SLOVENSKI STANDARD
SIST EN 14108:2003

01-november-2003

Derivati maġļ ob in olj - Metil estri maġļ obnih kislin (FAME) - Doloļ evanje natrija z atomsko absorpcijsko spektrometrijo

Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of sodium content by atomic absorption spectrometry

Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester (FAME) - Bestimmung des Natriumgehaltes durch Atomabsorptionsspektrometrie

Produits dérivés des corps gras - Esters méthyliques d'acides gras (EMAG) - Détermination de la teneur en sodium par spectrométrie d'absorption atomique

Ta slovenski standard je istoveten z: **EN 14108:2003**

ICS:

67.200.10	Rastlinske in ġivalske maġļ obe in olja	Animal and vegetable fats and oils

SIST EN 14108:2003 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14108

April 2003

ICS 67.200.10

English version

Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of sodium content by atomic absorption spectrometry

Produits dérivés des corps gras - Esters méthyliques d'acides gras (EMAG) - Détermination de la teneur en sodium par spectrométrie d'absorption atomique

Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester (FAME) - Bestimmung des Natriumgehaltes durch Atomabsorptionsspektrometrie

This European Standard was approved by CEN on 2 January 2003.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Foreword

This document (EN 14108:2003) has been prepared by Technical Committee CEN/TC 307 "Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis", 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 October 2003 and conflicting national standards shall be withdrawn at the latest by October 2003.

This document has been prepared under Mandate M/245 on Fatty Acid Methylester (FAME) given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

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

1 Scope

This European standard specifies a method for the determination of sodium contents equal to or greater than 1 mg/kg.

This method is applicable to fatty acid methyl esters intended for addition to mineral oils.

WARNING — The use of this method may involve hazardous equipment, materials and operations. This method does not purport to address to all of the safety problems associated with its use, but it is the responsibility of the user to search and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Principle

The vegetable oil methyl ester sample is diluted with a xylene solution.

The sodium content in the sample is directly determined by flame atomic absorption spectrometry at the wavelength of 589 nm. The calibration solutions used are prepared from a sodium organometallic salt dissolved in a mixture of xylene and stock oil. The addition of stock oil to the calibration solutions is necessary in order to improve their storage (the low element contents are unstable) and the linearity of the calibration.

WARNING — The ester sample shall be diluted at least 25 times with xylene so that the comparison of the measurements of the sample solution and standards is valid.

NOTE Xylene can be replaced by cyclohexane or light petroleum in those laboratories that are not authorized to use aromatic solvents.

3 Reagents

Use only reagents of recognised analytical grade, unless otherwise specified.

3.1 Recommendations for washing glassware

The glassware used for the preparation of the solutions shall be washed at least twice with an approximate 5 mol/l solution of hydrochloric acid, rinsed with distilled water then dried in order to avoid sodium pollution.

3.2 Xylene (mixture of isomers)

WARNING — Inflammable and noxious.

3.3 Stock oil 75 (75 mm^2/s)[1)]

3.4 Stock oil solution in xylene (200 g/l); dilute in a 200 ml volumetric flask, 40 g of stock oil (3.3) with xylene. Store this solution in a polypropylene bottle.

3.5 Sodium, solution in oil (5 000 mg/kg)[1)]. Ready-to-use solution of a sodium organometallic salt in stock oil, having a certified titre.

3.6 Sodium, solutions of intermediary dilutions for the preparation of the set of calibration solutions.

---

1) Products available commercially from CONOSTAN Standard, supplied by Conostan Division, Continental Oil Co, Ponca City, OK 74601 – USA.
SPEX Standard supplied by SPEX Industries, Inc. Chemical Sales Department, 3880 Park Avenue, Edison, NJ 08820 - USA.
This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of these products.

3.6.1 Sodium, solution in xylene (500 mg/l): weigh, to within 0,001 g, approximately 2,5 g of the sodium solution (3.5) in a 25 ml volumetric flask and dilute to the mark with xylene.

This solution may be stored for a month.

3.6.2 Sodium, solution in xylene (5 mg/l): sample, using a pipette, 0,50 ml of the sodium standard solution (3.6.1), transfer into a 50 ml volumetric flask and dilute to the mark with xylene.

Prepare a new solution each day.

NOTE The solutions of intermediary dilutions may be prepared in glass flasks (the sodium pollution being negligible at these contents).

4 Apparatus

4.1 Atomic absorption spectrometer

Any atomic absorption spectrometer may be used provided that it is equipped with:

4.1.1 A hollow sodium cathode lamp.

4.1.2 A nebulization system suited for the organic solutions, of which the materials are solvent-resistant.

4.1.3 A burner head capable of being used with organic solutions and an air-acetylene flame.

4.2 **Balance**, with an accuracy of 1 mg.

4.3 Glassware

4.3.1 25 ml and 50 ml volumetric flasks.

4.3.2 0,5 ml graduated precision pipette.

4.4 Polypropylene ware

4.4.1 50 ml and 200 ml volumetric flasks.

4.4.2 10 ml pipette.

4.4.3 250 ml bottles

4.4.4 Automatic pipette having a variable volume of 1 ml to 5 ml, fitted with ejectable and disposable polypropylene tips.

5 Procedure

WARNING — In order to avoid polluting the sodium solutions, it is recommended to prepare all the determination solutions in polypropylene flasks and to conduct all the sampling operations using polypropylene pipettes or pipettes having disposable polypropylene tips. It is however possible to use glassware taking cleaning precautions in order to avoid pollution by the elements being analysed.

5.1 Sampling

Sampling is not part of the method spacified in this European Standard. A recommended sampling method is given in EN ISO 5555 [1].

5.2 Preparation of the sample

5.2.1 Weigh to within 0,001 g approximately 2 g (mass m in g) of ester sample in a 50 ml volumetric flask (volume V in ml) and dilute to the mark with xylene.

5.2.2 Sample two test portions per sample.

5.3 Preparation of the set of calibration solutions

5.3.1 Prepare the calibrations solutions having the following sodium contents:

0,1 mg/l - 0,2 mg/l - 0,3 mg/l.

5.3.2 Using a variable volume automatic pipette, transfer 1,00 ml, 2,00 ml and 3,00 ml of the 5 mg/l sodium solution (3.6.2) into 50 ml volumetric flasks.

5.3.3 With a polypropylene pipette, add to each flask 10 ml of the 200 g/l solution of stock oil in xylene (3.4) and dilute to the mark with xylene.

5.3.4 Prepare the zero member or blank test in the same manner without adding any sodium solution.

5.3.5 Prepare the calibration solutions just prior to their measurement on account of their instability.

5.4 Spectrometric measurements

5.4.1 Preparation of the spectrometer

— Set the wavelength at 589,0 nm and the band pass at 0,5 nm;

— conduct the aspiration of the 0,3 mg/l calibration solution in order to optimize the different instrument settings. Seek the maximum response of the signal by adjusting:

  — the air-acetylene gaseous mixture;

  — the aspiration speed of the solution;

  — the burner position.

— conduct the aspiration of the xylene placed in a polypropylene bottle in order to set the instrument at zero absorbance.

5.4.2 Calibration

Conduct the aspiration of the blank (or zero member) and calibration solutions and carry out three measurements for each of them.

Calculate for each solution the arithmetical mean of the three measurements.

Plot the calibration curve from the means obtained.

By way of indication, the value of the optical density obtained for a 0,3 mg/l concentration is approximately 0,290 and the curve is more or less a straight line in the range considered.

5.4.3 Samples

Conduct the aspiration of the solutions of the samples and carry out the measurements in the same way as for the standards.

NOTE In the case where the measurement of the sample exceeds the set of calibration solutions given in 5.3, prepare standards having higher sodium contents, the maximum content being 1 mg/l.

6 Expression of results

Determine the sodium contents c_1 and c_2 in mg/l of the two test portions of the sample, by referring to the calibration curve.

Calculate the sodium contents C_1 and C_2 of the sample, expressed in mg/kg, using the equation:

$$C = c \times \frac{V}{m}$$

where

m is the mass (in grams) of the sample test portion;

V is the volume of the sample solution (in millilitres).

Calculate the mean content C of sodium in the sample from C_1 and C_2.

Express the result in mg/kg, to the nearest 0,1 mg/kg.

7 Precision

An interlaboratory test organized in 2000 at European level with the participation of 13 laboratories, each having carried out two determinations on each sample, gave the statistical results indicated in annex A.

7.1 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short time interval, shall not be greater than:

$r = 0{,}086\ X + 0{,}242$

more than once out of 20 determinations.

7.2 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, shall not be greater than:

$R = 0{,}263\ X + 1{,}355$

more than once out of 20 determinations.

8 Test report

The test report shall specify:

— all information necessary for the complete identification of the sample;

— the sampling method used if known;

— the test method used, with reference to this European standard;

— all operating details not specified in this European Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

— the test result(s) obtained, or if the repeatability has been checked, the final quoted result obtained.