SLOVENSKI STANDARD
SIST EN 14107:2003

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Derivati maġļ ob in olj - Metil estri maġļ obnih kislin (FAME) - Doloļ evanje fosforja z emisijsko spektrometrijo z induktivno sklopljeno plazmo (ICP)

Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of phosphorus content by inductively coupled plasma (ICP) emission spectrometry

Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester (FAME) - Bestimmung des Phosphorgehaltes durch Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP)

Produits dérivés des corps gras - Esters méthyliques d'acides gras (EMAG) - Détermination de la teneur en phosphore par spectrométrie d'émission de plasma induit par haute fréquence (méthode ICP)

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

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EUROPEAN STANDARD

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Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester (FAME) - Bestimmung des Phosphorgehaltes durch Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP)

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

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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.

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

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Foreword

This document (EN 14107:2003) 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 Standardshall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2003, and conflicting national standards shall be withdrawn at the latest by September 2003..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.

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 an inductively coupled plasma (ICP) emission spectrometry method for the determination of phosphorus content between 4 mg/kg and 20 mg/kg in Fatty Acid Methyl Esters, hereinafter referred as FAME.

This method aims to evaluate the FAME quality, in terms of transesterification by-products content such as phosphorus, whose concentration may affect the fuel behaviour.

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 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 ISO 661, *Animal and vegetable fats and oils - Preparation of test portion (ISO 661:1989).*

3 Terms and definitions

For the purposes of this European Standard, the following term and definition applies.

3.1
phosphorus content
residual phosphorus remaining in FAME from phospholipids after the vegetable oil transesterification reaction

4 Principle

A weighed test portion of sample is diluted in xylene. The standards are prepared from a phosphorus organic compound dissolved in a mineral oil and diluted in a mixture of xylene and stock oil. The addition of stock oil makes it possible to reduce the differences in viscosity between samples and standards and improves their storage.

The solutions are introduced in aerosol form into an inductively coupled argon plasma. The phosphorus content is determined by comparing the emission of the element in the solution of the test portion of the sample with the emission of the standards at the same wavelength.

NOTE 1 The wavelengths commonly used are 213,6 nm and 178,3 nm.

The sample shall be diluted at least ten times with xylene in order to allow a proper introduction of the aerosol into the plasma.

NOTE 2 Xylene may be replaced by other suitable solvents in case of instable plasma or specific laboratory safety use.

5 Reagents

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

5.1 Recommendation for washing glassware : in order to avoid phosphorus pollution due to the phosphates contained in the detergents used for washing the glassware, rinse the latter at least twice with an approximate 5 mol/l solution of hydrochloric acid. Next rinse with distilled water then dry.

5.2 Xylene (mixture of isomers).

WARNING Inflammable and noxious.

5.3 Stock oil 75[1)] (viscosity 75 mm^2/s).

5.4 Stabilizer [1)], optional, to stabilise the stock solution.

5.5 Phosphorus organic standard [1)], 1000 mg/kg.

NOTE Other suitable commercially available standard can also be used.

5.6 Phosphorus, intermediate dilution solution for preparation of the set of calibration solutions : 100 mg/l phosphorus solution containing 0,6 % of stabilizer (5.4) : weigh to within 0,001 g approximately 5 g of the phosphorus solution (5.5) in a 50 ml volumetric flask. Add approximately 0,3 g of stabilizer (5.4), dilute to the mark with xylene and homogenise the solution. The exact titre of this solution will be calculated and subsequently used for the standards. This solution may be kept during one month, if the stabilizer (5.4) is used.

In case stabilizer is not used, the intermediate dilution solution of phosphorus cannot be kept.

NOTE The volumetric flasks can be replaced by disposable non calibrated flasks; in this case, the dilutions of the samples are prepared and expressed in mass/mass instead of mass/volume.

6 Apparatus

6.1 An inductively coupled plasma emission spectrometer equipped with the following elements :

6.1.1 A quartz torch.

6.1.2 A nebulizer (device which transforms the solution into an aerosol).

6.1.3 A peristaltic pump is necessary. The pumping speed shall be between 0,5 ml/min and 3 ml/min inclusive. Viton®[2)] flow rate tubes are recommended in order to withstand the xylene.

6.2 Balance, with an accuracy of 0,1 mg.

6.3 Glassware : 25 ml and 50 ml volumetric flasks.

6.4 Graduated pipettes, of 1 ml and 5 ml capacity or Variable volume automatic pipettes fitted with disposable polypropylene tips.

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1) Possible suppliers :
CONOSTAN Division, Continental Oil Co, Ponca City, OK 74706 – USA
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. Equivalent products may be used if they can be shown to lead to the same results.

2) Viton is a trade name of flow rate tubes.
This information is given for the convenience of users of this European standard and does not constitute an endorsement by CEN of this product. Equivalent products may be used if they can be shown to lead to the same results.

7 Sampling

It is important that the laboratory receive a sample, which is truly representative and has not been damaged or changed during transport or storage.

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

8 Preparation of test portion

Prepare the test portion in accordance with EN ISO 661. The test portion shall not be heated and/or filtered.

9 Procedure

9.1 Sample preparation : in order to avoid phosphorus pollution of the solutions, all sampling operations are carried out using polypropylene pipettes or automatic pipettes having disposable tips.

Weigh to within 0,001 g approximately 2,5 g (mass m in g) of sample in a 25 ml volumetric flask (volume *V* in ml), dilute to the mark with xylene and shake manually to homogenise.

Take two test portions per sample.

NOTE The volumetric flasks may be replaced by disposable non calibrated flasks; in this case, the dilutions of the samples are prepared and expressed in mass/mass instead of mass/volume.

9.2 Preparation of the standards : prepare calibration solutions having the following phosphorus contents : 0 mg/l, 0,5 mg/l, 1 mg/l, 2 mg/l, 4 mg/l.

The exact contents of each standards shall be calculated according to the exact concentration of phosphorus intermediate solution (see 5.6).

NOTE 1 The following procedure is given as an example :

In five 100 ml volumetric flasks, weigh 10 g of stock oil (5.3). Using graduated pipettes, transfer 0 ml, 0,5 ml, 1 ml, 2 ml, 4 ml of the 100 mg/l phosphorus solution (see 5.6). Fill up to the mark with xylene and homogenise the solutions.

WARNING — The solutions shall be freshly prepared for each series of analyses.

NOTE 2 When dilutions of the samples are expressed in % (*m/m*) (see Note in 5.6), the calibration solutions are prepared in order to be expressed in the same way.

9.3 Preparation of the appliance : since appliances stemming from diverse manufacturers have different configurations and settings, it is difficult to specify an exact procedure. Follow the manufacturer's instructions for using the instrument with organic solvents.

The choice of the instrumental parameters is determined so as to obtain the best signal / background ratio.

9.4 Measurement : the measurement of the intensity of the analytical line corresponds to the counting of the maximum of the line deducted from the counting of the background. Certain instruments are equipped with software which allows the automatic correction of the background.

9.5 Calibration : conduct the aspiration of both the blank and calibration solutions. Carry out three measurements for each of them.

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

Construct the calibration curve from these measurements using linear regression, by plotting the emission intensity values against the values of the respective phosphorus concentrations expressed in mg/l.

NOTE If the plot of the emission intensity values against the values of the phosphorus contents is not linear, the procedure should be inspected for errors and, if necessary, the calibration procedure should be repeated starting with clause 9.2.

9.6 Sample analysis : conduct the aspiration of the solution of the sample. Carry out the measurements in the same way as for the standards.

NOTE The drift has to be checked regularly.

10 Results

Determine, according to the calibration curve, the phosphorus concentration c (mg/l) of the two test portions of a same sample.

Calculate the phosphorus concentration C of the sample, expressed in mg/kg, using the equation:

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

where

V is the volume of the sample solution (millilitres);

c is the phosphorus concentration of the sample solution (milligrams per litre);

m is the mass of the sample test portion (grams).

Calculate the phosphorus content C of the sample by taking the arithmetical mean of the two values obtained. Express the result in mg/kg to the nearest 0,1 mg/kg.

NOTE When the solutions are expressed in as mass fraction in percent (see Note in 5.6 and Note 2 in 9.2), the calculation of the phosphorus content C of the sample is made using the equation :

$$C = c_p \times \frac{P}{m}$$

where

c_p is the concentration of the sample solution (milligrams per kilogram);

P is the total mass of the solution (grams).

11 Precision

11.1 Interlaboratory test

Details of an interlaboratory are given in annex A. The values derived from these tests may not be applicable to concentration ranges and matrices other than those given.

11.2 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: 0,6 mg/kg more than once out of 20 determinations.

11.3 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:

$R = 0{,}192\ C + 0{,}025$

more than once out of 20 determinations.

12 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.