BRITISH STANDARD

BS EN
1014-4:2010

Wood preservatives — Creosote and creosoted timber — Methods of sampling and analysis

Part 4: Determination of the water-extractable phenols content of creosote

ICS 71.100.50

BSI
British Standards

National foreword

This British Standard is the UK implementation of EN 1014-4:2010. It supersedes BS EN 1014-4:1996 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/515, Wood preservation.

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

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Foreword

This document (EN 1014-4:2010) 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of an identical text or by endorsement, at the latest by December 2010, and conflicting national standards shall be withdrawn at the latest by December 2010.

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This document supersedes EN 1014-4:1995.

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 1014-3, *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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1 Scope

This European Standard 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 per kilogram of creosote.

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 extracted with water. The aqueous extract is analyzed using high performance 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 grade 1 of EN ISO 3696:1995.

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.

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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 six months although frequent use may result in faster ageing.

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 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.

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

5.9 Glass syringe of 1 ml 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

6.2.1 General

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.

6.2.2 Preparation of "large" test samples

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

Weigh accurately and directly into a 500 ml separating funnel 100,0 g to within 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 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.

6.2.3 Preparation of "small" test samples

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

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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 European Standard and does not constitute an endorsement by CEN of this product.

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.

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

7.3 Analyze 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).

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

7.5 Measure the height of the peaks for each individual phenol.

8 Calculation

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

$$P_{\mathrm{i}} = \frac{P_{\mathrm{c}} \times H_{\mathrm{s}}}{C_{\mathrm{c}} \times H_{\mathrm{c}}} \times 1\,000$$

where

P_{c} is the concentration of the phenol considered in the calibration solution nearest to the test sample, in milligrams per litre (mg/l);

H_{c} is the mean of the duplicated peak heights of the phenol considered, obtained with the calibration solution, in millimetres (mm);

C_{c} is the concentration of the aqueous extract of creosote in the test sample (6.2), in milligrams per litre (mg/l);

H_{s} is the peak height for the phenol under consideration obtained for the test sample (6.2), in millimetres (mm).

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:

$$P_{\mathrm{s1}} = \sum P_{\mathrm{i1}}$$

and

$$P_{\mathrm{s2}} = \sum P_{\mathrm{i2}}$$

9 Expression of results

Report the water-extractable phenols content P, of the laboratory sample as the average $P_{s1}$and P_{s2} in grams phenols per kilogram creosote, rounded to the nearest 1 g/kg.

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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.

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 grams of water-extractable phenols per kilogram of creosote (see Clause 9);

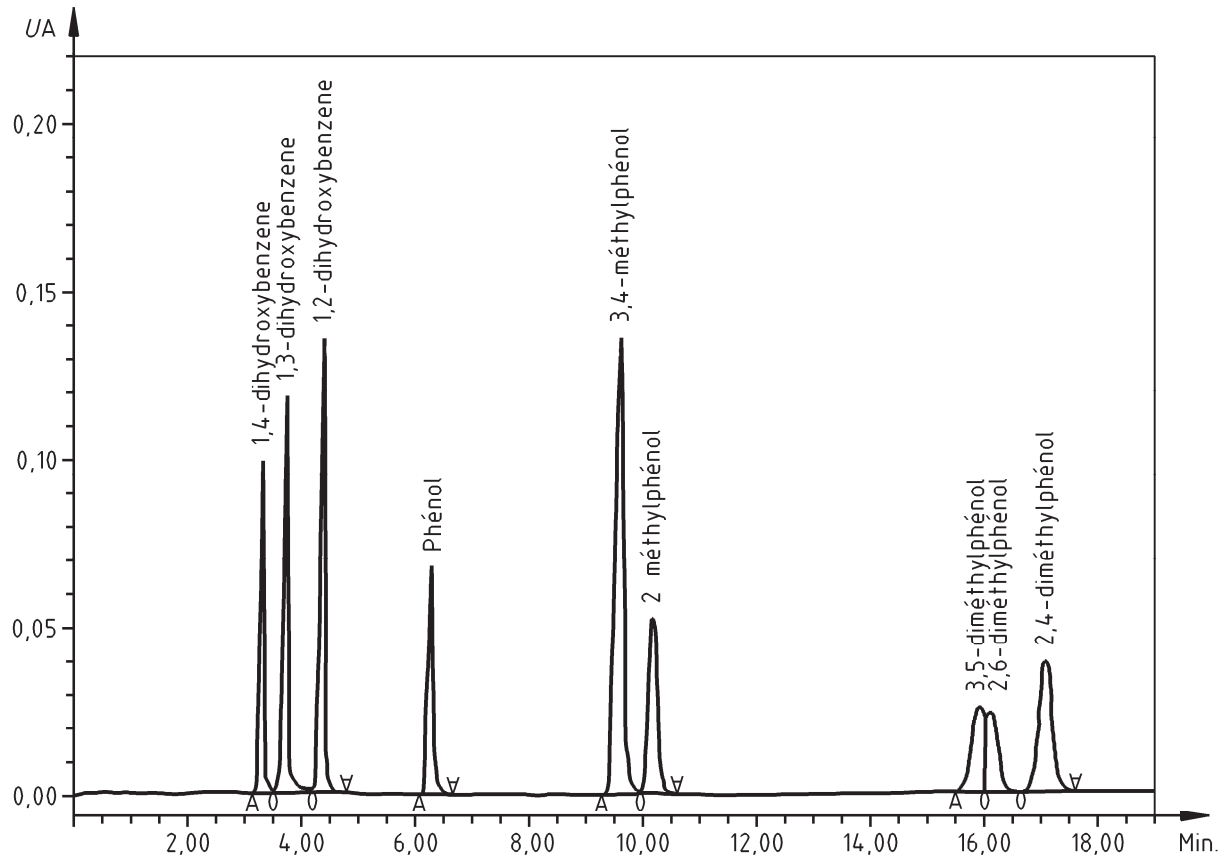
e) whether the repeatability has been verified;

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 results.

Annex A
(informative)

Example of chromatogram



Column: Packing C18-particle size 5 µm;

Length 250 mm;

Internal diameter 4,6 mm.

Operation conditions:

Flow: 1 ml/min;

Temperature: 24 °C.

NOTE Retention times depend on the column used and on the operational conditions.

Figure A.1 — Example of chromatogram

Annex B
(informative)

Interpretation of the test results

This method can e.g. be used to determine the water-extractable phenols content of creosote (sampled according to the procedure defined in EN 1014-1) or of creosote extracted from creosoted timber (according to the procedure defined in EN 1014-2).

In the latter case, however, care should be taken when drawing conclusions as to the water-extractable phenols content of the creosote with which the timber was originally treated. In the course of the service-life the chemical composition of the creosote in treated timber changes as a result of the preferential evaporation or leaching of certain components. As water-extractable phenols are one of these preferential components, the measured water-extractable phenols content (P) will be lower than the content in the original creosote.

Bibliography

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

[2] 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*

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