CERTIFICATION REPORT

The certification of selected chemical and physical properties in automotive diesel fuel containing a volume fraction of 7 % biodiesel: ERM®- EF003

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Abstract

This report describes the production of ERM®-EF003, which is an automotive diesel fuel material containing a volume fraction of 7 % fatty acid methyl ester (biodiesel) certified for the volume fraction of the fatty acid methyl ester content, the mass fraction of mono-aromatic hydrocarbon, di-aromatic hydrocarbon, polycyclic aromatic hydrocarbon, and total aromatic hydrocarbon content, and density, kinematic viscosity, and lubricity. This material was produced following ISO Guide 34:2009 and is certified in accordance with ISO Guide 35:2006.

The material is an automotive diesel fuel containing a volume fraction of approximately 7 % biodiesel that is based on rapeseed oil fatty acid methyl ester with the addition of 1 g/kg antioxidant (butylhydroxytoluene). It was provided by a producer in Germany. The material was filled in amber glass ampoules. Between unit-homogeneity was quantified and stability during dispatch and storage were assessed in accordance with ISO Guide 35:2006. The minimum sample intake is the required sample volume stipulated in the respective documentary standard.

The material was characterised by an interlaboratory comparison of laboratories of demonstrated competence and adhering to ISO/IEC 17025:2005. Technically invalid results were removed but no outlier was eliminated on statistical grounds only.

Uncertainties of the certified values were calculated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM) and include uncertainties related to possible inhomogeneity, instability and characterisation.

The material is intended for quality control and assessment of method performance. As with any reference material, it can be used for establishing control charts or validation studies. The certified reference material (CRM) is available in amber glass ampoules containing 27 mL of automotive diesel fuel material with a volume fraction of 7 % biodiesel closed under argon atmosphere.

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Disclaimer

Summary

This report describes the production of ERM®-EF003, which is an automotive diesel fuel material containing a volume fraction of 7 % fatty acid methyl ester (biodiesel) certified for the volume fraction of the fatty acid methyl ester content, the mass fraction of mono-aromatic hydrocarbon, di-aromatic hydrocarbon, polycyclic aromatic hydrocarbon, and total aromatic hydrocarbon content, and density, kinematic viscosity, and lubricity. This material was produced following ISO Guide 34:2009 [1] and is certified in accordance with ISO Guide 35:2006 [2].

The material is an automotive diesel fuel containing a volume fraction of approximately 7 % biodiesel that is based on rapeseed oil fatty acid methyl ester with the addition of 1 g/kg antioxidant (butylhydroxytoluene). It was provided by a producer in Germany. The material was filled in amber glass ampoules.

Between unit-homogeneity was quantified and stability during dispatch and storage were assessed in accordance with ISO Guide 35:2006 [2]. The minimum sample intake is the required sample volume stipulated in the respective documentary standard.

The material was characterised by an interlaboratory comparison of laboratories of demonstrated competence and adhering to ISO/IEC 17025:2005 [3]. Technically invalid results were removed but no outlier was eliminated on statistical grounds only.

Uncertainties of the certified values were calculated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM) [4] and include uncertainties related to possible inhomogeneity, instability and characterisation.

The material is intended for quality control and assessment of method performance. As with any reference material, it can be used for establishing control charts or validation studies. The certified reference material (CRM) is available in amber glass ampoules containing 27 mL of automotive diesel fuel material with a volume fraction of 7 % biodiesel closed under argon atmosphere.

The following values were assigned:

	Certified value 8)	Uncertainty 9)	Unit
Fatty acid methyl ester content 1)	6.88	0.17	% (V/V) 6)
Mono-aromatic hydrocarbon content 2)	18.8	0.7	% (m/m) 7)
Di-aromatic hydrocarbon content 2)	1.84	0.19	% (m/m) 7)
Polycyclic aromatic hydrocarbon content 2)	2.01	0.25	% (m/m) 7)
Total aromatic hydrocarbon content 2)	20.8	0.9	% (m/m) 7)
Density (at 15.0 °C) 3)	837.23	0.07	kg/m^3
Kinematic viscosity (at 40.0 °C) 4)	2.892	0.012	mm^2/s
Lubricity 5)	220	60	µm

1) As defined by EN 14078:2014; 2) As defined by EN 12916:2016; 3) As defined by EN ISO 12185:1996; 4) As defined by EN ISO 3104:1996 5) As defined by EN 12156-1:2016

6) As called in EN 14078:2014, which is equivalent to 10^{-2} mL/mL; 7) As called in EN 12916:2006, which is equivalent to 10^{-2} g/g

8) Certified values are values that fulfil the highest standards of accuracy and represent the unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory. The certified values and their uncertainties are traceable to the International System of Units (SI)

9) The uncertainty of the certified 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/IEC Guide 98-3, Guide to the Expression of Uncertainty in Measurement (GUM:1995), ISO, 2008

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Glossary

ASTM International	ASTM International (formerly American Society for Testing and Materials)
ANOVA	Analysis of variance
b	Slope in the equation of linear regression $y = a + bx$
CEN	European Committee for Standardization
CFPP	Cold filter plugging point
CI	Confidence interval
CRM	Certified reference material
DAH	Di-aromatic hydrocarbon
DCN	Derived cetane number
df	Degrees of freedom
EC	European Commission
EN	European norm (standard)
ERM®	Trademark of European Reference Materials
EU	European Union
FAME	Fatty acid methyl ester
GUM	Guide to the Expression of Uncertainty in Measurement
HFRR	High-frequency reciprocating rig
ISO	International Organization for Standardization
IU	International units
JRC	Joint Research Centre
k	Coverage factor
MAH	Mono-aromatic hydrocarbon
m/m	Mass fraction
MS_{between}	Mean of squares between-unit from an ANOVA
MS_{within}	Mean of squares within-unit from an ANOVA
n	Number of replicates per unit
n.a.	Not applicable
n.c.	Not calculated
Poly AH	Polycyclic aromatic hydrocarbon
QC	Quality control
rel	Index denoting relative figures (uncertainties etc.)
RM	Reference material
RSD	Relative standard deviation

RSE	Relative standard error (=RSD/$\sqrt{n}$)
s	Standard deviation
s_{bb}	Between-unit standard deviation; an additional index "rel" is added when appropriate
s_{between}	Standard deviation between groups as obtained from ANOVA; an additional index "rel" is added as appropriate
se	Standard error
SI	International System of Units
s_{meas}	Standard deviation of measurement data; an additional index "rel" is added as appropriate
s_{within}	Standard deviation within groups as obtained from ANOVA; an additional index "rel" is added as appropriate
s_{wb}	Within-unit standard deviation
T	Temperature
t	Time
t_i	Time point for each replicate
$t_{\alpha,\ \mathrm{df}}$	Critical t-value for a t-test, with a level of confidence of $1-\alpha$ and df degrees of freedom
t_{sl}	Proposed shelf life
t_{tt}	Proposed transport time
T+AH	Tri+-aromatic hydrocarbon
Total AH	Total aromatic hydrocarbon
u	Standard uncertainty
U	Expanded uncertainty
u^{*}_{bb}	Standard uncertainty related to a maximum between-unit inhomogeneity that could be hidden by method repeatability/intermediate precision; an additional index "rel" is added as appropriate
u_{bb}	Standard uncertainty related to a possible between-unit inhomogeneity; an additional index "rel" is added as appropriate
u_{c}	Combined standard uncertainty; 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_{rec}	Standard uncertainty related to possible between-unit inhomogeneity modelled as rectangular distribution; an additional index "rel" is added as appropriate
u_{sts}	Standard uncertainty of the short-term stability; an additional index "rel" is added as appropriate
α	Significance level
Δ_{meas}	Absolute difference between mean measured value and the certified value
$\nu_{s,meas}$	Degrees of freedom for the determination of the standard deviation s_{meas}
$\nu_{MSwithin}$	Degrees of freedom of MS_{within}
V/V	Volume fraction
$\bar{y}$	mean

1 Introduction

1.1 Background

The Fuel Quality Directive 98/70/EC [5] as amended by Directive 2009/30/EC [6], as regards the specification of petrol, diesel and gas-oil and introducing a mechanism to monitor and reduce greenhouse gas emissions, sets common fuel quality rules that are an important element for ensuring that air pollutant emissions from vehicles are optimally reduced, a single fuel market is established and vehicles operate correctly everywhere in the European Union.

In 1993 the European Committee for Standardization (CEN) was mandated by the European Commission and the European Free Trade Association [7] to develop a uniform standard that is defining product specifications and measurement methods for automotive diesel fuel, resulting in the documentary European Standard EN 590 [8]. This standard is designed to meet the needs of European business and industry, whilst also taking into account the legitimate concerns of consumers and other stakeholders, and the requirements of relevant European legislation [5, 6]. It is applicable to automotive diesel fuel for use in diesel engine vehicles designed to run on automotive diesel fuel containing a volume fraction of up to 7.0 % fatty acid methyl ester (FAME), so-called 'diesel (B7)'.

Many of the specified parameters in EN 590 [8] rely on 'method-specific' data obtained using standardised measurement procedures. However, the availability and use of these standard methods do not per se guarantee reliable measurement results. It is widely accepted that laboratories need to demonstrate their proficiency in the application of standard methods. ISO/IEC 17025 [3] explicitly states "*The laboratory shall confirm that it can properly operate standard methods before introducing the tests or calibrations. If the standard method changes, the confirmation shall be repeated*". In order to provide the analytical laboratories with the necessary tools for adequate quality assurance and quality control during the analysis of automotive diesel fuels, suitable CRMs are necessary.

ERM-EF003 is certified for selected parameters of EN 590 [8], i.e. the volume fraction of the fatty acid methyl ester (FAME), the mass fraction of the mono-aromatic hydrocarbon (MAH), di-aromatic hydrocarbon (DAH), polycyclic aromatic hydrocarbon (poly AH), total aromatic hydrocarbon (total AH) content, and density, kinematic viscosity, and lubricity. The provision of ERM-EF003 increases the comparability and reliability of measurements between laboratories, allowing laboratories to prove their competences/proficiency. Two other parameters, i.e. the cold filter plugging point and cloud point, are covered by ERM-EF004 [9].

1.2 Choice of the material

EN 590 [8] is applicable to automotive diesel fuel for use in diesel engine vehicles designed to run on automotive diesel fuel containing a volume fraction of up to 7 % fatty acid methyl ester. Hence, the chosen base material is a commercial automotive diesel fuel which was taken directly from the refinery blender unit without containing fatty acid methyl esters. This diesel was blended with biodiesel that is based on rapeseed oil fatty acid methyl ester with the addition of an antioxidant (butylhydroxytoluene), to achieve a volume fraction of 7 % biodiesel. The final blend, i.e. diesel (B7), was provided by a producer in Germany.

1.3 Design of the CRM project

The chosen parameters for this project were a selection of those listed in EN 590 [8]. Nine parameters (cetane number, cetane index, flash point, carbon residue, ash content, total contamination, copper strip corrosion, distillation, manganese content) had to be excluded for practical reasons, as their required sample intakes would have exceeded the 27 mL that was filled per unit. In total, 12 parameters were investigated, covering both chemical and physical properties (Table 1). For practical reasons, two other parameters, i.e. the cold filter plugging point and cloud point, are covered by ERM-EF004 [9]. The homogeneity and stability of the material were evaluated through studies involving measurement of all certified parameters using the documentary standards as listed in Table 1. The certified values were established by an intercomparison of different laboratories using all the same measurement methods for each parameter (Table 1).

Table 1: Selected parameters and corresponding documentary standards for measurements, and measurement principles (see Annex D, Table D1)

Parameter	Documentary standard	Method principle
Fatty acid methyl ester (FAME) content	EN 14078:2014 [10]	Infrared spectrometry method
Mono-aromatic hydrocarbon (MAH) content	EN 12916:2016 [11]	High performance liquid chromatography method with refractive index detection
Di-aromatic hydrocarbon (DAH) content	EN 12916:2016 [11]	
Tri+-aromatic hydrocarbon (T+AH) content	EN 12916:2016 [11]	
Polycyclic aromatic hydrocarbon (Poly AH) content	EN 12916:2016 [11]	
Total aromatic hydrocarbon (Total AH) content	EN 12916:2016 [11]	
Water content	EN 12937:2000 [12]	Coulometric Karl Fischer titration method
Sulfur content	ISO 20846:2011 [13]	Ultraviolet fluorescence method
Density at 15.0 °C	EN ISO 12185:1996 [14]	Oscillating U-tube method
Kinematic viscosity at 40.0 °C	EN ISO 3104:1996 [15]	Measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer.
Oxidation stability	EN 15751:2014 [16]	Accelerated oxidation method
Lubricity	ISO 12156-1:2016 [17]	High-frequency reciprocating rig (HFRR)

2 Participants

2.1 Project management and evaluation

European Commission, Joint Research Centre, Directorate F – Health, Consumers and Reference Materials, Reference Materials Unit, Geel, BE
(accredited to ISO Guide 34 for production of certified reference materials, BELAC No. 268-RM)

2.2 Processing

European Commission, Joint Research Centre, Directorate F – Health, Consumers and Reference Materials, Reference Materials Unit, Geel, BE
(accredited to ISO Guide 34 for production of certified reference materials, BELAC No. 268-RM)

2.3 Homogeneity study

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

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

2.4 Stability study

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

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

2.5 Characterisation

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

BfB Oil Research, Gembloux, BE

FUNDACIÓN CETENA, Noain, ES
(measurements partially under the scope of ISO/IEC 17025 accreditation ENAC 69/LE1062)

INNOVHUB - Stazioni Sperimentali per l'Industria, Milan, IT
(measurements partially under the scope of ISO/IEC 17025 accreditation ACCREDIA No. 0137)

INSPECTORATE Antwerp NV, Antwerp, BE
(measurements under the scope of ISO/IEC 17025 accreditation BELAC No 486-TEST)

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

INTERTEK - Immingham, Immingham, UK
(measurements under the scope of ISO/IEC 17025 accreditation UKAS No. 4162)

INTERTEK Iberica Spain, Bilbao, ES
(measurements under the scope of ISO/IEC 17025 accreditation DAC-N°40/LE911)

ITS Testing Services (UK) Limited (Teesside Laboratory), Cleveland, UK
(measurements partially under the scope of ISO/IEC 17025 accreditation UKAS No. 4106)

LABORATORIO DE COMBUSTIBLES – Universidade da Coruña, Ferrol, ES
(measurements under the scope of ISO/IEC 17025 accreditation ENAC No 814/LE1688)

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

SGS Belgium NV, Antwerp, BE
(measurements under the scope of ISO/IEC 17025 accreditation BELAC No. 005-TEST)

SGS ESPAÑOLA DE CONTROL, S.A.U., Barcelona, ES
(measurements under the scope of ISO/IEC 17025 accreditation ENAC 14/LE249 Rev.15)

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

3 Material processing and process control

3.1 Origin of the starting material

A commercial mixture of automotive diesel fuel and biodiesel based on rapeseed oil fatty acid methyl ester with the addition of about 1 g/kg of the antioxidant butylhydroxytoluene (supplier information) was selected as base material and provided by ASG Analytik-Service Gesellschaft mbH, Neusäss (DE). Three metal barrels containing in total 300 L of the material were delivered to the JRC at Geel. The compliance of the fuel with EN 590 [8] was verified by analysis of the fuel.

Table 2: Analysis of the starting material

Parameter	Unit	Result	Specification	Test method
Cetane number (DCN)	-	53.1	min. 51	EN 15195 [18]
Cetane Index	-	53.4	min. 46	EN ISO 4264 [19]
Density (15 °C)	[kg/m^3]	837.2	820 - 845	EN ISO 12185 [14]
Polycyclic aromatic hydrocarbon content	[% (m/m)]	2.3	max. 8	EN 12916 [11]
Sulfur content	[mg/kg]	7.7	max. 10	EN ISO 20884 [13]
Flash point	[°C]	74	>55	EN ISO 2719 [20]
Carbon residue (10 % Dist.)	[% (m/m)]	<0.10	max. 0.30	EN ISO 10370 [21]
Ash content (775 °C)	[% (m/m)]	0.005	max. 0.01	EN ISO 6245 [22]
Water content	[mg/kg]	30	max. 200	EN ISO 12937 [12]
Total contamination	[mg/kg]	3	max. 24	EN 12662 [23]
Copper strip corrosion	Korr.Grad	1	1	EN ISO 2160 [24]
Fatty acid methyl ester content	[% (V/V)]	6.8	max. 7	EN 14078 [10]
Oxidation stability	[g/m^3]	15	max. 25	EN ISO 12205 [25]
Filterable insolubles	[g/m^3]	2	-	EN ISO 12205 [25]
Adherent insolubles	[g/m^3]	13	-	EN ISO 12205 [25]
Oxidation stability	[h]	54.4	min. 20	EN 15751 [16]
HFRR (Lubricity)	[µm]	195	max. 460	EN ISO 12156-1 [17]
Kinematic viscosity (40 °C)	[mm^2/s]	2.888	2.00-4.50	EN ISO 3104 [15]
% (V/V) recovery at 250 °C	[% (V/V)]	31.9	<65	EN ISO 3405 [26]
% (V/V) recovery at 350 °C	[% (V/V)]	95.2	min. 85	EN ISO 3405 [26]
95 % (V/V) recovery	[°C]	349.5	max. 360	EN ISO 3405 [26]
CFPP	[°C]	-28	max. 0.5	EN 116 [27]
Manganese content	[mg/L]	<0.5	max. 120	EN 16576 [28]

3.2 Processing

Upon arrival at JRC at Geel the material was immediately stored at 18 °C in darkness until further treatment. Before ampouling, about 260 L of the material was transferred from the transport drums into a plastic drum over a 125 µm nylon filter to remove residual dust or particles. The material was mixed with an IKA Turbotron (Janke & Kunkel, Staufen, Germany) for 30 minutes and gently bubbled with nitrogen to homogenise the material. The main means of stabilisation were the addition of an antioxidant (butylhydroxytoluene) and application of an inert atmosphere. For the latter, nitrogen was gently bubbled through the material throughout the filling process. To remove most of the oxygen from the amber glass ampoules, they were flushed with argon over the headspace after filling with diesel (B7). Afterwards, the ampoules were flame-sealed. Ampouling was performed on a ROTA automatic ampouling machine, model R910/PA (ROTA Verpackungstechnik GmbH & Co.KG, Wehr, DE). The 30-mL amber glass ampoules were filled with 27 mL of diesel (B7). In total, 5000 ampoules were filled. In this report the term "unit" is used for each sample item / ampoule. Subsequently the ampoules were labelled in fill-order so that each unit is associated with a unique number.

3.3 Process control

During processing, 20 units were selected, two consecutive ampoules every 500th unit. Water measurements applying coulometric Karl Fischer titration were made on each unit. The water content did not show any trend in the filling sequence at a 99 % confidence level and was below 200 mg/kg, which was the predefined quality criterion. However, a trend in the filling sequence was detected at a 95 % confidence level. Due to the fact that the measured values were very low and close to the limit of quantification, it was decided to go ahead with the material, as the impact on other parameters was negligible as confirmed with the results obtained in the homogeneity study.

4 Homogeneity

A key requirement for any reference material aliquoted into units is equivalence between those units. In this respect, it is relevant whether the variation between units is significant compared to the uncertainty of the certified value, but it is not relevant if this variation between units is significant compared to the analytical variation. Consequently, ISO Guide 34 [1] requires RM producers to quantify the between unit variation. This aspect is covered in between-unit homogeneity studies.

The within-unit inhomogeneity does not influence the uncertainty of the certified value when the minimum sample intake is respected, but determines the minimum size of an aliquot that is representative for the whole unit. For all parameters the minimum sample intake is the required sample volume stipulated in the respective documentary standards.

4.1 Between-unit homogeneity

The between-unit homogeneity was evaluated to ensure that the certified values of the CRM are valid for all units of the material, within the stated uncertainties. The number of units selected for each parameter corresponds to approximately the cube root of the total number of units produced.

The units were selected using a random stratified sampling scheme covering the whole batch for the between-unit homogeneity test. For this, for each parameter the batch was divided into 18 groups (with a similar number of units) and one unit was selected randomly from each group. Two independent samples were taken from each selected unit, and analysed by using the respective standard methods of EN 590 [8] (Table 1).

A different design was used for the measurements of the density and kinematic viscosity, as the required sample intake only allow one analysis per unit. The principle of the setup is shown in Figure 1.

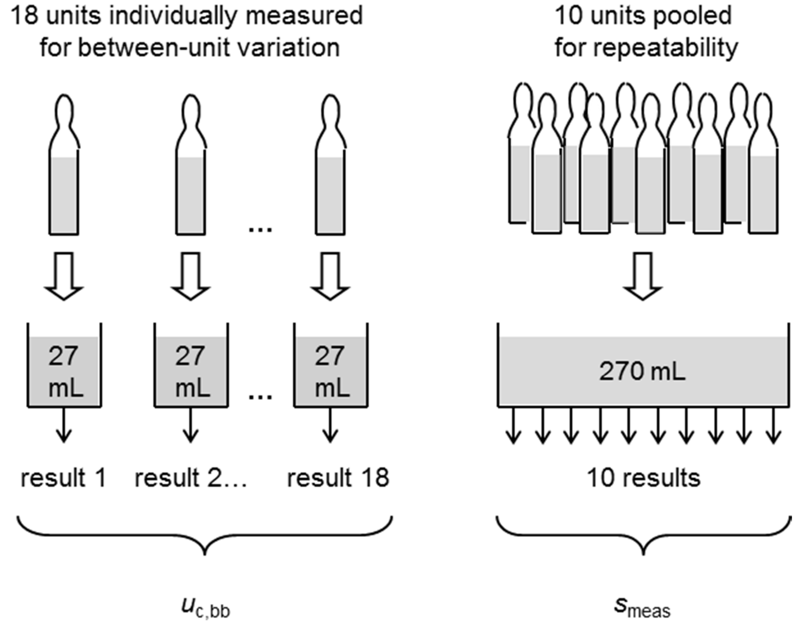

Figure 1: Setup of between homogeneity study for density and kinematic viscosity

As different units can only be measured once, the variability between results contains both repeatability and real between-unit variation. To obtain an assessment of the repeatability standard deviation of the laboratory, it was decided to pool 10 units, mix them and perform 10 replicate measurements from the pooled sample, whereas the between-unit measurements were done on the 18 individual units. Consequently, for each parameter, 28 units were selected using a random stratified sampling scheme. To this end, the batch was divided into groups (with a similar number of units) and one unit was selected randomly from each group.

The measurements for the fatty acid methyl ester content, mono-aromatic hydrocarbon, di-aromatic hydrocarbon, tri+-aromatic hydrocarbon, polycyclic aromatic hydrocarbon, total aromatic hydrocarbon content, sulfur content, density and kinematic viscosity were performed under repeatability conditions, and in a randomised manner to be able to separate a potential analytical drift from a trend in the filling sequence.

The measurements for the water content, oxidation stability and lubricity were performed under intermediate precision conditions (different days). Consequently, day-to-day effects can occur that could mask the between-bottle variation. Therefore, it had to be checked first if there is a significant difference between the day means using a t-test at a 95 % confidence level or ANOVA for the measurements spread over more than two days. Significant day to day effects were only present for lubricity, for which the measurements were spread over 9 days. Hence, a correction was applied by dividing every data point by the respective day mean in order to limit day-to-day effects in the between bottle uncertainty evaluation. The water content did not show a significant day-to-day effect. For the oxidation stability the measurements were spread over two days and on each day the measurements were done on three different instruments. In this case, it had to be first checked whether there is a significant difference between the instrument means using ANOVA. On both days significant instrument-to-instrument effects were present and consequently a correction was applied by dividing every data point by the respective instrument mean. Afterwards, a check of the normalised values for a significant difference between the day means using a t-test at a 95 % confidence level did not show any difference. All measurements were done in a randomised manner to be able to separate a potential analytical drift from a trend in the filling sequence. The results are shown as graphs in Annex A.

Regression analyses were performed to evaluate potential trends in the analytical sequence as well as trends in the filling sequence. No trends in the filling sequence or the analytical sequence were observed for the fatty acid methyl ester content, mono-aromatic hydrocarbon, di-aromatic hydrocarbon, tri+-aromatic hydrocarbon, polycyclic aromatic hydrocarbon, total aromatic hydrocarbon content, oxidation stability, and lubricity at a 95 % confidence level. A filling trend was detected for the water content. Some significant (95 % confidence level) trends in the analytical sequence were visible for the sulfur content, density, and kinematic viscosity, pointing at a changing parameter, e.g. a signal drift in the analytical system. The correction of biases, even if they are statistically not significant, was found to combine the smallest uncertainty with the highest probability to cover the true value [29]. Correction of trends is therefore expected to improve the sensitivity of the subsequent statistical analysis through a reduction in analytical variation without masking potential between-unit heterogeneities. As the analytical sequence and the unit numbers were not correlated, trends significant on at least a 95 % confidence level were corrected for sulfur, density and kinematic viscosity as shown below:

$$x_{i\_corr} = x_i - b \cdot i$$ **Equation 1**

b **=** slope of the linear regression

i **=** position of the result in the analytical sequence

All datasets (analytical trend corrected datasets for sulfur, density and kinematic viscosity) were assessed for consistency using Grubbs outlier tests at a confidence level of 99 % on the individual results and on the unit means. One outlying individual result was detected for kinematic viscosity. Since no technical reason for the outlier could be found, the data was retained for statistical analysis.

Quantification of between-unit inhomogeneity was undertaken by analysis of variance (ANOVA), which separates the between-unit variation (s_{bb}) from the within-unit variation (s_{wb}). The latter is equivalent to the method repeatability (fatty acid methyl ester content, mono-aromatic hydrocarbon, di-aromatic hydrocarbon, tri+-aromatic hydrocarbon, polycyclic aromatic hydrocarbon, total aromatic hydrocarbon content, sulfur content, density and kinematic viscosity) or intermediate precision (water content, oxidation stability and lubricity) if the individual samples were representative for the whole unit.

Evaluation by ANOVA requires mean values per unit, which follow at least a unimodal distribution and results for each unit that follow unimodal distributions with approximately the same standard deviations. The distribution of the mean values per unit was visually tested using histograms and normal probability plots. Too few data are available for the unit means to make a clear statement of the distribution. Therefore, it was checked visually whether all individual data follow a unimodal distribution using histograms and normal probability plots. Minor deviations from unimodality of the individual values do not significantly affect the estimate of between-unit standard deviations. The results of all statistical evaluations are given in Table 3.

Table 3: Results of the statistical evaluation of the homogeneity studies

Parameters	Trends [1)]		Outliers [2)]		Distribution	
	Analytical sequence	Filling sequence	Individual results	Unit means	Individual results	Unit means
FAME content	no	no	none	none	unimodal	unimodal
MAH content	no	no	none	none	unimodal	unimodal
DAH content	no	no	none	none	unimodal	unimodal
T+AH content	no	no	none	none	unimodal	unimodal
Poly AH content	no	no	none	none	unimodal	unimodal
Total AH content	no	no	none	none	unimodal	unimodal
Water content	no	yes	none	none	unimodal	unimodal
Sulfur content	yes	no	none	none	unimodal	unimodal
Density at 15.0 °C	yes	no	none	n.a. [5)]	unimodal	n.a. [5)]
Kinematic viscosity at 40.0 °C	yes	no	1-statistical reason (retained)	n.a. [5)]	unimodal	n.a. [5)]
Oxidation stability [3)]	no	no	none	none	unimodal	unimodal
Lubricity (HFRR) [4)]	no	no	none	none	unimodal	unimodal

[1)] 95 % confidence level
[2)] 99 % confidence level
[3)] Statistical evaluation done using instrument-to-instrument corrected data
[4)] Statistical evaluation done using day-to-day corrected data, due to non-repeatability conditions
[5)] n.a.: not applicable due to different study design

It should be noted that $s_{\mathrm{bb,rel}}$ and $s_{\mathrm{wb,rel}}$ are estimates of the true standard deviations and are therefore subject to random fluctuations. Therefore, the mean square between groups ($MS_{between}$) can be smaller than the mean squares within groups (MS_{within}), resulting in negative arguments under the square root used for the estimation of the between-unit variation, whereas the true variation cannot be lower than zero. In this case, u^*_{bb}, the maximum inhomogeneity that could be hidden by method repeatability, was calculated as described by Linsinger *et al.* [30]. u^*_{bb} is comparable to the limit of quantification of an analytical method, yielding the maximum inhomogeneity that might be undetected by the given study setup.

Method repeatability ($s_{wb,rel}$), between–unit standard deviation ($s_{bb,rel}$) and $u^*_{bb,rel}$ were calculated as:

$$s_{wb,rel} = \frac{\sqrt{MS_{within}}}{\overline{y}} \qquad \textbf{Equation 2}$$

$$s_{bb,rel} = \frac{\sqrt{\dfrac{MS_{between} - MS_{within}}{n}}}{\overline{y}} \qquad \textbf{Equation 3}$$

$$u^*_{bb,rel} = \frac{\sqrt{\dfrac{MS_{within}}{n}}\sqrt[4]{\dfrac{2}{\nu_{MSwithin}}}}{\overline{y}} \qquad \textbf{Equation 4}$$

MS_{within}	mean of squares within-unit from an ANOVA
$MS_{between}$	mean of squares between-unit from an ANOVA
$\overline{y}$	mean of all results of the homogeneity study
n	mean number of replicates per unit
$\nu_{MSwithin}$	degrees of freedom of MS_{within}

Due to the different study design used for the density the applied evaluation approach differed. To obtain the standard deviation between units (s_{bb}) the standard deviation from the 18 individual units ($u_{c,bb}$) must be corrected for the pure measurement standard deviation (s_{meas}) coming from the pooled sample as shown in Equation 5 [31].

$$s_{\text{bb,rel}} = \frac{\sqrt{u^2_{\text{c,bb}} - s^2_{\text{meas}}}}{\overline{y}} \qquad \textbf{Equation 5}$$

As in both cases $u_{c,bb}$ was smaller than s_{meas} the inhomogeneity that can be hidden by method repeatability is defined as follows, based on the recommendation of Reference [32]:

$$u^*_{\text{bb,rel}} = \frac{s_{\text{meas}*}\sqrt[4]{\dfrac{2}{\nu_{\text{s,meas}}}}}{\overline{y}} \qquad \textbf{Equation 6}$$

A different approach was adopted for kinematic viscosity for which an outlying value at the unit level was detected. In this case between-unit inhomogeneity was modelled as a rectangular distribution limited by the largest outlying unit value, and the rectangular standard uncertainty of homogeneity was estimated by:

$$u_{\text{rec}} = \frac{\left|outlier - \bar{y}\right|}{\sqrt{3} \cdot \bar{y}}$$ **Equation 7**

$\bar{y}$ mean of all results of the homogeneity study

When a trend in the filling sequence was significant at least at 99 % confidence level, the uncertainty was assessed in a different way. This applies for the water content. Here, u_{rec} was estimated using a rectangular distribution between the highest and lowest unit mean. The corrected uncertainty in those cases where there was a significant trend in the filling sequence is given in:

$$u_{rec} = \frac{\left|highest\ result\ \text{-}\ lowest\ result\right|}{2 \cdot \sqrt{3} \cdot \bar{y}}$$ **Equation 8**

The results of the evaluation of the between-unit variation are summarised in Table 4. The resulting values from the above equations were converted into relative uncertainties. In most cases, the uncertainty contribution for homogeneity was determined by the method repeatability.

Table 4: Results of the homogeneity studies

Parameter	$s_{\text{wb,rel}}$ [%]	$s_{\text{bb,rel}}$ [%]	$u^*_{\text{bb,rel}}$ [%]	$u_{\text{rec,rel}}$ [%]	$u_{\text{bb,rel}}$ [%]
FAME content	0.21	0.08	0.08	n.a. 2)	0.08
MAH content	2.50	0.49	1.02	n.a. 2)	1.02
DAH content	3.06	1.23	1.25	n.a. 2)	1.25
T+AH content	23.65	n.c. 1)	9.65	n.a. 2)	9.65
Poly AH content	3.59	n.c. 1)	1.46	n.a. 2)	1.46
Total AH content	2.43	n.c. 1)	0.99	n.a. 2)	0.99
Water content	3.28	11.70	1.34	12.15	12.15
Sulfur content	1.15	n.c. 1)	0.47	n.a. 2)	0.47
Density at 15.0 °C	0.0012	n.c. 1)	0.0008	n.a. 2)	0.0008
Kinematic viscosity at 40 °C	0.10	n.c. 1)	0.07	0.18	0.18
Oxidation stability	1.92	n.c. 1)	0.81	n.a. 2)	0.81
Lubricity	6.07	n.c. 1)	2.48	n.a. 2)	2.48

1) n.c.: cannot be calculated as $MS_{\text{between}} < MS_{\text{within}}$

2) n.a.: not applicable

The homogeneity study showed no outlying unit means or trends in the filling sequence for the fatty acid methyl ester content, polycyclic aromatic hydrocarbon content, sulfur content, density, oxidation stability and lubricity. Therefore the between-unit standard deviation s_{bb} can be used as estimate of u_{bb}. As u^*_{bb} sets the limits of the study to detect inhomogeneity, the larger value of s_{bb} and u^*_{bb} is adopted as uncertainty contribution to account for potential inhomogeneity.

One outlying unit value was found for kinematic viscosity. However, taking this extreme value into account, as it is representing the whole unit, the inhomogeneity as quantified as u_{rec} is still sufficiently small to make the material useful. Therefore, u_{rec} was used as estimate of u_{bb}.

For the water content a trend in the filling sequence was detected. In this case u_{rec}, calculated using the half-width of a rectangular distribution between the highest and lowest unit average, was used as estimate of $u_{bb.}$

4.2 Within-unit homogeneity and minimum sample intake

The within-unit homogeneity is closely correlated to the minimum sample intake. The minimum sample intake is the minimum amount of sample that is representative for the whole unit and thus should be used in an analysis. Using sample sizes equal or above the minimum sample intake guarantees the certified value within its stated uncertainty. The minimum sample intake is defined by the required sample volume stipulated in the respective documentary standards (Table 1).

5 Stability

Time, temperature, light and the presence of oxygen were regarded as the most relevant influences on stability of the material. Principal means of stabilisation were the addition of an antioxidant (butylhydroxytoluene), and creation of an inert atmosphere by flushing argon into the containment just before and after filling, removing the oxygen present, and by bubbling the material with nitrogen throughout the filling. The influence of ultraviolet or visible light was minimised by storing the material in containers which reduce light exposure. In addition, materials are stored in the dark and dispatched in boxes, thus removing any possibility of degradation by light. Therefore, only the influences of time and temperature needed to be investigated.

Stability testing is necessary to establish the conditions for storage (long-term stability) as well as the conditions for dispatch of the materials to the customers (short-term stability). During transport, especially in summer time, temperatures up to 60 °C can be reached and stability under these conditions must be demonstrated, if the samples are to be transported without any additional cooling.

The stability studies were carried out using an isochronous design [33]. In this approach, samples were stored for a particular length of time at different temperature conditions. Afterwards, the samples were moved to conditions where further degradation can be assumed to be negligible (reference conditions). At the end of the isochronous storage, the samples were analysed simultaneously whenever possible under repeatability conditions. Analysis of the material (after various exposure times and temperatures) under repeatability conditions greatly improves the sensitivity of the stability tests.

5.1 Short-term stability study

For the short-term stability study, samples were stored at -20 °C, 18 °C, 40 °C and 60 °C for 0, 1, 2 and 4 weeks (at each temperature). Storage at 60 °C mimics worst case conditions for transport during hot conditions, whereas storage at -20 °C checks whether first precipitates formed in the sample can be completely re-melted. The 18 °C and 40 °C studies acted as backup for the case that the material would degrade at 60 °C. The reference temperature was set to 4 °C. For all parameters, apart from density and kinematic viscosity, two units per storage time were selected using a random stratified sampling scheme. From each unit, two samples were measured using the standard methods as given in Table 1. For density and kinematic viscosity four units per storage time were selected using a random stratified sampling scheme, but only one measurement was done on each unit due to the higher sample amount needed. The measurements were performed under repeatability conditions for all parameters apart from lubricity, which were spread over four different working days due to the long time required for the measurements. All measurements were done in a randomised sequence to differentiate any potential analytical drift from a trend over storage time.

Significant (95 % confidence level) trends in the analytical sequence were visible for the sulfur content at all storage temperatures, for density at a storage temperature of 40 °C, and kinematic viscosity at a storage temperature of 18 °C, pointing at instability of the analytical systems. Hence, the data were corrected as described in Section 4.1 using Equation 1.

The data were evaluated individually for each temperature. The results were screened for outliers using the single and double Grubbs test on a confidence level of 99 %. Some outlying results were found (Table 5). As no technical reason for the outliers could be found all data were retained for statistical analysis. A tentative removal of the outliers did not change the outcome of the trend test. Only for the FAME content two outliers were detected and removed, as they were identified as technical outliers. The laboratory reported that a different measurement principle was applied compared to the remaining samples, as there was not enough sample left.

In addition, the data were evaluated against storage time, and regression lines of the determined parameters versus time were calculated, to test for potential increases/decrease of the individual parameters due to shipping conditions. The slopes of the regression lines were tested for statistical significance. None of the trends was statistically significant at a 95 % confidence level for any of the temperatures apart from the oxidation stability tested at 60 °C and the sulfur content tested at -20 °C.

The results of the measurements are graphically shown in Annex B. The results of the statistical evaluation of the short-term stability are summarised in Table 5.

Table 5: Results of the short-term stability tests

Parameters	Number of individual outlying results [1]				Trends [2]			
	-20 ºC	18 ºC	40 °C	60 °C	-20 ºC	18 ºC	40 °C	60 °C
FAME content	2 [4]	none	none	1 [5]	no	no	no	no
MAH content	none	1 [5]	1 [5]	none	no	no	no	no
DAH content	none	none	none	none	no	no	no	no
T+AH content	none	none	none	none	no	no	no	no
Poly AH content	none	none	none	none	no	no	no	no
Total AH content	none	1 [5]	none	none	no	no	no	no
Water content	none	2 [5]	none	none	no	no	no	no
Sulfur content	none	none	none	none	yes	no	no	no
Density at 15.0 °C	1 [5]	none	1 [5]	1 [5]	no	no	no	no
Kinematic viscosity at 40 °C	none	none	none	1 [5]	no	no	no	no
Oxidation stability	none	none	none	none	no	no	no	yes
Lubricity [3]	none	none	none	none	no	no	no	no

[1] 99 % confidence level
[2] 95 % confidence level
[3] day-to-day corrected data
[4] technical outliers (removed)
[5] statistical outliers (retained)

Statistical outliers were detected for the FAME content, MAH content, total AH content, water content, density and kinematic viscosity, and these were retained for the estimation of u_{sts}. For the majority of the parameters the trends were not statistically significant on a 95 % confidence level for any of the temperatures.

For the sulfur content a significant trend at -20 °C was found at a 95 % confidence level (not at a 99 % confidence level), but the material appeared to be stable at all the other temperatures. The analyte cannot be created in the sample as the ampoule is a closed system. The positive trend could indicate a degradation of the matrix. However, if that happened, the effect should be seen for all parameters, which is not the case. The observed trend was therefore regarded as statistical artefact.

The only positive trend at 60 °C was observed for the oxidation stability. At 40 °C none of the parameters showed instability.

Consequently, it was decided that the material can be dispatched under ambient conditions. However, a temperature indicator for 40 °C will be added to the shipment, to ensure that the shipping temperatures exceeding this temperature can be detected and remediated.

5.2 Long-term stability study

For the long-term stability study, samples were stored at 18 °C for 0, 4, 8 and 12 months. The reference temperature was set to 4 °C. For all parameters, apart from density and kinematic viscosity, two units per storage time were selected using a random stratified sampling scheme. From each unit, two samples were measured using the standard methods as given in Table 1. For density and kinematic viscosity four units per storage time were selected using a random stratified sampling scheme, but only one measurement was done on each unit due to the higher sample amount needed. The measurements were performed under repeatability conditions for all parameters apart from oxidation stability and lubricity, which were spread respectively over two and three different working days due to the long time required for the measurements. All measurements were done in a randomised sequence to differentiate any potential analytical drift from a trend over storage time.

A significant trend (95 % confidence level) in the analytical sequence was visible for the fatty acid methyl ester content, pointing at instability of the analytical system. Hence, the data were corrected as described in Section 4.1 using Equation 1.

The results were screened for outliers using the single and double Grubbs test at a confidence level of 99 %. Some outlying individual results were found for density and kinematic viscosity (Table 6). As no technical reason for the outliers could be found all data were retained for statistical analysis. A tentative removal of outliers did not change the outcome of the trend test.

In addition, the data were plotted against storage time and linear regression lines of the determined parameters versus time were calculated. The slopes of the regression lines were tested for statistical significance (loss/increase due to storage). No significant trend was detected for most parameters at a 95 % confidence level. A significant negative trend was detected for di-aromatic hydrocarbon, tri+-aromatic hydrocarbon, polycyclic aromatic hydrocarbon and total aromatic hydrocarbon content.

The results of the long-term stability measurements are shown in Annex C. The results of the statistical evaluation of the long-term stability study are summarised in Table 6.

Table 6: Results of the long-term stability tests at 18 °C

Parameter	Number of individual outlying results [1]	Trend [2]
FAME content	none	no
MAH content	none	no
DAH content	none	yes
T+AH content	none	yes
Poly AH content	none	yes
Total AH content	none	yes
Water content	none	no
Sulfur content	none	no
Density at 15 °C	1 statistical outlier (retained)	no
Kinematic viscosity at 40 °C	1 statistical outlier (retained)	no
Oxidation stability	none	no
Lubricity	none	no

[1] 99 % confidence level
[2] 95 % confidence level

Statistical outliers were observed for density and kinematic viscosity but for none of the other parameters. Trends were detected for di-aromatic hydrocarbon, tri+-aromatic hydrocarbon, polycyclic aromatic hydrocarbon and total aromatic hydrocarbon content. No technical explanation could be found for their decrease, and the observed trends were regarded as statistical artefact. Moreover, by taking the standard deviation from the homogeneity study, the whole range of the obtained values is covered. However, without additional evidence for their stability, their mass fractions are given with combined uncertainties with u_{lts} including potential degradation of the material. Consequently, the material can be stored at 18 ± 5 °C. When additional information becomes available as part of a two year long-term stability study, it may be possible to confirm stability.

5.3 Estimation of uncertainties

Due to the intrinsic variation of measurement results, no study can entirely rule out degradation of materials, even in the absence of statistically significant trends. It is therefore necessary to quantify the potential degradation that could be hidden by the method repeatability/intermediate precision, i.e. to estimate the uncertainty of stability. This means that, even under ideal conditions, the outcome of a stability study can only be that there is no detectable degradation within an uncertainty to be estimated.

The uncertainties of stability during dispatch and storage were estimated, as described in [32] for each parameter. In this approach, the uncertainty of the linear regression line with a slope of zero was calculated. The uncertainty contributions u_{sts} and u_{lts} were calculated as the product of the chosen transport time/shelf life and the uncertainty of the regression lines as:

$$u_{sts,rel} = \frac{s_{rel}}{\sqrt{\sum\left(t_i - \bar{t}\right)^2}} \cdot t_{tt}$$ **Equation 9**

$$u_{lts,rel} = \frac{s_{rel}}{\sqrt{\sum\left(t_i - \bar{t}\right)^2}} \cdot t_{sl}$$ **Equation 10**

s_{rel}	relative standard deviation of all results of the stability study
t_i	time elapsed at time point *i*
$\bar{t}$	mean of all t_i
t_{tt}	chosen transport time (1 week at 40 ºC)
t_{sl}	chosen shelf life (24 months at 18 ºC)

For a few parameters (di-aromatic hydrocarbon, tri+-aromatic hydrocarbon, polycyclic aromatic hydrocarbon and total aromatic hydrocarbon content), for which a significant trend was found, u_{lts} comprises two main contributions. A term due to the degradation mentioned in 5.2 corresponding to a bias (u_{b1}), calculated as a rectangular distribution of the slope (*b*), and a second term, which considers the uncertainty associated to such bias (u_{b2}) including potential degradation of the material, are given. The u_{lts}, within the chosen shelf life of the material (t_{sl} = 24 months at 18 °C), is estimated as follows:

$$u_{\mathrm{lts,rel}} = \sqrt{u_{b1}^2 + u_{b2}^2} \cdot t_{sl}$$ **Equation 11**

where,

$$u_{\mathrm{b1}} = \frac{b}{\sqrt{3}}$$ **Equation 12**

$$u_{\mathrm{b2}} = \frac{s_{rel}}{\sqrt{\sum\left(t_i - \bar{t}\right)^2}}$$ **Equation 13**

The following uncertainties were estimated:

$u_{sts,rel}$, the uncertainty of degradation during dispatch. This was estimated from the 40 °C studies. The uncertainty describes the possible change during a dispatch at 40 °C lasting for one week.

$u_{lts,rel}$, the stability during storage. This uncertainty contribution was estimated from the 18 °C studies. The uncertainty contribution describes the possible degradation during 24 months storage at 18 °C.

The results of these evaluations are summarised in Table 7.

Table 7: Uncertainties of stability during dispatch and storage. $u_{\mathrm{sts,rel}}$ was calculated for a temperature of 40 °C and 1 week; $u_{\mathrm{lts,rel}}$ was calculated for a storage temperature of 18 °C and 24 months

Parameters	$u_{\mathrm{sts\,,rel}}$ [%]	$u_{\mathrm{lts,rel}}$ [%]
	40 °C	18 °C
FAME content	0.02	0.58 $^{1)}$
MAH content	0.44	0.56
DAH content	0.42	3.26
T+AH content	2.33	26.26
Poly AH content	0.51	4.94
Total AH content	0.43	1.02
Water content	2.79	18.27
Sulfur content	0.17	6.98
Density at 15.0 °C	0.0003	0.0020
Kinematic viscosity at 40 °C	0.02	0.05
Oxidation stability	0.38	12.59
Lubricity	0.57	11.02

$^{1)}$ corrected for analytical trend

The material showed no significant degradation even at 60 °C apart for the oxidation stability, but for none of the parameters a significant degradation was observed for transport below 40 °C. Therefore, the material can be transported at ambient conditions. However, a temperature indicator for 40 °C will be added to the shipment, to ensure that the shipping temperatures exceeding this temperature can be detected and remediated.

The material can be stored at 18 ± 5 °C. Stability was confirmed by the outcome of a two year long-term stability study, which was completed just before release of the material. The trends detected for di-aromatic hydrocarbon, tri+-aromatic hydrocarbon, polycyclic aromatic hydrocarbon and total aromatic hydrocarbon content in the one year long-term stability study were not observed anymore in the two year long-term stability study.

After the certification study, the material will be included in the JRC's regular stability monitoring programme, to control its further stability.

6 Characterisation

The material characterisation is the process of determining the property values of a reference material. Because many of the parameters described in EN 590 [8] are operationally defined, certified values could only be obtained when a specific measurement protocol is strictly followed. In this case, the identity of the measurand would be defined by the applied standard method. Therefore, the material characterisation was based on an intercomparison of expert laboratories, i.e. the properties of the material were determined in different laboratories using all the same methods for the measurements (Table 8).

6.1 Selection of participants

For the interlaboratory comparison, between 10 to 14 laboratories were selected based on criteria that comprised both technical competence and quality management aspects. Each participant was required to operate a quality system and to deliver documented evidence of its laboratory proficiency for the respective parameters in the field of diesel measurements by submitting results for intercomparison exercises or method validation reports. Having a formal accreditation was not mandatory, but meeting the requirements of ISO/IEC 17025 [3] was obligatory. Where measurements are covered by the scope of accreditation, the accreditation number is stated in the list of participants (Section 2).

6.2 Study setup

For every parameter, apart from density and kinematic viscosity, each laboratory received three units of ERM-EF003 and was requested to provide six independent results, two per unit. For both, density and kinematic viscosity, they received six units of ERM-EF003 and were requested to provide six independent results, one per unit. The units for material characterisation were selected using a random stratified sampling scheme and covered the whole batch. The measurements had to be spread over at least three days to ensure intermediate precision conditions.

For the water content, oxidation stability, density and kinematic viscosity, each participant received samples of ERM-EF001 [34] as a blinded quality control (QC) sample. The results for this sample were used to support the evaluation of the characterisation results.

Laboratories were not requested to give estimations of the expanded uncertainties of the mean value of the six results, as the accuracy of the methods is described in the respective documentary standards. However, all laboratories were asked to follow strictly the standard test method protocols as provided in EN 590 [8]. Deviations thereof would result in a rejection of the submitted data set.

6.3 Methods used

All laboratories used for the individual parameters the same measurement methods as given in Table 8. A summary of the individual measurement methods, giving their scopes and principles, is listed in Annex D.

These documentary standards give information on expected repeatability and reproducibility limits. The absolute difference between two single test results obtained under repeatability conditions can be expected to be less than or equal to the value of r, the repeatability limit, with a certain probability (usually 95 %). A reproducibility limit, R, is similarly defined for test results obtained under reproducibility conditions [35]. A repeatability limit is calculated from:

$$r = t \times \sqrt{2} \times s_r \qquad \textbf{Equation 14}$$

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:

$R = t \times \sqrt{2} \times s_R$ **Equation 15**

where s_R is the reproducibility standard deviation.

As the standard deviation between laboratories (s_L) is [36]

$s_L = \sqrt{s_R^2 - s_r^2}$ **Equation 16**

and as the expanded measurement uncertainty (U_{meas}) of an average of n measurements is

$U_{meas} = 2 \cdot \sqrt{s_L^2 + \frac{s_r^2}{n}}$ **Equation 17**

expanded measurement uncertainties were estimated for n = 6 replicates (Annex D, Table D2).

Table 8: Measurement methods used and number of participating laboratories

Parameter	Methods used	No. of participants
FAME content	EN 14078:2014	11
MAH content	EN 12916:2016	11
DAH content	EN 12916:2016	11
T+AH content	EN 12916:2016	11
Poly AH content	EN 12916:2016	11
Total AH content	EN 12916:2020	11
Water content	EN 12937:2000	14
Sulfur content	ISO 20846:2011	14
Density at 15.0 °C	EN ISO 12185:1996	13
Kinematic viscosity at 40.0 °C	EN ISO 3104:1996	14
Oxidation stability	EN 15751:2014	12
Lubricity	ISO 12156-1:2016	10

6.4 Evaluation of results

The characterisation study resulted in different numbers of submitted datasets for the individual parameters (Table 8). All individual results of the participants, grouped per parameter are displayed in tabular and graphical form in Annex E.

6.4.1 Technical evaluation

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

- strictly follow documentary standard
- compliance with the analysis protocol: sample preparations and measurements performed on three days
- method performance,
    - agreement with performance criteria of documentary standards
        - 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 documentary standard (all parameters)
        - 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 documentary standard (all parameters)
    - agreement of the measurement results with the assigned value of the QC sample (water content, oxidation stability, density, and kinematic viscosity)
        - Datasets were rejected when the QC results did not agree with the assigned values of ERM-EF001 [34] according to ERM Application Note 1 [37], using for the uncertainty of the measured value the measurement uncertainties (u_{meas}) derived from the respective documentary standards as given in Annex D, Table D2

Based on the above criteria, the following datasets were rejected as not technically valid (Table 9).

Table 9: Datasets that showed non-compliances with the analysis protocol and technical specifications, and action taken

Parameter	Lab-method code	Description of problem	Action taken
FAME content	L8, L11	L8: r and R limit not met; wrong calibration range; L11: r limit not met	not used for evaluation
MAH content	L8, L13	L8: r limit not met; L13: R limit not met	not used for evaluation
DAH content	L8, L13	L8: r and R limit not met; L13: r limit not met	not used for evaluation
T+AH content	L8, L13	As T+AH are part of poly and total AHs, and both labs (L8 and L13) are excluded for the latter ones, they are removed for T+AH too	not used for evaluation
Poly AH content	L8, L13	L8: r limit not met; L13: R limit not met	not used for evaluation
Total AH content	L8, L13	L8: r limit not met; L13: R limit not met	not used for evaluation
Water content	L3, L14	L3: r limit not met; L14: failure to measure QC sample	not used for evaluation
Kinematic viscosity	L3	Failure to measure QC sample; R limit not met	not used for evaluation
Oxidation stability		Most of the reported values are out of the scope of the method	not used for evaluation

All laboratories complied with the analysis protocol and were strictly following the documentary standards. 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.

Laboratory 11 did not meet the repeatability limit for the FAME content. Laboratory 8 did not meet the repeatability and reproducibility limit for the FAME content. Moreover, laboratory 8 did not meet the method performance criteria for the poly AHs. This was also true for laboratory 13 for the poly AHs and laboratory 3 for the water content. As the laboratories confirmed that this was not a transcription error, the datasets were rejected.

The results of laboratory 14 for the water content and laboratory 3 for kinematic viscosity were not accepted, as the results of the QC sample did not agree with the actual assigned values. The laboratories confirmed that this was not a transcription error and consequently these datasets were rejected.

For most of the datasets for the oxidation stability the provided values were out of the limitation of the scope of the method, which is a maximum induction period of 48 h, reflecting the precision range of the method. The remaining laboratories provided datasets within the scope of the method. Due to this scattered picture, it was decided that no certified value will be assigned for this parameter. Hence, results in Annex E are only given in tabular form. However, an additional material information value is provided (Section 7.3).

6.4.2 Statistical evaluation

The datasets accepted based on technical reasons were tested for normality of dataset means using kurtosis/skewness tests (99 % confidence level) and 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 10.

Table 10: Statistical evaluation of the technically accepted datasets for ERM-EF003

p: number of technically valid datasets

Parameter	*p*	Outliers		Normally distributed	Statistical parameters				
		Means	Variances		Unit	Mean	*s*	s_{between}	s_{within}
FAME	9	none	yes (L5,7)	yes	[% (V/V)]	6.876	0.212	0.211	0.056
MAH	9	none	none	yes	[% (m/m)]	18.824	0.736	0.732	0.176
DAH	9	none	yes (L3)	yes	[% (m/m)]	1.8441	0.1986	0.1971	0.0594
T+AH	9	none	yes (L2,3,6)	yes	[% (m/m)]	0.17209	0.06141	0.06009	0.03092
Poly AH	9	none	yes (L3)	yes	[% (m/m)]	2.012	0.204	0.202	0.079
Total AH	9	none	yes (L3)	yes	[% (m/m)]	20.844	0.771	0.765	0.235
Water	12	none	none	yes	[% (m/m)]	0.00635	0.00061	0.00039	0.00117
Sulfur	14	none	yes (L6)	yes	[mg/kg]	7.508	0.846	0.844	0.148
Density	13	none	yes (L4)	yes	[kg/m^3]	837.231	0.105	0.104	0.041
Kinematic viscosity	13	none	none	yes	[mm^2/s]	2.8919	0.0075	0.0074	0.0033
Lubricity	10	none	none	yes	µm	223.507	39.176	38.360	19.486

For all parameters the laboratory means follow normal distributions. None of the data contains outlying means. The statistical evaluation flags some laboratories as outlying variance for the FAME content, DAH content, T+AH content, poly AH content, total AH content, sulfur content, density, and oxidation stability while their mean results for these parameters still agree with the other data. As all laboratories used the same methods, this demonstrates that the proficiency of these laboratories in applying the respective method is worse than the one of the other laboratories. Hence, all datasets were retained, as all results still agree well with the repeatability and reproducibility requirements of the respective documentary standards.

The uncertainty related to the characterisation (u_{char}) is estimated as the standard error of the mean of laboratory means ($s/\sqrt{p}$) (Table 11).

Table 11: Uncertainty of characterisation for ERM-EF003

Parameter	p	Unit	Mean	s	u_{char}
FAME content	9	[% (V/V)]	6.876	0.212	0.071
MAH content	9	[% (m/m)]	18.824	0.736	0.245
DAH content	9	[% (m/m)]	1.8441	0.1986	0.0662
T+AH content	9	[% (m/m)]	0.17209	0.06141	0.02047
Poly AH content	9	[% (m/m)]	2.012	0.204	0.068
Total AH content	9	[% (m/m)]	20.844	0.771	0.257
Water content	12	[% (m/m)]	0.00635	0.00061	0.000176
Sulfur content	14	[mg/kg]	7.508	0.846	0.226
Density at 15.0 °C	13	[kg/m^3]	837.231	0.105	0.029
Kinematic viscosity at 40 °C	13	[mm^2/s]	2.8919	0.0075	0.0021
Lubricity	10	[µm]	223.507	39.176	12.389

7 Value Assignment

Certified, indicative and informative values were assigned.

Certified values are values that fulfil the highest standards of accuracy. Procedures at the JRC require generally pooling of not less than 6 datasets to assign certified values. Full uncertainty budgets in accordance with the 'Guide to the Expression of Uncertainty in Measurement' [4] were established.

Indicative values are values where either the uncertainty is deemed too large or where too few independent datasets were available to allow certification. Uncertainties are evaluated according to the same rules as for certified values.

7.1 Certified values and their uncertainties

The unweighted mean of the means of the accepted datasets as shown in Table 11 was assigned as certified value for each parameter.

The assigned uncertainty consists of uncertainties relating to characterisation, u_{char} (Section 6), potential between-unit inhomogeneity, u_{bb} (Section 4.1), and potential degradation during transport, u_{sts}, and long-term storage, u_{lts} (Section 5.3). These different contributions were combined to estimate the relative expanded uncertainty of the certified value ($U_{\text{CRM, rel}}$) with a coverage factor k given as:

$$U_{\text{CRM,rel}} = k \cdot \sqrt{u_{\text{bb,rel}}^2 + u_{\text{sts,rel}}^2 + u_{\text{lts,rel}}^2 + u_{\text{char,rel}}^2}$$ **Equation 18**

u_{char} was estimated as described in Section 6

u_{bb} was estimated as described in Section 4.1

u_{sts} and u_{lts} were estimated as described in Section 5.3

Because of the sufficient numbers of the degrees of freedom of the different uncertainty contributions, a coverage factor k of 2 was applied, to obtain the expanded uncertainties. The certified values and their uncertainties are summarised in Table 12.

Table 12: Certified values and their uncertainties for ERM-EF003

Parameter	Unit	Certified value	$u_{\text{char, rel}}$ [%]	$u_{\text{bb, rel}}$ [%]	$u_{\text{sts, rel}}$ [%]	$u_{\text{lts, rel}}$ [%]	$U_{\text{CRM, rel}}$ [%]	U_{CRM} 1)
FAME content	[% (V/V)]	6.88	1.03	0.08	0.02	0.58	2.37	0.17
MAH content	[% (m/m)]	18.8	1.30	1.02	0.44	0.56	3.61	0.7
DAH content	[% (m/m)]	1.84	3.59	1.25	0.42	3.26	10.06	0.19
Poly AH content	[% (m/m)]	2.01	3.38	1.46	0.51	4.94	12.37	0.25
Total AH content	[% (m/m)]	20.8	1.23	0.99	0.43	1.02	3.86	0.9
Density at 15.0 °C	[kg/m^3]	837.23	0.0035	0.0008	0.0003	0.0020	0.0082	0.07
Kinematic viscosity at 40.0 °C	[mm^2/s]	2.892	0.07	0.18	0.02	0.05	0.39	0.012
Lubricity	[µm]	220	5.54	2.48	0.57	11.02	25.19	60

1) Expanded (k = 2) and rounded uncertainty

7.2 Indicative values and their uncertainties

Indicative values were assigned for the mass fraction of tri-aromatic hydrocarbon content, and the water content, as the estimated final uncertainties were considered too large for the final use of the CRM. Long-term stability uncertainty gives in both cases the highest contribution to the total uncertainty. However, as both of them were evaluated as all the other certified values, the results were regarded as sufficiently trustworthy to assign indicative values.

Additionally, an indicative value was assigned for the sulfur content, as the material used contains 7 % (V/V) FAME and the standard method only specifies a test method for the determination of the sulfur content of diesel fuels, including those containing up to 5 % (V/V) FAME. However, the standard says also that other products may be analysed and other sulfur contents may be determined. Moreover, the difference between the mean value of laboratory 7 and the other results is not covered by the measurement uncertainties (U_{meas}) according to ERM Application Note 1 [37]. However, as the difference between the mean value of laboratory 7 and the other results is only small, it was decided to increase the uncertainty of the assigned value to an extent that the results of laboratory 7 fulfil the condition of ERM Application Note 1 [37], and the results were regarded as sufficiently trustworthy to assign an indicative value.

An indicative value may not be used as certified value. The uncertainty budgets were set up as for the certified values and are listed together with the assigned value in Table 13.

Table 13: Indicative values and their uncertainties for ERM-EF003

Parameter	Unit	Indicative value	$u_{\text{char, rel}}$ [%]	$u_{\text{bb, rel}}$ [%]	$u_{\text{sts, rel}}$ [%]	$u_{\text{lts, rel}}$ [%]	$U_{\text{CRM, rel}}$ [%]	U_{CRM} 1)
T+AH content	[% (m/m)]	0.17	11.89	9.65	2.33	26.26	60.99	0.11
Water content	[% (m/m)]	0.0064	2.77	12.15	2.79	18.27	44.57	0.0029
Sulfur content	[mg/kg]	7.5	3.01	0.47	0.17	6.98	20.57	1.6 2)

1) Expanded (*k* = 2) and rounded uncertainty

2) Increased to an extent that the results of laboratory 7 fulfil the condition laid down in ERM Application Note 1 [37]

7.3 Additional material information

The data provided in this section should be regarded as informative only on the general properties of the material and cannot be, in any case, used as certified or indicative value.

An additional material information value was assigned for the oxidation stability measured according to EN 15751:2014 [16], as the determined value was outside the scope of the method. The value is the weighted mean value of means of 10 accepted datasets of data (see Table 14).

Table 14: Additional material information value for ERM-EF003

Parameter	Value [h]
Oxidation stability	52

8 Metrological traceability and commutability

8.1 Metrological traceability

Identity

All parameters are considered as method-defined measurands and can only be obtained by following the procedures specified in EN 590 [8]. The measurands are therefore operationally defined by the methods listed in Table 8.

Quantity value

Traceability of the obtained results is based on the traceability of all relevant input factors. Instruments in individual laboratories were verified and calibrated with tools ensuring traceability to the International System of units (SI). 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.

8.2 Commutability

Many measurement procedures include one or more steps which select specific (or specific groups of) analytes from the sample for the subsequent whole measurement process. Often the complete identity of these 'intermediate analytes' is not fully known or taken into account. Therefore, it is difficult to mimic all analytically relevant properties of real samples within a CRM. The degree of equivalence in the analytical behaviour of real samples and a CRM with respect to various measurement procedures (methods) is summarised in a concept called 'commutability of a reference material'. There are various definitions that define this concept. For instance, the CLSI Guideline C53-A [38] recommends the use of the following definition for the term *commutability*:

"The equivalence of the mathematical relationships among the results of different measurement procedures for an RM and for representative samples of the type intended to be measured."

The commutability of a CRM defines its fitness for use and is therefore a crucial characteristic when applying different measurement methods. When the commutability of a CRM is not established, the results from routinely used methods cannot be legitimately compared with the certified value to determine whether a bias does not exist in calibration, nor can the CRM be used as a calibrant.

As the material comes from an industrial, diesel producing plant, it is representative for other diesel samples and the analytical behaviour will be the same as for a routine diesel (B7) sample.

9 Instructions for use

9.1 Safety information

The classification is according to Regulation (EC) No. 1272/2008 [39] and the usual hazard and precautionary phrases for diesel apply:

H226 - Flammable liquid and vapour.
H304 - May be fatal if swallowed and enters airways.
H351 - Suspected of causing cancer.
H411 - Toxic to aquatic life with long lasting effects.

P210 - Keep away from heat/ /sparks/open flames/hot surfaces. No smoking.
P273 - Avoid release to the environment.
P280 - Wear protective gloves/protective clothing/eye protection/face protection.
P301+P310 - IF SWALLOWED: Immediately call a POISON CENTER or doctor/physician.
P308+P313 - IF exposed or concerned: Get medical advice/attention.

9.2 Storage conditions

The material should be stored at (18 ± 5) °C in the dark. Care should be taken once the units are open. The user should close any units immediately after taking a sample.

Please note that the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially for opened units.

9.3 Preparation and use of the material

The units shall be shaken by turning upside down for at least 2 min before opening to ensure material re-homogenisation.

9.4 Minimum sample intake

The minimum sample intake is the required sample volume stipulated in the respective documentary standard [8].

9.5 Use of the certified value

The main purpose of these materials is to assess method performance, i.e. for checking accuracy of analytical results/calibration. As any reference material, it can be used for establishing control charts or validation studies.

Comparing an analytical result with the certified value

A result is unbiased if the combined standard uncertainty of measurement and certified value covers the difference between the certified value and the measurement result (see also ERM Application Note 1, https://crm.jrc.ec.europa.eu/) [37].

When assessing the method performance, the measured values of the CRMs are compared with the certified values. The procedure is summarised here:

Calculate the absolute difference between mean measured value and the certified value (Δ_{meas}).

Combine the measurement uncertainty (u_{meas}) with the uncertainty of the certified value (u_{CRM}): $u_{\Delta} = \sqrt{u_{meas}^{2} + u_{CRM}^{2}}$

Calculate the expanded uncertainty (U_{Δ}) from the combined uncertainty ($u_{\Delta,}$) using an appropriate coverage factor, corresponding to a level of confidence of approximately 95 %

If $\Delta_{\text{meas}} \leq U_{\Delta}$ then no significant difference exists between the measurement result and the certified value, at a confidence level of approximately 95 %.

Use in quality control charts

The materials can be used for quality control charts. Using CRMs for quality control charts has the added value that a trueness assessment is built into the chart.

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Annexes

Annex A: Results of the homogeneity measurements

Annex B: Results of the short-term stability measurements

Annex C: Results of the long-term stability measurements

Annex D: Summary of methods used in the characterisation study

Annex E: Results of the characterisation measurements

Annex A: Results of the homogeneity measurements

Data points represent data as reported by the laboratories, unless indicated as "normalised" or "analytical trend corrected".

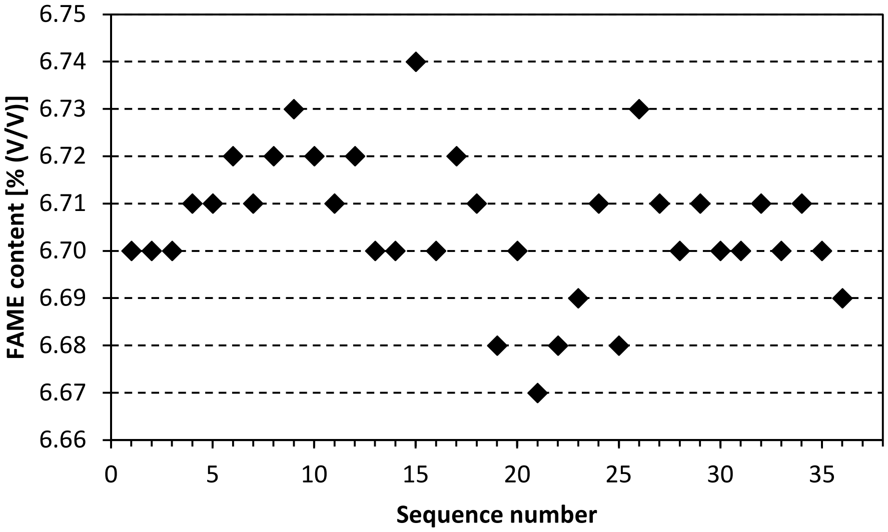

Figure A1: Individual measurement replicates for FAME content in the order of measurement.

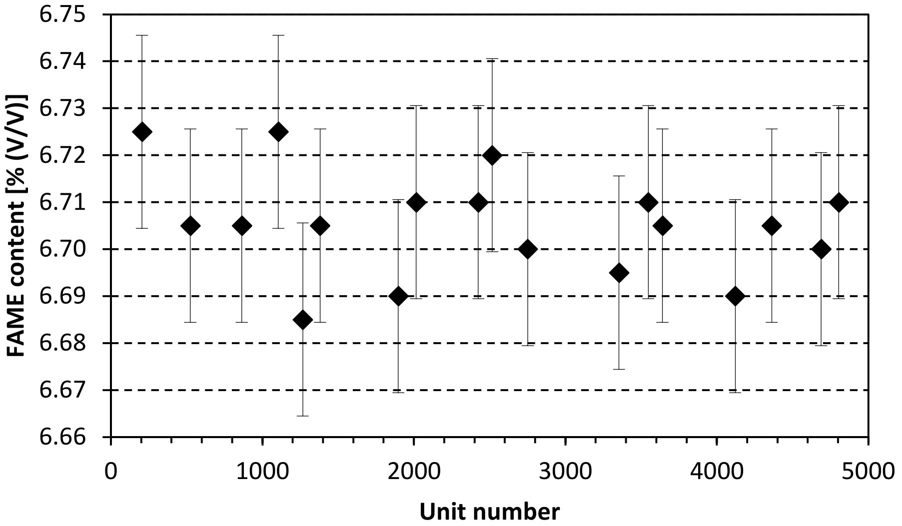

Figure A2: Unit means for FAME content, against unit number. Vertical bars are a 95 % confidence interval derived from s_{wb} from ANOVA for all units of the homogeneity study.

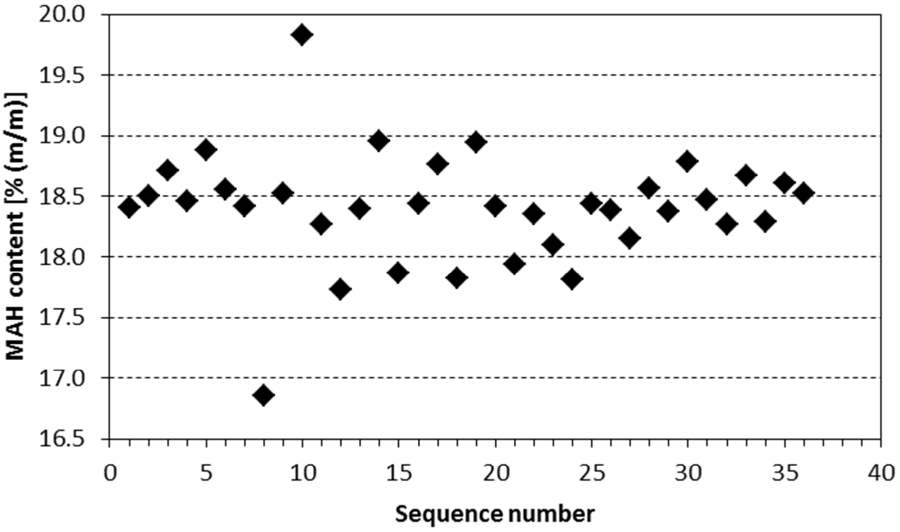

Figure A3: Individual measurement replicates for MAH content in the order of measurement.

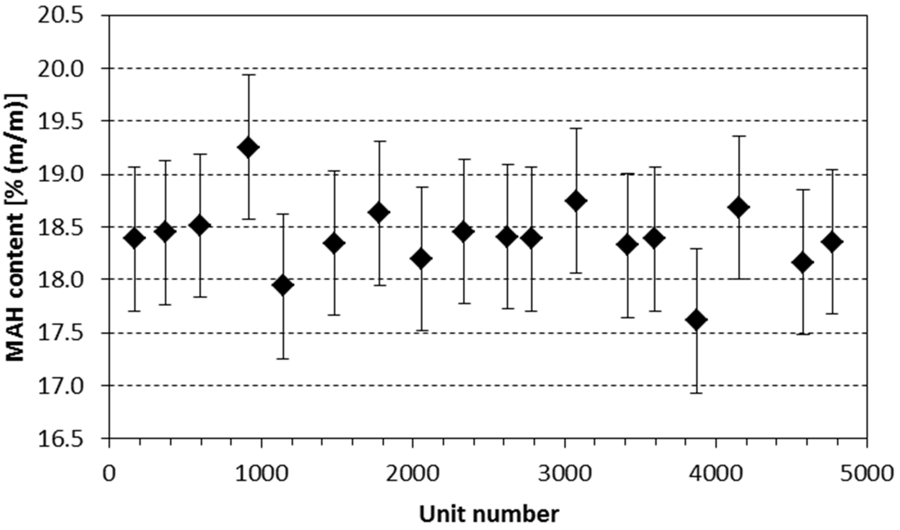

Figure A4: Unit means for MAH content, against unit number. Vertical bars are a 95 % confidence interval derived from s_{wb} from ANOVA for all units of the homogeneity study.

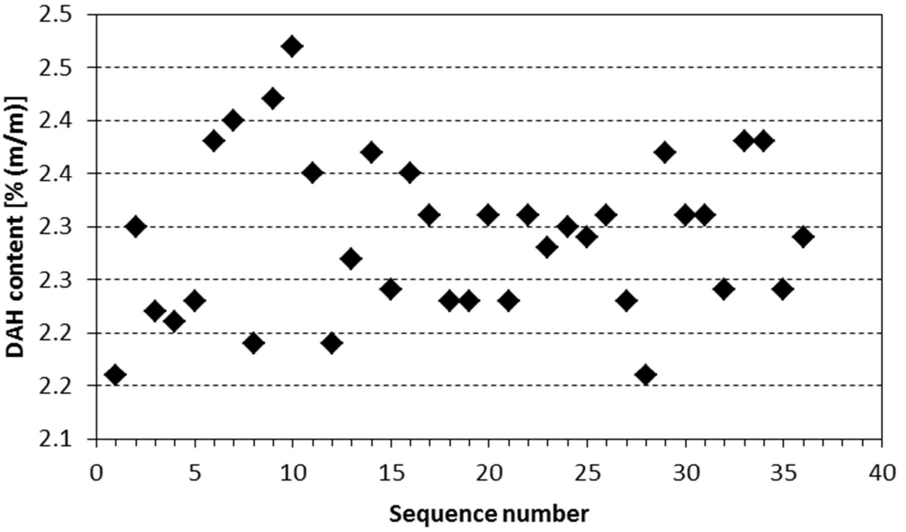

Figure A5: Individual measurement replicates for DAH content in the order of measurement.

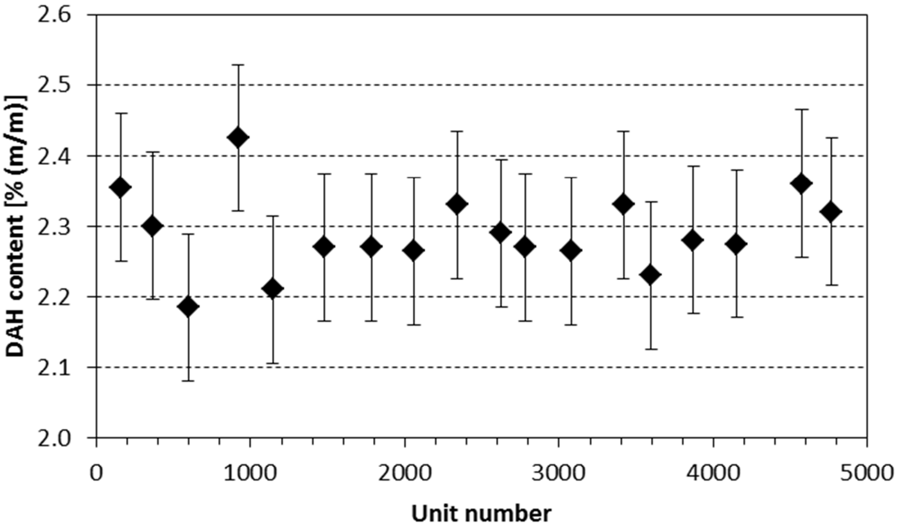

Figure A6: Unit means for DAH content, against unit number. Vertical bars are a 95 % confidence interval derived from s_{wb} from ANOVA for all units of the homogeneity study.

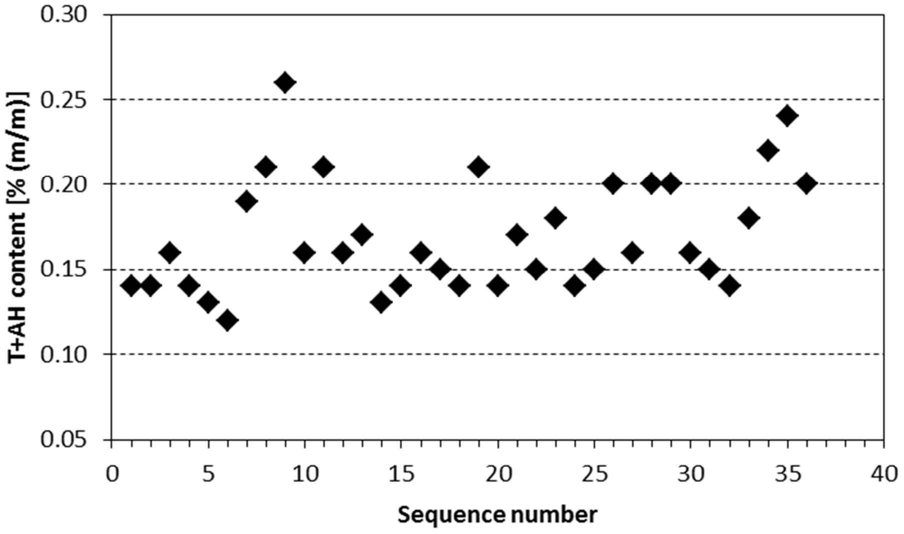

Figure A7: Individual measurement replicates for T+AH content in the order of measurement.

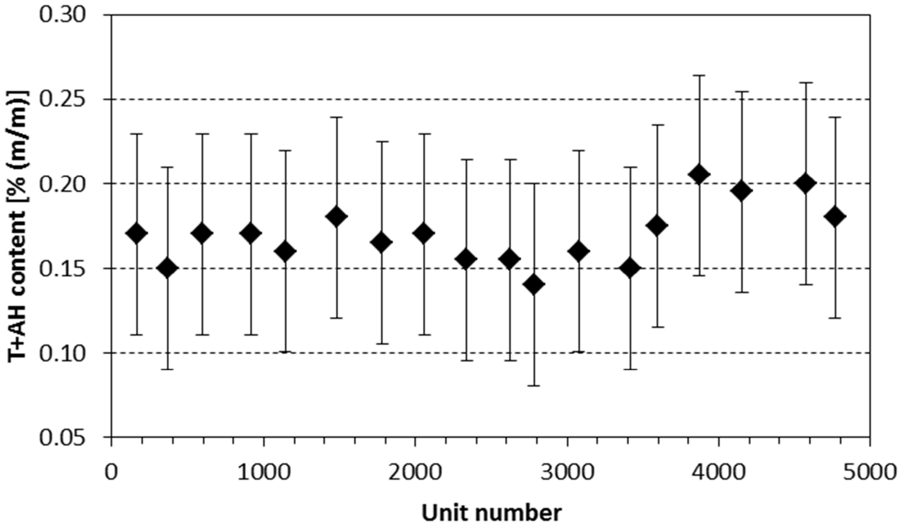

Figure A8: Unit means for T+AH content, against unit number. Vertical bars are a 95 % confidence interval derived from s_{wb} from ANOVA for all units of the homogeneity study.

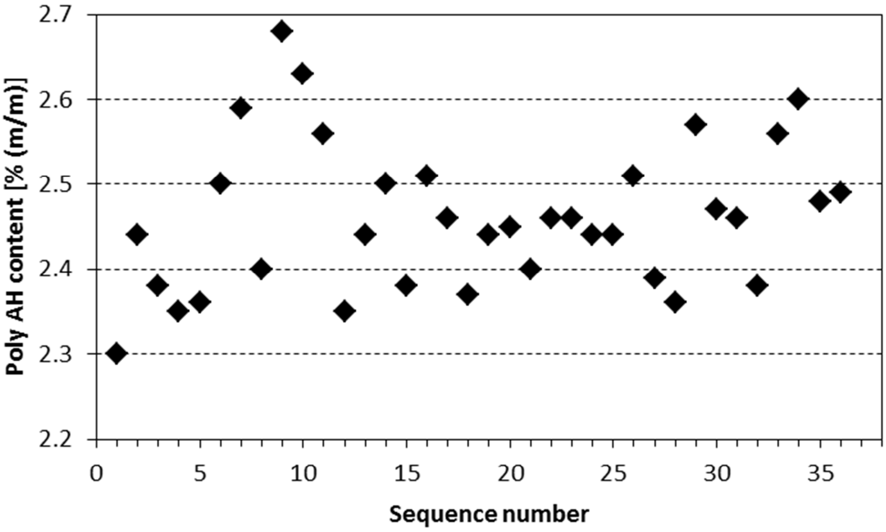

Figure A9: Individual measurement replicates for Poly AH content in the order of measurement.

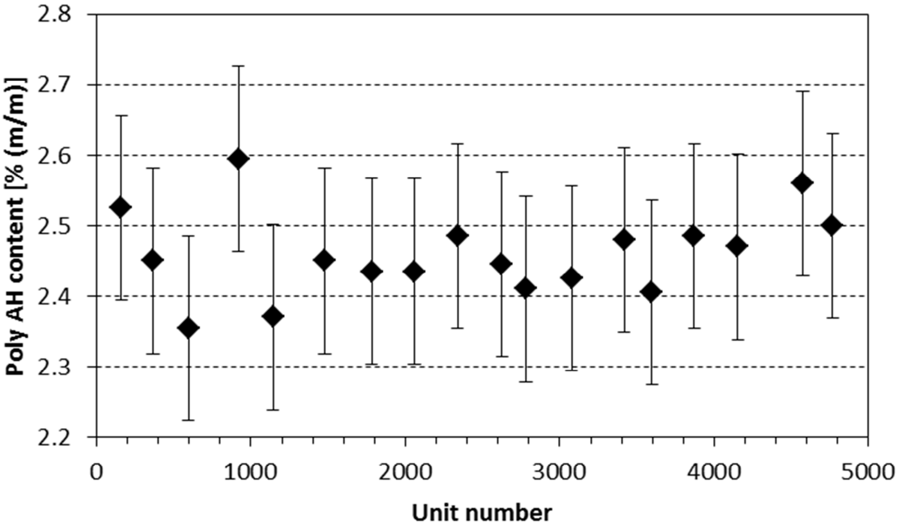

Figure A10: Unit means for Poly AH content, against unit number. Vertical bars are a 95 % confidence interval derived from s_{wb} from ANOVA for all units of the homogeneity study.

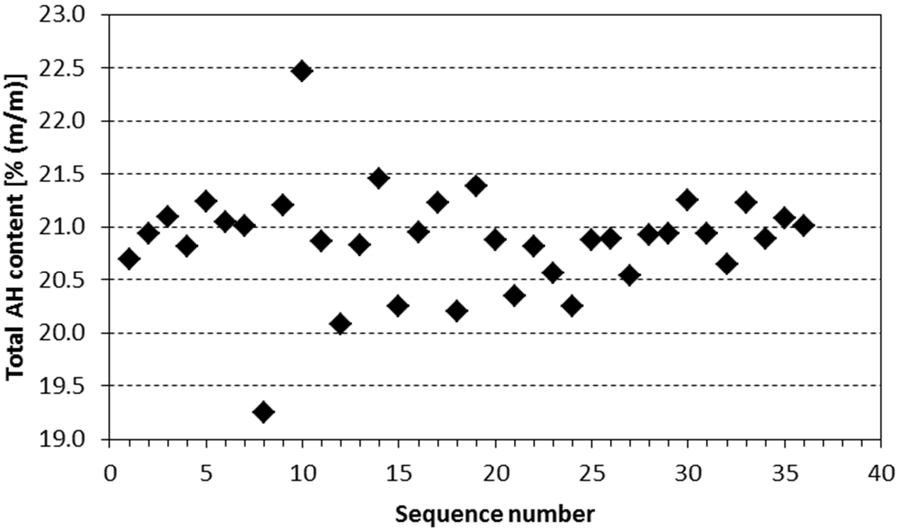

Figure A11: Individual measurement replicates for Total AH content in the order of measurement.

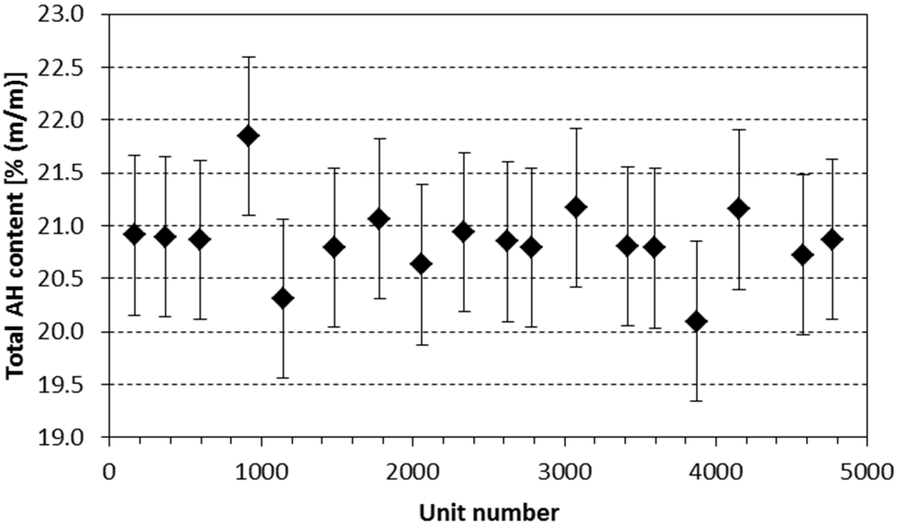

Figure A12: Unit means for Total AH content, against unit number. Vertical bars are a 95 % confidence interval derived from s_{wb} from ANOVA for all units of the homogeneity study.

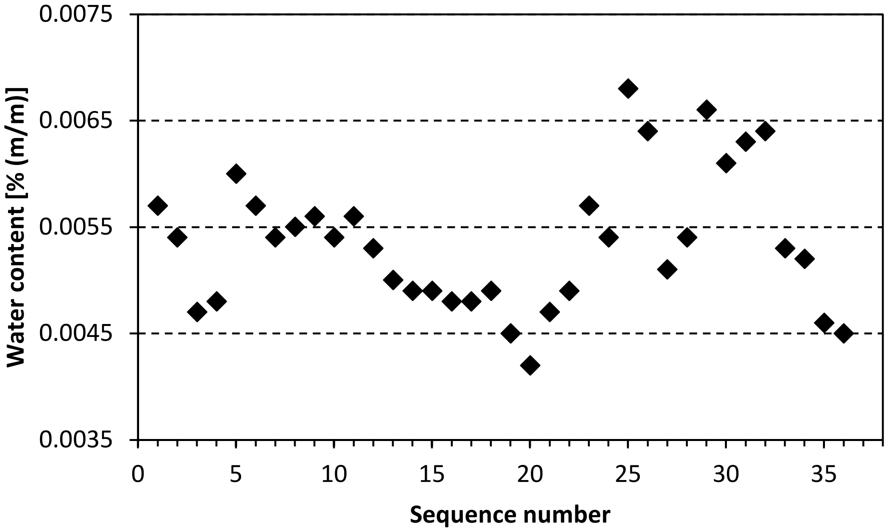

Figure A13: Individual measurement replicates for water content in the order of measurement.

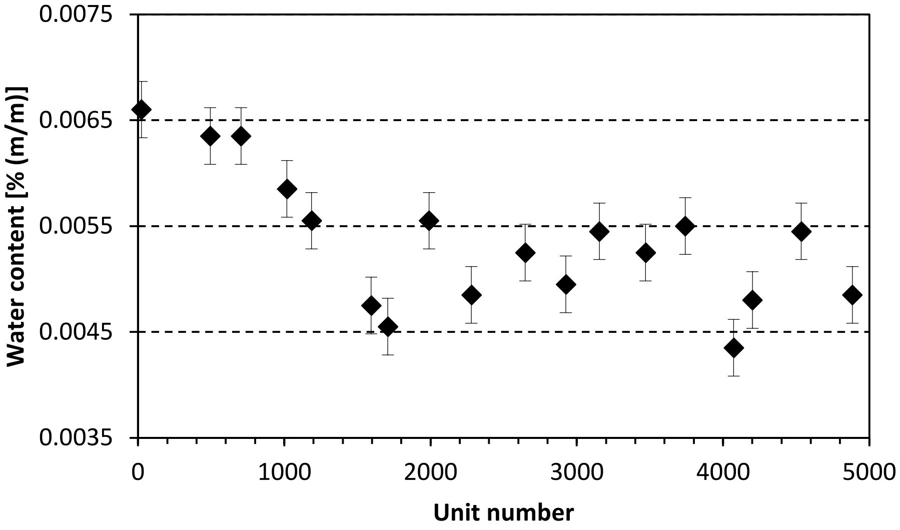

Figure A14: Unit means for water content, against unit number. Vertical bars are a 95 % confidence interval derived from s_{wb} from ANOVA for all units of the homogeneity study.

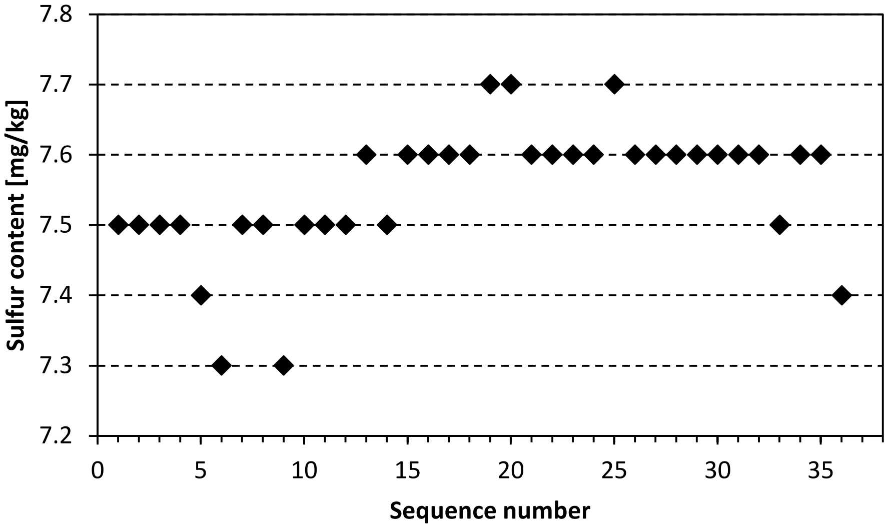

Figure A15: Individual measurement replicates for sulfur content in the order of measurement.

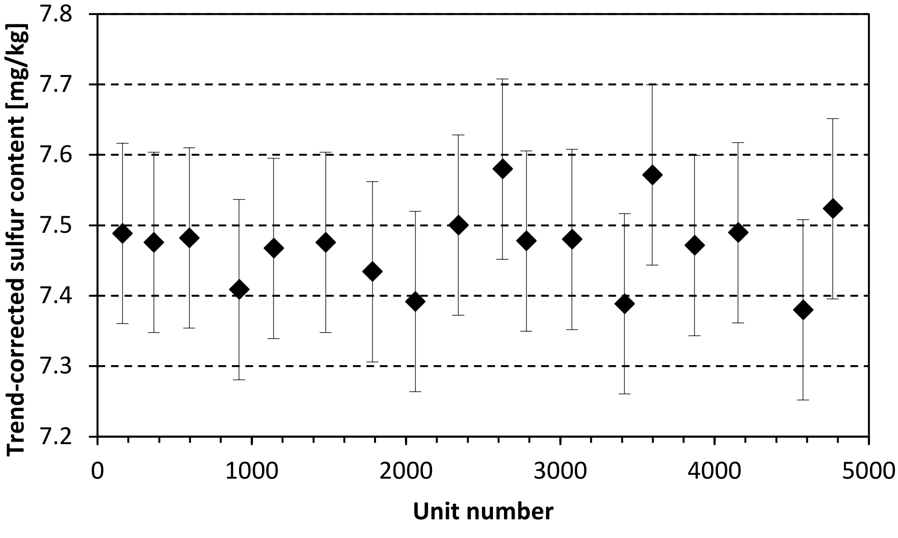

Figure A16: Analytical trend corrected unit means for sulfur content, against unit number. Vertical bars are a 95 % confidence interval derived from s_{wb} from ANOVA for all units of the homogeneity study.

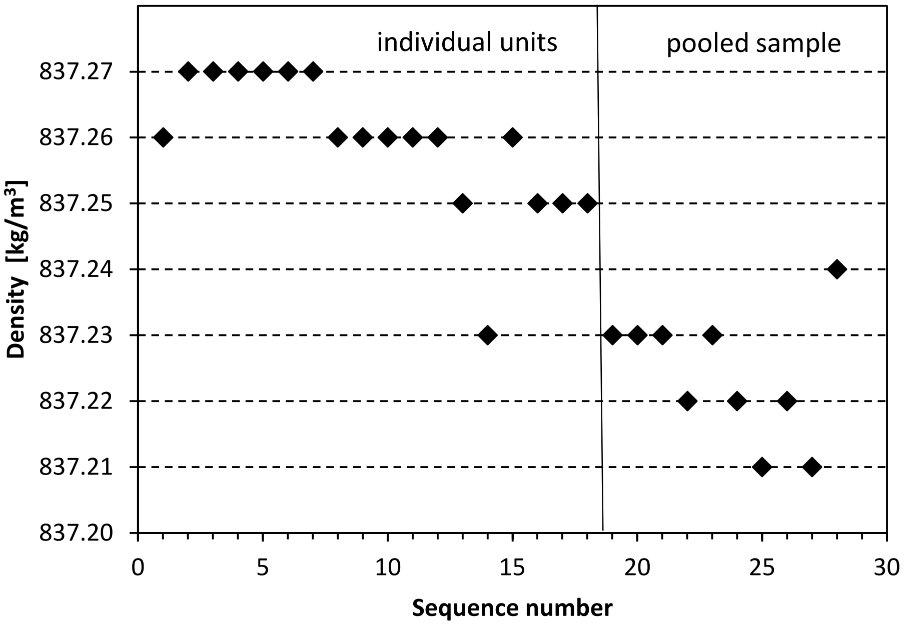

Figure A17: Individual measurements for density at 15.0 °C in the order of measurement. (Sequence number: measurements on 18 individual units and 10 measurements from pooled sample)

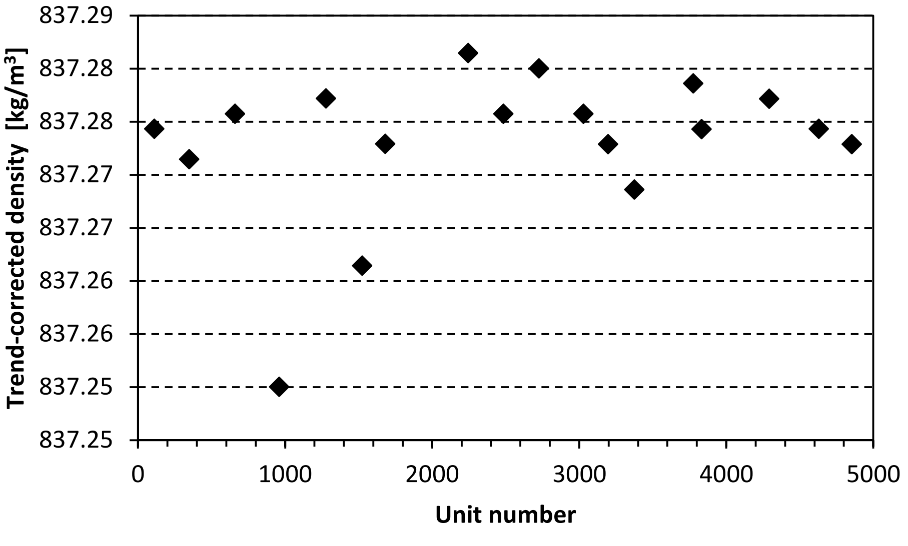

Figure A18: Analytical trend corrected unit values for density at 15.0 °C, against unit number.

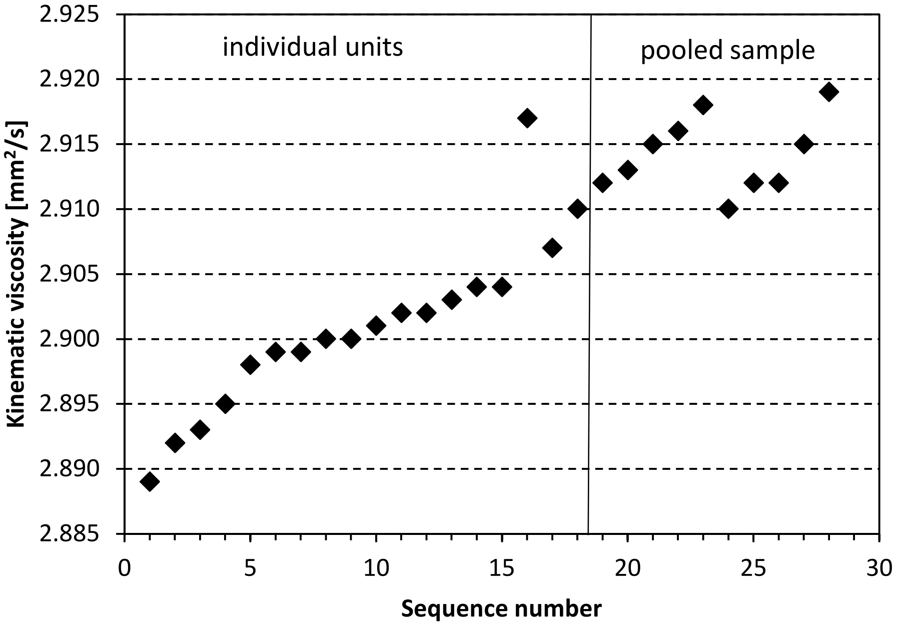

Figure A19: Individual measurements for kinematic viscosity at 40.0 °C in the order of measurement. (Sequence number: measurements on 18 individual units and 10 measurements from pooled sample)

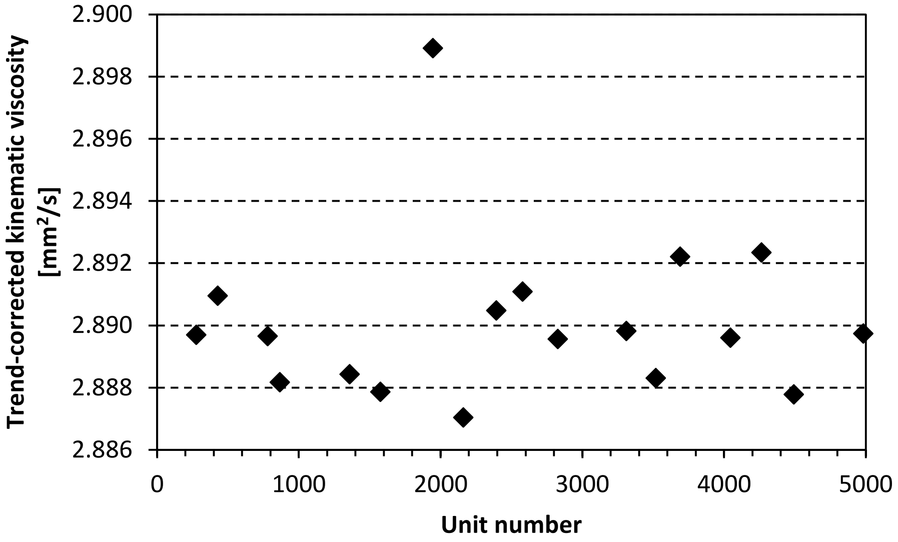

Figure A20: Analytical trend corrected unit values for kinematic viscosity at 40.0 °C, against unit number.

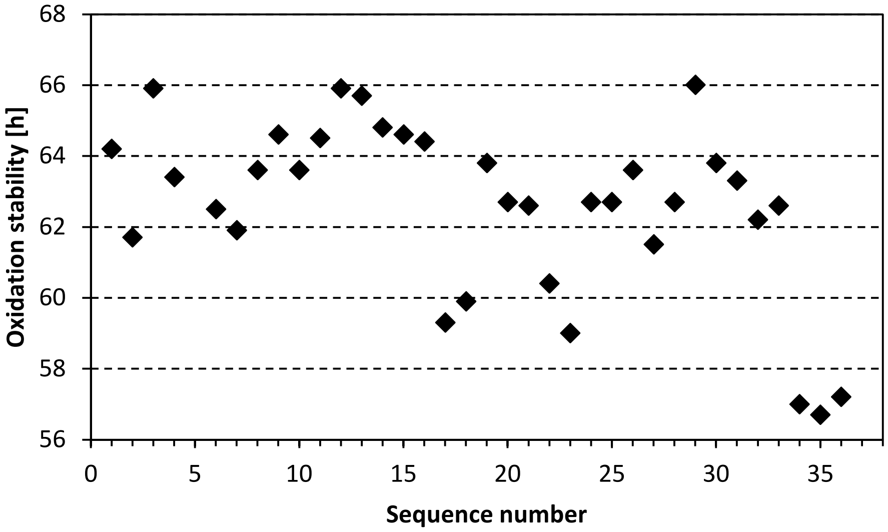

Figure A21: Individual measurement replicates for oxidation stability in the order of measurement.

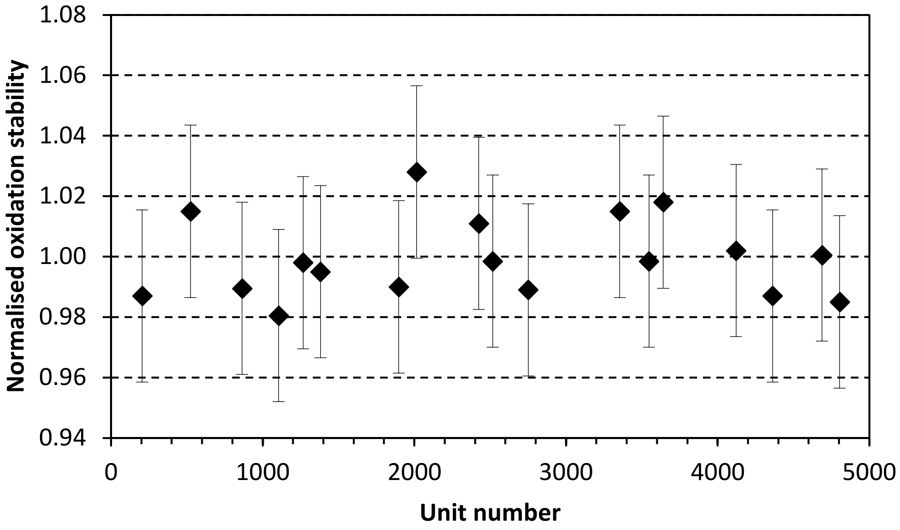

Figure A22: Normalised unit means for oxidation stability, against unit number. Vertical bars are a 95 % confidence interval derived from s_{wb} from ANOVA for all units of the homogeneity study.

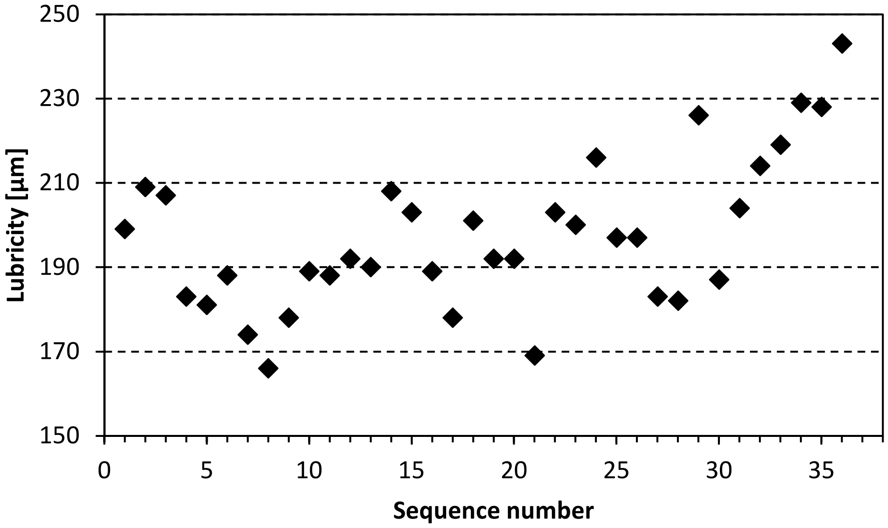

Figure A23: Individual measurement replicates for lubricity in the order of measurement.

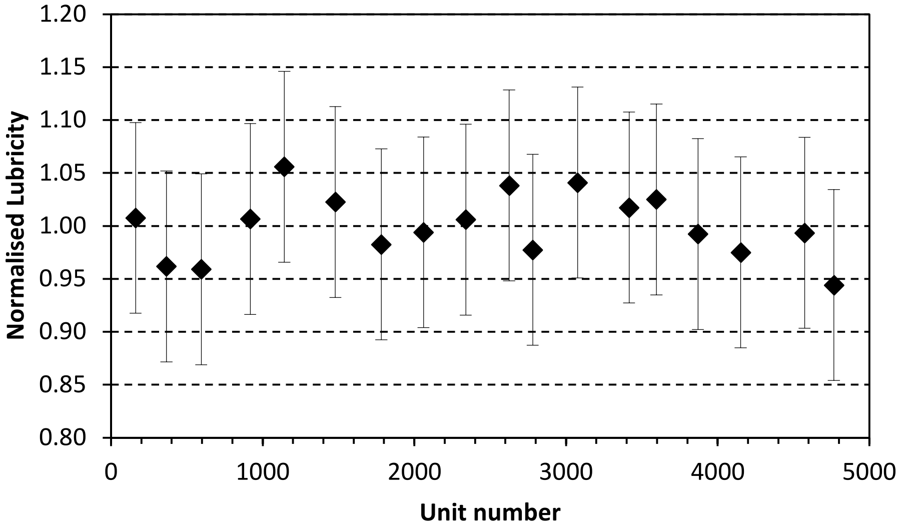

Figure A24: Normalised unit means for lubricity, against unit number. Vertical bars are a 95 % confidence interval derived from s_{wb} from ANOVA for all units of the homogeneity study.

Annex B: Results of the short-term stability measurements

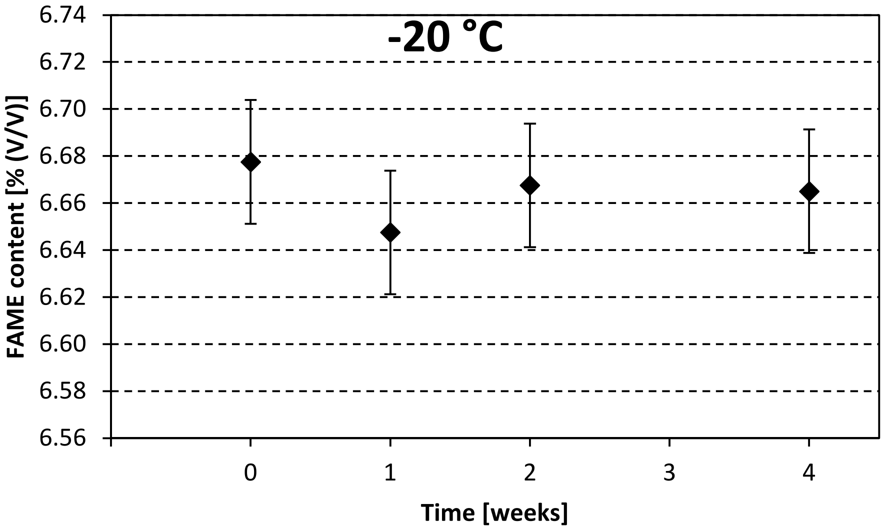

Figure B1: FAME content means measured at -20 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

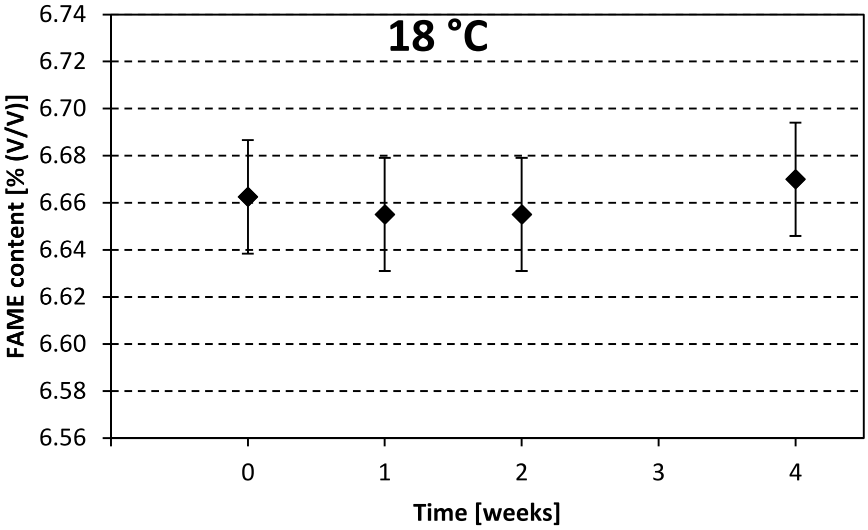

Figure B2: FAME content means measured at 18 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

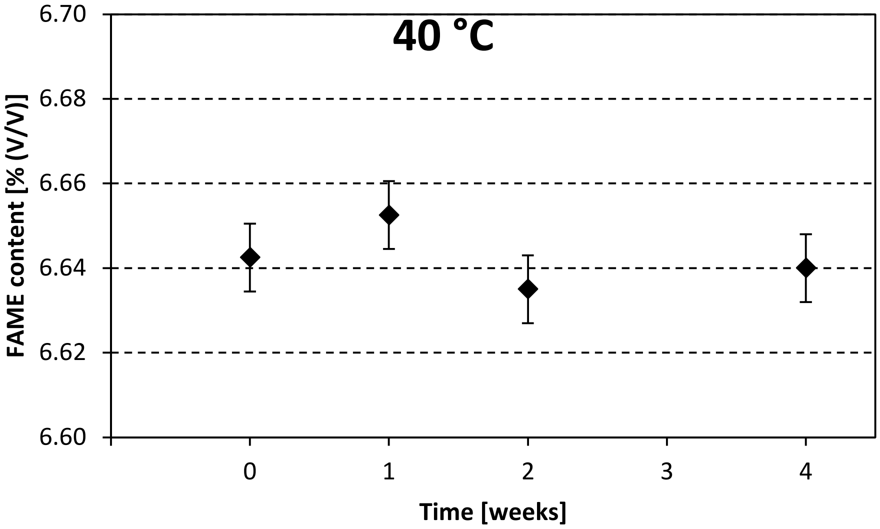

Figure B3: FAME content means measured at 40 °C each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

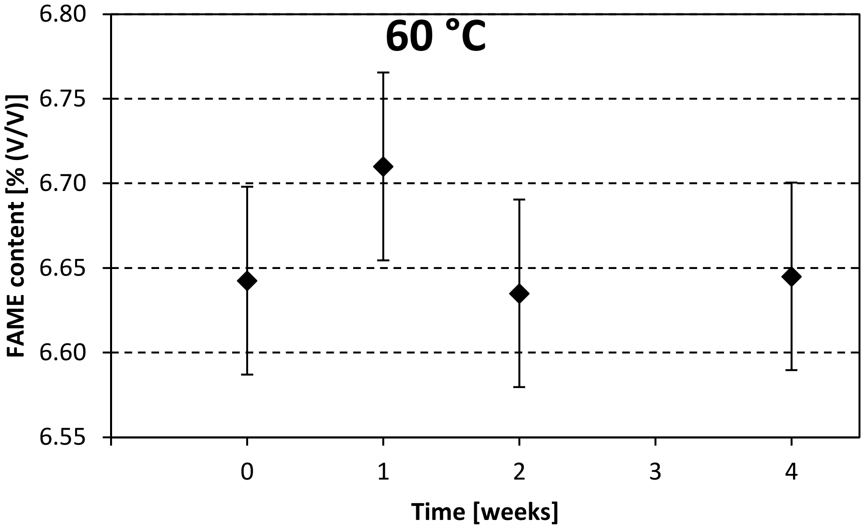

Figure B4: FAME content means measured at 60 °C each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

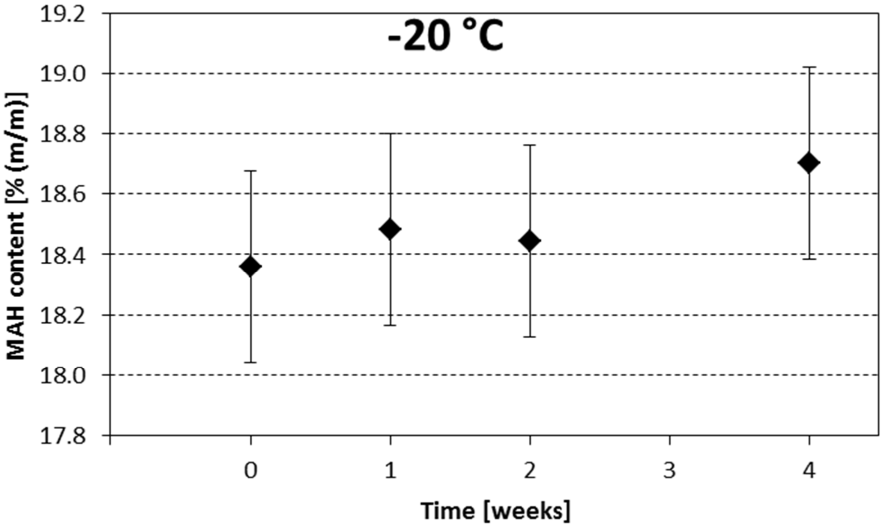

Figure B5: MAH content means measured at -20 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

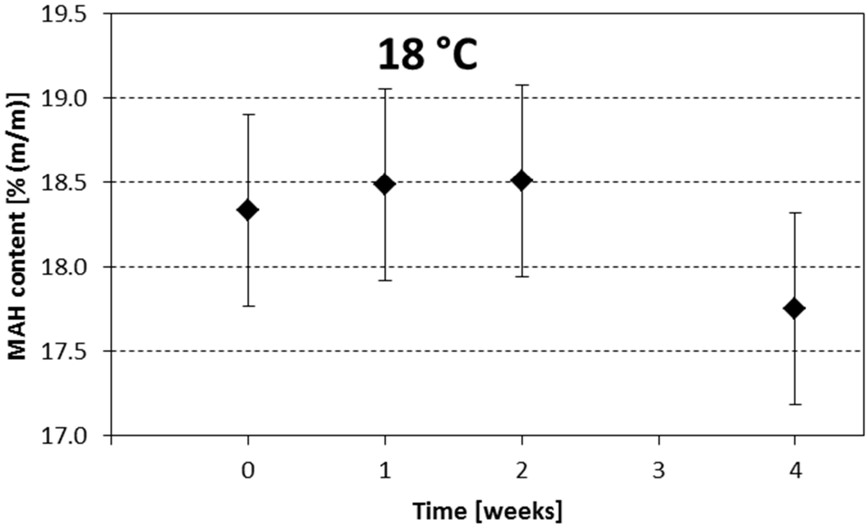

Figure B6: MAH content means measured at 18 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

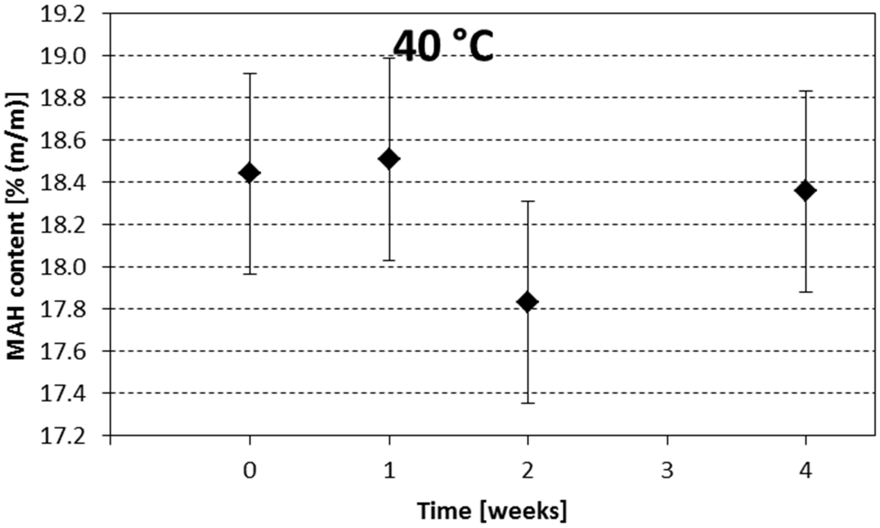

Figure B7: MAH content means measured at 40 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

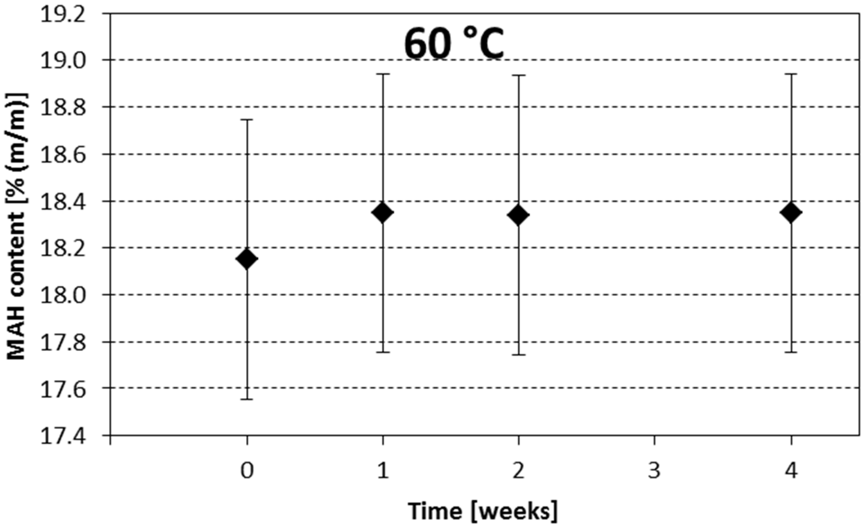

Figure B8: MAH content means measured at 60 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

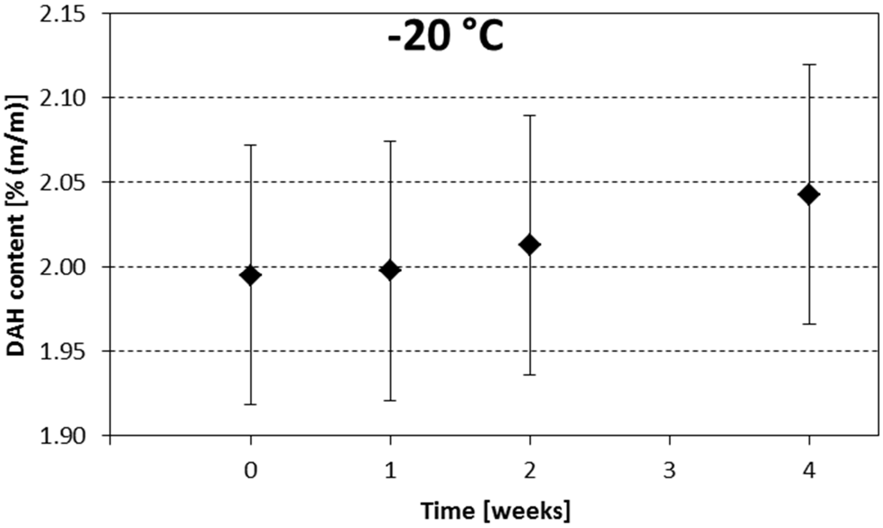

Figure B9: DAH content means measured at -20 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

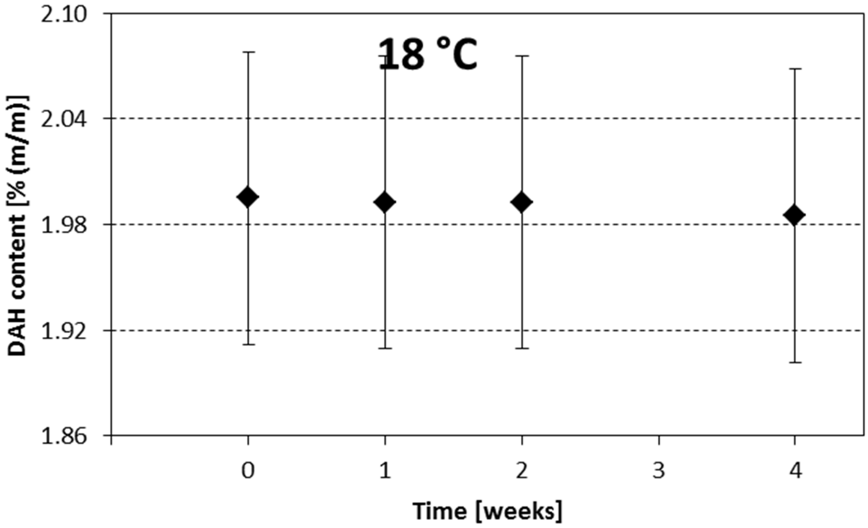

Figure B10: DAH content means measured at 18 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

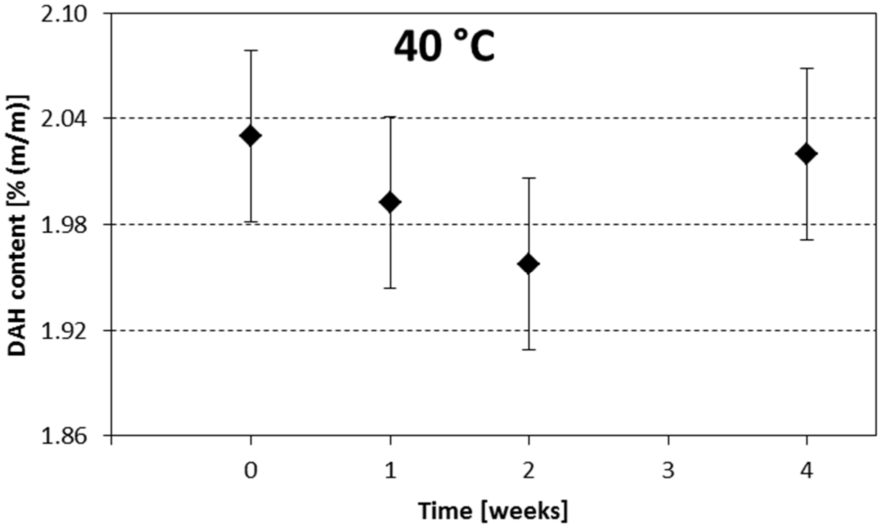

Figure B11: DAH content means measured at 40 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

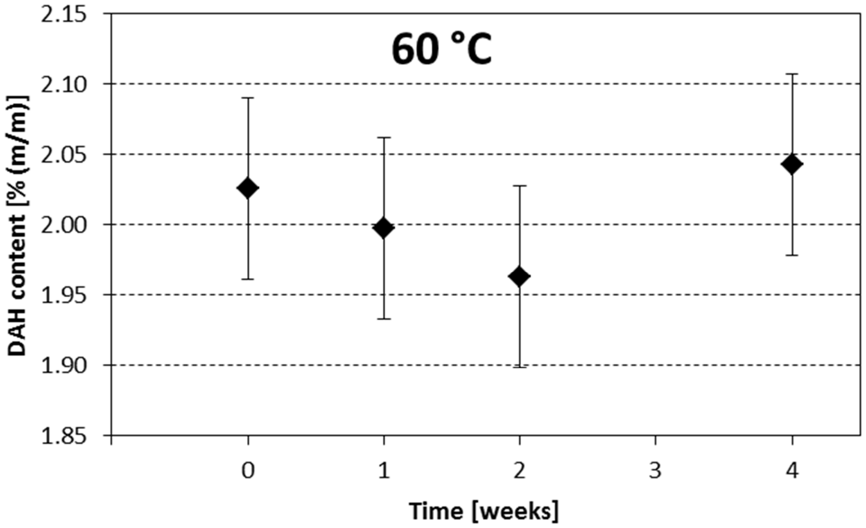

Figure B12: DAH content means measured at 60 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

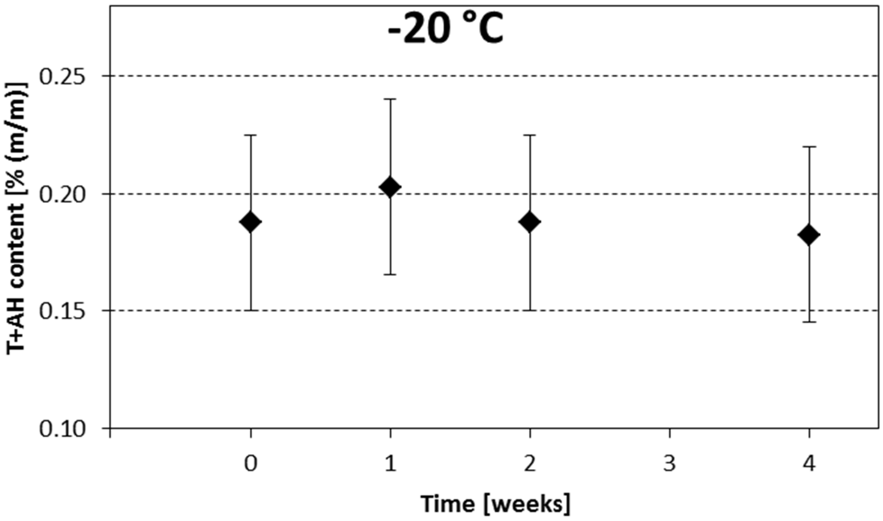

Figure B13: T+AH content means measured at -20 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

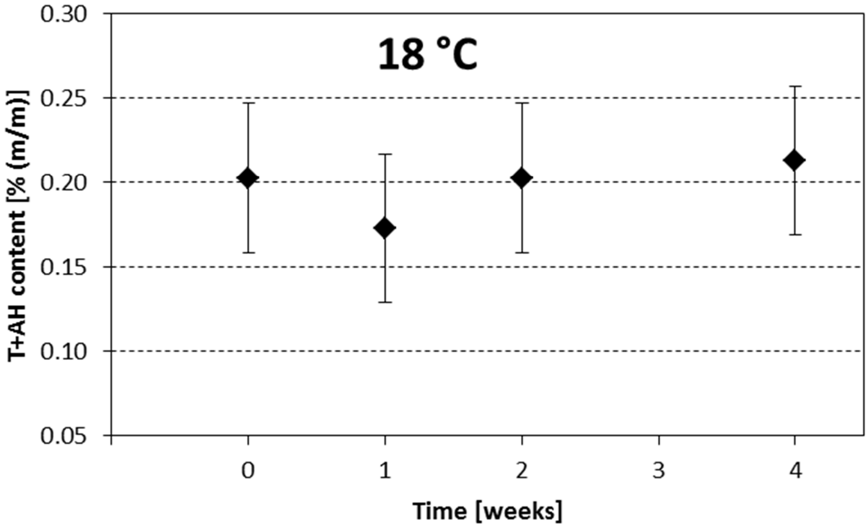

Figure B14: T+AH content means measured at 18 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

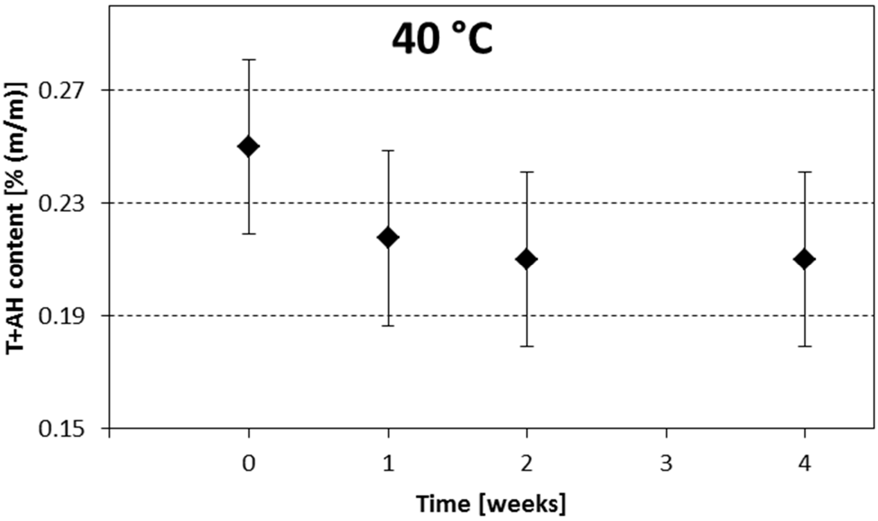

Figure B15: T+AH content means measured at 40 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

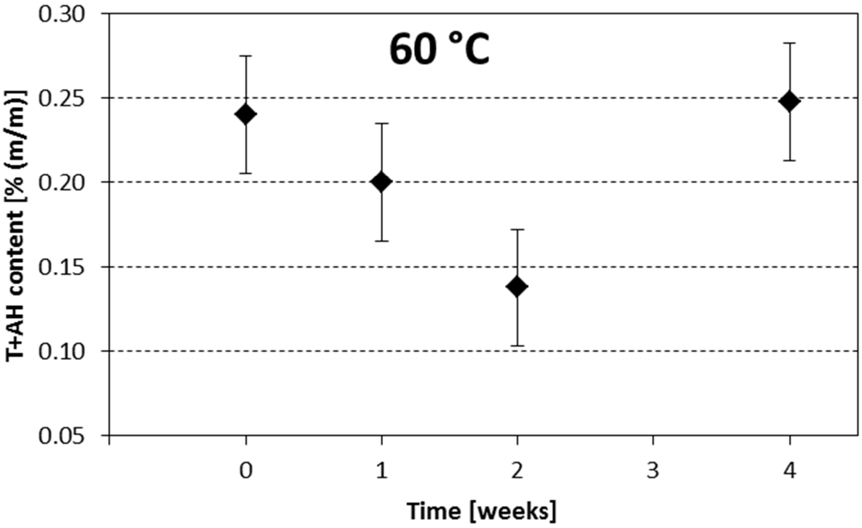

Figure B16: T+AH content means measured at 60 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

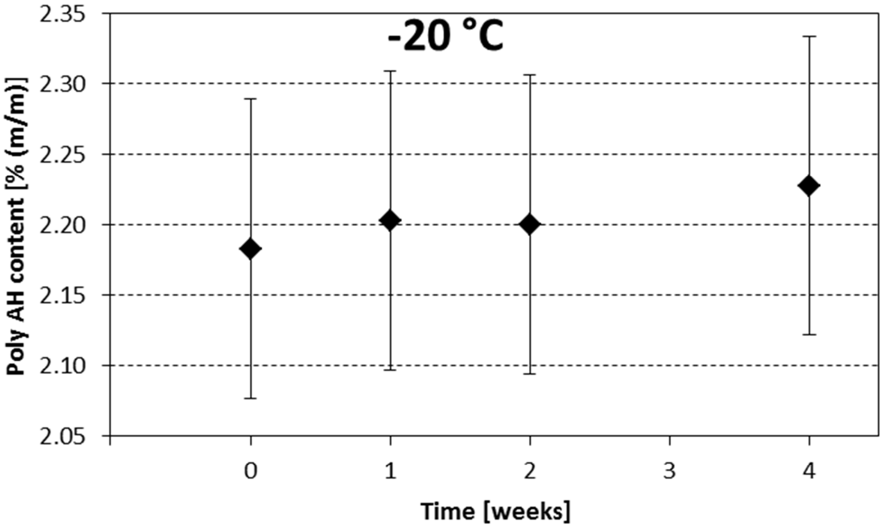

Figure B17: Poly AH content means measured at -20 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

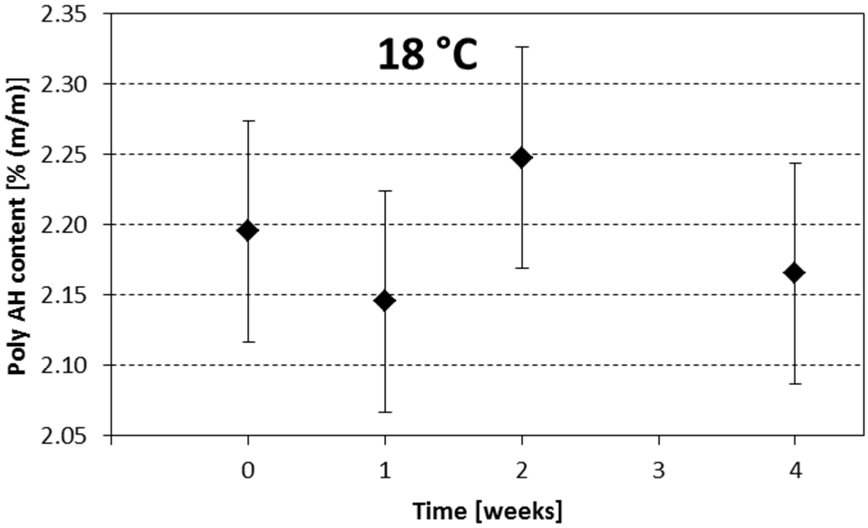

Figure B18: Poly AH content means measured at 18 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

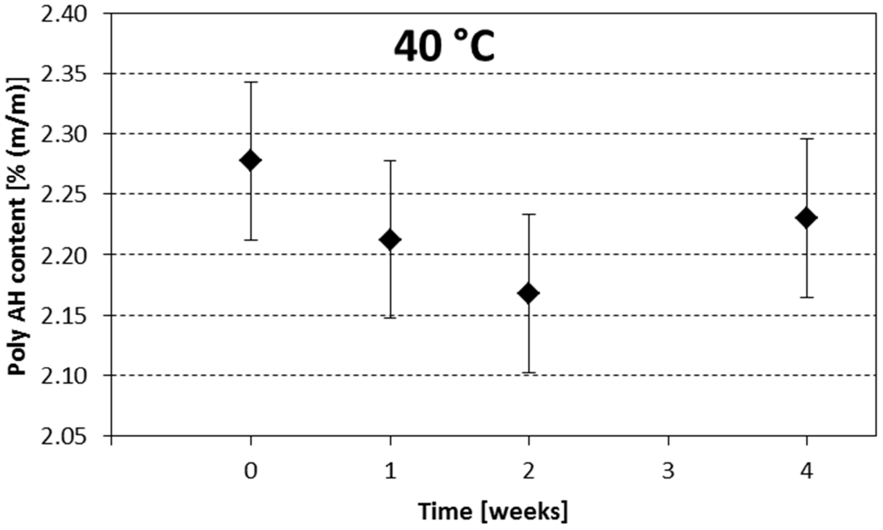

Figure B19: Poly AH content means measured at 40 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

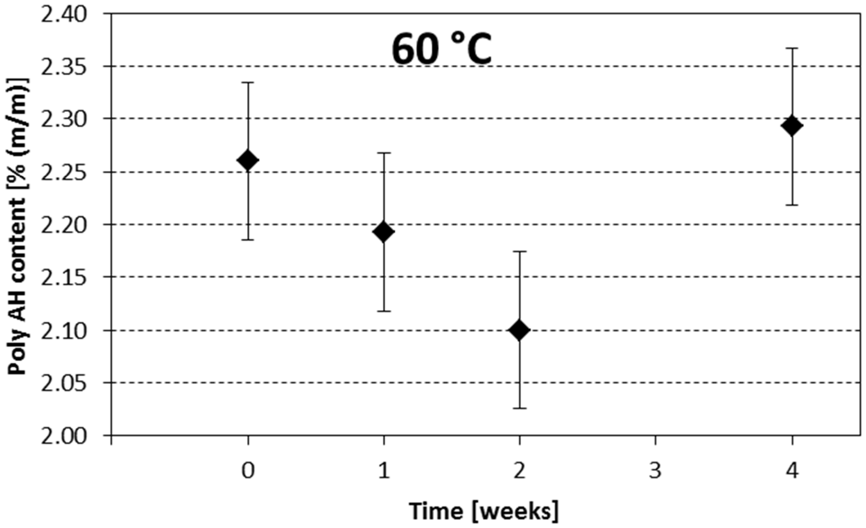

Figure B20: Poly AH content means measured at 60 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

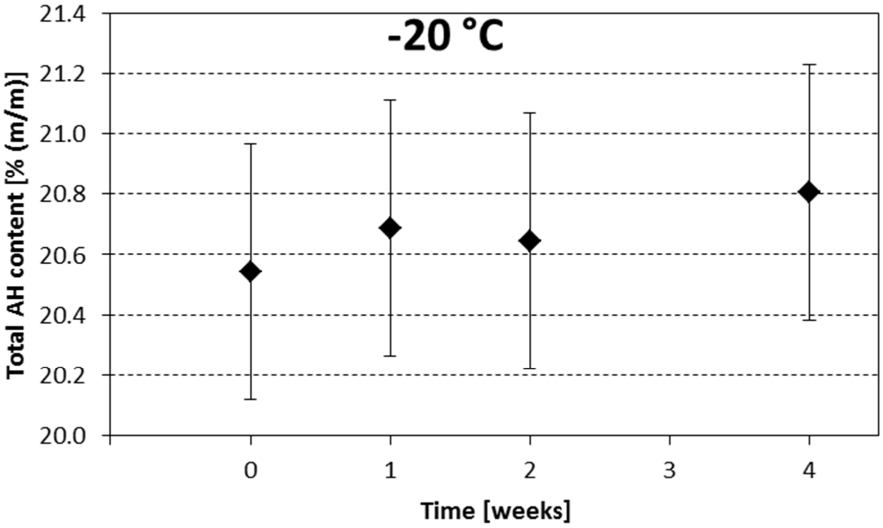

Figure B21: Total AH content means measured at -20 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

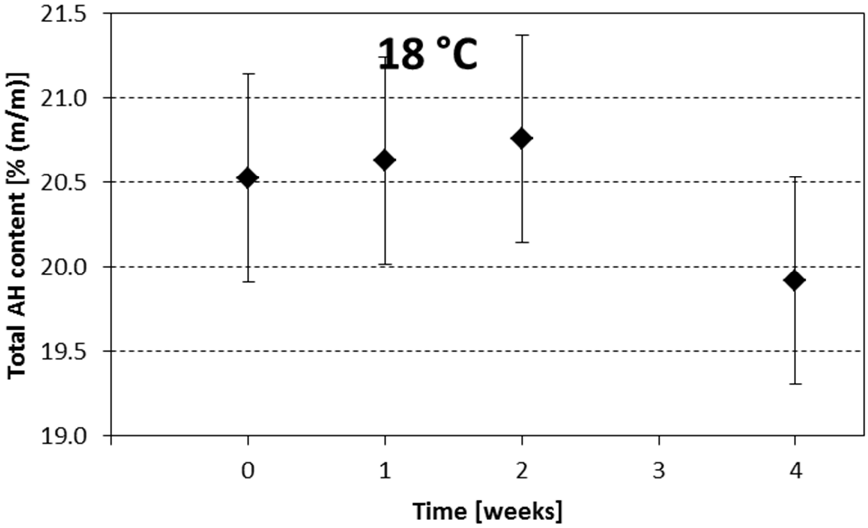

Figure B22: Total AH content means measured at 18 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

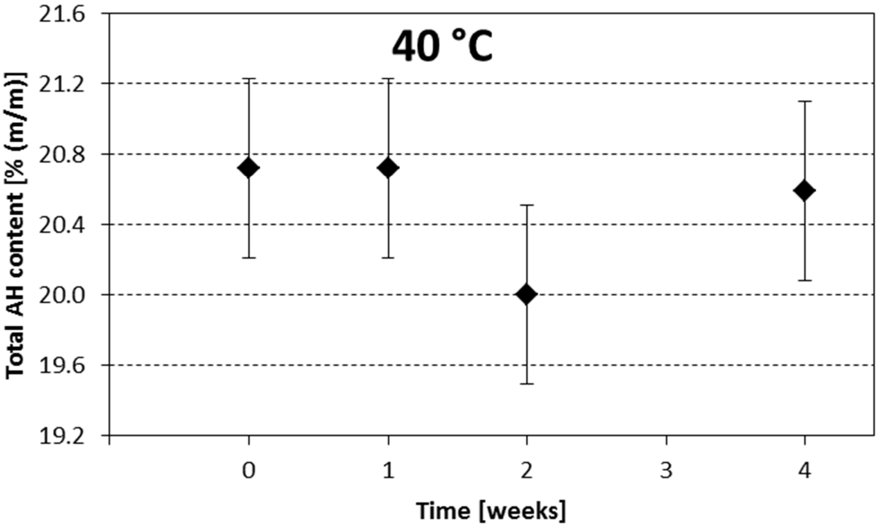

Figure B23: Total AH content means measured at 40 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

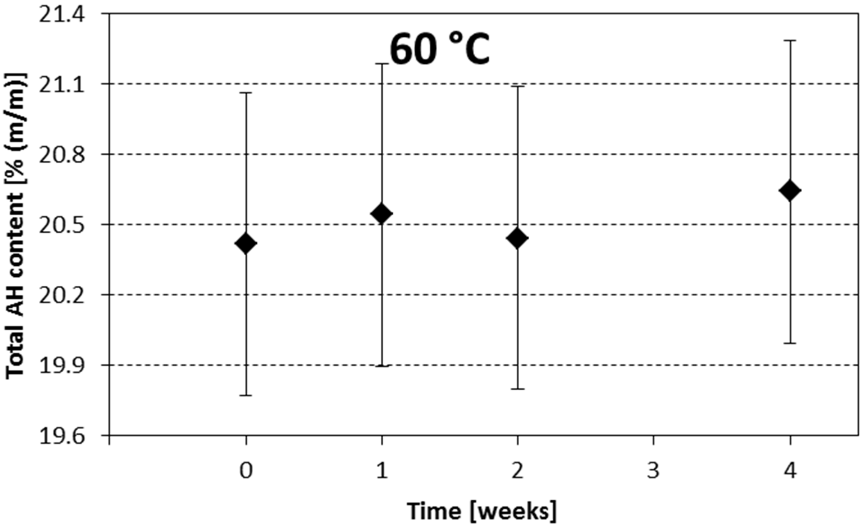

Figure B24: Total AH content means measured at 60 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

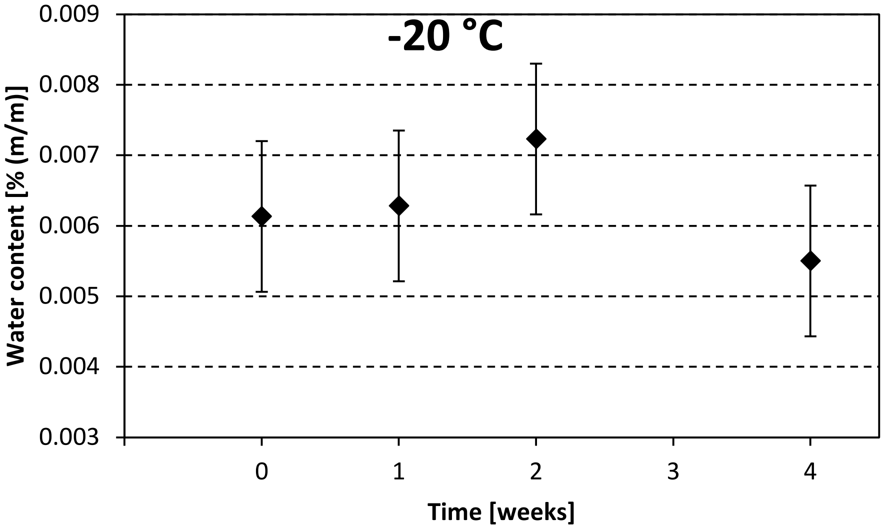

Figure B25: Water content means measured at -20 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

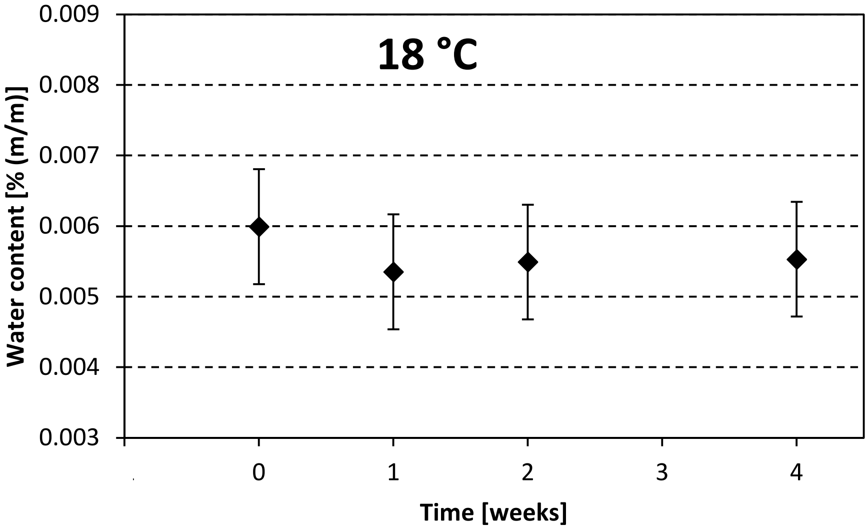

Figure 26: Water content means measured at 18 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

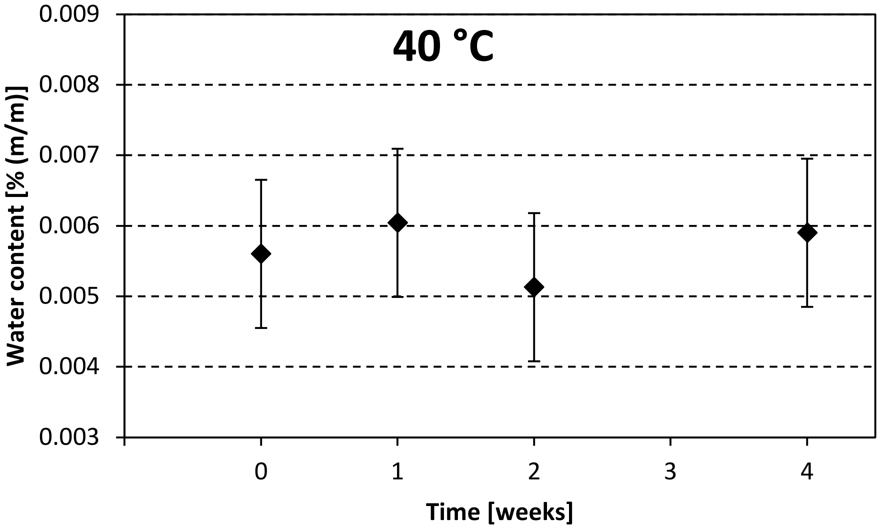

Figure B27: Water content means measured at 40 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

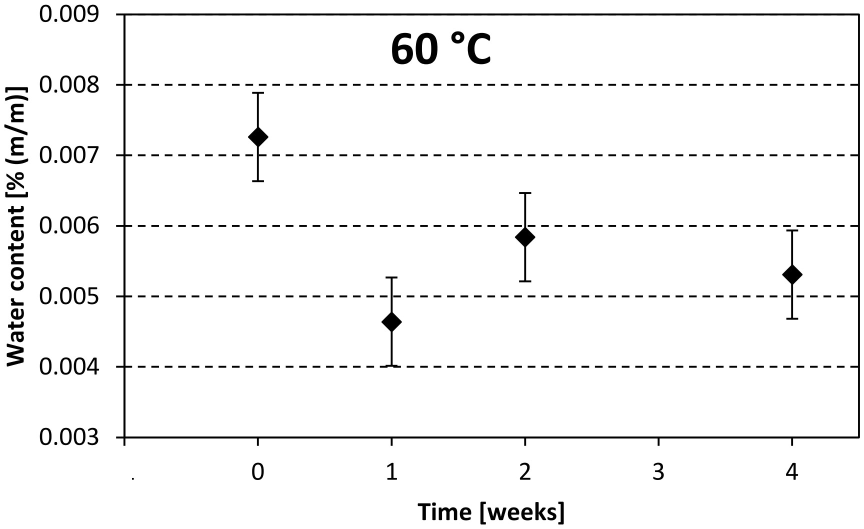

Figure B28: Water content means measured at 60 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

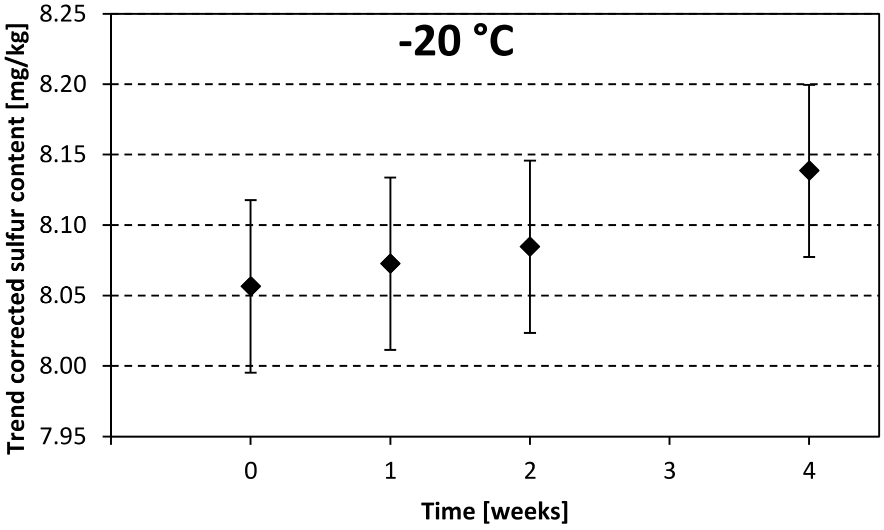

Figure B29: Analytical trend corrected sulfur content means measured at -20 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

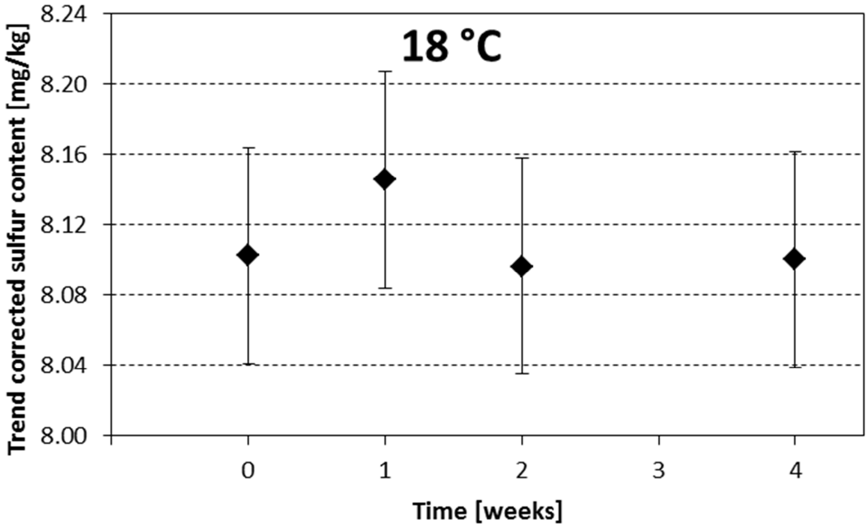

Figure B30: Analytical trend corrected sulfur content means measured at 18 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

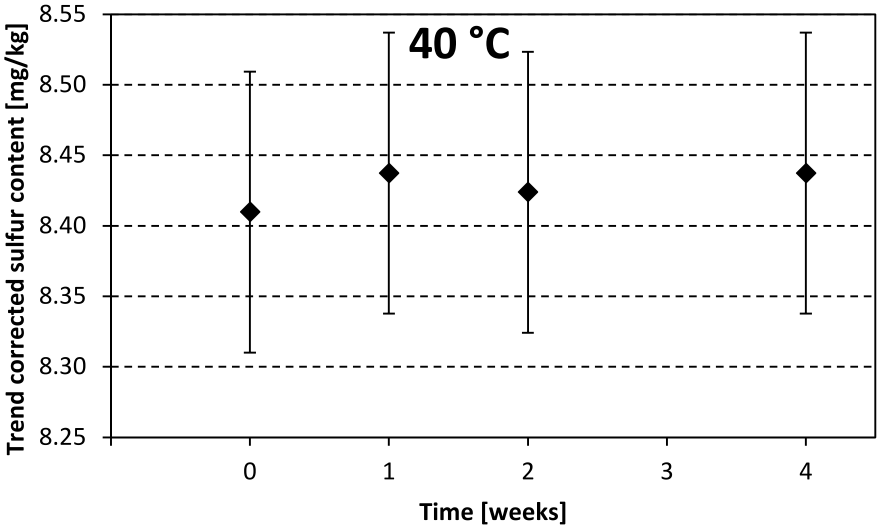

Figure B31: Analytical trend corrected sulfur content means measured at 40 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

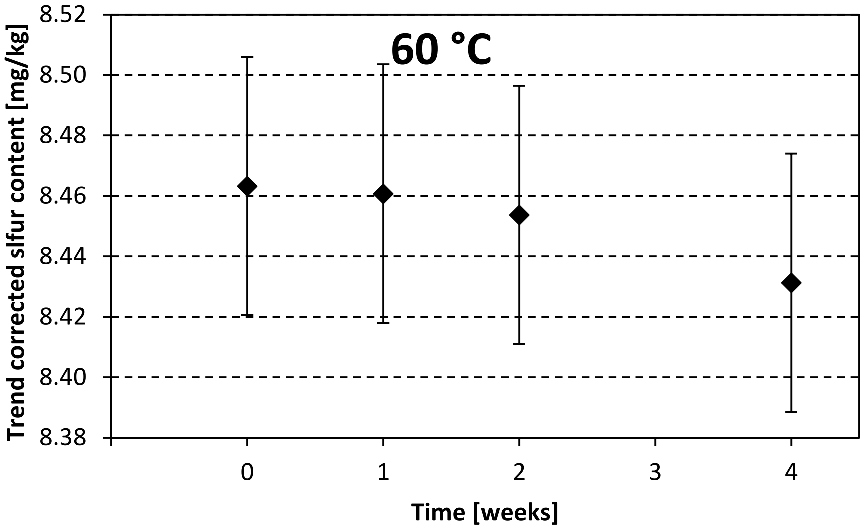

Figure B32: Analytical trend corrected sulfur content means measured at 60 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

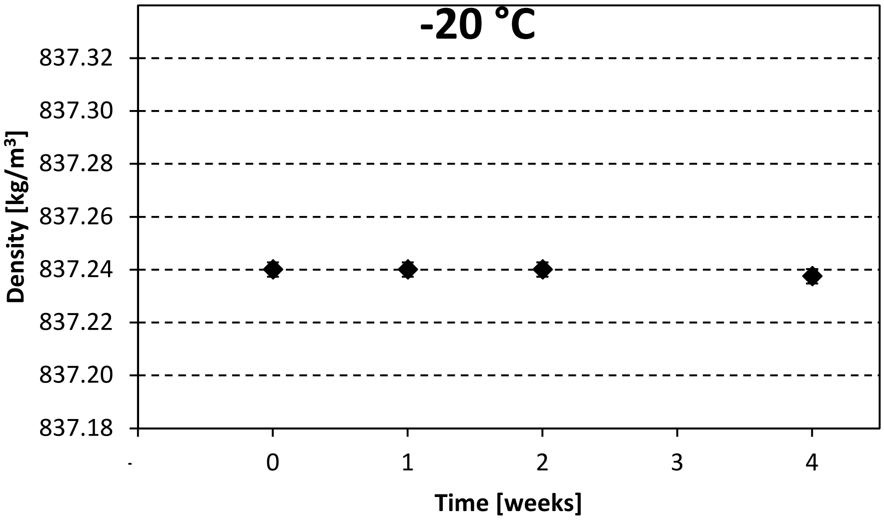

Figure B33: Density values measured at -20 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

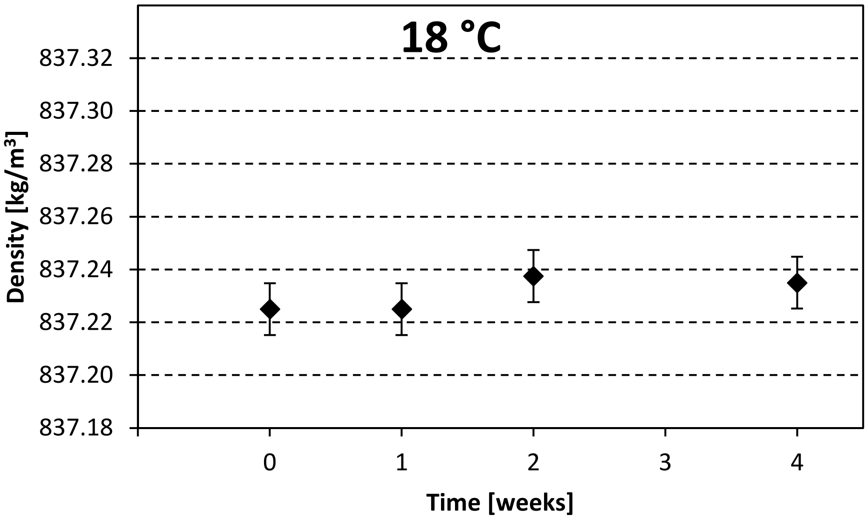

Figure B34: Density values measured at 18 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

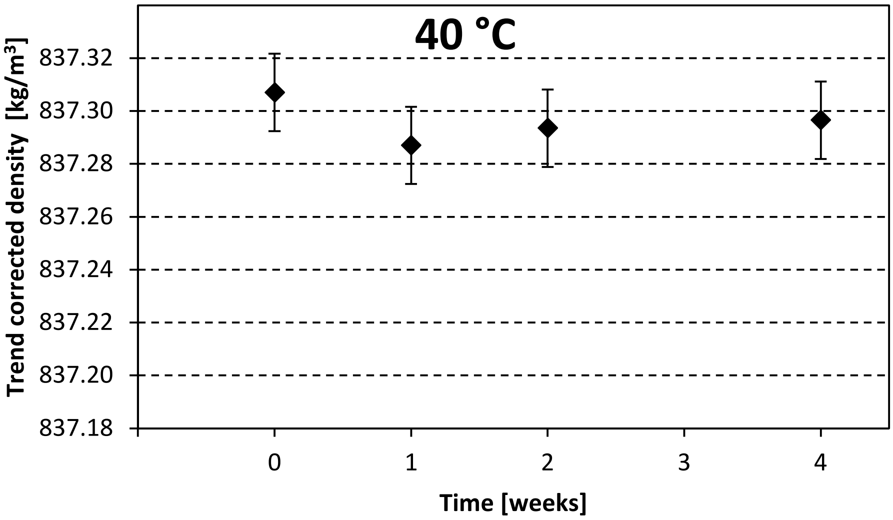

Figure B35: Analytical trend corrected density values measured at 40 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

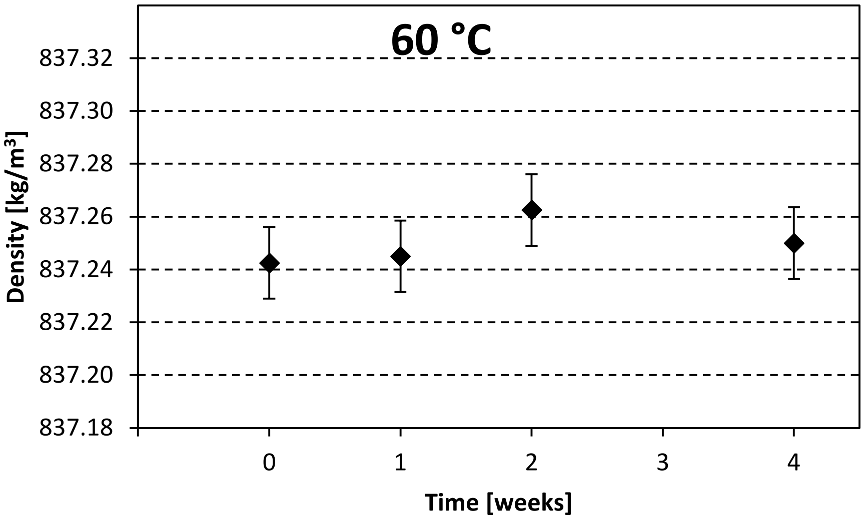

Figure B36: Density values measured at 60 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

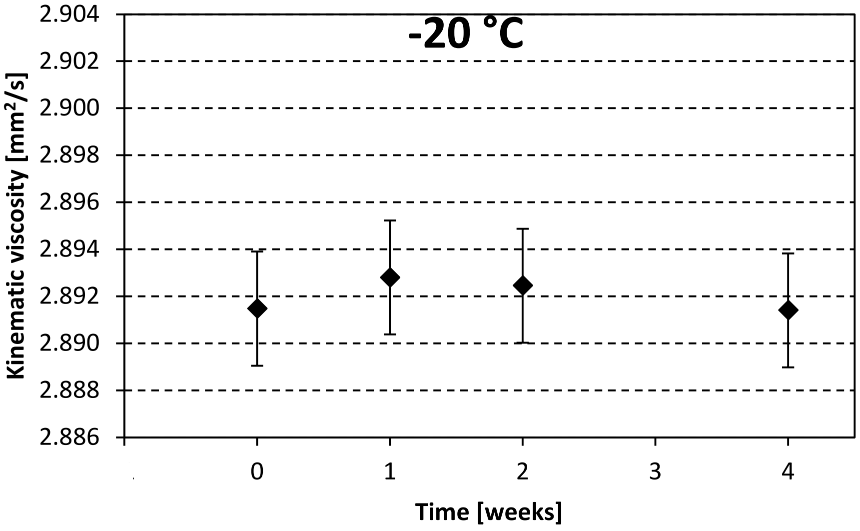

Figure B37: Kinematic viscosity values measured at -20 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

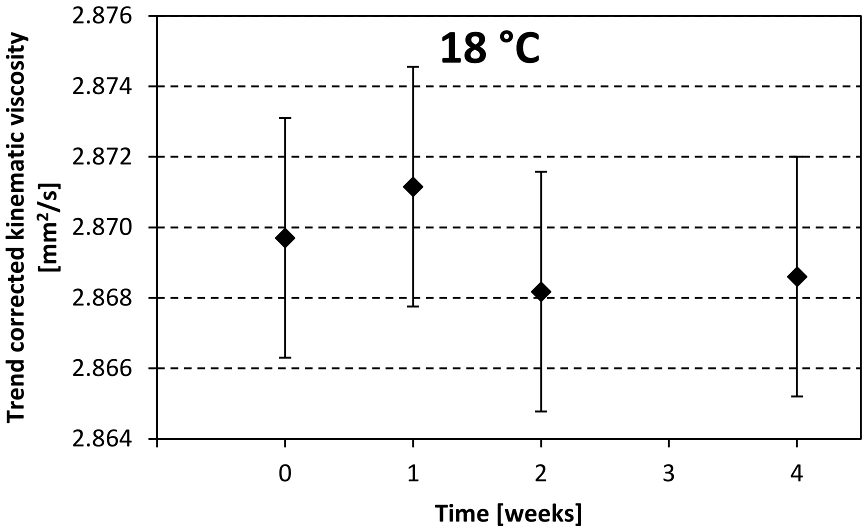

Figure B38: Analytical trend corrected kinematic viscosity values measured at 18 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

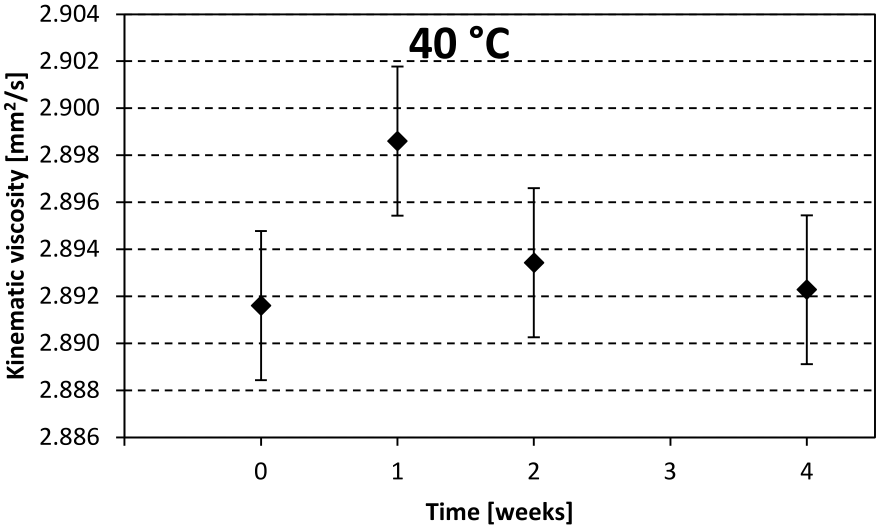

Figure B39: Kinematic viscosity values measured at 40 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

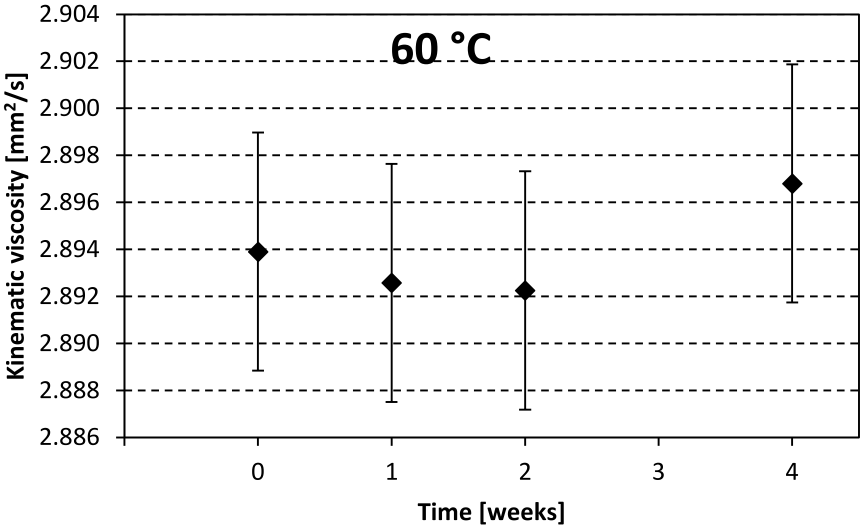

Figure B40: Kinematic viscosity values measured at 60 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

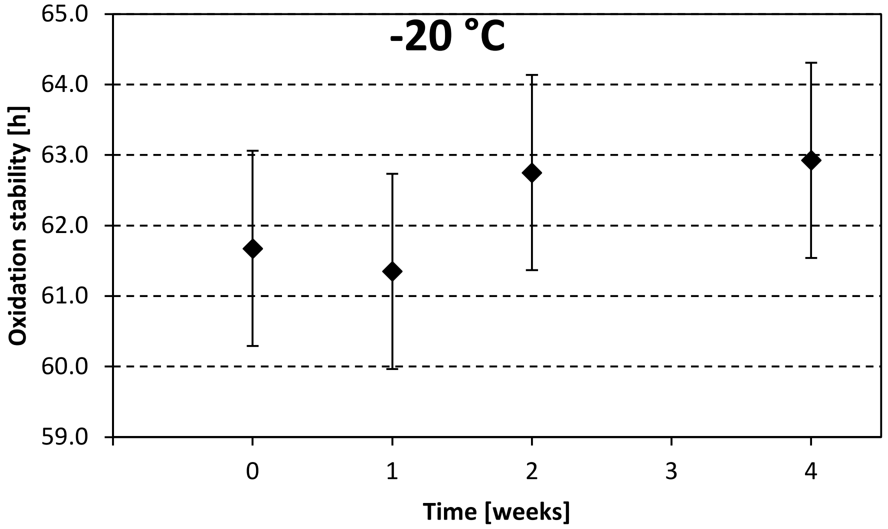

Figure B41: Oxidation stability means measured at -20 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

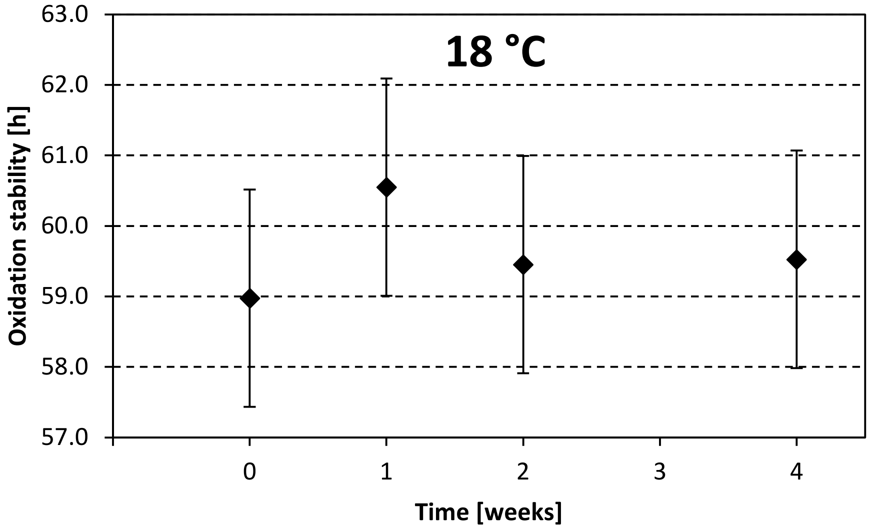

Figure B42: Oxidation stability means measured at 18 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

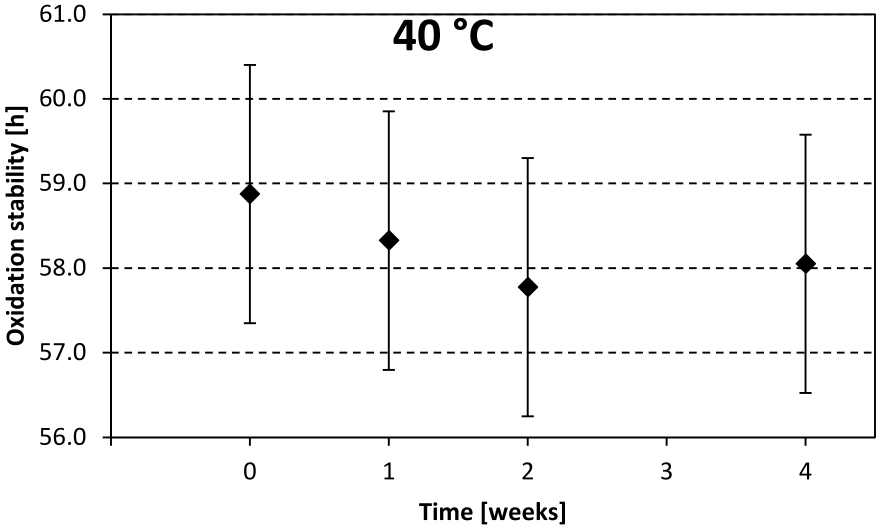

Figure B43: Oxidation stability means measured at 40 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

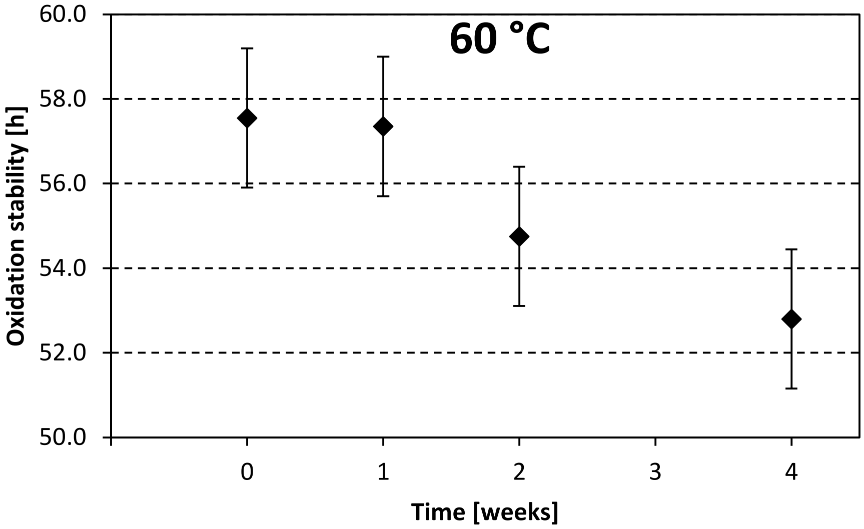

Figure B44: Oxidation stability means measured at 60 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

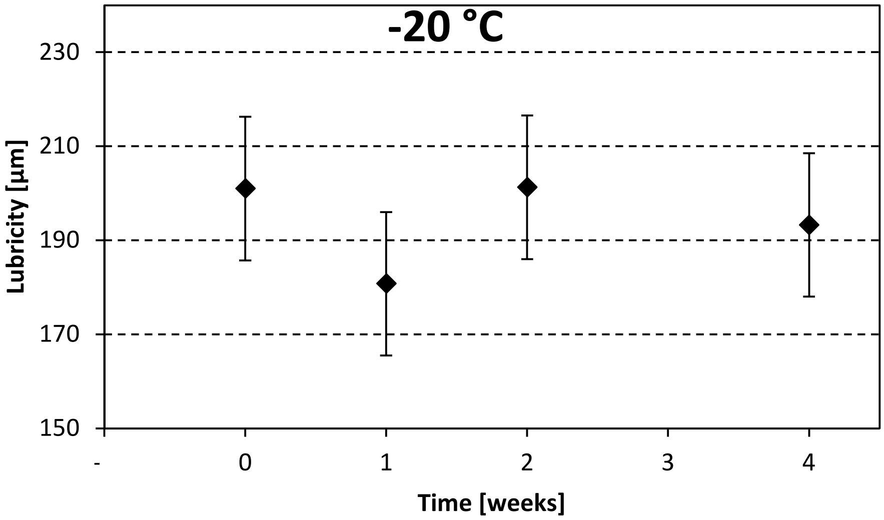

Figure B45: Lubricity means measured at -20 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

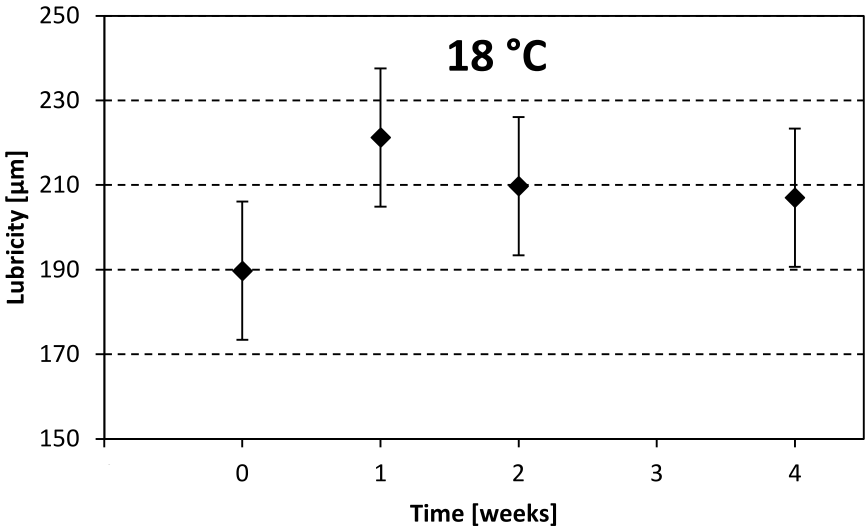

Figure B46: Lubricity means measured at 18 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

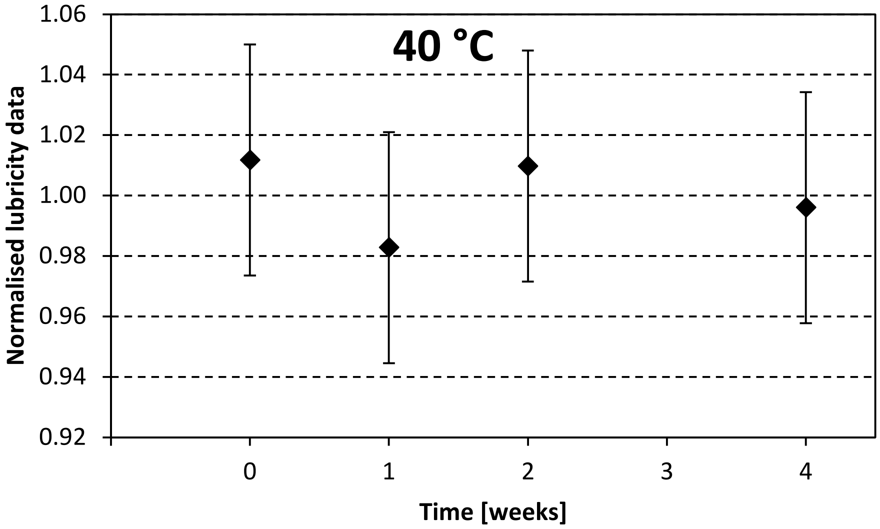

Figure B47: Normalised lubricity means measured at 40 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

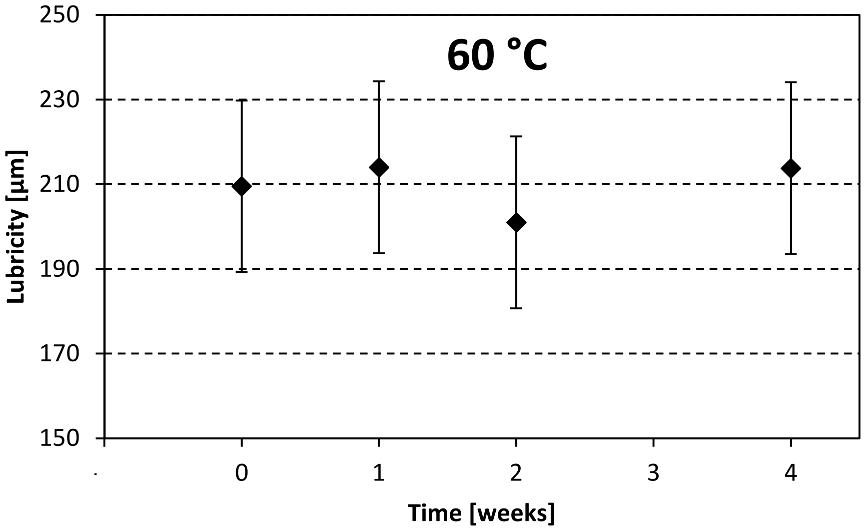

Figure B48: Lubricity means measured at 60 °C at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

Annex C: Results of the long-term stability measurements at 18 °C

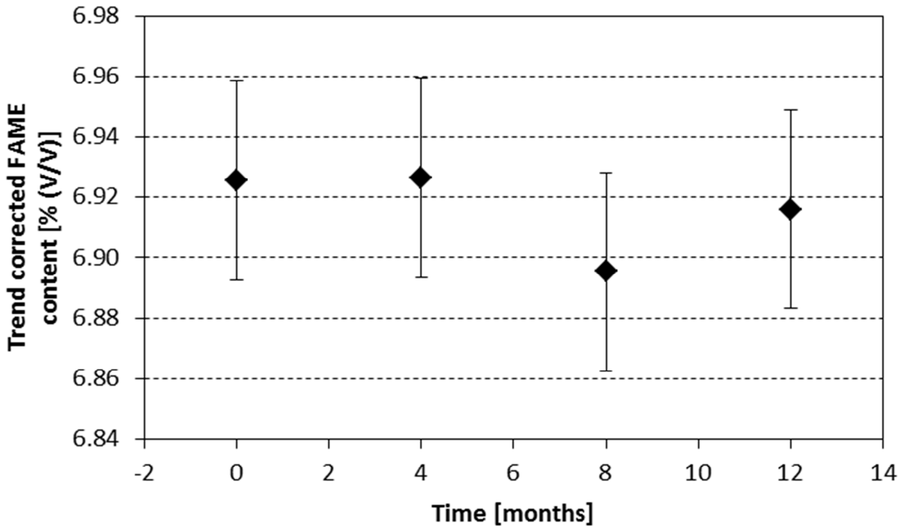

Figure C1: Analytical trend corrected FAME content means measured at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

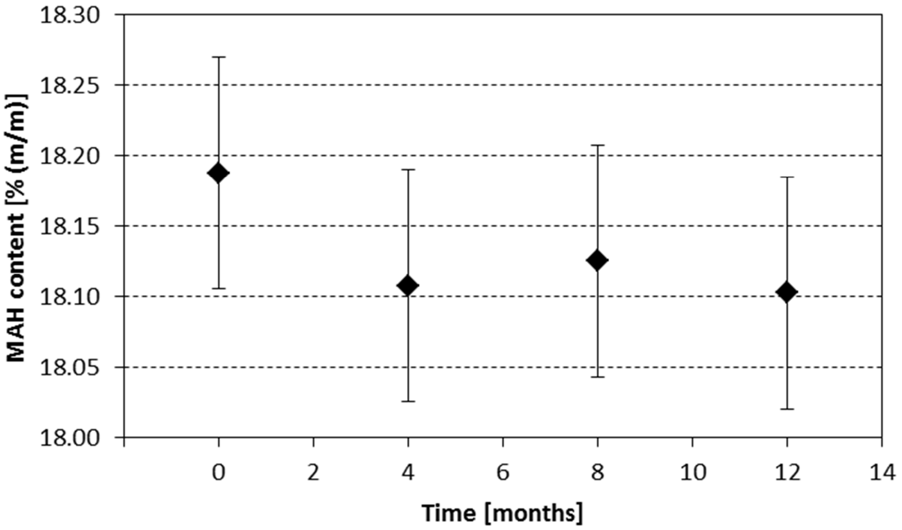

Figure C2: MAH content means measured at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

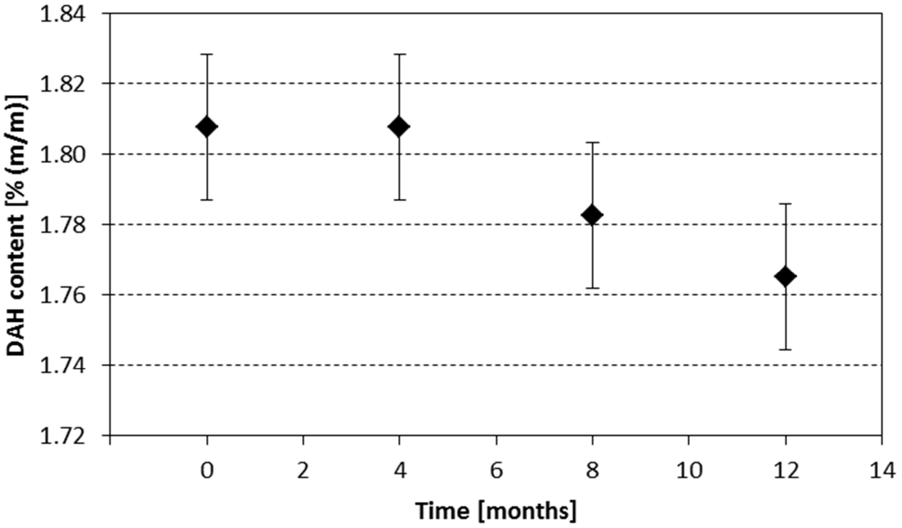

Figure C3: DAH content means measured at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

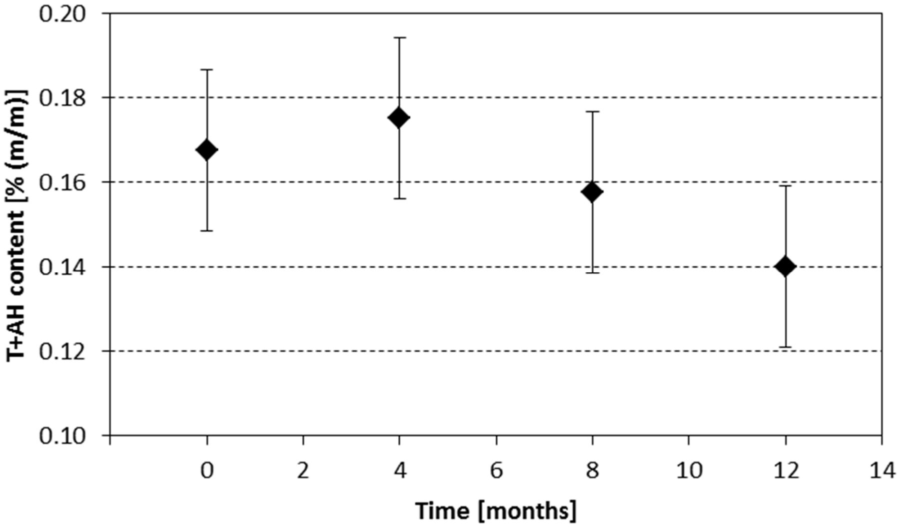

Figure C4: T+AH content means measured at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

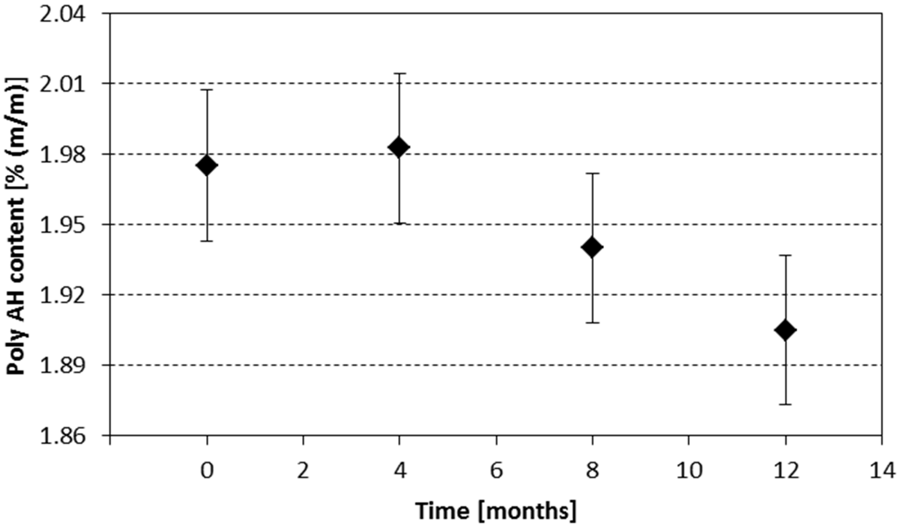

Figure C5: Poly AH content means measured at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

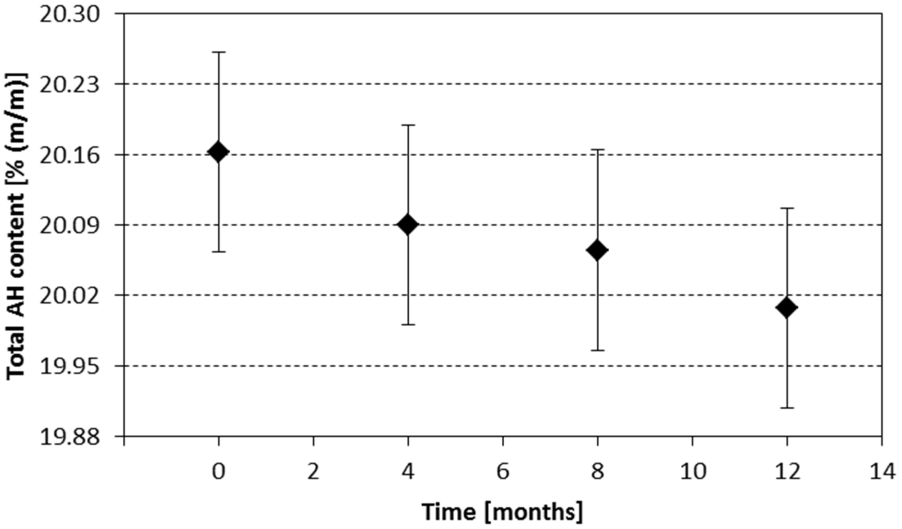

Figure C6: Total AH content means measured at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

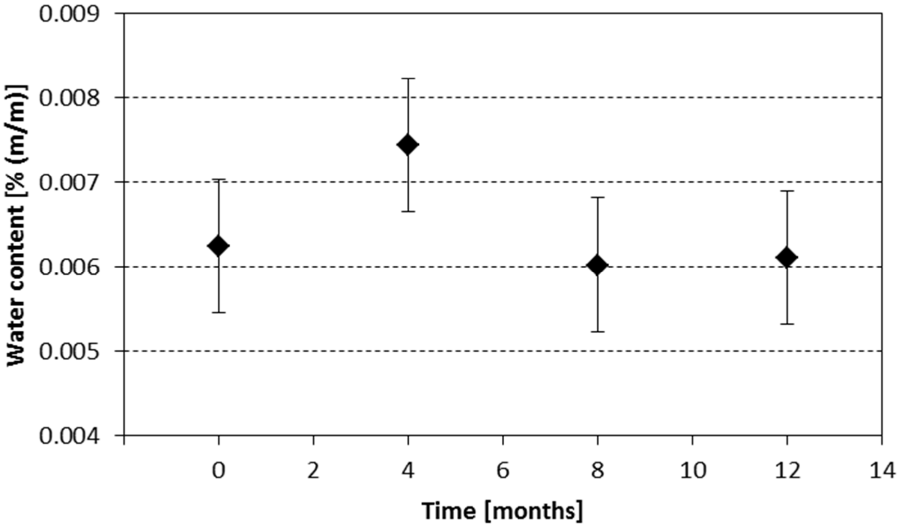

Figure C7: Water content means measured at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

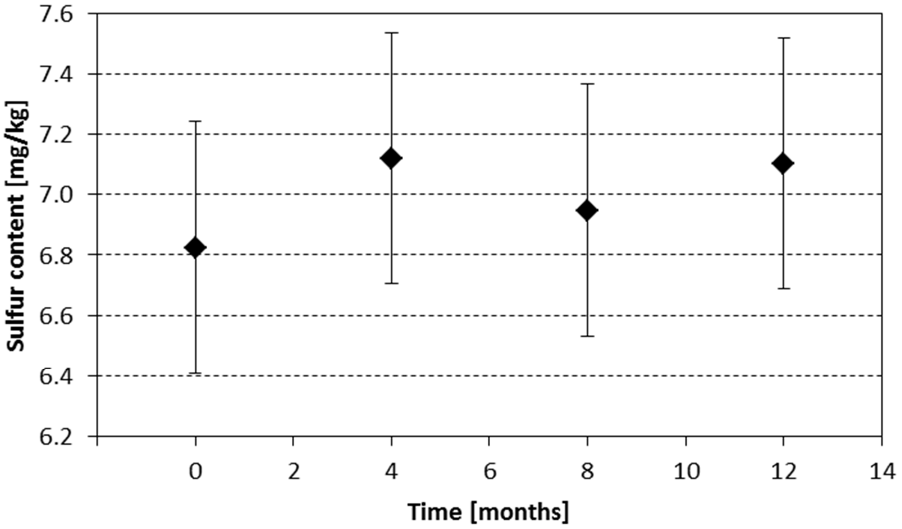

Figure C8: Sulfur content means measured at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

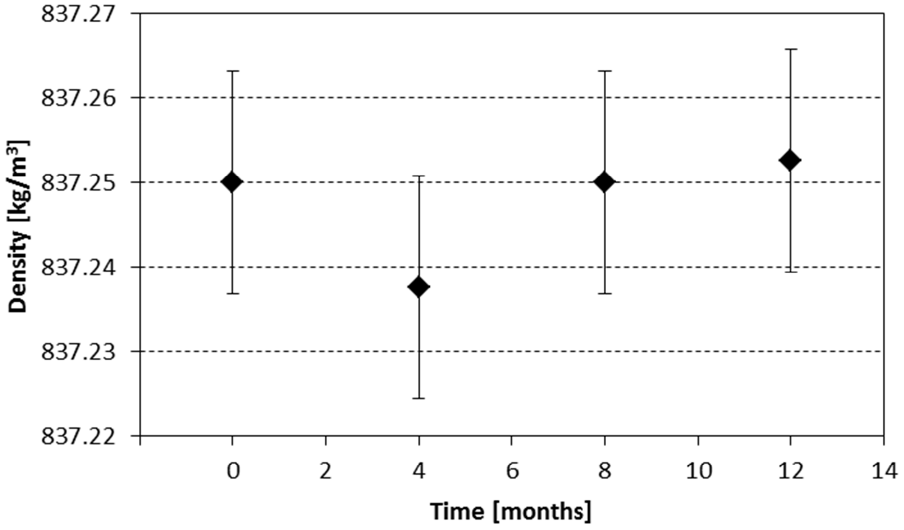

Figure C9: Density means measured at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

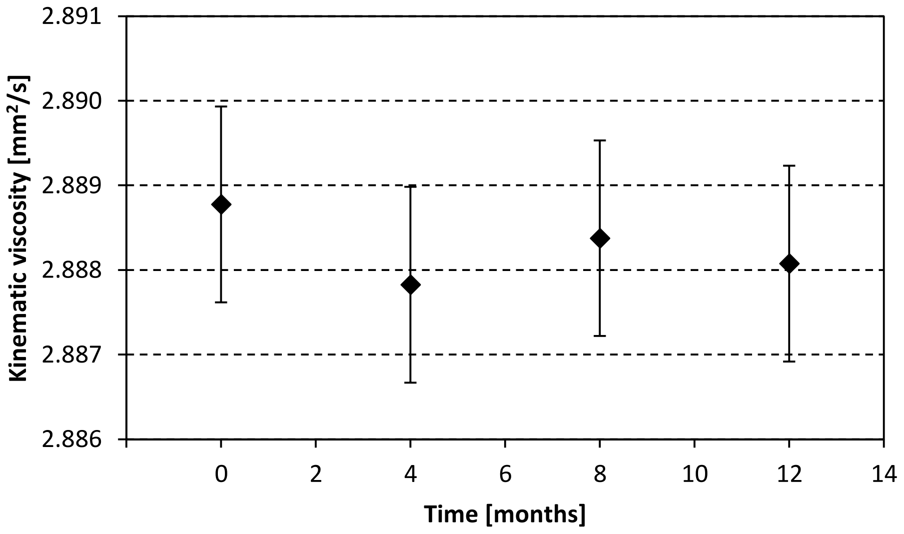

Figure C10: Kinematic viscosity means measured at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

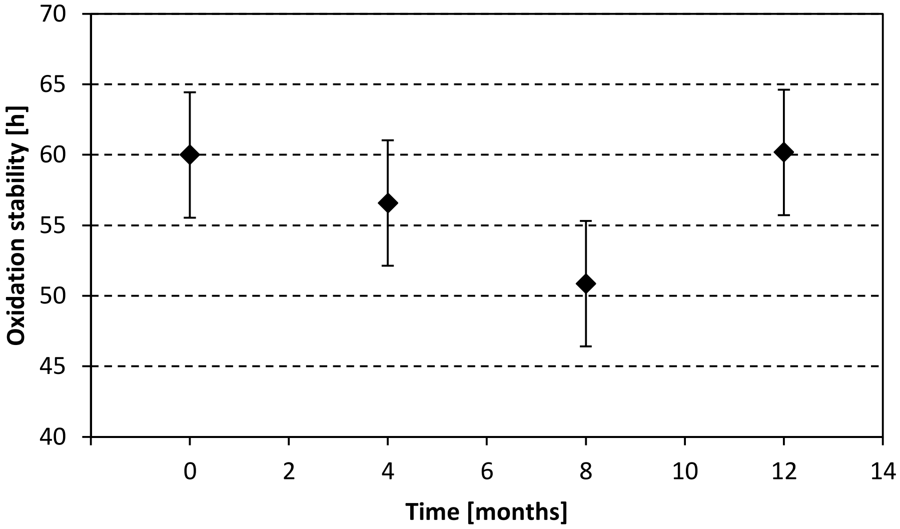

Figure C11: Oxidation stability means measured at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

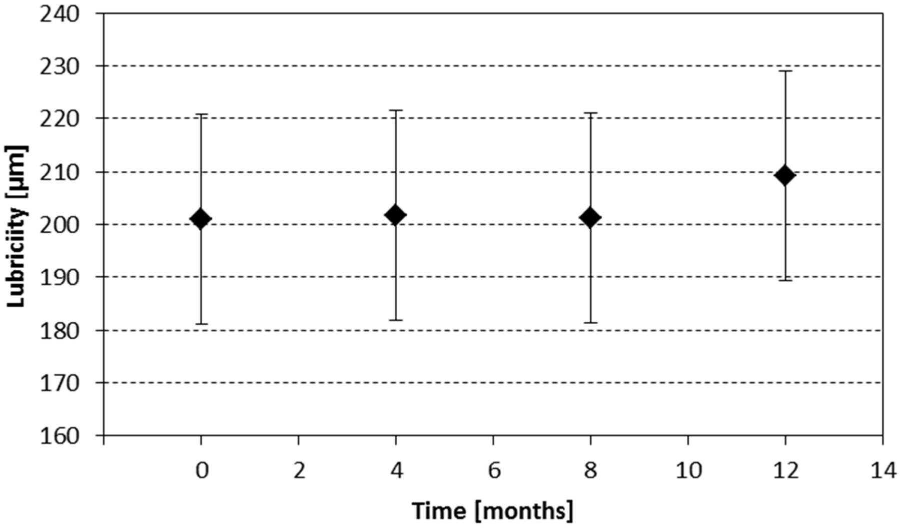

Figure C12: Lubricity means measured at each time-point. Vertical bars represent the 95 % confidence interval of the mean, based on the within-group standard deviation as obtained by single-factor ANOVA.

Annex D: Summary of methods used in the characterisation study

Table D1. Overview on scope and principles of documentary standards

Standard Reference	**EN 14078:2014**
Technical Body	CEN/TC 307 - Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis
Title	Liquid petroleum products - Determination of **fatty acid methyl ester (FAME) content** in middle distillates - Infrared spectrometry method
Scope	This European Standard specifies a test method for the determination of Fatty Acid Methyl Ester (FAME) content in diesel fuel or domestic heating fuel by mid infrared spectrometry, which applies to FAME contents of the three measurement ranges as follows: — range A: for FAME contents ranging from approx. 0,05 % (V/V) to approx. 3 % (V/V); — range B: for FAME contents ranging from approx. 3 % (V/V) to approx. 20 % (V/V); — range C: for FAME contents ranging from approx. 20 % (V/V) to approx. 50 % (V/V). This test method was verified to be applicable to samples which contain FAME conforming to EN 14214. Reliable quantitative results are obtained only if the samples do not contain any significant amounts of other interfering components, especially esters and other carbonyl compounds which possess absorption bands in the spectral region used for quantification of FAME. If such interfering components are present, this test method is expected to produce higher values.
Principle	The mid infrared absorption spectrum of a test portion of a sample which is diluted as appropriate if necessary with FAME-free solvent is recorded. The absorbance at the peak maximum of the typical absorption band for esters at about (1 745 ± 5) cm-1 is measured. Initially, calibration as well as evaluation of the data will be carried out as grams FAME per litre. For conversion of grams FAME per litre (g/l) to the reporting unit “% (V/V)”, a fixed density of FAME of 883,0 kg/m^3 (at 15 °C) is adopted.Three measurement ranges (A, B or C) have been chosen for which specific adjustments for the calibration and dilution need to be followed. Measurement should be conducted preferably without dilution for range A and a shorter path length of the measurement cell as well as an appropriate dilution for range B and C. Based on the absorbance measured at the maximum of the peak of the absorption band the FAME content is calculated by means of a calibration function which was determined by measuring calibration solutions for which the FAME content is known.

Standard Reference	**EN 12916:2016**
Technical Body	CEN/TC 307 - Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis
Title	Petroleum products - Determination of **aromatic hydrocarbon types** in middle distillates - High performance liquid chromatography method with refractive index detection
Scope	This European Standard specifies a test method for the determination of the content of mono-aromatic, diaromatic and tri+-aromatic hydrocarbons in diesel fuels that may contain fatty acid methyl esters (FAME) up to 5 % (V/V) and petroleum distillates in the boiling range from 150 °C to 400 °C. The polycyclic aromatic hydrocarbons content is calculated from the sum of di-aromatic and tri+-aromatic hydrocarbons and the total content of aromatic compounds is calculated from the sum of the individual aromatic hydrocarbon types. Compounds containing sulfur, nitrogen and oxygen may interfere in the determination; mono-alkenes do not interfere, but conjugated di-alkenes and polyalkenes, if present, may do so. The precision statement of the test method has been established for diesel fuels with and without FAME blending components, with a mono-aromatic content in the range from 6 % (m/m) to 30 % (m/m), a di-aromatic content from 1 % (m/m) to 10 % (m/m), a tri+-aromatic content from 0 % (m/m) to 2 % (m/m), a polycyclic aromatic content from 1 % (m/m) to 12 % (m/m), and a total aromatic content from 7 % (m/m) to 42 % (m/m).
Principle	A known mass of sample is diluted with heptane and a fixed volume of this solution injected into a high performance liquid chromatograph fitted with a polar column. This column has little affinity for non-aromatic hydrocarbons, whilst exhibiting a strong selectivity for aromatic hydrocarbons. As a result of this selectivity, the aromatic hydrocarbons are separated from the non-aromatic hydrocarbons and into distinct bands according to their ring structure, i.e. MAH, DAH and T+AH compounds. The column is connected to a refractive index detector which detects the components as they elute from the column. The electronic signal from the detector is continually monitored by a data processor. The amplitudes of the signals from the aromatics in the sample are compared with those obtained from calibration standards in order to calculate the mass fraction of MAHs, DAHs and T+AHs in the sample. The sum of the DAHs and T+AHs mass fractions is reported as the mass fraction of POLY-AH, and the sum of the MAHs, DAHs and T+AHs mass fractions is reported as the mass fraction of total aromatic hydrocarbons.

Standard Reference	**EN ISO 12937:2000**	**ISO 20846:2011**
Technical Body	CEN/TC 307 - Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis	CEN/TC 307 - Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis
Title	Petroleum products - Determination of **water** - Coulometric Karl Fischer titration method (ISO 12937:2000)	Petroleum products — Determination of **sulfur** content of automotive fuels — Ultraviolet fluorescence method
Scope	This International Standard specifies a method for the direct determination of water in petroleum products boiling below 390 °C. It covers the mass fraction range 0,003 % (m/m) to 0,100%(m/m). It is not applicable to products containing ketones or to residual fuel oils. This International Standard may be applicable to lubricating base oils. However, the precision has not been established for these materials. The precision given in clause 12 is based upon data obtained using dual-cell, dual-electrolyte systems.	This International Standard specifies an ultraviolet (UV) fluorescence test method for the determination of the sulfur content of motor gasolines containing up to 3,7 % (m/m) oxygen [including those blended with ethanol up to about 10 % (V/V)], and of diesel fuels, including those containing up to about 10 % (V/V) fatty acid methylester (FAME), having sulfur contents in the range 3 mg/kg to 500 mg/kg. Other products can be analysed and other sulfur contents can be determined according to this test method, however, no precision data for products other than automotive fuels and for results outside the specified range have been established for this International Standard. Halogens interfere with this detection technique at concentrations above approximately 3 500 mg/kg.
Principle	A sample is visually inspected (see 6.2.1). If clear and bright, and free from both water droplets and particulate matter on swirling, a weighed portion is injected into the titration vessel of a coulometric Karl Fischer apparatus in which iodine for the Karl Fischer reaction is generated coulometrically at the anode. When all the water has been titrated, excess iodine is detected by an electrometric end-point detector and the titration is terminated. Based on the stoichiometry of the reaction, one mole of iodine reacts with one mole of water, thus the quantity of water is proportional to the total integrated current according to Faraday's Law. If the sample is not clear and bright, or water droplets or particulate matter are observed on swirling, a portion of a solution of sodium dioctylsulfosuccinate is added prior to homogenizing with a mixer. A weighed portion is then treated as described above.	A hydrocarbon sample is injected into a UV fluorescence detector. The sample enters a high temperature combustion tube (1 000 °C to 1 100 °C), where the sulfur is oxidized to sulfur dioxide (SO_2) in an oxygen-rich atmosphere. Water produced during the sample combustion is removed and the sample combustion gases are exposed to UV light. The SO_2 absorbs the energy from the UV light and is converted to excited sulfur dioxide (SO_{2*}). The fluorescence emitted from the excited SO_{2*} as it returns to a stable state SO_2 is detected by a photomultiplier tube and the resulting signal is a measure of the sulfur contained in the sample.

Standard Reference	**ISO 12185:1996**	**IS0 3104:1994**
Technical Body	CEN/TC 307 - Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis	CEN/TC 307 - Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis
Title	Crude Petroleum and Petroleum products - Determination of **density** - Oscillating U-tube method	Petroleum products - Transparent and opaque liquids - Determination of **kinematic viscosity** and calculation of dynamic viscosity
Scope	This International Standard specifies a method for the determination, using an oscillating U-tube density meter, of the density of crude Petroleum and related products within the range 600 kg/m^3 to 1 100 kg/m^3 which tan be handled as Single-Phase liquids at the test temperature and pressure. This International Standard is applicable to liquids of any vapour pressure as long as suitable precautions are taken to ensure that they remain in Single Phase with no loss of light ends and subsequent changes in composition and density during both the Sample handling and the density determination.	This International Standard specifies a procedure 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, q, can be obtained by multiplying the measured kinematic viscosity by the density, *p*, of the liquid.
Principle	A small (typically less than 1 ml) Portion of the test Sample is introduced into a temperature-controlled Sample cell. The oscillation frequency is noted, and the density of the test Sample calculated using cell constants previously determined by measuring the oscillation frequencies when the cell is filled with calibration fluids of known density.	The time is measured for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer 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 viscometer.

Standard Reference	**EN 15751:2014**	**ISO 12156-1:2016**
Technical Body	CEN/TC 307 - Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis	CEN/TC 307 - Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis
Title	Automotive fuels - Fatty acid methyl ester (FAME) fuel and blends with diesel fuel - Determination of **oxidation stability** by accelerated oxidation method	Diesel fuel — Assessment of **lubricity** using the high-frequency reciprocatingrig (HFRR)
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).	This part of ISO 12156 specifies a test method using the high-frequency reciprocating rig (HFRR), for assessing the lubricating property of diesel fuels, including those fuels which may contain a lubricityenhancing additive. It applies to fuels used in diesel engines.
Principle	A stream of purified air is passed through the sample which has been heated to the target temperature which is 110 °C in the usual application of the method. Volatile compounds are formed during the oxidation process. They are, passed together with the air into a flask containing demineralised or distilled water, equipped with a conductivity electrode. The electrode is connected to a measuring and recording device. It indicates the end of the induction period by rapid increase of the conductivity due to the dissociation of volatile carboxylic acids produced during the oxidation process and absorbed in the water. For more details on the background of the method, see Annex A.	A sample of the fluid under test is placed in a test reservoir which is maintained at the specified test temperature. A fixed steel ball is held in a vertically mounted chuck and forced against a horizontally mounted stationary steel plate with an applied load. The test ball is oscillated at a fixed frequency and stroke length while the interface with the plate is fully immersed in the fluid. The metallurgies of the ball and plate, temperature, load, frequency, and stroke length are specified. The ambient conditions of temperature and humidity during the test are used to correct the size of the wear scar generated on the test ball to a standard set of ambient conditions. The corrected wear scar diameter is a measure of the fluid lubricity.

Table D2: Precision data as laid down in respective documentary standards and estimated expanded measurement uncertainties thereof

Parameter	Unit	r	R	U_{meas}
FAME content	[% (V/V)]	0.0166 x C - 0.0195	0.0793 x C - 0.0413	0.35
MAH content	[% (m/m)]	0.032 x C - 0.161	0.144 x C - 0.344	1.66
DAH content	[% (m/m)]	0.151 x C - 0.036	0.363 x C -0.087	0.38
T+AH content	[% (m/m)]	0.092 x C + 0.098	0.442 x C + 0.471	0.38
Poly AH content	[% (m/m)]	0.074 x C + 0.186	0.185 x C + 0.465	0.57
Total AH content	[% (m/m)]	0.040 x C - 0.070	0.172 x C - 1.094	1.70
Water content	[% (m/m)]	0.01874 x C^0.5	0.06877 x C^0.5	0.004
Sulfur content	[mg/kg]	0.0553 x C + 0.55	0.1120 x C + 1.12	1.25
Density at 15.0 °C	[kg/m^3]	0.4	1.5	1.04
Kinematic viscosity at 40.0 °C	[mm^2/s]	0.0043 x (C+1)	0.0082 x (C+1)	0.02
Oxidation stability	[h]	0.22027 + 0.04344 x C	0.37269 + 0.19038 x C	7.16
Lubricity	[µm]	50	80	46.93

[1] C=Determined result

Annex E: Results of the characterisation measurements

Note: For the measurement uncertainties of the individual laboratories the measurement uncertainties derived from the standard methods were taken.

Table E1: Volume fraction of FAME content in diesel (B7) as reported by each individual lab

Laboratory code	replicate 1 [% (V/V)]	replicate 2 [% (V/V)]	replicate 3 [% (V/V)]	replicate 4 [% (V/V)]	replicate 5 [% (V/V)]	replicate 6 [% (V/V)]	mean [% (V/V)]	RSD [%]
L2	6.62	6.62	6.65	6.65	6.64	6.64	6.64	0.21
L3	6.645	6.679	6.60	6.61	6.618	6.671	6.637	0.51
L4	6.6	6.6	6.6	6.6	6.6	6.5	6.6	0.62
L5	7.12	7.06	7.11	7.05	7.3	7.28	7.15	1.53
L6	6.79	6.82	6.86	6.87	6.87	6.860	6.845	0.48
L7	7.067	7.078	6.993	6.937	6.857	6.840	6.962	1.46
L9	7	7.01	6.94	6.93	6.96	6.94	6.96	0.49
L12	7.1	7.1	7.1	7.1	7.1	7.1	7.1	0.00
L13	6.99	7.04	6.98	7.03	6.97	6.99	7.00	0.40
Results not used for certification								
L8	*6.43*	*6.84*	*6.41*	*6.35*	*6.61*	*6.46*	*6.52*	*0.49*
L11	*7.00*	*7.00*	*7.20*	*7.30*	*7.20*	*7.30*	*7.17*	*1.91*

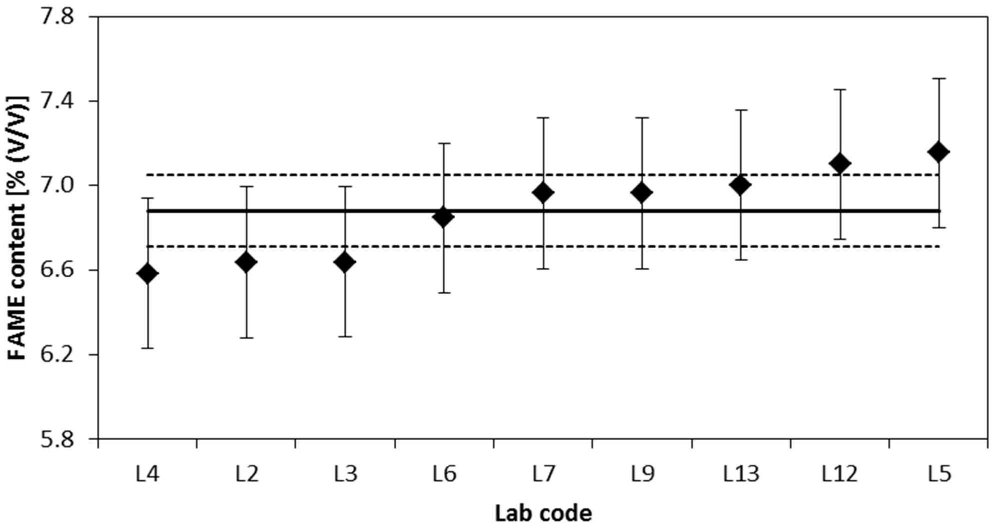

Figure E1: Results of the characterisation study for the volume fraction of FAME content in diesel (B7) measured using EN 14078 [10] (continuous line: certified value; dashed line: expanded uncertainty with k=2; error bars: expanded measurement uncertainty derived from EN 14078 and as given in Table D2)

Table E2: Mass fraction of MAH content in diesel (B7) 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 [%]
L1	18.1	18.2	18.1	18.2	18.2	18.2	18.1	0.27
L2	18.305	18.335	18.48	18.38	18.262	18.458	18.371	0.47
L3	18.2	18.6	18.9	19	18.7	18.3	18.6	1.71
L5	20.0	20.19	19.81	20.05	20.46	20.44	20.158	1.27
L6	18.0	18.0	18.2	18.2	18.3	18.5	18.2	0.99
L7	19.7	19.7	20.0	20.1	20.0	19.8	19.9	0.91
L9	18.63	18.66	18.62	18.63	18.54	18.6	18.61	0.22
L11	18.5	18.2	18.5	18.5	18.1	18.4	18.4	0.95
L12	19.1	19	19.1	19	19.1	19.1	19.1	0.27
Results not used for certification								
L8	*21.1*	*20.1*	*19.5*	*19.9*	*20.0*	*20.7*	*20.2*	*2.87*
L13	*21.5*	*21.1*	*19.3*	*19.4*	*18.1*	*18.0*	*19.6*	*7.46*

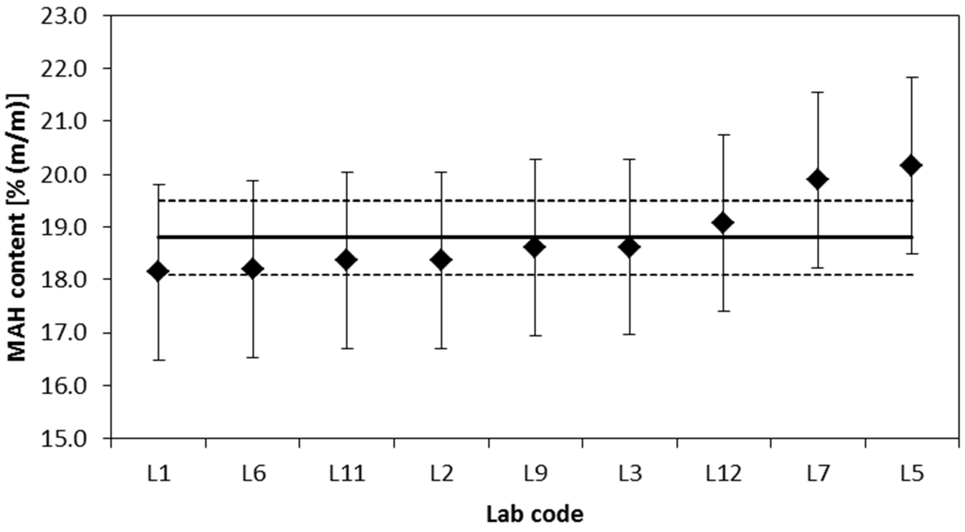

Figure E2: Results of the characterisation study for the mass fraction of MAH content in diesel (B7) measured using EN 12916 [11] (continuous line: certified value; dashed line: expanded uncertainty with k=2; error bars: expanded measurement uncertainty derived from EN 12916 and as given in Table D2)

Table E3: Mass fraction of DAH content in diesel (B7) 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 [%]
L1	1.76	1.75	1.77	1.77	1.73	1.73	1.75	1.05
L2	2.102	1.968	2.04	1.99	2.053	2.012	2.028	2.35
L3	1.80	1.90	2.20	2.10	1.90	1.90	1.97	7.66
L5	2.12	2.14	2.09	2.04	2.15	2.15	2.12	2.04
L6	1.78	1.83	1.83	1.78	1.83	1.85	1.82	1.62
L7	1.3991	1.3937	1.4851	1.4769	1.4732	1.4387	1.4445	2.80
L9	1.88	1.91	1.87	1.91	1.91	1.87	1.89	1.08
L11	1.9	1.9	1.9	1.9	1.8	1.9	1.9	2.17
L12	1.70	1.70	1.70	1.70	1.70	1.70	1.70	0.00
Results not used for certification								
L8	*1.9*	*2.4*	*1.9*	*2.3*	*1.6*	*2.3*	*2.1*	*15.20*
L13	*2.8*	*2.8*	*2.5*	*2.5*	*1.8*	*1.8*	*2.4*	*19.43*

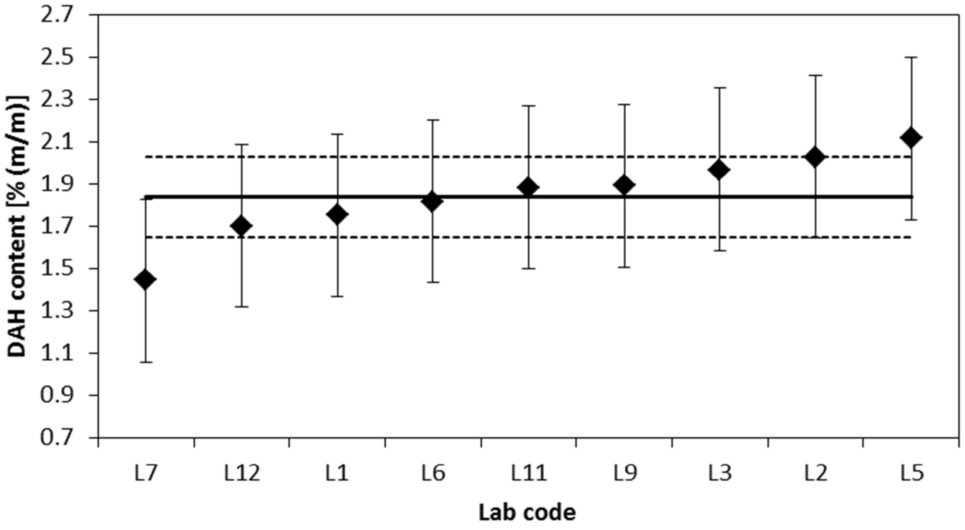

Figure E3: Results of the characterisation study for the mass fraction of DAH content in diesel (B7) measured using EN 12916 [11] (continuous line: certified value; dashed line: expanded uncertainty with k=2; error bars: expanded measurement uncertainty derived from EN 12916 and as given in Table D2)

Table E4: Mass fraction of T+AH content in diesel (B7) 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 [%]
L1	0.14	0.12	0.14	0.16	0.13	0.11	0.13	13.13
L2	0.276	0.227	0.191	0.166	0.279	0.192	0.222	21.32
L3	0.10	0.20	0.20	0.20	0.20	0.30	0.20	31.62
L5	0.24	0.24	0.23	0.23	0.24	0.250	0.238	3.16
L6	0.16	0.23	0.16	0.13	0.19	0.13	0.17	22.98
L7	0.2581	0.2453	0.2924	0.2816	0.2613	0.2431	0.2636	7.48
L9	0.13	0.13	0.11	0.14	0.13	0.11	0.13	9.80
L11	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.00
L12	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.00
Results not used for certification								
L8	*0.02*	*0.02*	*0.06*	*0.05*	*0.08*	*0.10*	*0.06*	*58.35*
L13	*0.181*	*0.176*	*0.133*	*0.127*	*0.041*	*0.040*	*0.116*	*53.87*

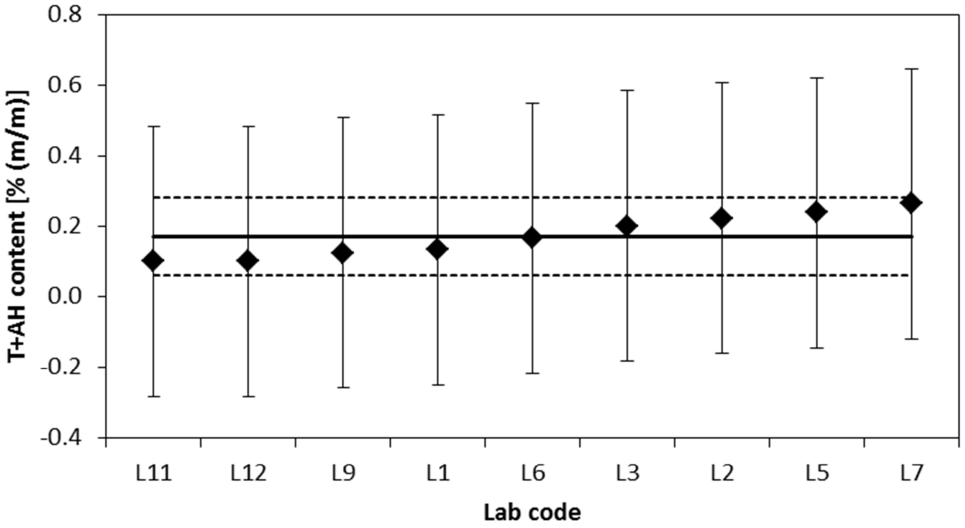

Figure E4: Results of the characterisation study for the mass fraction of T+AH content in diesel (B7) measured using EN 12916 [11] (continuous line: certified value; dashed line: expanded uncertainty with k=2; error bars: expanded measurement uncertainty derived from EN 12916 and as given in Table D2)

Table E5: Mass fraction of poly AH content in diesel (B7) 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 [%]
L1	1.90	1.87	1.91	1.93	1.86	1.84	1.89	1.80
L2	2.378	2.195	2.23	2.16	2.332	2.204	2.249	3.83
L3	1.90	2.00	2.40	2.30	2.00	2.10	2.12	9.17
L5	2.37	2.35	2.31	2.26	2.39	2.400	2.347	2.27
L6	1.94	2.06	1.99	1.91	2.02	1.98	1.98	2.72
L7	1.6572	1.6390	1.7775	1.7585	1.7345	1.6818	1.7081	3.32
L9	2.02	2.04	1.98	2.05	2.04	1.98	2.018	1.55
L11	2.0	2.0	2.0	2.0	2.0	2.0	2.0	0.00
L12	1.80	1.80	1.80	1.80	1.80	1.80	1.80	0.00
Results not used for certification								
L8	*1.9*	*2.4*	*1.9*	*2.3*	*1.7*	*2.4*	*2.1*	*14.44*
L13	*3.0*	*3.0*	*2.6*	*2.6*	*1.9*	*1.8*	*2.5*	*21.05*

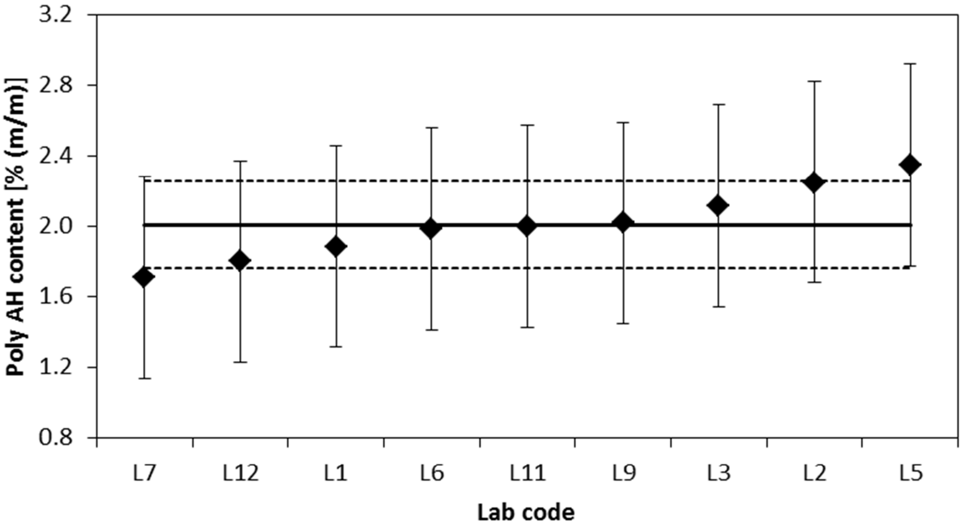

Figure E5: Results of the characterisation study for the mass fraction of poly AH content in diesel (B7) measured using EN 12916 [11] (continuous line: certified value; dashed line: expanded uncertainty with k=2; error bars: expanded measurement uncertainty derived from EN 12916 and as given in Table D2)

Table E6: Mass fraction of total AH content in diesel (B7) 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 [%]
L1	20.03	20.04	19.96	20.09	20.01	20.03	20.0	0.21
L2	20.683	20.53	20.71	20.54	20.594	20.662	20.620	0.37
L3	20.10	20.60	21.40	21.30	20.70	20.40	20.75	2.45
L5	22.36	22.57	22.13	22.32	22.85	22.840	22.512	1.30
L6	19.92	20.08	20.20	20.10	20.34	20.44	20.18	0.93
L7	21.3635	21.2975	21.8053	21.8406	21.7501	21.5136	21.5951	1.09
L9	20.65	20.7	20.6	20.69	20.58	20.58	20.63	0.26
L11	20.6	20.2	20.5	20.6	20.1	20.4	20.4	1.03
L12	20.90	20.80	20.90	20.80	21.00	20.90	20.88	0.36
Results not used for certification								
L8	*23.0*	*22.5*	*21.4*	*22.2*	*21.7*	*23.1*	*22.3*	*3.07*
L13	*24.5*	*24.1*	*22.0*	*22.0*	*20.0*	*19.8*	*22.1*	*8.91*

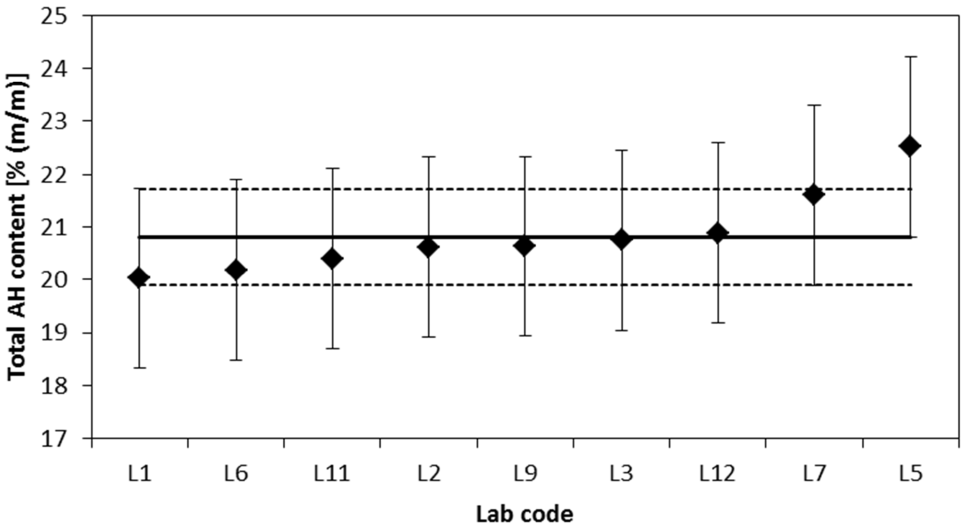

Figure E6: Results of the characterisation study for the mass fraction of total AH content in diesel (B7) measured using EN 12916 [11] (continuous line: certified value; dashed line: expanded uncertainty with k=2; error bars: expanded measurement uncertainty derived from EN 12916 and as given in Table D2)

Table E7: Mass fraction of water content in diesel (B7) 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 [%]
L1	0.00657	0.00642	0.00525	0.00513	0.00504	0.00525	0.00561	12.33
L2	0.00544	0.00549	0.00488	0.00491	0.00659	0.00661	0.00565	13.74
L4	0.00600	0.00570	0.00630	0.00570	0.00620	0.00570	0.00593	4.61
L5	0.00852	0.00837	0.00815	0.00755	0.00603	0.00604	0.00744	15.31
L6	0.00526	0.00507	0.00823	0.00843	0.00745	0.00719	0.00694	20.91
L7	0.00544	0.00569	0.00532	0.00524	0.00838	0.00781	0.00631	22.18
L8	0.00567	0.00588	0.00566	0.00573	0.00597	0.00625	0.00586	3.87
L9	0.00580	0.00550	0.00710	0.00690	0.00540	0.00530	0.00600	13.25
L10	0.00487	0.00490	0.00812	0.00786	0.00778	0.00746	0.00683	22.28
L11	0.00500	0.00500	0.00800	0.00800	0.00500	0.00500	0.00600	25.82
L12	0.00700	0.00700	0.00600	0.00600	0.00900	0.00800	0.00717	16.31
L13	0.00871	0.00866	0.00518	0.00519	0.00540	0.00556	0.00645	26.93
Results not used for certification								
L3	*0.0103*	*0.0095*	*0.0119*	*0.0098*	*0.0108*	*0.0091*	*0.0102*	*9.88*
L14	*0.0053*	*0.0054*	*0.0057*	*0.0055*	*0.0057*	*0.0054*	*0.0055*	*3.04*

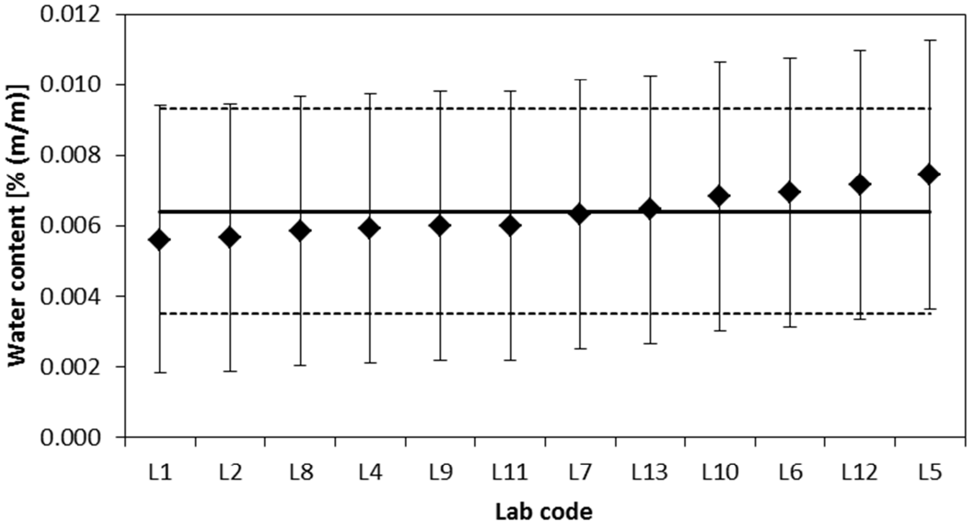

Figure E5: Results of the characterisation study for the mass fraction of water content in diesel (B7) measured using EN 12937 [12] (continuous line: certified value; dashed line: expanded uncertainty with k=2; error bars: expanded measurement uncertainty derived from EN 12937 and as given in Table D2)

Table E8: Mass fraction of sulfur content in diesel (B7) as reported by each individual lab

Laboratory code	replicate 1 [mg/kg]	replicate 2 [mg/kg]	replicate 3 [mg/kg]	replicate 4 [[mg/kg]	replicate 5 [mg/kg]	replicate 6 [mg/kg]	mean [mg/kg]	RSD [%]
L1	7.6	7.3	7.6	7.6	7.6	7.7	7.6	1.49
L2	7.57	7.73	7.50	7.56	7.48	7.70	7.59	1.36
L3	8.8	8.9	9.1	9.0	8.8	8.9	8.92	1.31
L4	8.07	7.95	7.98	7.91	8.16	7.78	7.98	1.65
L5	8.00	8.00	8.13	8.15	8.26	8.26	8.13	1.43
L6	6.7	6.4	7.0	7.2	6.5	6.5	6.7	4.60
L7	5.7	5.6	5.4	5.5	5.4	5.4	5.5	1.97
L8	7.54	7.51	7.72	7.79	7.66	7.70	7.65	1.42
L9	7.96	7.96	7.85	7.97	7.89	7.89	7.92	0.63
L10	6.31	6.48	6.82	6.56	6.3	6.22	6.45	3.43
L11	7.4	7.3	7.3	7.4	7	7.2	7.3	2.07
L12	7.6	7.7	7.7	7.6	7.4	7.5	7.6	1.54
L13	7.84	7.79	7.53	7.5	7.39	7.57	7.60	2.30
L14	8.21	8.30	8.34	8.31	8.24	8.23	8.27	0.63

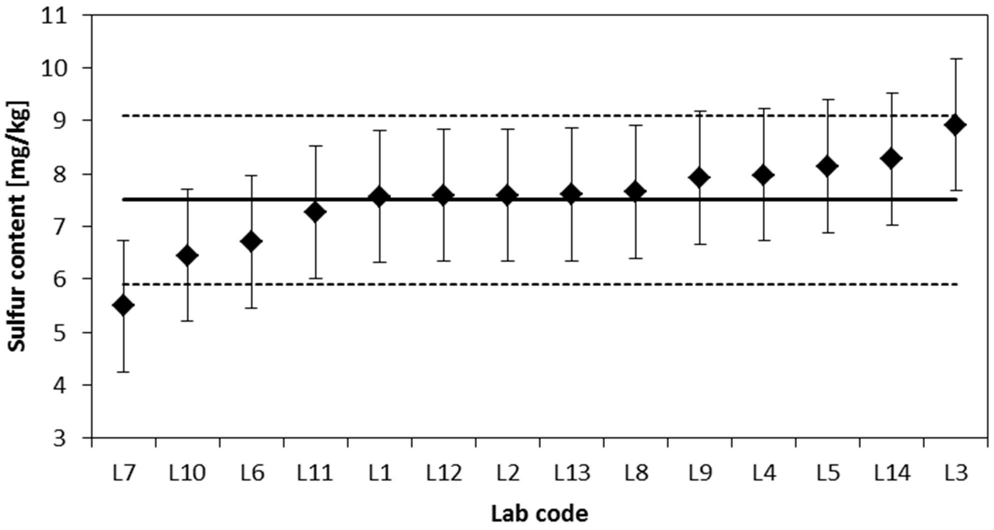

Figure E6: Results of the characterisation study for the mass fraction of sulfur content in diesel (B7) measured using ISO 20846 [13] (continuous line: certified value; dashed line: expanded uncertainty with k=2; error bars: expanded measurement uncertainty derived from ISO 20846 and as given in Table D2)

Table E9: Density in diesel (B7) as reported by each individual lab

Laboratory code	replicate 1 [kg/m^3]	replicate 2 [kg/m^3]	replicate 3 [kg/m^3]	replicate 4 [kg/m^3]	replicate 5 [kg/m^3]	replicate 6 [kg/m^3]	mean [kg/m^3]	RSD [%]
L1	837.30	837.31	837.39	837.31	837.31	837.31	837.32	0.004
L2	837.26	837.12	837.28	837.20	837.24	837.26	837.23	0.007
L3	837.10	837.10	837.10	837.10	837.00	837.10	837.08	0.005
L4	837.30	837.30	837.30	837.30	837.40	837.50	837.35	0.010
L5	837.34	837.33	837.30	837.34	837.32	837.34	837.33	0.002
L6	837.28	837.26	837.26	837.27	837.26	837.26	837.27	0.001
L7	837.12	837.12	837.12	837.12	837.10	837.10	837.11	0.001
L8	837.28	837.28	837.28	837.27	837.30	837.28	837.28	0.001
L9	837.27	837.26	837.33	837.31	837.21	837.22	837.27	0.006
L10	837.30	837.20	837.20	837.20	837.20	837.20	837.22	0.005
L11	837.00	837.00	837.00	837.00	837.00	837.00	837.00	0.000
L12	837.30	837.30	837.20	837.20	837.20	837.20	837.23	0.006
L13	837.30	837.40	837.30	837.30	837.30	837.30	837.32	0.005

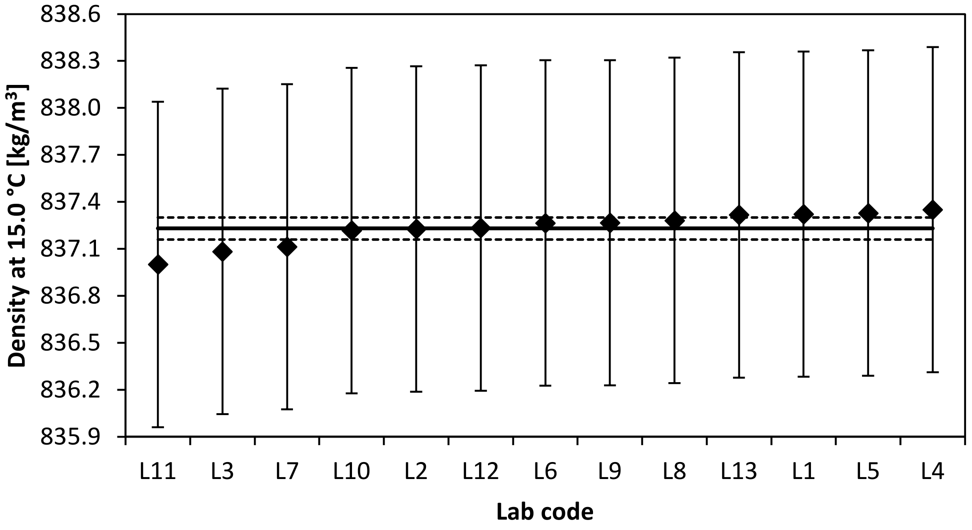

Figure E8: Results of the characterisation study for density in diesel (B7) measured using EN ISO 12185 [14] (continuous line: certified value; dashed line: expanded uncertainty with k=2; error bars: expanded measurement uncertainty derived from EN ISO 12185 and as given in Table D2)

Table E10: Kinematic viscosity in diesel (B7) as reported by each individual lab

Laboratory code	replicate 1 [mm^2/s]	replicate 2 [mm^2/s]	replicate 3 [mm^2/s]	replicate 4 [mm^2/s]	replicate 5 [mm^2/s]	replicate 6 [mm^2/s]	mean [mm^2/s]	RSD [%]
L1	2.887	2.887	2.886	2.887	2.890	2.887	2.887	0.05
L2	2.892	2.894	2.89	2.90	2.893	2.894	2.893	0.08
L4	2.9097	2.9061	2.9028	2.9133	2.9136	2.9028	2.9081	0.17
L5	2.9006	2.8975	2.8874	2.8882	2.8882	2.8913	2.8922	0.19
L6	2.8906	2.8886	2.8883	2.8886	2.8880	2.8886	2.8888	0.03
L7	2.8971	2.8957	2.8942	2.8942	2.8982	2.8966	2.8960	0.06
L8	2.8930	2.8941	2.8915	2.8912	2.8899	2.8906	2.8917	0.05
L9	2.8868	2.8870	2.8866	2.8891	2.8874	2.8874	2.8874	0.03
L10	2.8975	2.8889	2.8910	2.8900	2.8880	2.8880	2.8906	0.12
L11	2.880	2.881	2.881	2.881	2.880	2.882	2.881	0.03
L12	2.900	2.902	2.898	2.898	2.887	2.891	2.896	0.20
L13	2.901	2.902	2.900	2.904	2.900	2.903	2.902	0.06
L14	2.873	2.888	2.881	2.885	2.883	2.880	2.882	0.18
Results not used for certification								
L3	*2.9610*	*2.9590*	*2.9930*	*2.9580*	*2.9600*	*2.9740*	*2.9675*	*0.47*

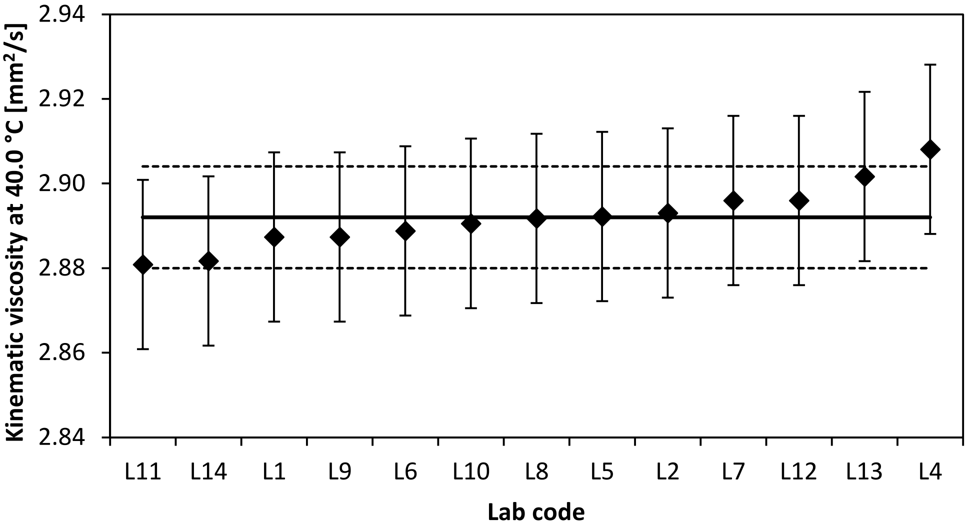

Figure E9: Results of the characterisation study for kinematic viscosity in diesel (B7) measured using EN ISO 3104 [15] (continuous line: certified value; dashed line: expanded uncertainty with k=2; error bars: expanded measurement uncertainty derived from EN ISO 3104 and as given in Table D2)

Table E11: Oxidation stability of diesel (B7) as reported by each individual lab

Laboratory code	replicate 1 [h]	replicate 2 [h]	replicate 3 [h]	replicate 4 [h]	replicate 5 [h]	replicate 6 [h]	mean [h]	RSD [%]
L1	56.23	58.09	56.33	58.11	60.15	59.68	58.10	2.81
L2	44.56	51.48	52.34	52.81	44.70	43.80	48.28	8.98
L3	43.10	44.30	48.20	45.40	44.20	46.50	45.28	4.06
L4	43.54	45.45	47.02	43.87	42.28	43.86	44.34	3.74
L5	43.59	44.33	45.98	45.76	46.15	/ *	45.16	2.52
L6	49.26	55.85	56.57	60.28	59.16	59.07	56.70	7.08
L7	60.97	60.89	58.82	62.35	58.73	58.95	60.12	2.50
L8	56.92	0.00	56.14	56.53	57.83	0.00	56.86	1.27
L9	56.88	56.94	56.86	56.81	54.46	54.13	56.01	2.38
L11	+ 40	/ *	+ 40	+ 40	+ 40	+ 40		
L12	> 48	> 48	> 48	47	>48	>48		
L13	49.96	50.30	43.92	45.77	49.14	48.13	47.87	5.28

* due to technical problem no result reported

Table E12: Lubricity of diesel (B7) as reported by each individual lab

Laboratory code	replicate 1 [µm]	replicate 2 [µm]	replicate 3 [µm]	replicate 4 [µm]	replicate 5 [µm]	replicate 6 [µm]	mean [µm]	RSD [%]
L1	271.0	278.0	267.0	272.0	291.0	278.0	276.2	3.05
L2	254.0	272.0	203.0	221.0	216.0	261.0	237.8	11.80
L3	294.00	298.00	272.00	282.00	314.00	306.00	294.33	5.24
L5	178.00	202.00	176.00	179.00	207.00	186.00	188.00	7.08
L6	172.0	200.0	224.5	187.5	198.0	198.0	196.7	8.75
L7	260.32	266.38	266.78	264.44	215.94	209.58	247.24	10.87
L8	225.00	194.00	249.00	247.00	207.00	188.00	218.33	12.03
L11	160	160	180	190	190	190	178	8.25
L12	200.0	200.0	200.0	200.0	210.0	200.0	201.7	2.02
L13	205.0	239.0	180.0	196.0	170.0	189.0	196.5	12.28

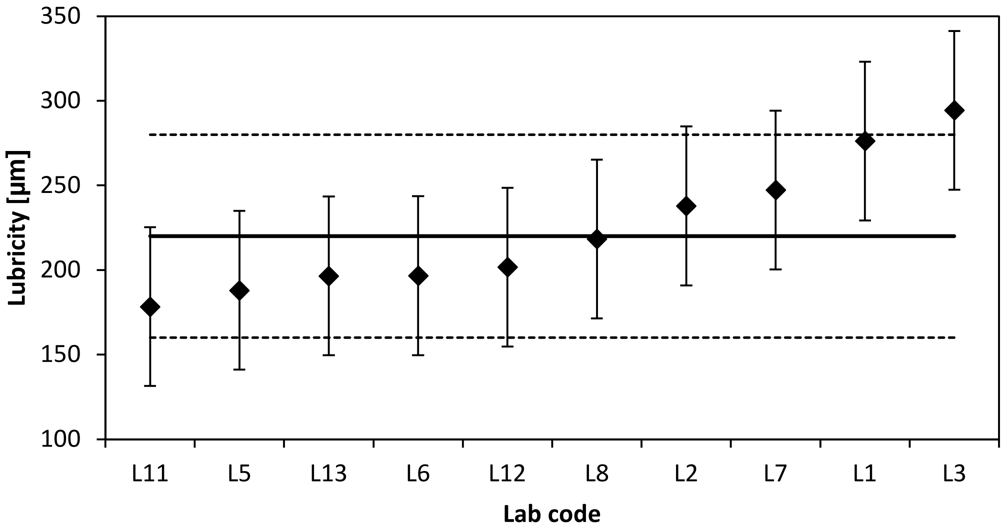

Figure E11: Results of the characterisation study for the lubricity of diesel (B7) measured using EN 12156-1 [17] (continuous line: certified value; dashed line: expanded uncertainty with k=2; error bars: expanded measurement uncertainty derived from EN 12156-1 and as given in Table D2)

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