, etc.

7.1 Additive for LC-eluent depending on method used, e.g. ammonium formate, formic acid, acetic acid.

7.2 Water, HPLC quality.

7.3 Dichloromethane, for biocide analysis.

7.4 Methanol, LC-MS/MS quality.

7.5 Internal standard (ISTD) solutions in methanol, mass concentration ρ = 10 µg/ml to 50 µg/ml[1].

Use as much as possible isotope labelled internal standards, if available and obtainable at reasonable price. The internal standard solution should be added at the extraction step and to standard solutions.

If no isotope labelled internal standards are used, it is recommended to check whether matrix compounds are co-eluting with the ISTD to ensure reliable results. Losses of the ISTD during clean-up will result in an overestimation of analyte concentration. Such losses should thus be minimal.

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