



SLOVENSKI STANDARD SIST EN 1014-3:2004

01-januar-2004

NUy]hbUgfYXghj UnU`Yg`E`?fYcnc]b`g`_fYcnc]ca `nUy]hb`Yg`E`A YrcXYj ncf Yb`U
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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

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

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

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Ta slovenski standard je istoveten z: EN 1014-3:1997

ICS:

71.100.50 S^ { ä æ Å Á ä å ä Å • æ Wood-protecting chemicals

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en

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EUROPEAN STANDARD

EN 1014-3

NORME EUROPÉENNE

EUROPÄISCHE NORM

August 1997

ICS 71.100.50

Supersedes ENV 1014-3:1995

Descriptors: Wood, wood preservatives, impregnating, creosote, chemical analysis, determination of content, high performance liquid chromatography

English version

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

iTech STANDARD PREVIEW

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

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This European Standard was approved by CEN on 1997-07-18. 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 Central Secretariat or to any CEN member.

The European Standards exist 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 Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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Foreword

This European Standard has been prepared by the Technical Committee CEN/TC 38 "Durability of wood and derived materials", the secretariat of which is held by AFNOR.

This European Standard supersedes ENV 1014-3:1995.

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

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 February 1998, and conflicting national standards shall be withdrawn at the latest by February 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This part of EN 1014 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

This European Standard incorporates by dated or undated reference provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in by amendment or revision. For undated references, the latest edition of the publication referred to applies:

EN ISO 3696 Water for analytical laboratory use - Specification and test methods.

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.

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4 Reagents

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4.1 Acetonitrile - water mixture. Add 220 ml of water (according to grade 1 of EN ISO 3696) to 780 ml of acetonitrile (CH₃CN, 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 (CH₃CN, 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 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 μ l 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 μ m; 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 μ l capacity.

6 Preparation of the calibration solutions and of the test samples.

6.1 Preparation of the calibration solutions.

Transfer by pipette (5.2) 1ml of the benzo[a]pyrene solution (4.2) to a 100ml one-mark volumetric flask and dilute to the mark with the acetonitrile-water mixture (4.1). To a series of 100ml one-mark volumetric flasks, transfer by pipette (5.2) 1ml, 2ml, 5ml and 20ml 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 exactly 30 min to ensure that the sample is completely liquid.

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

NOTE : Attention should be paid that no recrystallization 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 Analyse the test samples and the calibration solutions at the same temperature ($\pm 0,5^{\circ}\text{C}$).

At the same temperature (i.e. within $+0,5^{\circ}\text{C}$) 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, B_{s1} and B_{s2} , 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;

H_c is the mean of the duplicated benzo[a]pyrene peak heights obtained with the calibration solution in millimetres;

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

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

9 Expression of results

Report the benzo[a]pyrene content B_s 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 1mg/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.