



SLOVENSKI STANDARD

SIST EN 16300:2012

01-november-2012

Goriva za motorna vozila - Določevanje jodnega števila - Računska metoda iz podatkov plinske kromatografije

Automotive fuels - Determination of iodine value - Calculation method from gas chromatographic data

Kraftstoffe für Kraftfahrzeuge - Bestimmung der Iodzahl - Berechnung aus gaschromatografischen Daten

Carburants pour automobiles - Détermination de la valeur iodique - Méthode de calcul par dates de chromatographie en phase gazeuse

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ICS:

75.160.20 Tekoča goriva Liquid fuels

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EUROPEAN STANDARD

EN 16300

NORME EUROPÉENNE

EUROPÄISCHE NORM

September 2012

ICS 75.160.20

English Version

Automotive fuels - Determination of iodine value in fatty acid methyl esters (FAME) - Calculation method from gas chromatographic data

Carburants pour automobiles - Détermination de l'indice d'iode dans les esters méthyliques d'acides gras - Méthode de calcul à partir des données obtenues par chromatographie en phase gazeuse

Kraftstoffe für Kraftfahrzeuge - Bestimmung der Iodzahl in Fettsäure-Methylester (FAME) - Berechnung aus gaschromatographischen Daten

This European Standard was approved by CEN on 27 July 2012.

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 16300:2012) has been prepared by Technical Committee CEN/TC 19 "Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin", the secretariat of which is held by NEN.

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of an identical text or by endorsement, at the latest by March 2013, and conflicting national standards shall be withdrawn at the latest by March 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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Introduction

This European Standard has been developed in CEN/TC 19/JWG 1. This document is a self-contained European test method based on the provisions given in Annex B of EN 14214:2008 [2]. Some markets need a full test method to check FAME for compliance with EN 14214.

The method is not intended as a replacement for EN 14111 [1] (see also Scope).

The method is based on results for individual FAME components as determined by EN 14103. On the basis of EN 14103 up to the C24 esters can be determined and a calculation can be done. The correlation is well within the repeatability of the existing test method.

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1 Scope

This European Standard specifies a calculation procedure for the determination of Iodine value (“CIV” - “calculated iodine value”), of fatty acid methyl esters (FAME) to be used either as automotive or heating fuel for diesel engines as specified in EN 14214 [2] or as an extender for automotive fuel for diesel engines as specified in EN 590 [4].

This procedure has originally been described in Annex B of EN 14214:2008 [2]. The calculation procedure is now specified for methyl esters between C14 and C24. The calculation procedure uses as data entry the results from the gas chromatography determination (GC) according to EN 14103 of individual fatty acid methyl esters and is based on AOCS recommended practice Cd 1c – 85 for the determination of the iodine value of edible oil from its fatty acid composition. It is important to recognise that the latest version of EN 14103 is to be used for the determination of individual FAME components.

NOTE 1 Experience from the field and from several precision evaluation campaigns in Germany and elsewhere indicates that the results of the determination of iodine value by calculation specified here are very close to results obtained by titration with Wijs solvent according to EN 14111. Observed small differences were always found to be smaller than the reproducibility published in the actual EN 14111.

For informative purposes only, but not for cases of dispute, EN 14331 [5] may also be used to extract the FAME contents from FAME containing diesel fuels (like B5, B7, B30, etc.) and to use the contents of the individual FAME components from this method as data entry for the calculation specified in this European Standard.

In principle, other fatty acid *alkyl* esters can also be analysed. However, neither the close correlation to the titration method EN 14111 has been verified nor is any precision information available for such an extension of application range.

NOTE 2 For the purposes of this European Standard, the term “% (m/m)” is used to represent the mass fraction, μ , of a material.

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2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 14103, *Fat and oil derivatives — Fatty Acid Methyl Esters (FAME) — Determination of ester and linolenic acid methyl ester contents*

3 Procedure

The contents of individual fatty acid methyl esters (FAME) are determined according to the GC-determination of C18:3 which is specified in EN 14103. The contents, w_i , expressed in % (m/m), obtained for each of the individual FAMEs (i) are then used to calculate the contributions to the iodine value from each FAME component as given in formula (1), using the contribution factors given in Table 1. The calculated iodine value (CIV) is then determined as the sum of the individual contributions, disregarding (i.e. without inclusion of) the FAME component used as internal standard in EN 14103. An example for this calculation is given Table 2.

$$CIV = \sum_i \mu_i \cdot C_i \quad (1)$$

where

CIV is the calculated Iodine value of the sample, given in g Iodine per 100 g sample;

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μ_i is the content of individual FAME component i , given in % (m/m);

C_i is the contribution factor from FAME component i , listed in Table 1.

Table 1 — Contribution factors C_i for individual commonly appearing FAME components

FAME component ^a	FAME designator	Molecular mass M	Number of olefinic double bonds n_i	Contribution factor C_i
All saturated FAMEs	Cn:0	n.a.	0	0,000
Methyl myristoleate	C14:1	240,389	1	1,056
	C14:2	238,373	2	2,130
Methyl palmitoleate	C16:1	268,43	1	0,946
	C16:2	266,42	2	1,905
Methyl heptadecenoate	C17:1	282,46	1	0,899
	C17:2	280,45	2	1,810
Methyl oleate	C18:1	296,45	1	0,856
	C18:2	294,47	2	1,724
Methyl linoleate	C18:2	294,47	2	1,724
Methyl linolenate	C18:3	292,46	3	2,604
Methyl eicosenoate	C20:1	324,54	1	0,782
	C20:2	322,53	2	1,574
	C20:3	320,51	3	2,376
Methyl docosenoate	C22:1	352,59	1	0,720
	C22:2	350,58	2	1,448
	C22:3	348,56	3	2,184
Methyl tetracosenoate	C24:1	380,65	1	0,667
	C24:2 ^b	378,63	2	1,341
	C24:3 ^b	376,62	3	2,022
	C24:4 ^b	374,60	4	2,710

NOTE 1 Molecular weights are calculated using 12,01 (for C); 1,01 (for H); 16,00 (for O) and 253,81 (for I₂) (all in g/mol).

NOTE 2 Attention is also drawn to the fact that for many FAME components like e.g. C18:2, there may be more than one isomer in the sample. All present isomers should be securely identified and included in the calculation.

^a Other isomers are possible.

^b C 24:2, C24:3 and C24:4 are not determined by EN 14103. Factors are given for information only.

The contribution factors C_i listed in Table 1 for the individual FAME components i have been calculated according to Formula (2):

$$C_i = 253,81 \cdot n_i / M_i \quad (2)$$

where

253,81 is the molecular weight for the iodine molecule (I₂);

n_i is the number of olefinic double bonds in FAME component i ;

M_i is the molecular mass for the FAME component i (methyl ester).

Table 2 — Numeric example

Example of component / isomer	Double bond designation (FA Chain)	Contribution factor C_i	Content of FAME (i) μ_i % (m/m)	Effective contribution from FAME (i)
	C14:0	0,000	0,30	0,00
	C14:1	1,056	0,00	0,00
	C14:2	2,130	0,00	0,00
Methyl palmitoleate	C16:0	0,000	0,40	0,00
	C16:1	0,946	1,10	1,04
	C16:2	1,905	0,00	0,00
	C17:1	0,899	0,00	0,00
	C17:2	1,810	0,00	0,00
Methyl stearate	C18:0	0,000	2,00	0,00
Methyl oleate	C18:1	0,856	61,60	52,73
Methyl linoleate	C18:2	1,724	20,10	34,65
Methyl linolenate	C18:3	2,604	9,40	24,47
Methyl eicosenoate	C20:0	0,000	0,40	0,00
	C20:1	0,782	0,70	0,55
	C20:2	1,574	0,00	0,00
	C20:3	2,376	0,00	0,00
Methyl docosenoate	C22:0	0,000	0,70	0,00
	C22:1	0,720	1,10	0,79
	C22:2	1,448	0,00	0,00
	C22:3	2,184	0,30	0,66
Methyl tetracosenoate	C24:1	0,667	0,00	0,00
	C24:2	1,341	0,00	0,00
	C24:3	2,022	0,00	0,00
	C24:4	2,710	0,30	0,81
Total ester content, % (m/m)			98,40	
Calculated Iodine Value, (CIV), g I ₂ /100 g				115,7

4 Expression of results

The calculated iodine value (CIV) shall be reported rounded to the nearest 0,1 g Iodine/100 g.

5 Precision

5.1 Repeatability, r

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values given by Formula (3) in only one case in twenty.

$$r = 0,003 X + 0,544 7 \quad (3)$$