

SLOVENSKI STANDARD oSIST prEN 13991:2022

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Derivati pri pirolizi premoga - Olja iz premogovega katrana: kreozoti - Specifikacije in metode preskušanja

Derivatives from coal pyrolysis - Coal tar based oils: creosotes - Specifications and test methods

Derivate der Kohlenpyrolyse - Öle aus Steinkohlenteer: Kreosot - Anforderungen und Prüfverfahren

PREVIEW

Produits dérivés de la pyrolyse du charbon - Huiles de goudron de houille: Créosotes - Spécifications et méthodes d'essai na ards.iten.al)

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EUROPEAN STANDARD NORME EUROPÉENNE EUROPÄISCHE NORM

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January 2022

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Will supersede EN 13991:2003

English Version

Derivatives from coal pyrolysis - Coal tar based oils: creosotes - Specifications and test methods

Produits dérivés de la pyrolyse du charbon - Huiles de goudron de houille: Créosotes - Spécifications et méthodes d'essai Derivate der Kohlenpyrolyse - Öle aus Steinkohlenteer: Kreosot - Anforderungen und Prüfverfahren

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 317.

If this draft becomes a European Standard, 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.

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Recipients of this draft are the interest of the submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation $\frac{1}{77}$ by $\frac{1}{9}$ decreases $\frac{1}{9}$ and $\frac{1}{9}$ decreases $\frac{1}{9}$ de

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

prEN 13991:2022 (E)

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European foreword

This document (prEN 13991:2022) has been prepared by Technical Committee CEN/TC 317 "Derivatives from coal from pyrolysis", the secretariat of which is held by DIN.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 13991:2003.

A revision is required due to legal changes to the marketing of Creosote oils in Europe. According to Regulation 1907/2006/EC (Annex XVII, Entry 31, Restrictions), only Creosote oils with a benzo[a]pyrene content below 50 mg/kg may be placed on the market in Europe. This means that grade A quality with a benzo[a]pyrene content below 500 mg/kg is no longer allowed to stay on the market and is therefore deleted from the standard

Annexes A and B are normative. Annex C is informative.

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1 Scope

This document gives the specifications and the test methods for creosotes for industrial wood preservation.

Different grades of creosote are used depending on the desired properties of the treated wood.

WARNING — The use of this document may involve hazardous materials, operations and equipment. This document cannot address all of the safety implications associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices and assess the applicability of regulatory limitations prior to use. The warnings to use are covered in Annex C.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1014-1, Wood preservatives - Creosote and creosoted timber - Methods of sampling and analysis - Part 1: Procedure for sampling creosote

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

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

EN 15529, Derivatives from coal pyrolysis Terminology S.iteh.ai

BS 144:1997, Coal tar creosote for wood preservation

EN ISO 2719, Determination of flash point - Pensky-Martens closed cup method (ISO 2719)

ISO 386, Liquid-in-glass laboratory thermometers — Principles of design, construction and use

ISO 760, Determination of water — Karl Fischer method (General method)

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 15529 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

4 Sampling

Samples for the assessment of the parameters listed in Table 1 shall be taken in accordance with EN 1014-1.

5 Specifications and test methods

Specifications for creosote grade B and C and the relevant test methods are given in Table 1.

Grade B is intended for pressure impregnation. In contrast to grade A, the distillation residue is lowered in order to reduce the benzo[a]pyrene content. It is especially suitable for treatment of poles for overhead power and telecommunication lines, and for structural timbers where bleeding in service can occur.

Grade C excludes the lower boiling fraction allowable in the other types of creosote and because of the low volatility a reduction in odour is achieved.

Grade B and grade C are suitable for the hot-and-cold open-tank process.

Creosote in use should not contain more than 3 % of water.

Table 1 — Specifications for creosote grade B and C

Parameters		Grade B	Grade C	Test Method
Density (kg/m³) at 20/4 °C a		1,020 to 1,150	1,030 to 1,170	BS 144 Annex B
Water content (m/m) %		max. 1	max. 1	ISO 760 b
Crystallization temperature (°C)	h STAN	max. 23	max. 50	Annex A
Water-extractable phenols (m/m) %	PREV	max-3	max. 3	EN 1014-4
Insoluble matter in toluene (m/m) %	andards	max. 0,4 a	max. 0,4	BS144 Annex G
Boiling range (volume) %	oSIST prEN 13	991:2022		Annex B
Distillate to 235 °Cnttps://standard				
Distillate to 300 ©40-48b1-ac	59-d777b9d486′	77/o.40-60en-1	39 max.010	
Distillate to 355 °C		min.70	min. 65	
Benzo[a]pyrene content (mg/kg)		max. 50	max. 50	EN 1014-3
Flash point (°C)		min. 61	min. 61	EN ISO 2719

NOTE According to Regulation 1907/2006 / EC (Annex XVII, entry 31, Restrictions) only creosote oils with a Benzo[a]pyrene content lower than 50 mg/kg may be marketed in Europe. This means that former grade A quality with a Benzo[a]pyrene content of max. 500 mg/kg is no longer allowed to stay on the market. Grade A was therefore deleted.

^a The density is determined at a temperature above the crystallization temperature. For each degree Celsius above 20 °C, a fixed value of 0,7 kg/m 3 is added to the density reading.

b As an alternative, the distillation method of ISO 3733 can be used.

Annex A

(normative)

Determination of the crystallization temperature of coal tar oil

A.1 Scope

This method describes the procedure for the determination of the crystallization temperature of coal tar oil and is applicable to creosote, carbolineum, carbon black feedstock, fluxing oils and other coal tar based oils.

A.2 Sampling

Sampling shall follow the procedures described in EN 1014-1.

A.3 Apparatus: an example of the apparatus is given in Figure A.1

- **A.3.1** Porcelain dish, top diameter (105 ± 5) mm, height (60 ± 5) mm, capacity ca. 300 ml.
- **A.3.2** Porcelain dish, flat shape, diameter (250 ± 10) mm, capacity ca. 2 000 ml.
- **A.3.3** Electric heating plate, 500 W to 1 000 W.
- A.3.4 Thermometer, 0 °C to 100 °C, graduated in 0,5 °C as described in ISO 386.
- **A.3.5** Metal support ring.

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A.4 Procedure

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Heat the sample until completely free of crystals. The temperature shall be at least 10 °C above the specified maximum crystallization temperature.

Pour (150 ± 5) ml of the so prepared oil into the small porcelain dish.

Place the dish with oil on the support ring in the larger dish and fill the latter with water at a temperature about $10\,^{\circ}\text{C}$ lower than the specified maximum crystallization temperature.

The water surface shall be (10 ± 2) mm above the oil surface in the smaller dish.

Stir the oil gently with the thermometer, the oil shall be crystal free at the temperature indicated in Table 1.

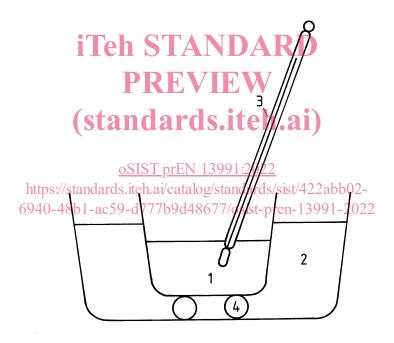
If it is required to know the crystallization temperature, repeat the determination with water at a lower temperature. Use some ice when necessary.

The temperature at which crystal formation starts is the crystallization temperature.

A.5 Test report

The test report shall at least include the following:

- type of tar oil;
- references of sample;
- the reference to this document, i.e. EN 13991;
- the date of determination;
- the name of the operator;
- the result of the determination, (Pass or Fail), the crystallization temperature if required, expressed in degrees Celsius;
- any particular points observed in the course of the sampling procedure and the test procedure.



Key

- 1 coal tar oil
- 2 water
- 3 thermometer
- 4 metal support ring

Figure A.1 — Determination of the crystallization temperature

Annex B

(normative)

Determination of the distillation range of coal tar oil

B.1 Scope

This method specifies requirements for components of an apparatus used in the determination of the distillation characteristics of coal tar oil. This method is applicable to creosotes, carbolineum, carbon black feedstock, fluxing oils and others coal tar based oils.

B.2 Sampling

Sampling shall follow the procedures described in EN 1014-1.

B.3 Apparatus

- B.3.1 Distillation flask as specified in Figure B.1.
- **B.3.2** Condenser tube as specified in Figure B.2. R.V. R.W.
- **B.3.3** Distillation cabinet as shown in Figure B.3.
- **B.3.4** 0 °C to 360 °C thermometer, graduated in 1 °C divisions, certified as in ISO 386.
- 100 ml measuring cylinder, scale ca. 140 mm to 150 mm, calibrated in 1 ml divisions. B.3.5
- Tripod or retort standand/ringdards.iteh.ai/catalog/standards/sist/422abb02-
- 160 mm × 160 mm gauze, without as best 0s 9d48677/osist-pren-13991-2022 **B.3.7**
- **B.3.8** Corks, beads.
- **B.3.9** Spirit lamp or equivalent heat-source.
- **B.3.10** Bunsen burner, or hot-plate (700 W, open coil and 25 Ω to 30 Ω rheostat).

B.4 Preparing the distillation

100 ml of prepared oil are used in the distillation. Since about 2 ml stick to the inner wall when the oil is poured out of the measuring cylinder, 102 ml of oil are measured out and then placed in the distillation flask with a few beads. The distillation flask is sealed with a drilled bung which holds the thermometer. Care shall be taken to ensure that the bung is firmly seated and the thermometer vertical. There shall be no oil on the neck of the flask or on the thermometer. The thermometer shall reach into the flask so that the mercury bulb is 12 mm to 13 mm above the surface of the liquid. The thermometer shall not be moved during distillation. Two gauze supports on the tripod are used when heating by gas or equivalent, and one gauze on the hot-plate when heating by electricity. The clean measuring cylinder is placed under the condenser tube outlet.