SLOVENSKI STANDARD
oSIST prEN 16300:2023

01-februar-2023

Goriva za motorna vozila - Določanje jodnega števila v metilnih estrih maščobnih kislin (FAME) - Računska metoda iz podatkov plinske kromatografije

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

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

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

Ta slovenski standard je istoveten z: **prEN 16300**

ICS:

75.160.20	Tekoča goriva	Liquid fuels

oSIST prEN 16300:2023 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 16300

January 2023

ICS 75.160.40

Will supersede EN 16300:2012

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 des esters méthyliques d'acides gras (EMAG) - 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 draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 19.

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

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European foreword

This document (prEN 16300:2023) 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 document is currently submitted to the CEN Enquiry.

This document will supersede EN 16300:2012.

In comparison with the previous edition, the following technical modifications have been made:

a) addition of a calculation of the pattern of fatty acid methyl esters;

b) re-calculated precision statement following the revision of EN 14103;

c) document revised editorially.

1 Scope

This document 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 fuel for diesel engines as specified in EN 14214 [2] or heating fuel or as an extender for automotive fuel for diesel engines as specified in EN 590 [3]. Ethyl esters or esters made from fish oil and mixtures thereof are not covered by this procedure.

The calculation procedure is specified for methyl esters between C6 and C24:1. 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 recognize that the latest version of EN 14103 is intended to be used for the determination of individual FAME components.

NOTE 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 the calculation specified here are very close to results obtained by titration with Wijs solvent according to EN 14111 [1]. 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 [4] 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 document.

This calculation method can be used only if the evaluated sample fulfils the requirement for ester content as reported in EN 14214.

The precision statement of this test method was determined by calculation from a Round Robin exercise with iodine values in the range of 16 g iodine/100 g to 126 g iodine/100 g.

The test method is also applicable for higher iodine values; however, the precision statement is not established for iodine values above 126 g iodine/100 g.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at https://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

4 Procedure

The contents of individual fatty acid methyl esters (FAME) shall be determined according to the GC-determination which is specified in EN 14103. Annex C of EN 14103 describes how the calculation of the pattern of fatty acid methyl esters and therefore each FAME shall be done. The contents, L_x, expressed in area %, obtained for each of the individual FAMEs (x) shall be 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 A.1. The calculated iodine value (CIV) shall be determined as the sum of the individual contributions, without inclusion of the FAME component used as internal standard in EN 14103. Unknown FAMEs are not taken into account because it is not clear if they are unsaturated. In case of any doubt, i.e. content of unknown FAMEs exceeds 3 %, a measurement according EN 14111 is recommended. An example for this calculation is given Table 1.

$$CIV = \sum_{x} L_x \cdot C_x \tag{1}$$

where

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

L_x is the content of individual FAME component x, given in area %;

C_x is the contribution factor from FAME component x, listed in Table 1.

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

FAME	Molecular mass Mx	Number of olefinic double bonds n_x	Contribution factor C_x
Cn:0	n.a.	0	0,000
C14:1	240,389	1	1,056
C14:2	238,373	2	2,130
C16:1	268,43	1	0,946
C16:2	266,42	2	1,905
C17:1	282,46	1	0,899
C17:2	280,45	2	1,810
C18:1	296,45	1	0,856
C18:2	294,47	2	1,724
C18:3	292,46	3	2,604
C20:1	324,54	1	0,782
C20:2	322,53	2	1,574
C20:3	320,51	3	2,376
C22:1	352,59	1	0,720
C22:2	350,58	2	1,448
C22:3	348,56	3	2,184
C24:1	380,65	1	0,667
C24:2 [a]	378,63	2	1,341
C24:3 [a]	376,62	3	2,022
C24:4 [a]	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 I_2) (all in g/mol).

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

[a] C24:2, C24:3 and C24:4 are not determined by EN 14103. Factors are given for information only.

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

$$Cx = 253,81 \cdot \frac{nx}{Mx} \quad (2)$$

where

253,81 is the molecular weight for the iodine molecule (I_2);

n_x is the number of olefinic double bonds in FAME component *x*;

M_x is the molecular mass for the FAME component *x* (methyl ester).

An example for the calculation is shown in Annex A.

5 Expression of results

Report the calculated iodine value (*CIV*) rounded to the nearest 1 g iodine/100 g.

6 Precision

6.1 General

The ILS conditions did not meet all requirements of EN ISO 4259 due to the fact that the samples did not cover the full range of the scope. However, field experience indicates that the precision statement is correct for the full range of the scope. The precision statement will be monitored and verified by field data.

6.2 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.

$$r = 0,0028\,X + 0,1342 \quad (3)$$

where

X is the result or the mean of two results to be compared, in units of g iodine/100 g.

6.3 Reproducibility, *R*

The difference between two single and independent test results, obtained by different operators working in different laboratories 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 (4) in only one case in twenty.

$$R = 0,0323\,X + 1,5485 \quad (4)$$

where

X is the result or the mean of two results to be compared, in units of g iodine/100 g.

7 Test report

The test report shall contain the following information:

a) a reference to this document, i.e. prEN 16300:2023;

b) the type and complete identification of the product tested;

c) result of the test (see Clause 5);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of the test.