SLOVENSKI STANDARD
SIST EN 1014-4:2004

01-januar-2004

Zaġļitna sredstva za les ï Kreozot in s kreozotom zaġļiten les ï Metode vzorļenja in analize ï 4. del: Ugotavljanje deleġa vodotopnih fenolov v kreozotu

Wood preservatives - Creosote and creosoted timber - Methods of sampling and analysis - Part 4: Determination of the water-extractable phenols content of creostote

Holzschutzmittel - Teerimprägnieröl und damit imprägniertes Holz - Probenahme und Analyse - Teil 4: Bestimmung des Gehaltes an wasserextrahierbaren Phenolen in Teerimprägnieröl

Produits de préservation du bois - Créosote et bois créosoté - Méthodes d'échantillonnage et d'analyse - Partie 4: Détermination de la teneur en phénols extractibles a l'eau de la créosote

Ta slovenski standard je istoveten z: **EN 1014-4:1995**

ICS:

71.100.50 Kemikalije za zaġļito lesa Wood-protecting chemicals

SIST EN 1014-4:2004 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 1014-4

July 1995

ICS 71.100.50

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

English version

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

Produits de préservation du bois - Créosote et bois créosoté - Méthodes d'échantillonage et d'analyse - Partie 4: Détermination de la teneur en phénols extractibles à l'eau de la créosote

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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.

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CEN

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

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Foreword

This European Standard has been prepared by the Technical Committee CEN/TC 38 "Durability of wood-based products", of which the secretariat 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of an identical text or by endorsement, at the latest by January 1995, and conflicting national standards shall be withdrawn at the latest by January 1995.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;
- prEN 1014-2 : Wood preservatives - Creosote and creosoted timber - Methods of sampling and analysis - Part 2 : Procedure for sampling creosoted timber and the extraction of creosote from the sample;
- prENV 1014-3: Wood preservatives - Creosote and creosoted timber - Methods of sampling and analysis - Part 3 : Determination of the benzo[a]pyrene content of creosote.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, 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 high performance liquid chromatographic (HPLC) method for the determination of the water-extractable phenols content of creosote.

For reasons of precision, this standard is applicable to the determination of the water-extractable phenols content of creosotes containing more than 10 g of water-extractable phenols/kg of creosote.

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.

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

3 Principle

The creosote sample is extracted with water. The aqueous extract is analyzed using high performance liquid chromatography (HPLC) at constant temperature with a reversed phase packed column and isocratic elution. The result is compared with that from a known reference standard containing various phenols known to be extracted by water from creosote.

4 Reagents

4.1 **acetonitrile**, HPLC grade.

4.2 **water**, according to ISO 3696-grade 1.

4.3 **methanol**, HPLC grade.

4.4 **acetic acid**, HPLC grade.

4.5 **phenol** purity 98 % (m/m) minimum.

4.6 **2-methylphenol** (o-cresol) purity 98 % (m/m) minimum.

4.7 **3-methylphenol** (m-cresol) purity 98 % (m/m) minimum.

4.8 **4-methylphenol** (p-cresol) purity 98 % (m/m) minimum.

4.9 **1,2-dihydroxybenzene** (catechol) purity 98 % (m/m) minimum.

4.10 **1,3-dihydroxybenzene** (resorcinol) purity 98 % (m/m) minimum.

4.11 1,4-dihydroxybenzene (hydroquinone) purity 98 % (m/m) minimum.

4.12 2,4-dimethylphenol purity 98 % (m/m) minimum.

4.13 2,6-dimethylphenol purity 98 % (m/m) minimum.

4.14 3,5-dimethylphenol purity 98 % (m/m) minimum.

4.15 acetonitrile/methanol mixture. Add 500 ml of acetonitrile (4.1) to 500 ml methanol (4.3) and mix thoroughly.

4.16 water/acetic acid mixture. Add 10 ml of acetic acid (4.4) to 990 ml water (4.2) and mix thoroughly.

4.17 HPLC eluent. Add together 180 ml acetonitrile (4.1), 180 ml methanol (4.3) and 640 ml of the water/acetic acid mixture (4.16), and mix thoroughly.

NOTE : If the HPLC equipment has a solvent mixing system where separate containers can hold the acetonitrile, the methanol, and the water/acetic acid mixture, the preparation of 4.17 is unnecessary.

4.18 standard phenols solution. Weigh 200,0 mg to within 0,1 mg of each of the phenols (4.5 to 4.14) into a single 100 ml one-mark volumetric flask. Add 36 ml of the acetonitrile/methanol mixture (4.15). Make up to the mark with the water/acetic acid mixture (4.16). Transfer the solution to brown glass storage flasks (5.6). Store the flasks in the dark below 10° C.

WARNING: Care should be taken to avoid any skin contact with the phenols.

NOTE : Under these storage conditions the solution is stable for 6 months although frequent use may result in faster ageing.

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 marked pipettes of 5 ml, 10 ml and 25 ml capacity.

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

- a solvent delivery pump with constant flow regulation;
- 10 μl loop injector;
- reversed 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;
- ultraviolet detector capable of being set at an absorption wavelength of 276 nm;
- an integrator or potentiometric recorder.

NOTE : As an alternative, any other HPLC configuration giving at least the same resolution (see annex A) could be used.

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

5.5 **Laboratory balance**, capable of weighing to 0,1 g.

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

When only very small amounts of creosote (a few grams) are available, the following additional apparatus is necessary:

5.7 **Glass screw-topped phial** of 10 ml capacity.

5.8 **Phase separation one-side silicone-treated filter papers**, with a diameter of 70 mm[1].

5.9 **Glass syringe** of 1ml capacity.

6 Preparation of the calibration solutions and of the test samples

6.1 Preparation of calibration solutions

Transfer by pipette (5.2) 25 ml, 10 ml and 5 ml of the standard phenols solution (4.18) to a series of 100 ml one-mark volumetric flasks. Make up to the mark with HPLC eluent (4.17).

Transfer the calibration solutions to brown glass storage flasks (5.6). Store the flasks in the dark below 10° C.

The calibration solutions should be prepared each day.

6.2 Preparation of the test sample

Prepare duplicate test samples.

Ensure that the sample of creosote consists of a single phase. If the laboratory sample derives from creosote in which crystals appear at ambient temperatures, heat the sample to a temperature at which it forms a single phase.

6.2.1 Preparation of "large" test samples

NOTE 1 : This procedure should be followed if a sufficient amount of creosote (e.g. 250 g) is available.

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[1] Whatman 1 PS is an example of a suitable product available commercially. The information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of this product.

Weigh accurately and directly into a 500 ml separating funnel 100,0 g to within 0,1 mg of the creosote sample. Add the same amount of water (4.2) weighed to an accuracy of 0,1 g. Stopper the funnel and agitate it vigorously for 15 min, venting the funnel from time to time. Allow to stand until the two layers separate (approximately 10 min.). Filter the aqueous layer through a filter paper[2)] until it is clear.

NOTE 2 : Several passes through the filter paper may be required.

Weigh 5,0 g of the clear aqueous extract to an accuracy of 0,01 g into a 10 ml one-mark volumetric flask. Make up to the mark with water (4.2) to produce the test sample.

NOTE 3 : It may be necessary to use a larger quantity of the clear aqueous extract if the phenolic content is very low.

6.2.2 Preparation of "small" test samples

NOTE : This procedure should be followed in case only a small amount of creosote (e.g. 2 to 5 grams) is available.

Weigh accurately and directly into the screw-topped phial (5.7) 1 g of the creosote sample to an accuracy of 1 mg. Add twice this amount of water (4.2), weighed to an accuracy of 0,02 g, agitate vigorously for 15 min.
Filter on the phase separation filter (5.8).

Take approximately 1 ml of the aqueous phase retained on the filter with the syringe (5.9), taking care not to include any creosote.

7 Procedure

7.1 Set up the apparatus (5.3) in accordance with the manufacturer's instructions. Adjust the UV detector to a wavelength of 276 nm.

Under isocratic conditions set the flow rate through the column to 1,0 ml/min using the HPLC eluent (4.17).

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

Inject successively the series of calibration solutions and then the two test samples into the chromatograph (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 height of the peaks for each individual phenol.

[2)] Whatman No 1 is an example of a suitable product available commercially. This information is given for the convenience of users of this standard and does not consitute an endorsement by CEN of this product.

8 Calculation

Calculate the content of each individual phenol in the two test samples (P_{i1} and P_{i2}) in grams phenol/kilogram creosote, using the equation :

$$P_i = \frac{P_c x H_s}{C_c x H_c} x 1000$$

Where :
P_c is the concentration of the phenol considered in the calibration solution nearest to the test sample, in milligrams per litre;
H_c is the mean of the duplicated peak heights of the phenol considered, obtained with the calibration solution, in millimetres;
C_c is the concentration of the aqueous extract of creosote in the test sample (6.2), in milligrams per litre;
H_s is the peak height for the phenol under consideration obtained for the test sample (6.2), in millimetres.

Calculate the total water-extractable phenols content in the two test samples (P_{S1} and P_{S2}) in grams per kilogram of the laboratory sample, using the following equation:

$$P_{S1} = \Sigma P_{i1}$$

and

$$P_{S2} = \Sigma P_{i2}$$

9 Expression of results

Report the water-extractable phenols content P_s of the laboratory sample as the average of P_{S1} and P_{S2} in grams phenols per kilogram creosote, rounded to the nearest 1 g/kg.

10 Precision

NOTE : The precision data are derived from an inter-laboratory test with "large" test samples (6.2.1). The modifications introduced for "small" test samples (6.2.2) imply that the precision might be lower than given in 10.1 and 10.2.

10.1 Repeatability

Duplicate results obtained by the same operator shall be considered suspect if they differ by more than 5 % of the smaller.

10.2 Reproducibility

Single results obtained by two laboratories shall be considered suspect if they differ by more than 15 % of the smaller