BRITISH STANDARD

BS EN
13991:2003

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

The European Standard EN 13991:2003 has the status of a British Standard

ICS 71.100.50; 75.140

National foreword

This British Standard is the official English language version of EN 13991:2003.

The UK participation in its preparation was entrusted to Technical Committee CII/56, Derivatives from coal pyrolysis, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 15 and a back cover.

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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

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Contents

Foreword

This document (EN 13991:2003) has been prepared by Technical Committee CEN /TC 317, "Derivatives from coal pyrolysis", the secretariat of which is held by IBN.

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of an identical text or by endorsement, at the latest by February 2004, and conflicting national standards shall be withdrawn at the latest by February 2004.

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

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, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard 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 European Standard may involve hazardous materials, operations and equipment. This standard cannot address all of the safety implications associated with its use. It is the responsibility of the user of this standard 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

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 it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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 12303:2000, *Coal tar based oils - Terminology*.

BS 144:1997, Coal tar creosote for wood preservation.

EN ISO 2719, *Determination of flash point - Pensky-Martens closed cup method (ISO 2719:2002)*

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 European Standard, the following terms and definitions given in EN 12303:2000 apply.

4 Sampling

Sampling of creosotes and preparation of samples.

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 A, B, C and the relevant test methods are given in Table 1.

Table 1 — Specifications for creosote grade A, B and C

Parameters	Grade A	Grade B	Grade C	Test Method
Density (kg/m^3) at 20/4 °C	1,040-1,150	1,020-1,150	1,030-1,170	BS144-annex B
Water content (m/m) %	max. 1	max. 1	max. 1	ISO 760 [a]
Crystallization temperature (°C)	max. 23	max. 23	max. 50	Annex A
Water-extractable phenols (m/m) %	max. 3	max. 3	max. 3	EN 1014-4
Insoluble matter in toluene (m/m) %	max. 0.4	max. 0.4	max. 0.4	BS144-annex G
Boiling range (volume) %				annex B
Distillate to 235 °C	max. 10	max. 20	-	
Distillate to 300 °C	20-40	40-60	max. 10	
Distillate to 355 °C	55-75	min.70	min. 65	
Benzo[a]pyrene content (mg/kg)	max. 500	max. 50	max. 50	EN 1014-3
Flash point (°C)	min. 61	min. 61	min. 61	EN ISO 2719

[a] As an alternative, the distillation method of ISO 3733 can be used.

NOTE 1 Grade A is intended for treatment of timber by pressure impregnation.

Grade B is also 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.

NOTE 2 All types of creosotes are suitable for the hot-and-cold open-tank process.

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

NOTE 4 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.

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 - 1 000) W.

A.3.4 Thermometer, (0° - 100) °C, graduated in 0,5 °C as described in ISO 386.

A.3.5 Metal support ring.

A.4 Procedure

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 °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 ;

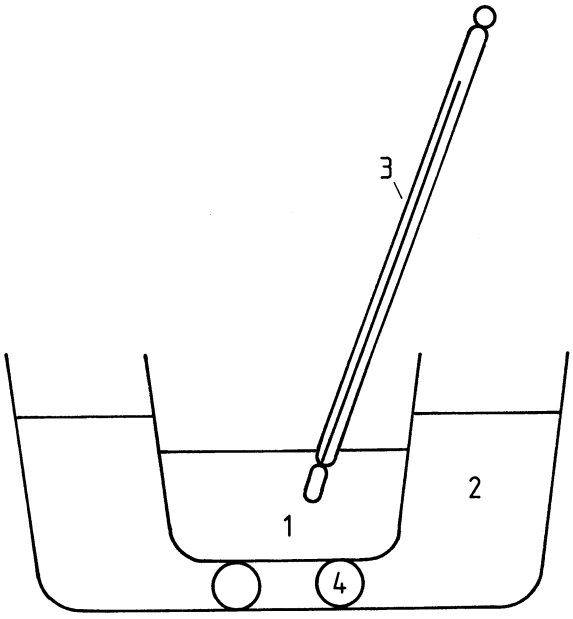
— the reference to this European Standard ;

— 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.

B.3.3 Distillation cabinet as shown in Figure B.3.

B.3.4 (0 - 360) °C thermometer, graduated in 1 °C divisions, certified as in ISO 386.

B.3.5 100 ml measuring cylinder, scale ca. (140 - 150) mm, calibrated in 1 ml divisions.

B.3.6 Tripod or retort stand and ring.

B.3.7 (160 x 160) mm gauze, without asbestos.

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.

B.5 Method (see Figures B.4 – B.5)

The distillation flask is first heated gently using a small flame or a low heat setting on the hot-plate, in order to prevent bumping and foaming of the water which is always present. When the water has evaporated, the oil starts to boil gently. The heat should be controlled such that the first drop of oil falls into the measuring cylinder after 5 to a maximum of 15 minutes. The distillation should then proceed so that 1-2 drops fall into the measuring cylinder every second. If solid deposits form in the condenser tube they should be liquefied by gently heating, e.g. by using the spirit lamp. As soon as the thermometer shows the temperature given in the specification, the amount collected in the measuring cylinder is measured.

The amounts measured in ml correspond to the percentages in volume %.

NOTE For comparison analyses, the effect of barometric pressure should be taken into consideration (cf. ASTM D 246-73).

Precision

Repeatability : 2,0 vol. % absolute for the individual fraction.

Reproducibility : 4,0 vol. % absolute for the individual fraction.

B.6 Test report

The test report shall at least include the following :

— type of tar oil ;

— reference of sample ;

— the reference to this European Standard ;

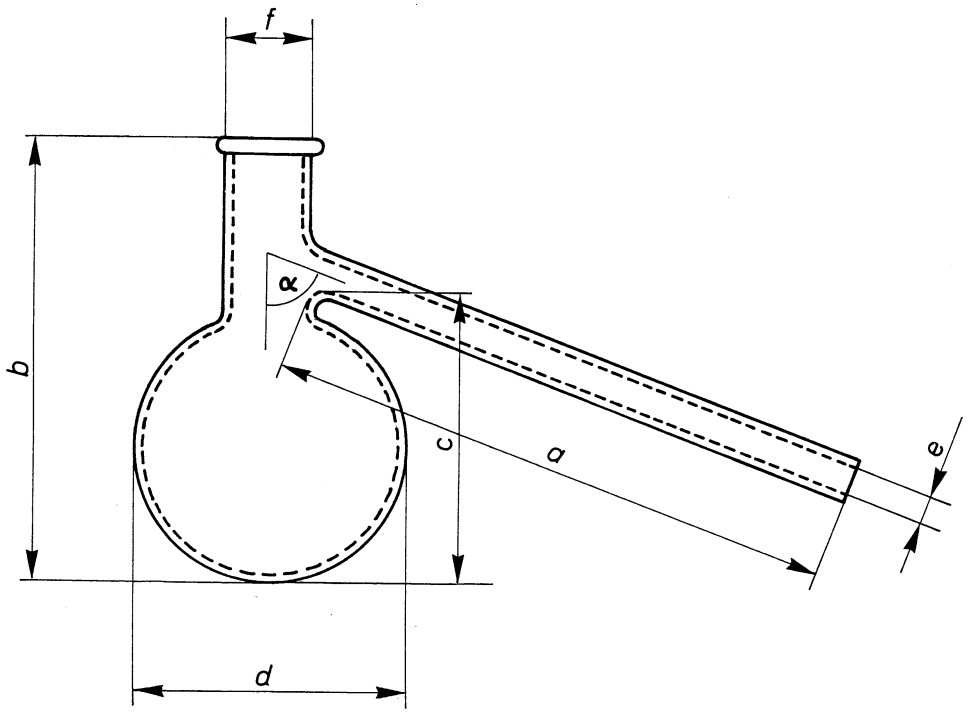
— the date of determination ;

— the name of the operator ;

— the result of determination ;

— any particular points observed in the course of the sampling procedure and the test procedure.

Dimensions in millimeters

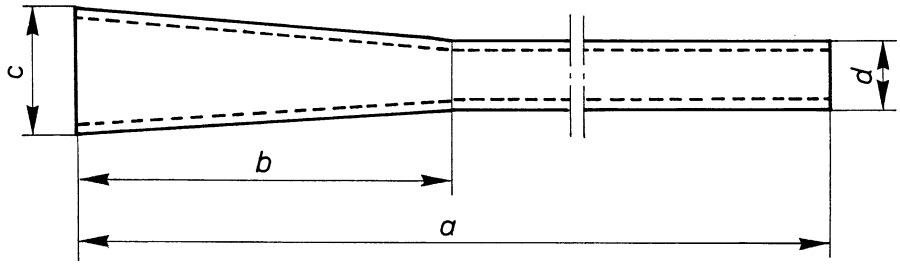


Key

a = 200 ± 5,0
b = 135 ± 1,5
c = 93 ± 1,5
d = 86 ± 1,5
e = 10 ± 0,5
f = 22 ± 1,0
∝ = 73 ± 2,0
(in degrees)

Figure B.1 — Distillation flask

Dimensions in millimeters

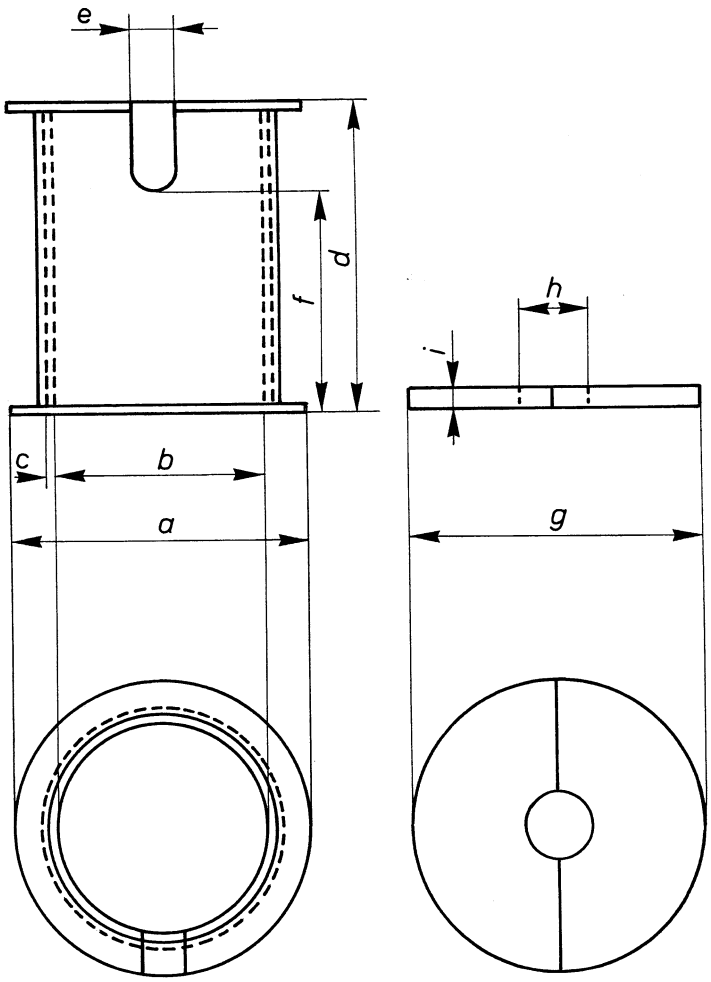


Key

a = 360 ± 6,0
b = 100 ± 5,0
c = 28,5 ± 3,0
d = 12,5 ± 1,5

Figure B.2 — Condenser tube

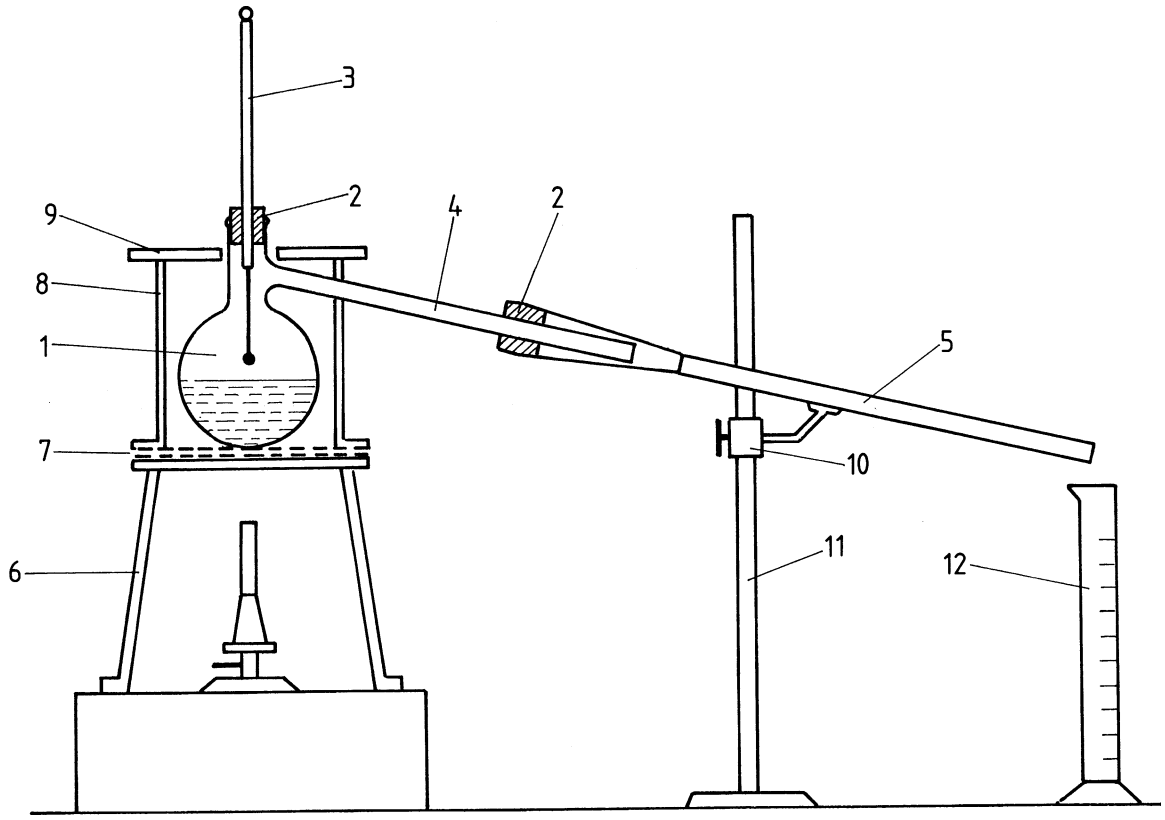
Dimensions in millimeters



Key

a = 136 ± 3
b = 105 ± 2
c = 3,2 ± 0.3
d = 111 ± 3
e = 16 ± 2
f = 76 ± 2
g = 136 ± 3
h = 27 ± 1
i = 6,6 ± 0,5

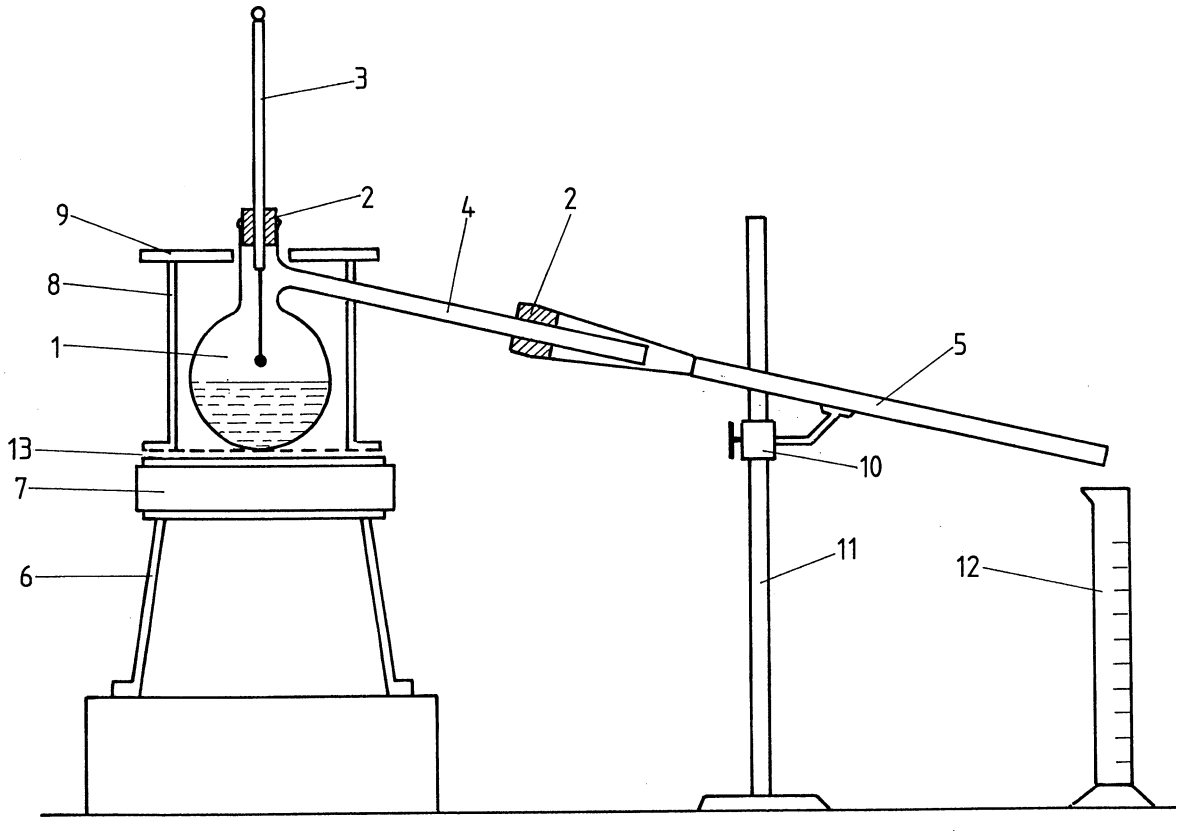
Figure B.3 — Protective screen



Key

1 Distillation flask
2 Bung
3 Thermometer
4 Flask nozzle
5 Condenser tube
6 Tripod
7 Double gauze
8 Protective screen
9 Lid
10 Retort clamp
11 Retort stand
12 Clean measuring cylinder

Figure B.4 — Determination of the distillation range with a bunsen burner



Key

1 Distillation flask
2 Bung
3 Thermometer
4 Flask nozzle
5 Condenser tube
6 Tripod
7 Regulated hot plate
8 Protective screen
9 Lid
10 Retort clamp
11 Retort stand
12 Clean measuring cylinder
13 Gauze

Figure B.5 — Determination of the distillation range with electric hot plate

Annex C
(informative)

Warning to use[1]

C.1 Composition - Data on components

- Chemical characterization :

- **CAS. N° Designation**: 8001-58-9 Creosote.

- Identification number (s) :

- EINECS Number : 2322875.

- EU Number : 648-101-00-4.

- Chemical characterization :

- Description : Complex mixture of polynuclear aromatic and heterocyclic hydrocarbons.

- Dangerous components : Void.

C.2 First aid measures

- General information: No special measures required.

- After inhalation: Supply fresh air ; consult doctor in case of symptoms.

- After skin contact: Instantly wash with water and soap and rinse thoroughly.

- After eye contact: Rinse opened eye for several minutes under running water.

- After swallowing: In case of persistent symptoms consult doctor.

C.3 Handling and storage

- Handling.

- **Information for safe handling:** No special measures required; precautions against electrostatic charging.

- **Information about protection against explosions and fires:** No special measures required.

- Storage

- Requirements to be met by storerooms and containers: No special requirements.

- Information about storage in one common storage facility: Not required.

- Further information about storage conditions: None.

C.4 Disposal considerations

- Product :

- European waste catalogue: The waste code classification is to be carried out according to the European Waste Catalogue (EWC) specifically for each branch of industry and each type of process.

- Uncleaned packagings :

- Recommandation : Disposal should be made according to official regulations.

- Recommended cleaning agent : Water, if necessary with cleaning agent.

[1] The latest edition of the publication of the Material Safety Data Sheet of the product applies

Bibliography

ASTM D246-95, *Standard test method for distillation of creosote and creosote-coal tar solution.*

91/155/EEC Directive: Commission Directive of 5 March 1991 defining and laying down the detailed arrangements for the system of specific information relating to dangerous preparations in implementation of Article 10 of Directive 88/379/EEC

2001/58/EC Directive: Commission Directive of 27 July 2001modifying the 91/155/EEC Directive, defining and laying down the detailed arrangements for the system of specific information relating to dangerous preparations in implementation of Article 14 of Directive 1999/45/EEC and defining and laying down the detailed arrangements for the system of specific information relating to dangerous substances in implementation of Article 27 of Directive 67/548/EEC.

ISO 3733 , *Petroleum products and bituminous materials - Determination of water - Distillation method*.

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