BRITISH STANDARD

BS EN
14109:2003

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

The European Standard EN 14109:2003 has the status of a British Standard

ICS 67.200.10

National foreword

This British Standard is the official English language version of EN 14109:2003.

The UK participation in its preparation was entrusted to Technical Committee AW/307, Oilseeds animal & vegetable fats and oils, 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.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the *BSI Catalogue* under the section entitled "International Standards Correspondence Index", or by using the "Search" facility of the *BSI Electronic Catalogue* or of British Standards Online.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 15 May 2003

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 9 and a back cover.

The BSI copyright notice displayed in this document indicates when the document was last issued.

Amendments issued since publication

Amd. No.	Date	Comments

ISBN 0 580 41841 3

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14109

April 2003

ICS 67.200.10

English version

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

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

Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester (FAME) - Bestimmung des Kaliumgehaltes 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

Ref. No. EN 14109:2003 E

Foreword

This document (EN 14109:2003) 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 potassium contents equal to or greater than 0,5 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 and a stabilizer.

The potassium content in the sample is directly determined by flame atomic absorption spectrometry at the wavelength of 766,5 nm. The calibration solutions used are prepared from a potassium organometallic salt dissolved in a mixture of xylene and stabilizer. The addition of a stabilizer 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 ten 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 which are not authorized to use aromatic solvents.

3 Reagents

Use only reagents of recognized 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 potassium pollution.

3.2 Xylene, mixture of isomers.

WARNING — Inflammable and noxious.

3.3 Stabilizer, supplied by CONOSTAN[1)].

3.4 Stabilizer solution in xylene, 100 g/l.

Dilute in a 200 ml polypropylene volumetric flask, 20 g of stabilizer (3.3) with xylene. Store this solution in a polypropylene bottle.

3.5 Potassium, solution in oil, 5 000 mg/kg.

Ready-to-use solution of a potassium organometallic salt in stock oil, having a certified titre [2)].

1) Products available commercially from CONOSTAN Standard, supplied by Conostan Division, Continental Oil Co, Ponca City, OK 74601 – USA.

2) Conostan (see above) or 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 Potassium, solution of intermediary dilutions for the preparation of the set of calibration solutions.

3.6.1 Potassium, solution in xylene, 500 mg/l.

Weigh, to within 0,001 g, approximately 2,5 g of the potassium solution (3.6) in a 25 ml volumetric flask and dilute to the mark with xylene. This solution can be stored for a month.

3.6.2 Potassium, solution in xylene, 5 mg/l.

Sample, using a pipette, 0,50 ml of the potassium 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 potassium 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 potassium 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 5 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 potassium 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 specified in this European Standard. A recommended sampling method is given in EN ISO 5555 [1].

5.2 Preparation of the sample

Weigh to within 0,001 g approximately 5,0 g (mass *m* in g) of ester sample in a 50 ml volumetric flask (volume *V* in ml).

Using a polypropylene pipette, add to the flask 5,0 ml of the 100 g/l solution of stabilizer in the xylene (see 3.4) and dilute to the mark with xylene.

Sample two test portions per sample.

5.3 Preparation of the set of calibration solutions

Prepare the calibrations solutions having the following potassium contents:

0,1 mg/l - 0,2 mg/l - 0,3 mg/l - 0,5 mg/l

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

With a polypropylene pipette, add to each flask 5 ml of the 100 g/l solution of stabilizer in xylene (see 3.4) and dilute to the mark with xylene.

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

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 766,5 nm and the bandpass at 1 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 0,095 and the curve is more or less a straight line the range being exploited.

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 potassium contents, the maximum content being 2 mg/l.

6 Expression of results

Determine the potassium 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 potassium 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 potassium 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 = 0,3 mg/kg 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 = 0,505 X + 0,522

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.

Annex A
(informative)

Results of an interlaboratory trial

A European collaborative test involving 13 laboratories in 6 countries was carried out on 5 samples:

Sample 1 : FAME prepared from mixture of sunflower and rapeseed oils (40:60);

Sample 2 : FAME prepared from rapeseed oil;

Sample 3 : FAME prepared from sunflower oil;

Sample 4 : FAME prepared from mixture of sunflower and rapeseed oils (80:20);

Sample 5 : FAME prepared from rapeseed oil.

The test was organized by CEN TC 307/WG1 in 2000 and the results obtained were subjected to statistical analysis in accordance with EN ISO 4259 [2] to give the precision data shown in Table A.1.

Table A.1

Sample	1	2	3	4	5
N° of participating laboratories	13	13	13	13	13
N° of participating laboratories after eliminating outliers	8	11	12	12	12
Mean value (mg/kg)	**0,15**	**0,50**	**5,60**	**4,42**	**2,49**
Repeatability standard deviation (mg/kg)	0,13	0,07	0,08	0,09	0,06
Reproducibility standard deviation (mg/kg)	0,14	0,26	1,04	0,92	0,64
Repeatability limit, r (mg/kg)	0,43	0,21	0,23	0,28	0,19
Reproducibility limit R (mg/kg)	0,45	0,80	3,21	2,83	1,98

Bibliography

[1] EN ISO 5555, *Animal and Vegetable fats and oils – Sampling* (ISO 5555:2001).

[2] EN ISO 4259, *Petroleum products - Determination and application of precision data in relation to methods of test (ISO 4259:1992/Cor 1:1993).*

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: +44 (0)20 8996 9000. Fax: +44 (0)20 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: +44 (0)20 8996 9001. Fax: +44 (0)20 8996 7001. Email: orders@bsi-global.com. Standards are also available from the BSI website at http://www.bsi-global.com.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: +44 (0)20 8996 7111. Fax: +44 (0)20 8996 7048. Email: info@bsi-global.com.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: +44 (0)20 8996 7002. Fax: +44 (0)20 8996 7001. Email: membership@bsi-global.com.

Information regarding online access to British Standards via British Standards Online can be found at http://www.bsi-global.com/bsonline.

Further information about BSI is available on the BSI website at http://www.bsi-global.com.

Copyright

BSI
389 Chiswick High Road
London
W4 4AL