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Wood preservatives – Creosote and creosoted timber – Methods of sampling and analysis – Part 3: Determination of the benzo(a)pyrene content of creosote

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Europastandarden EN 1014-3:2010 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN 1014-3:2010.

Denna standard ersätter SS-EN 1014-3, utgåva 1.

The European Standard EN 1014-3:2010 has the status of a Swedish Standard. This document contains the official English version of EN 1014-3:2010.

This standard supersedes the Swedish Standard SS-EN 1014-3, edition 1.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

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Denna standard är framtagen av kommittén för Beständighet, SIS/TK 182/AG 3.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 1014-3

June 2010

ICS 71.100.50

Supersedes EN 1014-3:1997

English Version

Wood preservatives - Creosote and creosoted timber - Methods of sampling and analysis - Part 3: Determination of the benzo(a)pyrene content of creosote

Produits de préservation du bois - Créosote et bois créosoté - Méthodes d'échantillonnage et d'analyse - Partie 3: Détermination de la teneur en benzo[a]pyrène dans la créosote

Holzschutzmittel - Kreosot (Teerimprägnieröl) und damit imprägniertes Holz - Probenahme und Analyse - Teil 3: Bestimmung des Gehaltes an Benzo(a)pyren im Kreosot

This European Standard was approved by CEN on 12 May 2010.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 1014-3:2010: E

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Foreword

This document (EN 1014-3:2010) 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 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 December 2010, and conflicting national standards shall be withdrawn at the latest by December 2010.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 1014-3:1997.

This standard forms part of a series of standards relating to the sampling and analysis of creosote and creosoted timber. The other standards of the series are:

EN 1014-1, *Wood preservatives — Creosote and creosoted timber — Methods of sampling and analysis — Part 1: Procedure for sampling creosote*

EN 1014-2, *Wood preservatives — Creosote and creosoted timber — Methods of sampling and analysis — Part 2: Procedure for obtaining a sample of creosote from creosoted timber for subsequent analysis*

EN 1014-4, *Wood preservatives — Creosote and creosoted timber — Methods of sampling and analysis — Part 4: Determination of the water-extractable phenols content of creosote*

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination of benzo(a)pyrene in creosote using high performance liquid chromatography (HPLC).

This standard is only applicable to creosotes containing more than 30 mg/kg benzo(a)pyrene.

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 ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Principle

The creosote sample is diluted to an appropriate concentration with an acetonitrile/water mixture. The diluted sample is analyzed using high performance liquid chromatography (HPLC) at constant temperature with a reverse phase packed column and isocratic elution. The result is compared with that from a known reference standard.

4 Reagents

4.1 Acetonitrile − water mixture. Add 220 ml of water (according to grade 1 of EN ISO 3696:1995) to 780 ml of acetonitrile (CH_3CN, HPLC grade) and mix thoroughly. The mixture shall be at room temperature before use.

4.2 Benzo(a)pyrene solution, containing approximately 0,1 mg/ml of benzo(a)pyrene. Weigh to the nearest 0,1 mg, approximately 0,01 g of benzo(a)pyrene with a purity of min. 98 % and transfer it quantitatively to a 100 ml one-mark volumetric flask. Add 50 ml of the acetonitrile (CH_3CN, HPLC grade). Dissolve the benzo(a)pyrene and make up to the mark with the acetonitrile-water mixture (4.1). Store the prepared solution in the stoppered brown glass storage flasks (5.6) in the dark, at a temperature below 10 °C.

CAUTION — Care should be taken to prevent benzo(a)pyrene contacting the skin.

NOTE 1 Under normal conditions, this solution is stable for six months, although frequent use may result in faster ageing.

NOTE 2 Commercially available certified standard solutions, including those containing additional components, may be used.

5 Apparatus

Usual laboratory apparatus and glassware together with the following:

5.1 Volumetric glassware, which shall have an accuracy of at least 0,5 %.

5.2 Single mark pipettes class A of 1 ml, 2 ml, 5 ml and 20 ml capacity.

5.3 High performance liquid chromatograph (HPLC) which shall consist of

— a solvent delivery pump with constant flow regulation;

— on-line degasser;

— 20 µl loop injector;

— reverse phase stainless steel column, 250 mm in length with an internal diameter of 4 mm, packed with C18 bonded silica stationary phase, having a particle size of 5 µm; the use of a column specially designed for polyaromatic hydrocarbons analysis is recommended;

— fluorescence detector capable of being set at (380 ± 3) nm excitation wavelength and (403 ± 5) nm emission wavelength;

— a potentiometric recorder.

NOTE As an alternative, any other HPLC configuration giving at least the same resolution (see Figures A.1 and A.2 or A.3) could be used.

5.4 Analytical balance, capable of weighing to 0,1 mg.

5.5 Ultrasonic bath capable of containing a 100 ml volumetric flask.

5.6 Brown glass storage flasks of 100 ml capacity, fitted with ground glass stoppers.

5.7 Glass or metal receptacles fitted with stoppers.

5.8 Glass syringe for HPLC, of 50 µl capacity.

6 Preparation of the calibration solutions and of the test samples

6.1 Preparation of the calibration solutions

Transfer by pipette (5.2) 1 ml of the benzo(a)pyrene solution (4.2) to a 100 ml one-mark volumetric flask and dilute to the mark with the acetonitrile-water mixture (4.1). To a series of 100 ml one-mark volumetric flasks, transfer by pipette (5.2) 1 ml, 2 ml, 5 ml and 20 ml of this solution and dilute to the mark with acetonitrile-water mixture (4.1). This provides calibration solutions containing 0,01 mg/l, 0,02 mg/l, 0,05 mg/l and 0,20 mg/l benzo(a)pyrene. Store the calibration solutions in stoppered brown glass storage flasks (5.6.) in the dark at a temperature below 10 °C.

NOTE Under normal conditions, the calibration solutions are stable for three months, although frequent use may result in faster ageing.

6.2 Preparation of the test samples

Prepare duplicate test samples.

The laboratory sample shall be in a closed receptacle (5.7). Heat the closed receptacle to 70 °C for (30 ± 2) min to ensure that the sample is completely liquid.

Weigh to the nearest 0,1 mg, 30,0 mg of the laboratory sample into a 100 ml one-mark volumetric flask.

NOTE Attention should be paid that no re-crystallization occurs during this transfer.

Record the masses taken as m_1 and m_2. Add approximately 80 ml of the acetonitrile-water mixture (4.1). Place the volumetric flask in the ultrasonic bath (5.5) for 5 min to dissolve the sample.

When the sample has dissolved completely, make up to the mark with the acetonitrile-water mixture (4.1).

7 Procedure

7.1 Set-up the HPLC (5.3) in accordance with the manufacturer's instructions.

Adjust the fluorescence detector to the following wavelengths:

— excitation: (380 ± 3) nm;

— emission: (403 ± 5) nm.

The fluorescence detector shall be fine-tuned to maximize the signal for benzo(a)pyrene.

Under isocratic conditions set the flow rate through the column to 1,2 ml/min using the acetonitrile-water mixture (4.1) as the eluent.

7.2 Analyze the test samples and the calibration solutions at the same temperature (± 0,5 °C).

At the same temperature (i.e. within ± 0,5 °C) and using the syringe (5.8) fill up the loop injector and inject successively the series of calibration solutions (6.1) and then the two test samples into the HPLC apparatus (5.3).

7.3 Repeat 7.2 in reverse order by successively injecting portions of the two test samples followed by the calibration solutions.

7.4 Measure the peaks heights for benzo(a)pyrene produced by the HPLC recorder for all test samples and calibration solutions.

8 Calculation

Calculate the benzo(a)pyrene content of the two samples, Bs_1 and Bs_2, expressed in milligrams benzo(a)pyrene per kilogram creosote, using the equation:

$$B_s = \frac{B_c \times H_s}{C_c \times H_c} \times 10^6$$

where

B_c is the concentration of the benzo(a)pyrene calibration solution nearest to the test sample in milligrams per litre (mg/l);

H_c is the mean of the duplicated benzo(a)pyrene peak heights obtained with the calibration solution in millimetres (mm);

C_c is the concentration of creosote in the test sample (6.2) in milligrams per litre (mg/l);

H_s is the peak height obtained for the test sample (6.2) in millimetres (mm).

9 Expression of results

Report the benzo(a)pyrene content B, of the laboratory sample as the average of B_{s1} and B_{s2} in milligrams per kilogram benzo(a)pyrene in creosote, rounded to the nearest 1 mg/kg (see Annex B).

10 Precision

10.1 Repeatability

Results obtained by the same operator shall be considered suspect if the duplicates differ by more than 10 % of the lower in the concentration range up to 100 mg/kg benzo(a)pyrene in creosote and 5 % of the lower above 100 mg/kg benzo(a)pyrene in creosote.

10.2 Reproducibility

Single results obtained by two laboratories shall be considered suspect if they differ by more than 30 % of the lower.

11 Test report

The test report shall include at least the following information:

a) the number and date of this European Standard;

b) full identification of the sample tested and details of its preparation for analysis;

c) the date of the test;

d) the results of the analysis, expressed as milligrams per kilogram benzo(a)pyrene in creosote (see Clause 9);

e) whether the repeatability has been verified;

f) any particular points observed in the course of the test;

g) any operations not specified in the method or regarded as optional which might have affected the result.