



SLOVENSKI STANDARD

SIST EN 14719:2005

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Pulp, paper and board - Determination of the Diisopropyl-naphthalene (DIPN) content by solvent extraction

Faserstoff, Papier und Karton - Bestimmung des Gehaltes an Diisopropyl-naphthalin (DIPN) mittels Lösemittel-extraktion

Pâtes, papiers et cartons - Détermination de la teneur en diisopropyl-naphtalène (DIPN) par extraction au solvant

Ta slovenski standard je istoveten z: EN 14719:2005

ICS:

85.040	Vlaknine	Pulps
85.060	Papir, karton in lepenka	Paper and board

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en

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EUROPEAN STANDARD
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ICS 85.040; 85.060

English version

Pulp, paper and board - Determination of the Diisopropylnaphthalene (DIPN) content by solvent extraction

Pâtes, papiers et cartons - Détermination de la teneur en
diisopropylnaphtalène (DIPN) par extraction au solvant

Faserstoff, Papier und Karton - Bestimmung des Gehaltes
an Diisopropylnaphthalin (DIPN) mittels
Lösemittelextraktion

This European Standard was approved by CEN on 27 June 2005.

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Foreword

This European Standard (EN 14719:2005) has been prepared by Technical Committee CEN/TC 172 "Pulp, paper and board", the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by February 2006, and conflicting national standards shall be withdrawn at the latest by February 2006.

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

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EN 14719:2005 (E)**1 Scope**

This European Standard describes a method for the determination of the content of diisopropylnaphthalene (DIPN) in paper, board and pulp by solvent extraction. The content of diisopropylnaphthalene (DIPN) is expressed as mg of total diisopropylnaphthalene DIPN isomers per kilogram of paper, board or pulp. This test method is appropriate for the quantitative determination of the content of diisopropylnaphthalene (DIPN) with a limit of determination of about 0,6 mg/kg.

2 Principle

The content of total diisopropylnaphthalene (DIPN) is determined by solvent extraction of the paper, board or pulp sample and analysed by gas chromatography with mass selective detection (GC-MS), using diethylnaphthalene as an internal standard.

NOTE It may be worthwhile to state that diisopropylnaphthalene (DIPN) can be present as a contaminant in pulp, paper and board. For the time being it cannot be avoided that diisopropylnaphthalene (DIPN) is present when using recovered fibres. It may be present as either adsorbed onto the test piece or present in an encapsulated form. Acetone is an effective solvent for complete extraction of both physical forms of diisopropylnaphthalene (DIPN).

3 Reagents

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3.1 General

All reagents shall be of recognised analytical quality unless otherwise stated.

3.2 Standards

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3.2.1 Diisopropylnaphthalene ¹⁾ (DIPN) (technical mixture of isomers)

The purity shall be given by the supplier or 2,6-Diisopropylnaphthalene.

3.2.2 Diethylnaphthalene (DEN), with a purity of 97 % or more**3.3 Chemicals, Acetone****3.4 Calibration stock solutions****3.4.1 Stock solution of diisopropylnaphthalene (DIPN) in acetone with an accurately known concentration of approximately 1 200 mg/l**

Weigh accurately about 30 mg of diisopropylnaphthalene (DIPN) (3.2.1) into a 25 ml volumetric flask. Fill the volumetric flask to approximately half the volume with acetone (3.3), shake to dissolve and dilute to the mark with acetone.

Calculate the concentration of diisopropylnaphthalene (DIPN) in mg/l, by using the exact mass taken and the purity.

1) Diisopropylnaphthalene is a name of a product supplied by Fisher. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the product. Equivalent products from other manufacturers may be used if they can be shown to lead to the same results.

3.4.2 Standard stock solution (A) with an accurately known concentration of approximately 12 mg/l

Transfer 1 ml of the 1 200 mg/l stock solution (3.4.1) into a 100 ml volumetric flask and dilute to the mark with acetone.

3.4.3 Standard stock solution (B) with an accurately known concentration of approximately 120 mg/l

Transfer 10 ml of the 1 200 mg/l stock solution (3.4.1) into a 100 ml volumetric flask and dilute to the mark with acetone.

3.5 Internal standard stock solution

Stock solution of diethylnaphthalene (DEN) (3.2.2) in acetone with a concentration of approximately 1 000 mg/l.

Weigh accurately approximately 50 mg of diethylnaphthalene (DEN) (3.2.2) into a 50 ml volumetric flask and dilute to the mark with acetone.

4 Apparatus and auxiliary aids

NOTE An instrument or item of apparatus is listed only if it is special, or made to a particular specification, usual laboratory glassware and equipment being assumed to be available.

4.1 Gas chromatograph

Equipped with a mass selective detector.

4.2 Gas chromatograph analytical capillary column

Appropriate operating conditions shall be established for the specific equipment used for the determination.

An example of suitable chromatographic conditions is given below:

Column	5 % phenylmethyl siloxane 30 m x 0,25 mm ID 0,25 µm film thickness
Carrier gas:	Helium, 1 ml/min
Temperature program:	isothermal at 70 °C for 2 min, from 70 °C to 250 °C at 10 °C/min isothermal at 250 °C for 15 min
Injector:	Splitless, 240 °C
Transferline:	280 °C
Detection:	MSD (Mass Selective Detector) Ions, diisopropylnaphthalene (DIPN) m/z 155, 197, 212 diethylnaphthalene (DEN) m/z 169

The following retention times have been observed under these conditions:

- diisopropylnaphthalene (DIPN) 6 major isomers 15,5 min to 16,1 min;
- diethylnaphthalene (DEN) 14,8 min.

A separation of diisopropylnaphthalene (DIPN) into 7 isomers was observed with other columns, which did not influence the result.

EN 14719:2005 (E)**4.3 Ultrasonic bath****4.4 Microsyringes, 100 µl, 250 µl****4.5 Graduated pipettes, 1 ml, 2 ml, 10 ml****4.6 40 ml screw top vials, with PTFE (polytetrafluorethylene) lined septa****5 Sampling**

Wrap test pieces for test individually in aluminium foil. Avoid direct contact to plastic films. Use the test pieces without humidity-conditioning or drying.

6 Preparation of test pieces

Carry out the test in duplicate.

Cut (avoiding edges) a piece (approx. 5 g) of the paper, board or pulp test piece into test pieces with an area of approximately 2 cm². At the same time weigh a specimen for determination of the dry content.

Accurately weigh (2,00 ± 0,01) g of the prepared test piece into a 40 ml screw top vial, add 100 µl of internal standard stock solution (3.5) and allow it to soak in. Then add by measuring cylinder 25 ml acetone. Cap the vial and leave to stand for about 16 h at room temperature and then put the vials to the ultrasonic bath for 15 min.

Remove a small portion of the extract for analysis by GC-MS (Gas Chromatography Mass Spectrometry).

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7 Calibration**7.1 Calibration standards****7.1.1 General**

Prepare two sets of calibration standards in the ranges of 0,05 mg/l to 1,0 mg/l (calibration A) and 1 mg/l to 10 mg/l (calibration B).

NOTE There are two calibration standards for two different values.

7.1.2 Calibration A

Add 0,1 ml, 0,2 ml, 0,5 ml, 1,0 ml and 2,0 ml of standard stock solution A (3.4.2) and 100 µl internal standard stock solution (3.5) into five 25 ml volumetric flasks by syringe or pipette. Dilute to the mark with acetone to give calibration standard solutions nominally containing 1,2 µg, 2,4 µg, 6 µg, 12 µg and 24 µg of diisopropyl-naphthalene (DIPN).

7.1.3 Calibration B

Add 0,2 ml, 0,5 ml, 1,0 ml and 2,0 ml of standard stock solution B (3.4.3) and 100 µl internal standard stock solution (3.5) into four 25 ml volumetric flasks by syringe or pipette. Dilute to the mark with acetone to give calibration standard solutions nominally containing 24 µg, 60 µg, 120 µg and 240 µg of diisopropyl-naphthalene (DIPN).

Calculate the exact amounts of diisopropyl-naphthalene (DIPN) added to the calibration standard solutions.

NOTE The calibration standard solutions can be stored in a refrigerator up to 3 month.

7.2 Blank value

Follow the procedure described in Clause 6 but without addition of the test piece.

7.3 Control standard

Prepare a control standard from the standard stock solution A (3.4.2) by adding 2 ml into a 25 ml volumetric flask and 100 μ l internal standard stock solution (3.5) and diluting to the mark with acetone. Calculate the amount of diisopropylnaphthalene (DIPN) added in μ g.

8 Procedure

8.1 GC-MS analysis

Maintain the same operating conditions of the GC-MS system throughout the measurements of all solutions prepared in Clauses 6 to 7.3.

8.2 Calibration

Inject the calibration standards prepared in 7.1 together with the control standard (7.3) for GC-MS analysis.

Obtain the integrated peak areas of the DIPN and DEN peaks. Construct the calibration line by plotting the total peak area ratio of DIPN/DEN versus the mass of DIPN (μ g) added to the calibration solutions.

NOTE The correlation coefficients of the calibration lines should be 0,996 or better.

The control standard should give a result from the calibration graph within 8 % of the true value.

If the correlation coefficients are less than 0,996, then fresh intermediate standard stock solutions and calibration solutions should be prepared from the original stock solutions.

8.3 Test piece analysis

8.3.1 Test piece extracts

Inject the test piece extracts and blank as prepared in Clauses 6 and 7.2 for GC-MS analysis.

8.3.2 Determination of DIPN

Identify the six DIPN peaks on the basis of the retention time and measure the total peak areas. Also measure the DEN peak area and calculate the DIPN/DEN area ratio.

8.4 Evaluation of data

8.4.1 Interferences

No interferences with the DIPN or DEN peaks arising from the extraction procedure have so far been observed using the method and the GC-MS conditions specified in (4.2).

NOTE If DIPN is found in the blank (7.2) it is recommended to check for the source of contamination.

8.4.2 Determination of the DIPN concentration in the test pieces

8.4.2.1 Graphical determination

Read from the calibration graph (see Clause 8) the quantity of DIPN in μ g in the test piece extract by interpolation of the total peak area ratios DIPN/DEN to the x-axis.