SLOVENSKI STANDARD
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Trajnost lesa in lesnih proizvodov – Kvantitativno določanje pentaklorofenola v lesu – Plinska kromatografska metoda

Durability of wood and wood-based products - Quantitative determination of pentachlorophenol in wood - Gas chromatographic method

Dauerhaftigkeit von Holz und Holzprodukten - Quantitative Bestimmung von Pentachlorphenol in Holz - Gaschromatographische Verfahren

Durabilité du bois et des matériaux dérivés du bois - Analyse quantitative du pentachlorophénol dans le bois - Méthode par chromatographie en phase gazeuse

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Durability of wood and wood-based products – Quantitative determination of pentachlorophenol in wood – Gas chromatographic method

Durabilité du bois et des matériaux dérivés du bois – Analyse quantitative du pentachlorophénol dans le bois – Méthode par chromatographie en phase gazeuse

Dauerhaftigkeit von Holz und Holzprodukten – Quantitative Bestimmung von Pentachlorphenol in Holz – Gaschromatographische Verfahren

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (CEN/TR 14823:2003) has been prepared by Technical Committee CEN/TC 38 “Durability of wood and wood-based products”, the secretariat of which is held by AFNOR.

This status of this document as Technical Report has been chosen because this method described is an example of a laboratory method validated by a ring test. It is recognized that, for example, different techniques of extraction might be employed by the analyst without loss of analytical performance and quality. However, when using different techniques the comparability to the method described in this Technical Report should be demonstrated, e.g. by using a certified reference material (CRM).

Introduction

At present, no standardised method for the analysis of pentachlorophenol (PCP) in wood is recognized in Europe. Only a few national standards are available world wide, e g BS 5666, Part 6 and AWPA Standard A-5 and these methods are designed for the analysis of timber treated with levels of PCP that are necessary to prevent degradation of the treated timber by fungi.

This Technical Report has been issued in order to facilitate the analysis of PCP-treated wood and panels and in particular low levels of PCP that can be present in packaging timbers and pallets. Low levels of PCP and other chlorophenols, and the anisoles derived from them, can cause taints in foodstuffs. PCP can be present as residues from old anti-sapstain treatment or some timber treated against decay could inadvertently have been incorporated into the panel product.

1 Scope

This Technical Report specifies a laboratory method of determining the pentachlorophenol content of wood. The method is applicable to all types of PCP- treated wood and wood-based materials as well as for the analysis of waste timber with respect to its PCP content.

The method has a quantification limit corresponding to 100 µg PCP per kilogram of wood material expressed as dry matter. The method described has a measurement range up to PCP contents of 25 mg/kg of dry matter. These figures refer to the given example (where an aliquot of 1 ml of the extract is used for acetylation, see 8.4).

NOTE 1 If lower quantification limits are required, a higher volume of extract aliquot can be used for derivatisation.

NOTE 2 This method could have some modifications with some wood species as hardwoods.

2 References

EN 212, *Wood preservatives - General guidance on sampling and preparation for analysis of wood preservatives and treated timber.*

EN 322, *Wood-based panels – Determination of moisture content.*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987).*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ISO 11465, *Soil quality - Determination of dry matter and water content on a mass basis - Gravimetric method.*

3 Safety precautions

Persons using this method should be familiar with normal analytical laboratory procedures and practice.

This method does not purport to address all the safety problems, if any, associated with its use.

It is the responsibility of the user to establish safety and health practices and to ensure compliance with any European or national regulatory conditions.

4 Principle

Pentachlorophenol is extracted from the wood material using methanol. The extracted pentachlorophenol is transformed to pentachlorophenol acetate by derivatisation of an aliquot of the extract in aqueous potassium carbonate solution with acetic anhydride. The acetate derivative formed is extracted from this aqueous solution with n-hexane and analysed by gas chromatography with electron capture detection.

5 Reagents

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade which have been checked in advance as to not interfere with the analytical results, and water complying with grade 3 as defined in EN ISO 3696.

5.1 Acetic anhydride ($C_4H_6O_3$).

5.2 Methanol (CH_3OH)**.**

NOTE Other solvents can be used instead of methanol, some extraction difficulties can occur with some wood species (e.g. hardwoods). It is recommended to cross check the extraction efficiency of any other solvent or solvent mixture with that of methanol.

5.3 n-Hexane (C_6H_{14}).

5.4 Potassium carbonate, solution, c($K CO_3$) = 0,1 mol/l.

5.5 Sodium sulfate, anhydrous (Na_2SO_4).

5.6 Pentachlorophenol (PCP, (C_6Cl_5OH)) of certified purity (> 99%), e.g. IPO 560[1)].

5.7 2,4,6-tribromophenol (TBP, ($C_6H_2Br_3OH$)) of certified purity (> 98%) as an internal standard.

5.8 Purified sand, (ignited at 900 °C after acid treatment)

NOTE An untreated wood sample (of the same wood species as the sample for analysis) known to be free of pentachlorophenol can also be used instead of purified sand but before use it should be checked for interference with the analytical method.

6 Apparatus

Ordinary laboratory apparatus and the following.

NOTE Glassware should be thoroughly cleaned prior to use.

6.1 Analytical balance, accurate to 0,01 mg.

6.2 Ultra-sonic bath equipped with a thermostat capable of controlling and maintaining a temperature of 40 °C.

6.3 Disposable Pasteur pipettes made of glass, 2,0 ml capacity.

6.4 Separating funnel, 150 ml capacity.

6.5 Volumetric pipettes, 1,0 ml and 10 ml or 20 ml capacity.

6.6 Microlitre syringes, 25 µl, 100 µl and 500 µl capacity.

6.7 Volumetric flasks, 10 ml, 25 ml and 50 ml capacity.

6.8 Conical flasks 100 ml capacity, with screw caps provided with an insert of polytetrafluoroethylene (PTFE) or alternatively with standard ground stoppers provided with PTFE standard ground sockets and conical joint clips.

6.9 Gas chromatograph equipped with a splitless/split or a non-discriminating injection system and an electron capture detector (ECD)

6.10 Separation column. One capillary column, or preferably two, with stationary phase of different polarity e.g. DB 1, DB 5, DB 17[2)]; length: 25 m to 30 m; internal diameter: 0,2 mm to 0,32 mm; film thickness: 0,25 µm to 0,33 µm.

1) IPO 560 is an example of a suitable product available commercially, supplied by Promochem. This information is given for the convenience of users of this CEN Technical Report and does not constitute and endorsement by CEN of this product.

6.11 Pre-column coated with deactivated silica.

7 Preparation of the test sample

Collect at least 10 g of the sample material taken according to e.g. EN 212. This sample material is preferably ground under mild conditions (e.g. cryogenic grinding) to chips with an approximate particle size of 0,5 mm to 1,0 mm diameter.

NOTE 1 Wood shavings can also be made for analysis (especially if other compounds e.g. trichlorophenols or trichloroanisols have to be analysed at the same time).

Homogenise the ground sample material to obtain a representative sample, and store it in a brown glass vessel with screw caps with a PTFE insert. This is the test sample.

NOTE 2 If possible, the sample for analysis should be prepared from a minimum of five independent pieces or sections representatively taken from the quantity of wood material to be analysed.

8 Procedure

8.1 General

It is recommended to carry out at least two parallel analyses. If results differ by more than 10 % an additional analysis shall be carried out.

8.2 Standard solutions

8.2.1 Preparation of stock solutions (calibrant and internal standard)

Prepare the stock solutions by weighing about 12,5 mg of PCP (5.6) and TBP (5.7) separately to the nearest of 0,01 mg and dissolve each in 25 ml methanol (5.2).

NOTE These stock solutions with nominal concentrations of 0,5 mg/ml can be stored in the dark at –18 °C for three months.

8.2.2 Preparation of working solutions

Transfer by pipette (6.5) 1,00 ml of stock PCP solution (8.2.1) to a 10 ml one-mark volumetric flask and make up to the mark with methanol (5.2) to give a solution having a nominal concentration of 50 µg/ml PCP. Repeat with the TBP stock solution to give a solution having a nominal concentration of 50 µg/ml TBP.

8.2.3 Preparation of calibration standards

To a series of five conical flasks (6.8) containing (5,0 ± 0,1) g of purified sand (5.8), transfer 25 µl, 50 µl, 100 µl, 250 µl and 500 µl of PCP working solution (8.2.2). To each of the five flasks and another flask containing sand but no added PCP, add 250 µl of TBP working solution (8.2.2) to give a series of standards containing 0,0 µg, 1,25 µg, 2,5 µg, 5 µg, 12,5 µg and 25 µg of PCP with 12,5 µg TBP as internal standard in each.

NOTE This procedure assumes that 1 ml of the methanol extract will be taken for preparation of the calibration solutions. If a greater volume of extract is to be taken, for example 5 ml, the above standards should be prepared from working solutions each with a nominal concentration of 10 µg/ml .

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2) DB1, DB5, DB17 are examples of a suitable product available commercially, supplied by J & W. This information is given for the convenience of users of this CEN Technical Report and does not constitute an endorsement by CEN of these products.