ADDENDUM TO:

The certification of the mass fraction of the ester, linolenic acid methyl ester, monoglyceride, diglyceride, triglyceride, total glycerol and water content, density, viscosity, oxidation stability, acid value, iodine value and flash point of biodiesel: ERM®- EF001

Certification report

EUR 26711 EN

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Directorate F – Health, Consumers and Reference Materials

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Disclaimer

Certain commercial equipment, instruments, and materials are identified in this report to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the European Commission, nor does it imply that the material or equipment is necessarily the best available for the purpose.

Summary

This addendum to the certification report EUR 26711 EN [1] is concerning the update of the certificate of the certified biodiesel reference material ERM-EF001, whose properties are defined by measurement procedures that were partly outdated and adapted to newly revised measurement procedures. It describes the measures taken either to confirm the validity of certified values for editorially changed measurement procedures, or to certify properties with measurement procedures that were subject to fundamental technical changes or were newly published.

New certified values and uncertainties were assigned for the ester content, the linolenic acid methyl ester content, viscosity, and the iodine value. All properties were characterised by an interlaboratory comparison of laboratories of demonstrated competence using the newly revised measurement procedures and adhering to ISO/IEC 17025:2017 [2]. Technically invalid results were removed but no outlier was eliminated unless a technical reason for the deviation was found.

Uncertainties of the certified values were calculated in accordance with ISO 17034:2016 [3] and ISO Guide 35:2017 [4] and include uncertainties related to possible inhomogeneity and instability as reported in EUR 26711 EN [1] and uncertainties related to characterisation reported in this addendum.

Old measurement procedures on the certificate were replaced by new measurement procedures in the case of purely editorial modifications, with the assigned certified values and their uncertainties remaining unchanged. This applied to the oxidation stability, the flash point and the methanol content.

Before release of the updated certificate, the project was subjected to an internal peer-review.

The following values were assigned (implemented changes given in bold):

	Certified value [10]	Uncertainty [11]	Unit
Ester content [1]	**97.4**	**0.6**	[% (m/m)] [9]
Linolenic acid methyl ester content [1]	**8.52**	**0.09**	[% (m/m)] [9]
Ester content [2]	98.9	1.7	[% (m/m)] [9]
Linolenic acid methyl ester content [2]	8.82	0.16	[% (m/m)] [9]
Density (at 15 °C) [3]	883.20	0.04	[kg/m^3]
Viscosity (at 40 °C) [4]	**4.474**	**0.006**	[mm^2/s]
Oxidation stability (at 110 °C) [5]	9.8	0.5	[h]
Iodine value [6]	112	4	[g iodine/100 g]
Iodine value [7]	**107.3**	**1.9**	[g iodine/100 g]
Flash point [8]	181	14 [12]	[°C]

1) As defined by **EN 14103:2020**
2) As defined by EN 14103:2011
3) As defined by EN ISO 12185:1996
4) As defined by **EN ISO 3104:2020**
5) As defined by **EN 15751:2014** and **EN 14112:2020**
6) As defined by EN 14111:2003
7) As defined by **EN 16300:2012**
8) As defined by **EN ISO 3679:2015**
9) As called in EN 14103:2011 and **EN 14103:2020**, which is equivalent to 10^{-2} g/g
10) Certified values are values that fulfil the highest standards of accuracy. The given values represent the unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory. The certified value and its uncertainty are traceable to the International System of Units (SI).
11) The uncertainty of the certified value is the expanded uncertainty with a coverage factor $k = 2$ corresponding to a level of confidence of 95 %, estimated in accordance with ISO 17034:2016 and ISO Guide 35:2017.
12) The uncertainty of the certified value is the expanded uncertainty with a coverage factor $k = 2.8$ corresponding to a level of confidence of 95 %, estimated in accordance with ISO 17034:2016 and ISO Guide 35:2017.

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Glossary

ANOVA	Analysis of variance
c_{CRM}	Certified value
CEN	European Committee for Standardization
c_{meas}	Mean measured value
CRM	Certified reference material
EN	European norm (standard)
ERM®	Trademark owned by the European Commission; used by the JRC for reference materials
FAME	Fatty acid methyl ester
GC	Gas chromatography
GUM	Guide to the Expression of Uncertainty in Measurement
ISO	International Organization for Standardization
JRC	Joint Research Centre of the European Commission
k	Coverage factor
n	Number of replicate analysis per unit
p	Number of technically valid datasets
rel	Index denoting relative figures (uncertainties etc.)
RSD	Relative standard deviation
r	Repeatability limit
R	Reproducibility limit
$s_{between}$	Standard deviation between groups as obtained from ANOVA; an additional index "rel" is added as appropriate
SI	International System of Units
s_L	Standard deviation between laboratories
s_r	Repeatability standard deviation
s_R	Reproducibility standard deviation
s	Standard deviation
s_{within}	Standard deviation within groups as obtained from ANOVA; an additional index "rel" is added as appropriate
t	Two-tailed Student t value at the 95 % confidence level
u_{bb}	Standard uncertainty related to a possible between-unit inhomogeneity; an additional index "rel" is added as appropriate
u_{char}	Standard uncertainty of the material characterisation; an additional index "rel" is added as appropriate
u_{CRM}	Combined standard uncertainty of the certified value; an additional index

	"rel" is added as appropriate
U_{CRM}	Expanded uncertainty of the certified value; an additional index "rel" is added as appropriate
u_{Δ}	Combined standard uncertainty of measurement result and certified value
u_{lts}	Standard uncertainty of the long-term stability; an additional index "rel" is added as appropriate
u_{meas}	Standard measurement uncertainty
U_{meas}	Expanded measurement uncertainty
u_{sts}	Standard uncertainty of the short-term stability; an additional index "rel" is added as appropriate
Δ_{meas}	Absolute difference between mean measured value and the certified value
VMR	Verification measurement results

1 Introduction

1.1 Background

In 2014, ERM-EF001, a biodiesel material based on 100 % rapeseed oil fatty acid methyl ester, was certified for selected parameters of EN 14214:2012 [5]. The certified properties are operationally defined measurands and can only be obtained by following the measurement procedures specified on the corresponding certificate of the certified reference material (CRM), i.e.

- the ester and linolenic acid methyl ester content as defined by EN 14103: 2011 [6];
- density as defined by EN ISO 12185:1996 [7];
- viscosity as defined by EN ISO 3104:1996 [8];
- oxidation stability as defined by EN 14112:2003 [9];
- iodine value as defined by EN 14111:2003 [10];
- flash point as defined by EN ISO 3679:2004 [11];
- methanol as defined by EN 14110:2003 (indicative value) [12].

These measurement procedures can always be subject to revision. Two of the measurement procedures remained unchanged since the release of ERM-EF001, i.e.

- density as defined by EN ISO 12185:1996 [7];
- iodine value as defined by EN 14111:2003 [10].

The other measurement procedures were either revised, i.e.

- the ester and linolenic acid methyl ester content as defined by EN 14103: 2020 [13];
- viscosity as defined by EN ISO 3104:2020 [14];
- oxidation stability as defined by EN 14112:2020 [15];
- flash point as defined by EN ISO 3679:2015 [16];
- methanol as defined by EN 14110:2019 [17];

or new measurement procedures related to some of the certified properties were published during the lifetime of the original project, or immediately thereafter, i.e.

- oxidation stability as defined by EN 15751:2014 [18];
- iodine value as defined by EN 16300:2012 [19].

The modifications in a new revision can often only be editorial, but sometimes a measurement procedure can also undergo fundamental technical changes that can have an impact on the measurement results. In both cases, the certificate should be updated to the new measurement

procedures in order to keep the material fit for purpose, whereby the update process requires different measures depending on the nature of the modification.

If there is only an editorial change, it should be checked that measurement results obtained with the newly revised measurement procedure are in agreement with the certified value as defined by the previous edition. If this is confirmed, the old measurement procedure on the certificate can be replaced by the new measurement procedure, without changing the certified value and its uncertainty.

In case of a fundamental technical change, a completely new certified value must be assigned, with the material being re-characterised in an interlaboratory comparison using the newly revised measurement procedure.

This addendum describes the measurements and evaluations carried out

- for the confirmation of the validity of the certified values for editorially changed measurement procedures,
- and for the certification of properties with measurement procedures that were subject to fundamental technical changes or were newly published.

1.2 Outline of the CRM project

The production of a CRM as defined in ISO 17034 [3] is a project comprising planning, processing of the material, homogeneity and stability testing, characterisation and assigning of the property values and finally distribution and post-certification monitoring to control stability. ERM-EF001, a biodiesel material certified for selected parameters of EN 14214 [5], was released in 2014 following the above steps.

Some of the originally certified properties were retracted in 2018 as a result of the post certification monitoring to control stability, namely monoglyceride content, diglyceride content, total glycerol content, water content, and acid value. For this reason, it was decided to retract the triglyceride content as well from the current certificate, since its relevance is no longer given by the withdrawal of the other glycerides. All these properties will not be discussed further in this addendum.

The current project is regarding the update of the certificate of the existing ERM-EF001. Many processes, usually part of the production of a CRM, are therefore not dealt with here, only the measures taken to adapt ERM-EF001 to the newly revised measurement procedures are addressed (Figure 1).

In order to be able to classify the extent of the modifications made in a newly revised measurement procedures, these were first compared editorially with the former editions.

The changes found were then discussed with an expert laboratory to confirm, from a practical point of view, whether the modifications made in the measurement procedure could have any impact on the final measurement result or are only of an editorial nature without affecting the measurement result.

In case of a purely editorial change, the newly revised measurement procedure was assessed by a series of verification measurements performed by an expert laboratory. The verification measurement results obtained with the newly revised measurement procedure should not differ from the certified value as defined by the previous edition. If this was confirmed, the outdated measurement procedure on the certificate was replaced by the new measurement procedure, without changing the assigned certified value and its uncertainty.

In case of a fundamental technical modification, a completely new certified value was assigned, with the material being re-characterised in an interlaboratory comparison using the newly revised measurement procedure. Uncertainties from this new characterisation study were combined with the uncertainties from homogeneity and stability studies from the original project in 2014. Finally, certified values as defined by the newly revised measurement procedures and uncertainties were implemented in the certificate.

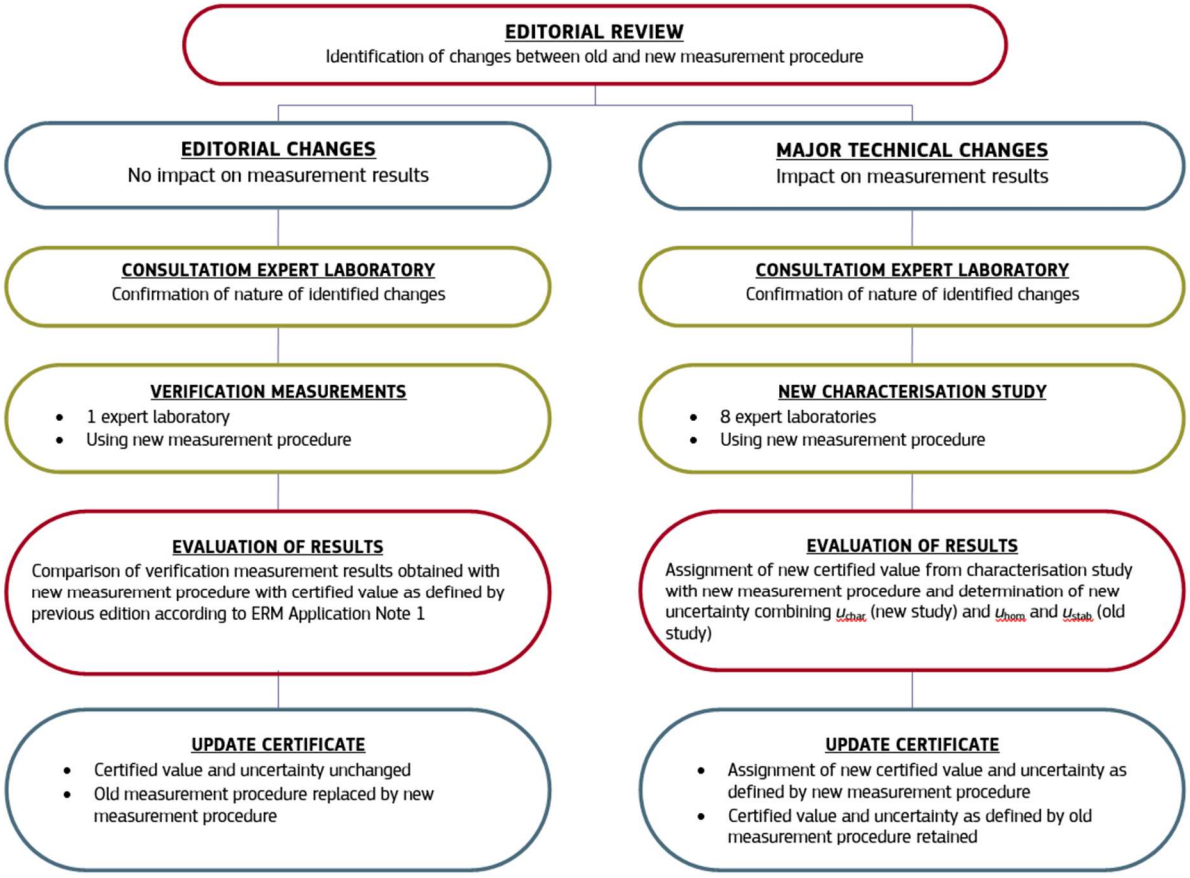

Figure 1: Process to update the certificate of ERM-EF001 to newly revised measurement procedures

Uncertainties of certified and indicative values were estimated in compliance with ISO 17034 [1], which implements the basic principles of ISO/IEC Guide 98 (GUM) [20].

The current project, including the outcome of the review process of the measurement procedures, the evaluation of the obtained measurement data from the verification study for editorially changes measurement procedures, the characterisation study for technically changed measurement procedures and the assignment of certified values and uncertainties, was subjected to an internal peer-review.

2 Participants

2.1 Project management and data evaluation

European Commission, Joint Research Centre, Directorate F – Health, Consumers and Reference Materials, Geel, BE
(accredited to ISO 17034:2016 for production of certified reference materials, BELAC No. 268-RM)

2.2 Editorial review of measurement procedures

European Commission, Joint Research Centre, Directorate F – Health, Consumers and Reference Materials, Geel, BE

2.3 Verification measurements

EESTI KESKKONNAUURINGUTE KESKUS OÜ (Estonian Environmental Research Centre), Tallinn, EE
(measurements under the scope of ISO/IEC 17025:2017 accreditation EAK L008)

2.4 Characterisation measurements

ASG Analytik-Service AG, Neusäss, DE
(measurements under the scope of ISO/IEC 17025:2017 accreditation D-PL-11334-01-00)

EESTI KESKKONNAUURINGUTE KESKUS OÜ (Estonian Environmental Research Centre), Tallinn, EE
(measurements under the scope of ISO/IEC 17025:2017 accreditation EAK L008)

INNOVHUB - Stazioni Sperimentali per l'Industria, Milan, IT

INTERTEK BELGIUM NV, Antwerp, BE
(measurements partially under the scope of ISO/IEC 17025 accreditation BELAC; No. 105-TEST)

ITERG - Département Analyse & Expertise, Canéjan, FR

NAITEC - Fundación I+D Automoción y Mecatrónica, Noain, ES

SGS ESPAÑOLA DE CONTROL, S.A., Barcelona, ES
(measurements under the scope of ISO/IEC 17025:2017 accreditation ENAC 14/LE249)

VÚRUP, a.s., Bratislava, SK
(measurements under the scope of ISO/IEC 17025:2017 accreditation SNAS No. S-119)

All laboratories are identified by a code (e.g. L01). The numbering is not in the alphabetical order presented above.

3 Editorial review of measurement procedures

To classify the extent of the modifications made in the newly revised measurement procedures, the new version of the measurement procedures were first compared with the former editions by the JRC (Geel, BE).

EN 14103:2020 [13] describes the determination of the mass percentage of total methyl esters of fatty acids and the mass percentage of linolenic acid methyl ester present in the sample, by gas chromatography (GC) according to a procedure using internal calibration (nonadecanoic acid methyl ester) (see Annex A Table A1). EN 14103:2020 [13] supersedes EN 14103:2011 [6]. In comparison with the previous edition, the following technical modifications have been made: a) note on natural nonadecanoic acid methyl ester added in the scope; b) new procedure to check nonadecanoic acid methyl ester purity, with new GC conditions, and reduction of the minimum GC purity (99.5 to 99.0 % (m/m)); c) calculation of results revised by incorporation of theoretical flame ionization detector correction factor, which gives a better accuracy of the calculated contents in case of presence of methyl esters with short chains; d) new interlaboratory study conducted and precision adopted; e) new sample chromatograms recorded and added; f) calculation of the pattern of fatty acid methyl esters (FAMEs) incorporated as informative Annex C; g) modification of the way of integration by taking all the peaks into consideration whereas in the previous edition all the peaks identified as FAMEs were taken into consideration; h) increase of the FAME sample test portion to 250 mg whereas in the previous edition the sample test portion was 100 mg; i) document revised editorially. It was concluded that the modifications have an impact on the measurement results.

EN ISO 12185:1996 specifies a measurement procedure for the determination of density. This measurement procedure remained unchanged since the release of ERM-EF001. Hence, no further action was taken.

EN ISO 3104:2020 [14] specifies a measurement procedure to measure the time for a fixed volume of liquid to flow under gravity through the glass capillary of a calibrated viscosimeter under a reproducible driving head and at a known and closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscosimeter (see Annex A Table A2). EN ISO 3104:2020 [14] supersedes EN ISO 3104:1996 [8]. In comparison with the previous edition, the following modifications have been made: (a) precision data have been updated to all actual fuels on the market. (NOTE: no changes for FAMEs), (b) biodiesel blends and paraffinic diesel have been included in the scope (NOTE: not relevant in this context), (c) the procedure description and allowance of automated techniques have been included (NOTE: Previous edition allowed already automated viscosimeters, i.e. automated viscometers, which have been shown to measure kinematic viscosity within the limits of precision given in clause 14, are acceptable alternatives.) It was concluded that none of these changes should have an impact on the measurement result for ERM-EF001.

EN 14112:2020 [15] specifies a measurement procedure for the determination of the oxidation stability of FAMEs at 110 °C, by means of measuring the induction period up to 48 h. EN 15751:2014 [18] describes a similar measurement procedure for oxidation stability determination of pure FAMEs and of blends of FAME with petroleum based diesel (see Annex A Table A3). In principle, EN 15751 is based on EN 14112, which was specifically adapted for the determination of oxidation stability of FAMEs. At the time of development the measurement procedure was applicable for FAME fuel according to EN 14214 [5], but questions remained on the accuracy

towards blends of FAME and diesel fuel. The goal was to have one single measurement procedure for FAME fuel, diesel/FAME blends and pure diesel fuels. Although the modifications cover FAME fuel and diesel/FAME blends, the European Committee for Standardization (CEN/TC 307) decided that it was better to retain EN 14112 for methyl esters and publish a separate standard for all automotive fuel and heating oil applications, as the use of 'diesel and diesel blends' falls out the scope of CEN/TC 307. EN 14112:2020 [15] supersedes EN 14112:2003 [9]. In comparison with the previous edition, the following modifications have been made: (a) change of Figure 2, removal of dimension between air inlet and heating block; (b) introduction removed, (c) document revised editorially. It was concluded that none of these changes should have an impact on the measurement result and that EN 15751 is equivalent to EN 14112 for this purpose.

EN 14111:2003 specifies a measurement procedure for the determination of the iodine value. This measurement procedure remained unchanged since the release of ERM-EF001. Hence, no further action was taken.

EN ISO 3679:2015 [16] is used to determine whether a product will or will not flash at a specified temperature (flash no-flash Procedure A) or the flash point of a sample (Procedure B) (see Annex A Table A4). EN ISO 3679:2015 [16] supersedes EN ISO 3679:2004 [11]. In comparison with the previous edition, the following modifications have been made: (a) incorporation of ISO 3680 flash point technique into flash/no flash technique as a separate procedure due to the fact that many apparatus on the market combine both tests (NOTE: the flash no- flash procedure is not relevant for ERM-EF001; the certified property is the flash point of biodiesel); (b) title change (NOTE: not relevant), (c) revision of temperature measuring device requirements (NOTE: not relevant), (d) new precision covering both gas and electric ignition (NOTE: in fact only precision data for gas ignition given; previous edition gives only gas ignition). It was concluded that none of these changes should have an impact on the measurement result for ERM-EF001.

EN 14110:2019 [17] specifies a measurement procedure for the determination of the methanol content of FAME for use as diesel fuel and domestic heating fuel. The sample is heated at 80 °C in a hermetically sealed vial to allow desorption of contained methanol into the gas phase. When the equilibrium is reached a defined part of the gas phase is injected into a GC, where methanol is detected with a flame ionization detector (see Annex A Table A5). The amount of methanol can be determined either by internal calibration (procedure A) or by external calibration (procedure B). EN 14110:2019 [17] supersedes EN 14110:2003 [12]. In comparison with the previous edition, the following modifications have been made: (a) addition of formula (1) - resolution between methanol and 2-propanol, (b) correction of the formula to calculate the methanol content based on external calibration, (c) addition of Clause 2 - Normative References, (d) addition of Clause 7 - Sampling. It was concluded that none of these changes should have an impact on the measurement result for ERM-EF001.

The identified changes were discussed with an expert laboratory holding an ISO/IEC 17025 accreditation for the concerned measurements, to assess whether the modifications made in the measurement procedure could have any impact on the final measurement result or are just editorial in nature.

A fundamental technical change was confirmed for EN 14103:2020 [13]. The application of the newly revised measurement procedure will have an impact on the measurement results of the ester and linolenic acid methyl ester content. Hence, it was decided that new certified values and uncertainties are assigned, with the material being re-characterised by an interlaboratory

comparison of laboratories of demonstrated competence using the newly revised measurement procedure (Section 5).

A purely editorial change was confirmed for viscosity (EN ISO 3104:2020 [14]), the oxidation stability (EN 14112:2020 [15] and EN 15751:2014 [18]), flash point (EN ISO 3679:2015 [16]), and methanol content (EN 14110:2019 [17]). To confirm this assessment, it was decided to test these properties by another expert laboratory using the newly revised measurement procedures (verification measurements).

Additionally, it was decided to include a newly published measurement procedure for the iodine value (EN 16300:2012 [19]), which, according to the expert laboratory, is being used more and more in practice (see Annex A Table A6). This European Standard specifies a calculation procedure for the determination of the iodine value. A new certified value and uncertainty will be assigned, with the material being characterised by an interlaboratory comparison of laboratories of demonstrated competence using EN 16300:2012 (Section 5).

4 Verification measurements

4.1 Study setup

The verification measurements were performed by the Estonian Environmental Research Centre (Tallinn, EE) using the newly revised measurement procedures (Table 1).

Table 1: Measurement procedures used for certified values of ERM-EF001 and for verification measurements

Property	Measurement procedures used for certified values	Measurement procedures used for verification measurements
Viscosity	EN ISO 3104:1996 [8]	EN ISO 3104:2020 [14]
Oxidation stability	EN 14112:2003 [9]	EN 15751:2014 [18], equivalent to EN 14112:2020 [15]
Flash point	EN ISO 3679:2004 [11]	EN ISO 3679:2015 [16]
Methanol content (indicative value)	EN 14110:2003 [12]	EN 14110:2019 [17]

The different sample intakes required for the individual measurands resulted in a different number of units being made available for the measurements. The laboratory received

- two units of ERM-EF001 for the measurements of the oxidation stability and was requested to provide six independent results, three per unit,
- three units of ERM-EF001 for the measurements of the flash point and was requested to provide six independent results, two per unit,
- and six units of ERM-EF001 for each, the methanol content and viscosity and was requested to provide six independent results, one per unit.

All measurements, apart from viscosity, were performed under intermediate precision conditions (different working days) due to the required analysis times.

4.2 Evaluation of verification measurement results

The individual verification measurement results (VMR) obtained with the newly revised measurement procedures are displayed in Table 2.

Table 2: Datasets as reported for each property

Property	Viscosity [mm^2/s]	Oxidation stability [h]	Flash point [°C]	Methanol content [% (m/m)]
Replicate 1	4.4710	10.70	174.9	0.04432
Replicate 2	4.4719	10.71	174.8	0.04314
Replicate 3	4.4727	10.40	175.1	0.04703
Replicate 4	4.4733	10.83	175.4	0.05379
Replicate 5	4.4750	10.56	176.7	0.05431
Replicate 6	4.4710	10.44	176.6	0.05294

The assessment of the agreement between verification measurement results obtained with the newly revised measurement procedure and the certified value as defined by the previous edition was carried out according to the procedure described in the ERM Application Note 1 [21].

The difference between the verification measurements results and the certified value is compared with its uncertainty, i.e. the combined uncertainty of the two values, where the difference between the verification measurements results and the certified value of ERM-EF001, Δ_{meas}, is calculated as

$$\Delta_{meas} = |c_{meas} - c_{CRM}| \qquad \textbf{Equation 1}$$

c_{meas} mean measured value obtained with the newly revised measurement procedure
c_{CRM} certified value from ERM-EF001 obtained with the previous edition

The uncertainty of Δ_{meas} is calculated as:

$$U_{\Delta} = k \cdot \sqrt{u_{meas}^2 + u_{CRM}^2} \qquad \textbf{Equation 2}$$

U_{Δ} expanded combined uncertainty of the verification measurement results and the certified value
u_{meas} standard uncertainty of verification measurement results derived from the newly revised measurement procedure
u_{CRM} uncertainty of certified value
k coverage factor of 2 corresponding to a confidence level of approximately 95 %

The expanded uncertainty of the verification measurement results (U_{meas}) is derived from the respective revised measurement procedures. They give information on expected performance figures, i.e. repeatability and reproducibility limits (Table 3).

A repeatability limit, *r*, is the value of the maximum absolute difference between two single test results obtained under repeatability conditions that can be expected at a certain probability (usually 95 %). A reproducibility limit, *R*, is similarly defined for test results obtained under reproducibility conditions [22]. A repeatability limit is calculated from:

$$r = t \cdot \sqrt{2} \cdot s_r \qquad \textbf{Equation 3}$$

where t (1.96) is the two-tailed Student t value at the 95 % confidence level and s_r is the repeatability standard deviation.

Similarly, the reproducibility limit is calculated from:

$$R = t \cdot \sqrt{2} \cdot s_R \qquad \textbf{Equation 4}$$

where s_R is the reproducibility standard deviation.

The final expanded measurement uncertainty (U_{meas}) for the verification measurements was estimated using n=6 for the replicate measurements

$$U_{meas} = 2 \cdot \sqrt{s_L^2 + \frac{s_r^2}{n}} \qquad \textbf{Equation 5}$$

and the performance figures of the measurement procedure where the standard deviation between laboratories (s_L) is calculated as follows

$$s_L = \sqrt{s_R^2 - s_r^2} \qquad \textbf{Equation 6}$$

Table 3: Performance figures as laid down in respective measurement procedures and estimated expanded measurement uncertainties thereof

Measurand	Unit	*r*	*R*	U_{meas}
Viscosity as defined by EN ISO 3104:2020	[mm^2/s]	0.010	0.021	0.013
Viscosity as defined by EN ISO 3104:1994	[mm^2/s]	0.005	0.029	0.020
Oxidation stability as defined by EN 15751:2014	[h]	0.7	2.4	1.7
Oxidation stability as defined by EN 14112:2003	[h]	1.0	2.8	1.9
Flash point as defined by EN ISO 3679:2015	[°C]	1.9	15.0	10.6
Flash point as defined by EN ISO 3679:2004	[°C]	1.9	15.0	10.6
Methanol as defined by EN 14110:2019	[% (/m/m)]	0.004	0.014	0.010
Methanol as defined by EN 14110:2003	[% (/m/m)]	0.003	0.012	0.008

If the absolute difference between the values obtained from the verification measurements and the certified value is equal or smaller than the expanded combined uncertainty of the values from the verification measurements and the certified value, $\Delta_{meas} \leq U_{\Delta}$, then there is no significant difference between the verification measurement results obtained with the newly revised measurement procedure and the certified value as defined by the previous edition. This has been proven for all properties (Table 4).

Table 4: Comparison of verification measurement results with certified values using ERM Application Note 1 [21]

Property	Viscosity	Oxidation stability	Flash point	Methanol content
Unit	[mm^2/s}	[h]	[°C]	[% (m/m)]
c_{CRM}	4.465	9.8	181	0.041
U_{CRM}	0.005	0.5	14	0.016
c_{meas} [1)]	4.472	10.6	176	0.049
U_{meas}	0.013	1.7	11	0.010
Δ_{meas}	0.007	0.8	5	0.008
U_{Δ}	0.014	1.7	18	0.019
$\Delta_{meas} \leq U_{\Delta}$	YES	YES	YES	YES

Graphical depictions of the verification measurement results obtained with the newly revised measurement procedures and the results of the individual laboratories obtained in the characterisation study of ERM-EF001 are given in Figures 2 to 5.

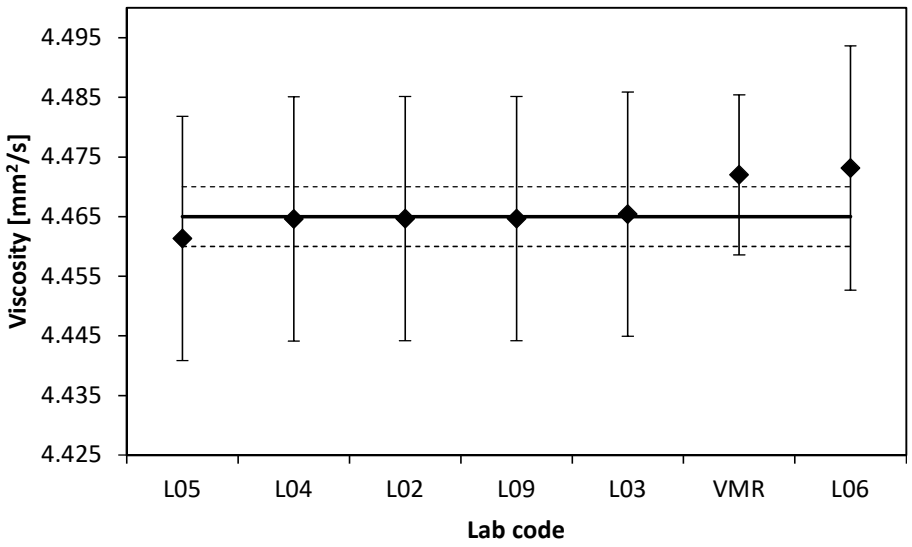

Figure 2: Results for viscosity (continuous line: certified value as defined by EN ISO 3104:1994; dashed line: expanded uncertainty of certified value with k = 2; error bars: expanded measurement uncertainty (U_{meas}) derived from EN ISO 3104:1994 for L02 to L09, and EN ISO 3104:2020 for VMR)

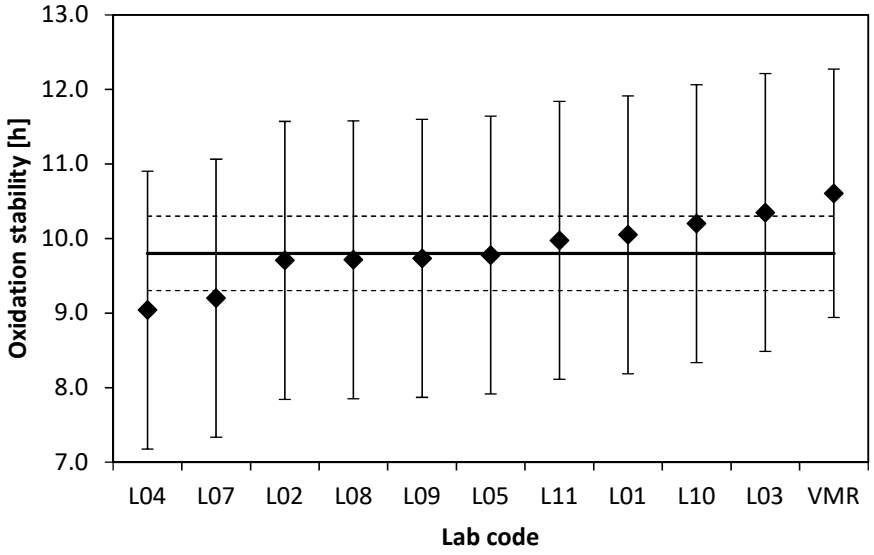

Figure 3: Results for the oxidation stability (continuous line: certified value as defined by EN 14112:2003; dashed line: expanded uncertainty of certified value with k = 2; error bars: expanded measurement uncertainty (U_{meas}) derived from EN 14112:2003 for L01 to L11, and EN 15751:2014 for VMR)

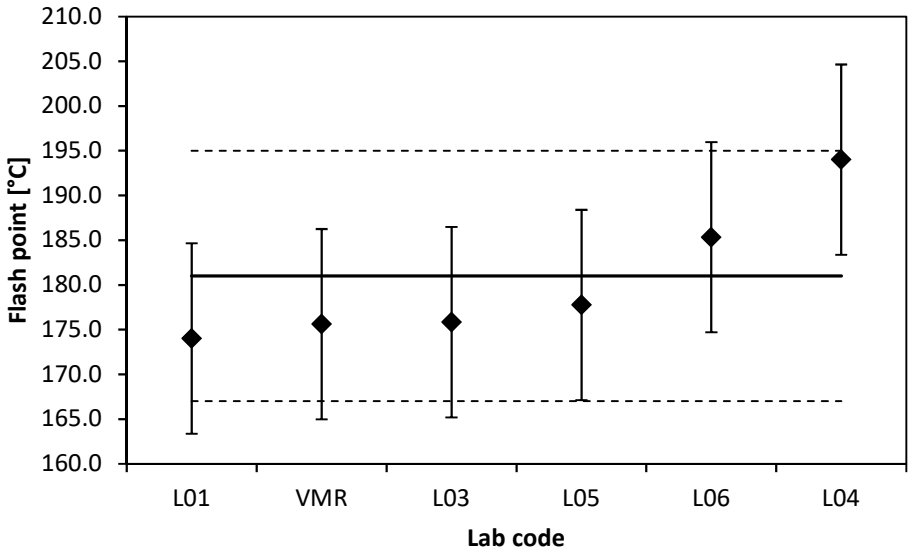

Figure 4: Results for flash point (continuous line: certified value as defined by EN ISO 3679:2004; dashed line: expanded uncertainty of certified value with k = 2; error bars: expanded measurement uncertainty (U_{meas}) derived from EN ISO 3679:2004 for L01 to L06, and EN ISO 3679:2015 for VMR)

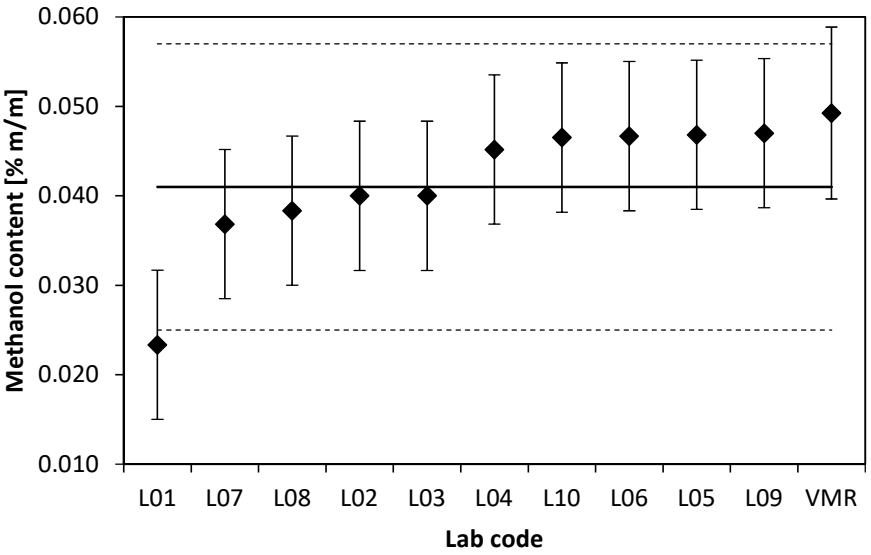

Figure 5: Results for methanol content (continuous line: certified value as defined by EN 14110:2003; dashed line: expanded uncertainty of certified value with k = 2; error bars: expanded measurement uncertainty (U_{meas}) derived from EN 14110:2003 for L01 to L10 and EN 14110:2019 for VMR)

For all properties, the verification measurement results obtained with the newly revised measurement procedure did not differ from the certified value as defined by the previous edition. Hence, the old measurement procedures on the certificate are replaced by the latest editions, without changing the assigned certified values and their uncertainties (Table 5).

Table 5: Resulting updates to the certificate for editorially changed measurement procedures

	Certified value [5]	Uncertainty [6]	Unit
Oxidation stability (at 110 °C) [1]	9.8	0.5	[h]
Flash point [2]	181	14	[°C]
	Indicative value [7]	Uncertainty [8]	Unit
Methanol content [3]	0.041	0.016	[% (m/m)] [4]

1) As defined by **EN 15751:2015** and **EN 14112:2020**
2) As defined by **EN ISO 3679:2015**
3) As defined by **EN 14110:2019**
4) As called in **EN 14110:2020**, which is equivalent to 10^{-2} g/g
5) Certified values are values that fulfil the highest standards of accuracy. The given values represent the unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory. The certified value and its uncertainty are traceable to the International System of Units (SI).
6) The uncertainty of the certified value is the expanded uncertainty with a coverage factor k = 2 corresponding to a level of confidence of 95 %, estimated in accordance with ISO 17034:2016 and ISO Guide 35:2017.
7) Indicative values are values where either the uncertainty is deemed too large or where too few independent datasets are available to allow certification and are therefore less reliable than certified values. Great caution should be used when using these values. The given value is an unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory. The indicative value and its uncertainty are traceable to the International System of Units (SI).
8) The uncertainty of the indicative value is the expanded uncertainty with a coverage factor k = 2 corresponding to a level of confidence of about 95 % estimated in accordance with ISO 17034:2016 and ISO Guide 35:2017.

Although the changes in the newly revised measurement procedure for viscosity were deemed to have no impact on the final measurement result, a different approach was chosen for its update on the certificate. Based on stability monitoring data received, which were gathered at the same time as the verification measurement results, it was decided to subject this property to a completely new characterisation and assign a new certified value and uncertainty (Section 5). The stability monitoring showed a trend towards increasing viscosity. If this trend were to continue in the next stability monitoring, viscosity would have to be withdrawn from the certificate, whereas a completely new re-characterisation will allow a longer use of the certified property.

5 Characterisation

The material characterisation is the process of determining the property values of a reference material.

The outcome of the editorial review (Section 3) was to re-characterise the ester and linolenic acid methyl ester content according to EN 14103:2020 [13].

In addition, viscosity was included for a complete re-characterisation using EN ISO 3104:2020 [14], although the changes in the newly revised measurement procedure were deemed to have no impact on the final measurement result (see Section 3.3). The decision to perform a complete re-characterisation was based on observing changes in the certified value for viscosity during stability monitoring.

Moreover, the iodine value according to EN 16300:2012 [19] was included. EN 16300:2012 [19] specifies a calculation procedure for the determination of the iodine value. The calculation procedure uses as data entry the results from the gas chromatography determination according to EN 14103 of individual fatty acid methyl esters for the determination of the iodine value. It is important to recognise that the latest version of EN 14103 is to be used for the determination of individual FAME components. The measurement procedure is not intended as a replacement for EN 14111 [10].

The re-characterisation was based on an interlaboratory comparison of expert laboratories, i.e. the properties of the material were determined in different laboratories to demonstrate the absence of a measurement bias. Due to the nature of the measurands all participants used the same measurement procedures for the measurements. This approach converts the systematic bias of each laboratory into a random variable, the combined effect of which is reduced by averaging over several laboratories.

5.1 Selection of participants

Eight laboratories were selected based on criteria that comprised both technical competence and quality management aspects. Each participating laboratory was required to operate a quality system. Laboratory proficiency in the field of biodiesel measurements was demonstrated by all laboratories through their successful participation in the interlaboratory comparison for the initial characterisation of ERM-EF001. Having a formal accreditation was not mandatory, but meeting the requirements of ISO/IEC 17025 [2] was obligatory. Where measurements are covered by the scope of accreditation, the accreditation number is stated in the list of participants (Section 2).

5.2 Study setup

Each laboratory received three units of ERM-EF001 for the measurements of the ester content, the linolenic acid methyl ester content and iodine value and was requested to provide six independent results, two per unit. Furthermore, each laboratory received six units of ERM-EF001 for the measurements of viscosity and was requested to provide six independent results, one per unit.

The sample preparations and measurements had to be done on three days to ensure intermediate precision conditions. An independent calibration was performed for each result whenever possible.

Laboratories were not requested to submit measurement uncertainties. Instead, the performance figures specified in the documentary standards were used, which give information on expected repeatability and reproducibility limits (see Section 4.2).

5.3 Measurement procedures used

All laboratories used the same measurement procedures for the selected measurands, i.e.

- ester content as defined by EN 14103:2020 [13];
- linolenic acid methyl ester content as defined by EN 14103:2020 [13];
- viscosity at 40 °C as defined by EN ISO 3104:2020 [14];
- iodine value as defined by EN 16300:2012 [19].

5.4 Evaluation of results

The characterisation study resulted in eight datasets for the ester content, linolenic acid methyl ester content, and iodine value and seven datasets for viscosity. All individual results of the participating laboratories, grouped per measurand, are displayed in tabular and graphical form in Annex B.

5.4.1 Technical evaluation

The obtained data were first checked for compliance with the requested instructions and for their validity based on technical reasons. The following criteria were considered during the evaluation:

- compliance with the instructions given: sample preparations and measurements performed on three days
- method performance, i.e. agreement of the measurement results with performance figures of the measurement procedure (see Section 4.2 and Table 6)
    - Datasets were rejected when the absolute difference between two independent test results from the same unit exceeded the repeatability limit (r) as laid down in the measurement procedure
    - Datasets were rejected when the absolute difference between two independent test results from two different units exceeded the reproducibility limit (R) as laid down in the measurement procedure.

Table 6: Performance figures as laid down in respective measurement procedures and estimated expanded measurement uncertainties thereof

Measurand	Unit	r	R	U_{meas}
Ester content	[% (m/m)]	1.65	2.45	1.38
Linolenic acid methyl ester content	[% (m/m)]	0.11	0.23	0.15
Viscosity at 40 °C	mm^2/s	0.010	0.021	0.013
Iodine value	[g iodine/100 g]	0.87	6.81	4.83

All laboratories complied with the instructions and were strictly following the measurement procedures. Method performance for most of the laboratories was in agreement with the repeatability and reproducibility limits, despite the fact that the measurements were performed on three days. Based on the above criteria, the following datasets were rejected as not technically valid (Table 7).

Table 7: Datasets that showed non-compliance with the instructions given and technical specifications, and action taken

Measurand	Lab code	Description of problem	Action taken
Ester content	L06	Technical problem with GC	Not used for evaluation
Linolenic acid methyl ester content	L06	Technical problem with GC	Not used for evaluation
Viscosity	L07	Reproducibility limit not met	Not used for evaluation
Iodine value	L06; L07	L06: Technical problem with GC; L07: Repeatability limit not met	Not used for evaluation

Laboratory 07 did not meet the repeatability limit for the iodine value and the reproducibility limit for viscosity. As the laboratory confirmed that this was not a transcription error, the datasets were rejected.

The datasets for the ester content, the linolenic acid methyl ester content, and iodine value from laboratory L06 were not used for the evaluation as the laboratory reported a technical issue while using the GC that caused incorrect results. The lab retracted the results.

5.4.2 Statistical evaluation

The datasets accepted based on technical reasons were tested for normality of dataset means using normal probability plots and were tested for outlying means using the Grubbs test and using the Cochran test for outlying standard deviations (both at a 99 % confidence level). Standard deviations within (s_{within}) and between ($s_{between}$) laboratories were calculated using one-way ANOVA. The results of these evaluations are shown in Table 8.

Table 8: Statistical evaluation of the technically accepted datasets for ERM-EF001. *p*: number of technically valid datasets

Measurand	*p*	Outliers		Normally distributed	Statistical parameters				
		Means	Variances		Unit	Mean	*s*	$s_{between}$	s_{within}
Ester content	7	none	none	yes	[% (m/m)]	97.387	0.603	0.587	0.333
Linolenic acid methyl ester	7	none	none	yes	[% (m/m)]	8.515	0.100	0.099	0.039
Viscosity	6	none	yes	yes	[mm^2/s]	4.4739	0.0065	0.0064	0.0024
Iodine value	6	none	none	yes	[g iodine/100 g]	107.289	0.905	0.896	0.317

For all parameters the laboratory means follow normal distributions. None of the data contains outlying means.

The statistical evaluation flags laboratory 3 as outlying variance for viscosity while its mean result for this measurand still agrees with the other data. Laboratory 3 was using a manual glass viscometers (procedure A) whereas the others used a glass capillary viscometers in an automated assembly (procedure B), both specified in the measurement procedure. Finally, all datasets were retained, as all results still agree well with the repeatability and reproducibility requirements of the respective measurement procedure.

The uncertainty related to the characterisation (u_{char}) is estimated as the standard error of the mean of laboratory means ($s/\sqrt{p}$) (Table 9).

Table 9: Uncertainties of characterisation for ERM-EF001. *p*: number of technically valid datasets

Measurand	*p*	Unit	Mean	*s*	u_{char}
Ester content	7	[% (m/m)]	97.387	0.603	0.228
Linolenic acid methyl ester content	7	[% (m/m)]	8.515	0.100	0.038
Viscosity	6	[mm^2/s]	4.4739	0.0065	0.0027
Iodine value	6	[g iodine/100 g]	107.289	0.905	0.369

6 Value Assignment

Certified values are values that fulfil the highest standards of accuracy. Procedures at JRC Directorate F recommend pooling of at least six datasets to assign certified values. Full uncertainty budgets in accordance with ISO 17034 [3] and ISO Guide 35 [4] were established.

New certified values were assigned for the ester content, linolenic acid methyl ester content, viscosity, and iodine value.

6.1 Certified values and their uncertainties

The unweighted mean of the means of the accepted datasets as shown in Table 8 were assigned as certified values for the ester and linolenic acid methyl ester content according to EN 14103:2020 [13], viscosity at 40 °C according to EN ISO 3104:2020 [14], and the iodine value according to EN 16300:2012 [19].

The assigned uncertainty consists of uncertainties relating to characterisation (u_{char}) reported in this addendum, whereas potential between-unit inhomogeneity (u_{bb}), and potential degradation during transport (u_{sts}), and long-term storage (u_{lts}) derive from EUR 26711 EN [1]. These different contributions were combined to estimate the relative expanded uncertainty of the certified value ($U_{CRM, rel}$) with a coverage factor (k) given as:

$$U_{CRM, rel} = k \cdot \sqrt{u^2_{bb, rel} + u^2_{sts, rel} + u^2_{lts, rel} + u^2_{char, rel}} \qquad \textbf{Equation 7}$$

- u_{char} was estimated as described in Section 5 of this addendum.
- u_{bb} was estimated as described in EUR 26711 EN [1] in Section 4.
- u_{sts} and u_{lts} were estimated as described in EUR 26711 EN [1] in Section 5.

The choice of the coverage (k) factor was based on the number of effective degrees of freedom as calculated using the Welch-Satterthwaite equation [20]. Applying this equation, the effective degrees of freedom shown in Table 10 were obtained.

Table 10: Effective degrees of freedom calculated using the Welch-Satterthwaite equation

Certified property	Effective degrees of freedom
Ester content	10
Linolenic acid methyl ester content	9
Iodine value	33
Viscosity (at 40 °C)	8

The JRC's procedures for assigning uncertainties to certified values stipulate that for more than five effective degrees of freedom a coverage (k) factor of 2 can be chosen. Therefore, a k-factor of 2 was applied to obtain the expanded uncertainties. The certified values and their uncertainties are summarised in Table 11.

Table 11: Certified values and their uncertainties for ERM-EF001

Certified property	Unit	Certified value	$u_{char, rel}$ [%]	$u_{bb, rel}$ [%]	$u_{sts, rel}$ [%]	$u_{lts, rel}$ [%]	$U_{CRM, rel}$ [%]	U_{CRM} [1)]
Ester content	[% (m/m)]	97.4	0.234	0.057	0.001	0.178	0.60	0.6
Linolenic acid methyl ester content	[% (m/m)]	8.52	0.444	0.068	0.001	0.208	1.0	0.09
Viscosity (at 40 °C)	[mm^2/s]	4.474	0.0593	0.0143	0.00019	0.028	0.134	0.006
Iodine value	[g iodine/100 g]	107.3	0.344	0.478	0.005	0.657	1.77	1.9

[1)] Expanded (k = 2) and rounded uncertainties; uncertainties are always rounded up [23] and in a way that the rounding error corresponds to 3 % to 30 % of the uncertainty

7 Metrological traceability

7.1 Metrological traceability

Identity – Editorially changed measurement procedures

Certified and indicative values and their uncertainties remained unchanged for the oxidation stability, the flash point and the methanol content. The verification measurements for editorially changed measurement procedures confirmed the validity of the assigned certified values and their uncertainties for the oxidation stability as defined by EN 15751:2014 [18], which is equivalent to EN 14112:2020 [15], the flash point as defined by EN ISO 3679:2015 [16], and the assigned indicative value for the methanol content as defined by EN 14110:2019 [17]. Consequently, the new identity statements are as follows:

Oxidation stability is an operationally defined measurand and can only be obtained by following the measurement procedures specified in EN 15751:2014 [18] and EN 14112:2020 [15].

Flash point is an operationally defined measurand and can only be obtained by following the measurement procedure specified in EN ISO 3679:2015 [16].

Methanol content is an operationally defined measurand and can only be obtained by following the measurement procedure specified in EN 14110:2019 [17].

A new certified value and uncertainty was assigned for viscosity although the changes in the newly revised measurement procedure were deemed to have no impact on the final measurement result. The update was prompted by stability issues that required the assignment of a new certified value. In this case, the certified value as defined by EN ISO 3104:1996 [8] is no longer valid and withdrawn from the certificate. Consequently, the new identity statement is as follows:

Viscosity is an operationally defined measurand and can only be obtained by following the measurement procedure specified in EN ISO 3104:2020 [16].

Identity – Technically changed measurement procedures

New certified values and uncertainties were assigned for technically changed measurement procedures, i.e. ester and linolenic acid methyl ester content as defined by 14103:2020 [13], and iodine value as defined by EN 16300:2012 [19]. The identity statements for the new certified values and uncertainties are as follows:

Ester and linolenic acid methyl ester content, and iodine value are operationally defined measurands and can only be obtained by following the measurement procedures specified in EN 14103:2020 [13] and EN 16300:2012 [19].

In addition, the certified values and uncertainties as defined by their corresponding old measurement procedures are retained on the certificate to be used independently from the new measurement procedure.

Quantity value - Technically changed measurement procedures

Traceability of the obtained results is based on the traceability of all relevant input factors. Investigation of the measurement procedure and measurement details of the individual results show that all relevant input parameters of each technically accepted dataset have been properly calibrated. All technically accepted datasets are therefore traceable to the same reference, namely the SI. This traceability to the same reference is also confirmed by the agreement of results within their respective uncertainties. As the assigned values are combinations of agreeing results individually traceable to the SI, the assigned quantity values themselves are traceable to the SI as well.

8 Acknowledgements

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Furthermore, the author would like to thank ASG Analytik-Service AG (Neusäss, DE) for the fruitful discussion on the modifications made in the measurement procedures.

9 References

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6 EN 14103:2011, Fat and oil derivates - Fatty acid methyl esters (FAME) - Determination of ester and linolenic acid methyl ester contents. European Committee for Standardization, Brussels, Belgium

7 EN ISO 12185:1996, Crude petroleum and petroleum products -Determination of density - Oscillating U-tube method. European Committee for Standardization, Brussels, Belgium

8 EN ISO 3104:1996, Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity. European Committee for Standardization, Brussels, Belgium

9 EN 14112:2003, Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of oxidation stability (accelerated oxidation test). European Committee for Standardization, Brussels, Belgium

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11 EN ISO 3679:2004, Determination of flash point - Rapid equilibrium closed cup method. European Committee for Standardization, Brussels, Belgium

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Annexes

Annex A: Scope of measurement procedures used for the verification measurements and the characterisation study

Table A1: Measurement procedure for the ester and linolenic acid methyl ester contents

Standard Reference	**EN 14103:2020**
Technical Body	CEN/TC 307 - Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis
Title	Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of **ester and linolenic acid methyl ester contents**
Scope	The purpose of this document is to describe a procedure for the determination of the ester content in fatty acid methyl esters (FAME) intended for incorporation into diesel oil. It also allows determining the linolenic acid methyl ester content. It allows verifying that the ester content of FAME is greater than 90 % (m/m) and that the linolenic acid methyl ester content is between 1 % (m/m) and 15 % (m/m). The precision was established using FAMEs with an ester content of 95 % (m/m) and 100 % (m/m) only, thus covering the range of the limit value. The method is also suitable outside of this range; however, precision for lower concentrations is subject to further work. This method is suitable for FAME which contains methyl esters between C6 and C24. NOTE 1 For the purposes of this document, the term "% (m/m)" is used to represent the mass fractions. This method was elaborated for FAME samples from usual raw material. For FAME sample from unidentified raw material, a solution of the test sample is prepared without any internal standard addition, in order to verify the absence of natural nonadecanoic acid methyl ester or other unknown substances co-eluting with the IS. NOTE 2 The calculation method of the pattern of fatty acid methyl esters is given in Annex C.

Table A2: Measurement procedure for viscosity

Standard Reference	**EN ISO 3104:2020**
Technical Body	ISO/TC 28 Petroleum and related products, fuels and lubricants from natural or synthetic sources
Title	Petroleum products — Transparent and opaque liquids — Determination of **kinematic viscosity** and calculation of dynamic viscosity
Scope	This document specifies Procedure A, using manual glass viscometers, and Procedure B, using glass capillary viscometers in an automated assembly, for the determination of the kinematic viscosity, v, of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity, n, is obtained by multiplying the measured kinematic viscosity by the density, ρ, of the liquid. The range of kinematic viscosities covered in this test method is from 0.2 mm^2/s to 300 000 mm^2/s over the temperature range 20 °C to +150 °C. NOTE The result obtained from this document is dependent upon the behaviour of the sample and is intended for application to liquids for which primarily the shear stress and shear rates are proportional (Newtonian flow behaviour). If, however, the viscosity varies significantly with the rate of shear, different results can be obtained from viscometers of different capillary diameters. The procedure and precision values for residual fuel oils, which under some conditions exhibit non-Newtonian behaviour, have been included.

Table A3: Measurement procedure for the oxidation stability

Standard Reference	**EN 15751:2014**
Technical Body	CEN/TC 19 Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin
Title	Automotive fuels - Fatty acid methyl ester (FAME) fuel and blends with diesel fuel - Determination of **oxidation stability** by accelerated oxidation method
Scope	This European Standard specifies a test method for the determination of the oxidation stability of fuels for diesel engines, by means of measuring the induction period of the fuel up to 48 h. The method is applicable to fatty acid methyl esters (FAME) intended for the use as pure biofuel or as a blending component for diesel fuels, and to blends of FAME with diesel fuel containing 2 % (V/V) of FAME at minimum. NOTE 1 EN 14112 [1] describes a similar test method for oxidation stability determination of pure fatty acid methyl esters (see the Introduction to this European Standard). NOTE 2 For induction periods higher than 48 h the precision is not covered by the precision statement of this method. The limit values of the relevant fuel standards are well within the scope of this test method. NOTE 3 The presence of cetane improver can reduce the oxidation stability determined by this test method. Limited studies with EHN (2-ethyl hexyl nitrate) indicated, however, that the stability is reduced to an extent which is within the reproducibility of the test method. NOTE 4 For the purposes of this European Standard, the term "% (V/V)" is used to represent the volume fraction (φ) of a material.

Table A4: Measurement procedure for flash point

Standard Reference	**EN ISO 3679:2015**
Technical Body	ISO/TC 28 Petroleum and related products, fuels and lubricants from natural or synthetic sources
Title	Determination of flash no-flash and **flash point** — Rapid equilibrium closed cup method
Scope	ISO 3679:2015 specifies procedures for flash point tests, within the temperature range of -30 °C to 300 °C, for paints, including water-borne paints, varnishes, binders for paints and varnishes, adhesives, solvents, petroleum, and related products. The procedures are used to determine whether a product will or will not flash at a specified temperature (flash no-flash Procedure A) or the flash point of a sample (Procedure B). When used in conjunction with a flash detector, ISO 3679:2015 is also suitable to determine the flash point of fatty acid methyl esters (FAME).

Table A5: Measurement procedure for methanol content

Standard Reference	**EN 14110:2019**
Technical Body	CEN/TC 307 - Oilseeds, vegetables and animal fats and oils and their by-products - Methods of sampling and analysis
Title	Fat and oil derivatives - Fatty Acid Methyl Esters - Determination of methanol content
Scope	This document specifies a method for the determination of the methanol content of fatty acid methyl esters (FAME) for use as diesel fuel and domestic heating fuel. The method is applicable to methanol contents between 0.01 % (m/m) and 0.5 % (m/m). The method is not applicable to mixtures of FAME containing other low boiling components. (NOTE For the purposes of this document, the terms "% (m/m)" and "% (V/V)" are used to represent respectively the mass fraction and the volume fraction) WARNING - The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to application of the standard, and fulfil statutory and regulatory requirements for this purpose.

Table A6: Measurement procedure for the iodine value

Standard Reference	**EN 16300:2012**
Technical Body	CEN/TC 19 Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin
Title	Automotive fuels - Determination of **iodine value** in fatty acid methyl esters (FAME) - Calculation method from gas chromatographic data
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 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.

Annex B: Results of the characterisation measurements

Table B1: Mass fraction of the ester content in ERM-EF001 as reported by each individual lab

Laboratory code	replicate 1 [% (m/m)]	replicate 2 [% (m/m)]	replicate 3 [% (m/m)]	replicate 4 [% (m/m)]	replicate 5 [% (m/m)]	replicate 6 [% (m/m)]	mean [% (m/m)]	RSD [%]
L01	97.10	97.52	97.01	97.20	97.35	96.96	97.19	0.22
L02	98.3	98.4	97.8	98.1	98.1	97.9	98.10	0.23
L03	96.37	96.33	96.24	96.55	96.24	96.58	96.39	0.15
L04	97.5	97.4	96.7	96.9	97.8	97.4	97.3	0.42
L05	97.21	96.9	97.19	97.24	96.51	97.05	97.02	0.29
L07	97.5	98.6	98.6	97.3	98.2	97.6	98.0	0.59
L08	97.4	98.0	97.4	98.0	97.9	97.9	97.8	0.29
Results not used for value assignment								
L06	*94.6*	*95.1*	*93.2*	*93.9*	*93.4*	*93.4*	*93.9*	*0.81*

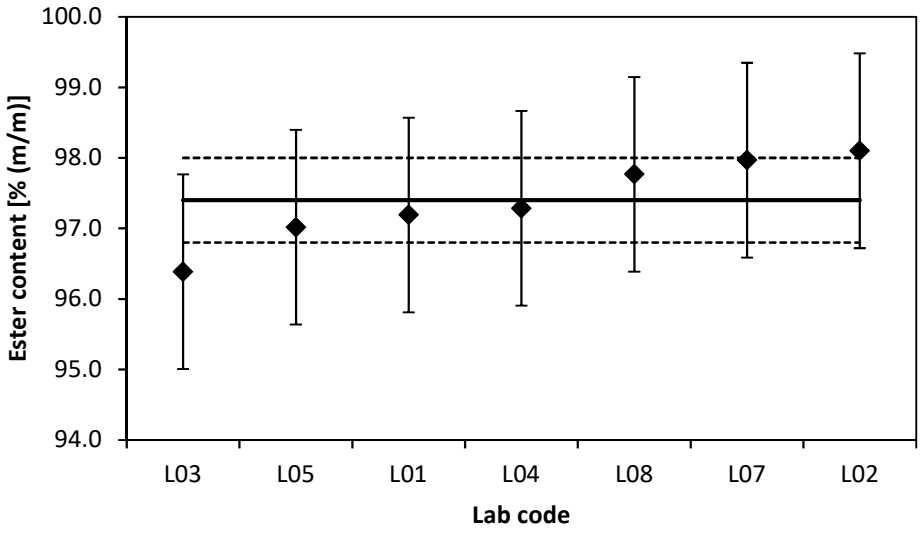

Figure B1: Results of the characterisation study for the mass fraction of the ester content in ERM-EF001 as defined by EN 14103:2020 (continuous line: certified value; dashed line: expanded uncertainty of certified value with k=2; error bars: expanded measurement uncertainty (U_{meas}) derived from EN 14103:2020)

Table B2: Mass fraction of linolenic acid methyl ester content in ERM-EF001 as reported by each individual lab

Laboratory code	replicate 1 [% (m/m)]	replicate 2 [% (m/m)]	replicate 3 [% (m/m)]	replicate 4 [% (m/m)]	replicate 5 [% (m/m)]	replicate 6 [% (m/m)]	mean [% (m/m)]	RSD [%]
L01	8.48	8.52	8.47	8.49	8.50	8.46	8.49	0.25
L02	8.7	8.7	8.6	8.7	8.6	8.6	8.7	0.63
L03	8.38	8.38	8.38	8.31	8.35	8.39	8.37	0.36
L04	8.5	8.5	8.4	8.4	8.5	8.5	8.5	0.61
L05	8.47	8.46	8.46	8.47	8.4	8.45	8.45	0.31
L07	8.55	8.61	8.64	8.53	8.6	8.55	8.58	0.50
L08	8.57	8.63	8.57	8.64	8.60	8.61	8.60	0.34
Results not used for value assignment								
L06	*7.4*	*7.3*	*7.2*	*7.2*	*7.1*	*7.0*	*7.2*	*1.96*

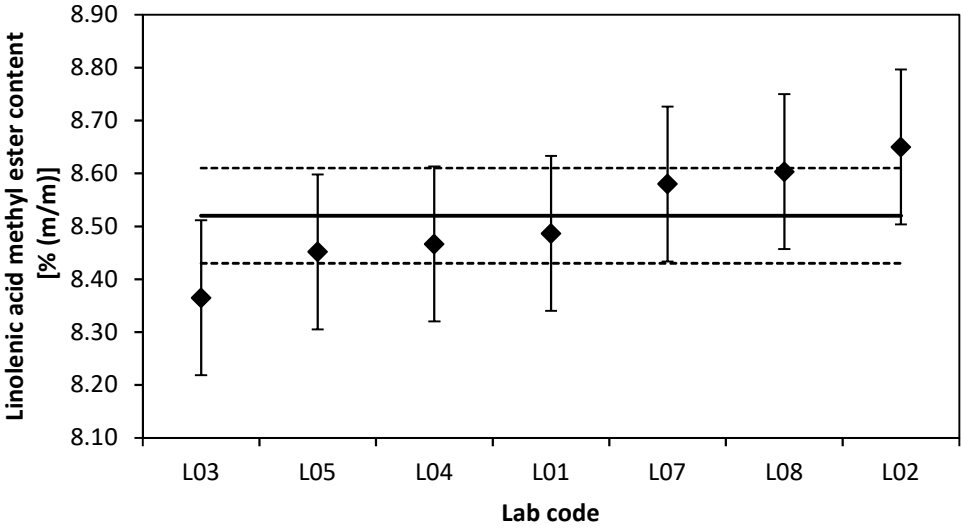

Figure B2: Results of the characterisation study for the mass fraction of the linolenic acid methyl ester content in ERM-EF001 as defined by EN 14103:2020 (continuous line: certified value; dashed line: expanded uncertainty of certified value with k=2; error bars: expanded measurement uncertainty (U_{meas}) derived from EN 14103:2020)

Table B3: Viscosity of ERM-EF001 as reported by each individual lab

Laboratory code	replicate 1 [% (m/m)]	replicate 2 [% (m/m)]	replicate 3 [% (m/m)]	replicate 4 [% (m/m)]	replicate 5 [% (m/m)]	replicate 6 [% (m/m)]	mean [% (m/m)]	RSD [%]
L01	4.474	4.479	4.479	4.475	4.473	4.473	4.476	0.06
L02	4.468	4.469	4.468	4.469	4.469	4.471	4.469	0.02
L03	4.48389	4.48839	4.48001	4.49215	4.48693	4.48281	4.48570	0.10
L04	4.471	4.472	4.472	4.471	4.471	4.472	4.472	0.01
L05	4.4674	4.4674	4.4674	4.4692	4.4678	4.4678	4.4678	0.02
L06	4.474	4.473	4.469	4.476	4.475	4.476	4.474	0.06
Results not used for value assignment								
L07	*4.466*	*4.47*	*4.528*	*4.507*	*4.534*	*4.47*	*4.496*	*0.692*

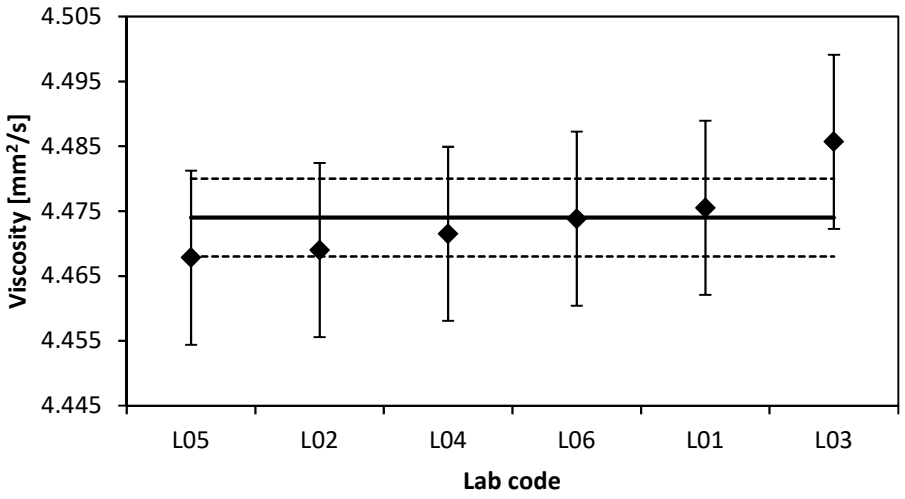

Figure B3: Results of the characterisation study for viscosity in ERM-EF001 as defined by EN ISO 3104:2020 (continuous line: certified value; dashed line: expanded uncertainty of certified value with k=2; error bars: expanded measurement uncertainty (U_{meas}) derived from EN ISO 3104:2020)

Table B4: Iodine value of ERM-EF001 as reported by each individual lab

Laboratory code	replicate 1 [% (m/m)]	replicate 2 [% (m/m)]	replicate 3 [% (m/m)]	replicate 4 [% (m/m)]	replicate 5 [% (m/m)]	replicate 6 [% (m/m)]	mean [% (m/m)]	RSD [%]
L01	107.2	107.6	107.1	107.2	107.4	107.0	107.3	0.20
L02	108.9	109.0	108.4	108.7	108.7	108.5	108.7	0.21
L03	106.5	106.6	106.4	106.5	106.2	106.8	106.5	0.19
L04	107.1	106.9	106.3	106.3	107.5	107	106.9	0.44
L05	106.6	106.5	106.6	106.6	105.8	106.4	106.4	0.29
L08	107.5	108.3	107.6	108.4	108.1	108.2	108.0	0.35
Results not used for value assignment								
L06	*104.1*	*104.5*	*103.6*	*102.8*	*103.0*	*102.6*	*103.4*	*0.74*
L07	*108.4*	*109.5*	*109.5*	*108.1*	*109.1*	*108.4*	*108.8*	*0.56*

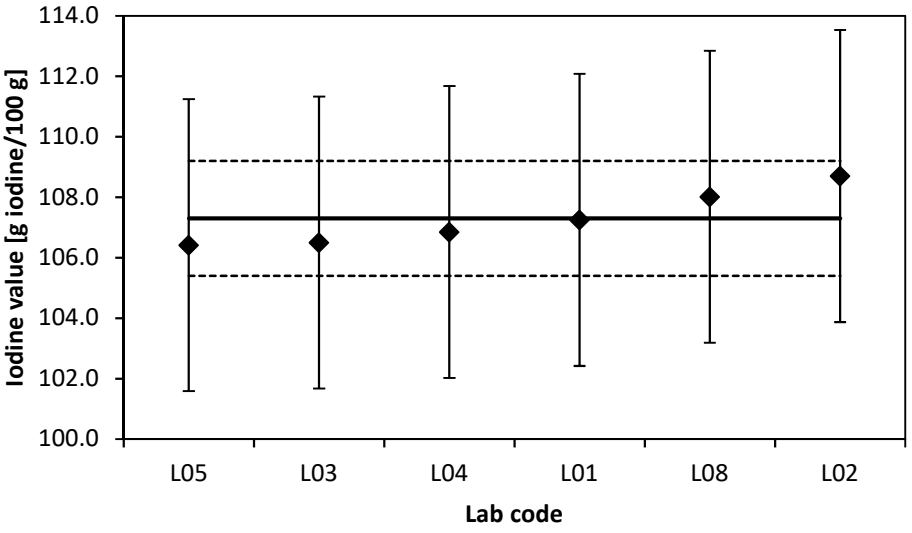

Figure B4: Results of the characterisation study for the iodine value in ERM-EF001 as defined by EN 16300:2012 (continuous line: certified value; dashed line: expanded uncertainty of certified value with k=2; error bars: expanded measurement uncertainty (U_{meas}) derived from EN 16300:2012)