SLOVENSKI STANDARD
SIST EN 13405:2003

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Povrġinsko aktivne snovi - Doloļ evanje dialkil-tetralinov v linearnem alkilbenzenu s tekoļ insko kromatografijo visoke loļ ljivosti (HPLC)

Surface active agents - Determination of dialkyl-tetralins content in linear alkylbenzene by high performance liquid chromatography (HPLC)

Grenzflächenaktive Stoffe - Bestimmung des Gehaltes von Dialkyltetralin in linearem Alkylbenzol mittels Hochleistungs-Flüssigchromatographie (HPLC)

Agents de surface - Détermination de la teneur en dialkyltétralines dans les alkylbenzenes linéaires par chromatographie liquide a haute performance (CLHP)

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Surface active agents - Determination of dialkyl-tetralins content in linear alkylbenzene by high performance liquid chromatography (HPLC)

Agents de surface - Détermination de la teneur en dialkyltétralines dans les alkylbenzènes linéaires par chromatographie liquide à haute performance (CLHP)

Grenzflächenaktive Stoffe - Bestimmung des Gehaltes von Dialkyltetralin in linearem Alkylbenzol mittels Hochleistungs-Flüssigchromatographie (HPLC)

This European Standard was approved by CEN on 23 October 2002.

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

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Contents

Foreword

This document (EN 13405:2002) has been prepared by Technical Committee CEN /TC 276, "Surface active agents" 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of an identical text or by endorsement, at the latest by June 2003, and conflicting national standards shall be withdrawn at the latest by June 2003.

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

1 Scope

This European Standard specifies a method for the determination of dialkyl-tetralins (DAT), being 1,4-dialkyl-2,3-dihydro-naphthalene in linear alkylbenzene (LAB) in the range of the mass fraction of 0,5 % to 10 %.

2 Normative reference

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 ISO 3696:1995, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987).*

3 Principle

The sample is analysed by isocratic high performance liquid chromatography (HPLC) using a micro-particulate silica stationary phase, 2,2,4-methylpentane mobile phase and ultra-violet absorbance detection at 254 nm. Quantification is made by using a "modified" external standard technique. Pure standard dialkyl-tetralins (DAT) are not available, therefore the molar response of DAT is assumed to be the same as the commercially available tetrahydronaphthalene (THN) used as external standard.

4 Reagents

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade and have been checked in advance as to not interfere with the analytical results.

4.1 Water, complying with grade 3 as defined in EN ISO 3696:1995.

4.2 2,2,4-trimethylpentane (iso-octane), C_8H_{18}, HPLC grade, with a water content less than 100 mg/kg.

NOTE Care should be taken when using iso-octane, which is highly flammable.

4.3 Tetrahydronaphthalene (THN), $C_{10}H_{12}$, minimum purity 96 % (determination by gas chromatography area standard technique).

NOTE Care should be taken when using tetrahydronaphthalene which can be irritating to eyes and skin.

4.4 Hexane, C_6H_{14}.

4.5 Toluene, C_7H_8.

5 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

5.1 High performance liquid chromatograph, suitable for analysis according to the operating instructions given in Table 1.

5.2 Chromatographic column, normal phase, 250 mm length, 4 mm internal diameter containing micro-particulate silica.

5.3 Suitable means, for determining peak area, e.g. integrator or computer.

5.4 Disposable filter, 0,5 µm pore diameter.

6 Procedure

6.1 Preliminary test

The column length (1 column or 2 columns in series) and the flow rate shall be arranged in order to optimise the efficiency and resolution (see Table 2 for column details). One way to test this is to run an HPLC chromatogram using a standard solution containing approximately 100 mg of toluene (4.5) and 10 mg of tetrahydronaphthalene (4.3) (THN) in 100 ml of hexane (4.4). An example of a chromatogram is given in Figure 1.

Do not wash the columns with any polar solvent otherwise the original separation factor is not to be restored any more.

From this chromatogram determine the net retention time (t') and the capacity factor (k') of both THN and toluene and calculate the relative separation factor (α).

NOTE Typical values for two Lichrosorb Si 60 columns and 1 ml/min flow rate are shown in Table 1.

Table 1 - Net retention time and capacity factor for toluene and THN for two Lichrosorb Si 60 columns and a flow rate of 1 ml/min

Reagent	Net retention time (min) $t' = t - t_o$	Capacity factor $k' = \frac{t - to}{t_o}$
Toluene	4,8	0,94
THN	6,3	1,24
NOTE t_o is the duration of elution at the dead volume. A typical value of t_o for n-hexane is 5,1 min.		

The calculated separation factor, $\alpha = \frac{k_2'}{k_1'}$, is:

$$\alpha = \frac{k'_{THN}}{k'_{Toluène}} = 1{,}32$$

A reproducible and correct DAT analysis in LAB samples requires that the HPLC conditions are set up so that α is greater than or equal to 1,20.

6.2 Calibration

Prepare a solution of 0,1 g/l of THN in 2,2,4-trimethylpentane (4.2) by weighing 100 mg of the tetrahydronaphthalene (4.3) to the nearest 0,1 mg and making up to volume in an 1 l volumetric flask or by dilution of a more concentrated solution.

NOTE A reference sample of LAB containing 1,2 % DAT is available from ECOSOL [1)] for checking the calibration of the method.

Analyse the solution by HPLC according to the conditions given in Table 2. The peak area for the THN peak is used for the calculation in clause 7.

If the mean molar mass is unknown, determine the carbon number distribution of the *LAB* sample to be analysed by a gas chromatography peak area normalisation technique, for example, as described in Annex A.

Calculate the average molar mass, M_{DAT}, of the DAT in the sample as follows:

$$M_{DAT} = \frac{100}{\sum_{i=8}^{15} \frac{W_{LAB}(i)}{M_{DAT}(i)}} \qquad (1)$$

where

$W_{LAB(i)}$ is the mass fraction of LAB at carbon number i of the alkyl chain, in percent;

$M_{DAT(i)}$ is the molar mass of DAT at carbon number i of the alkyl chain, in grams per mole.

6.3 Determination

Prepare a solution of 10 g/l of the sample in 2,2,4-trimethylpentane (4.2) by weighing 250 mg of the sample to the nearest 0,1 mg and making up to volume in a 25 ml volumetric flask.

Filter approximately 5 ml of the above solution through the disposable filter (5.4).

Analyse the filtered solution by HPLC according to the conditions given in Table 2.

NOTE An example of a chromatogram is given in Figure 2.

Use tangent skim technique for determining the area of the DAT peak.

1) ECOSOL is a sector group of CEFIC/CESIO (Avenue E. VAN NIEUWENHUYSE 4, bte 2 B - 1160 Brussels, Belgium).

Table 2 - Operating instructions

Column	
Material	Stainless steel
Length x internal diameter	250 mm x 4 mm
Packing	Microparticulate silica, 5 µm
Mobil phase	
Solvent	2,2,4-trimethylpentane
Flow rate	0,5 ml/min - 1 column (see clause 6)
	1,0 ml/min - 2 columns (see clause 6)
Programme	Isocratic for 30 min
Detector system	
Type	Ultra-violet absorbance
Wavelength	254 nm
Temperature	Room temperature
Injection volume	20 µl