Results of Proficiency Test Biodiesel 100% FAME (B100) May 2017

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1 Introduction

Since 1991, the Institute for Interlaboratory Studies organises every year proficiency tests (PT) for Fatty Acid Methyl Esters (FAME) used as Biodiesel B100. In the annual proficiency testing program of 2016/2017, it was decided to continue the May proficiency tests on Biodiesel B100 in accordance with the latest applicable version of ASTM D6751 specification and additional tests (e.g. EN14214:2012+A1:2014/AC:2014). In these interlaboratory studies, in total, 44 laboratories from 23 different countries registered for participation. See appendix 2 for a list of number of participants per study and per country. In this report the results of the 2017 Biodiesel B100 ASTM proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

In this proficiency test on Biodiesel B100, a sample of Rapeseed methyl ester was used. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. In this proficiency test, the participants received, depending on the registration, from one up to five different samples of Biodiesel B100, see table below.

Samples	Amount in L	Purpose	Spiked
#17065	#17065 1.5 For regular analysis		
#17066	#17066 2 Cetane Number &DCN		
#17067	0.1	Analysis of Phosphorus, Potassium, Sodium and Calcium & Magnesium	Phosphorus, Sodium, Calcium
#17068	#17068 1 Total Contamination test		Arizona dust
#17069	0.5	Cold Soak Test / Filter Blocking	

Table 1: five different Biodiesel B100 samples used in iis17G02ASTM

The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). These PTs fall under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

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2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

The necessary bulk material, 2 drums, with in total approximately 400 L of Biodiesel (100% RME) for this PT was obtained from an European producer.

Biodiesel B100 #17065

After fit-for-use testing and homogenisation of one drum, 64 amber glass bottles of 1L and 64 amber glass bottles of 0.5L, were filled, closed with inner and outer caps and labelled #17065. The homogeneity of the subsamples #17065 was checked by the determination of Density in accordance with ASTM D4052 on 8 stratified randomly selected samples:

	Density at 15°C in kg/m ³
sample #17065-1	881.92
sample #17065-2	881.90
sample #17065-3	881.91
sample #17065-4	881.91
sample #17065-5	881.90
sample #17065-6	881.90
sample #17065-7	881.89
sample #17065-8	881.89

Table 2: homogeneity test results of subsamples #17065

From the above test results, the calculated repeatability was calculated and compared with 0.3 times the reproducibilities (R) of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table:

	Density at 15°C in kg/m ³	
r (observed)	0.03	
reference test method	ISO12185:96	
0.3 x R (ref. test method)	0.15	

Table 3: evaluation of the repeatability of subsamples #17065

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Biodiesel B100 Cetane Number #17066

After fit-for-use testing and homogenisation of the second drum, 68 amber glass bottles of 1L were filled, closed with inner and outer caps and labelled #17066. The homogeneity of the subsamples #17066 was checked by the determination of Density in accordance with ASTM D4052 on 8 stratified randomly selected samples:

	Density at 15°C in kg/m ³
sample #17066-1	882.93
sample #17066-2	882.92
sample #17066-3	882.93
sample #17066-4	882.93
sample #17066-5	882.93
sample #17066-6	882.92
sample #17066-7	882.93
sample #17066-8	882.92

Table 4: homogeneity test results of subsamples #17066

From the above test results, the calculated repeatability was calculated and compared with 0.3 times the reproducibilities (R) of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table:

	Density at 15°C in kg/m ³	
r (observed)	0.01	
reference test method	ISO12185:96	
0.3 x R (ref. test method)	0.15	

Table 5: evaluation of the repeatability of subsamples #17066

Metals in Biodiesel B100 #17067

From the remaining batch used for sample #17065, approx. 6 litres (5.3 kg) were spiked with Calcium (approx. 6 mg/kg), Phosphorus (approx. 9 mg/kg) and Sodium (approx. 6 mg/kg). After homogenisation, the material was subsequently divided over 59 HDPE bottles of 0.1L and labelled #17067. The homogeneity of the subsamples #17067 was checked by determination of Phosphorus in accordance with EN14107 and Sodium in accordance with EN14214 on 8 stratified randomly selected samples:

	Phosphorus in mg/kg	Sodium in mg/kg
sample #17067-1	8.4	5.2
sample #17067-2	8.6 5.0	
sample #17067-3	8.6	5.0
sample #17067-4	8.3	5.1
sample #17067-5	8.5	5.0
sample #17067-6	8.4	5.0
sample #17067-7	8.4	5.0
sample #17067-8	8.4	5.1

Table 6: homogeneity test results of subsamples #17067

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From the above test results, the calculated repeatability was calculated and compared with 0.3 times the reproducibilities (R) of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table:

	Phosphorus in mg/kg	Sodium in mg/kg
r (observed)	0.3	0.2
reference test method	EN14107:03	EN14214:14
0.3 x R (ref. test method)	0.5	1.1

Table 7: evaluation of the repeatability of subsamples #17067

Total Contamination #17068

Into 41 amber glass bottles 1 ml of a freshly prepared and ultrasonically homogenized 15 g/kg Arizona Dust in oil suspension was pipetted. The addition was checked by weighing each bottle before and after the addition of the oil suspension. Subsequently, each bottle with inner and outer caps was filled with one litre Biodiesel B100, coming from the remaining batch used for sample #17065. The bottles were labelled #17068.

Cold Soak Test / Filter Blocking Tendency #17069

From the remaining batch used for the Cetane Number of Biodiesel, 25 litres were used to fill 48 bottles of 0.5 litre amber glass bottles and labelled #17069. The homogeneity of the subsamples was checked by the determination of density in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Density at 15°C in kg/m ³
sample #17069-1	882.92
sample #17069-2	882.92
sample #17069-3	882.91
sample #17069-4	882.92
sample #17069-5	882.92
sample #17069-6	882.92
sample #17069-7	882.93
sample #17069-8	882.93

Table 8: homogeneity test results of subsamples #17069

From the above test results, the calculated repeatability was calculated and compared with 0.3 times the reproducibilities (R) of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table:

	Density at 15°C in kg/m ³	
r (observed)	0.02	
reference test method	ISO12185:96	
0.3 x R (ref. test method)	0.15	

Table 9: evaluation of the repeatability of subsamples #17069

The calculated repeatabilities for sample #17065, #17066, #17067, #17068 and #17069 were less than 0.3 times the corresponding reproducibility of the respective reference method. Therefore, homogeneity of the subsamples was assumed.

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Depending on the registration of the participant, one 1 litre bottle and 0.5 litre bottle both labelled #17065, two 1 litre bottles labelled #17066, a 100mL bottle labelled #17067, a 1 litre bottle labelled #17068 and/or a 0.5 litre bottle labelled #17069, were dispatched to each of the participating laboratories on April 12, 2017.

2.5 STABILITY OF THE SAMPLES

The stability of the Biodiesel B100, packed in the brown glass bottles, was checked. The material has been found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The tests methods to be used by the participating laboratories should be in accordance with the requirements of ASTM D6751:15 and additional tests (e.g. EN14214:12+A1:14/AC:14):

Parameter	ASTM D6751:15	Parameter	EN14214:12
Acid Number	ASTM D664	Acid Value *)	EN14104 *)
Carbon Residue on 100% FAME	ASTM D4530		
		CFPP	EN116
Cloud Point	ASTM D2500		
Copper Strip Corrosion	ASTM D130	Copper Strip Corrosion	ISO2160
		Density at 15°C	ISO12185
Flash Point	ASTM D93	Flash Point (Recc) *)	ISO3679 *)
		Flash Point (PMcc)	ISO2719
		Iodine Value	EN14111
Kin. Visc. at 40°C	ASTM D445	Kin. Visc. at 40°C	ISO3104
Oxidation Stability	EN15751	Oxidation Stability	EN14112
Sulphated Ash	ASTM D874	Sulphated Ash	ISO3987
Sulphur	ASTM D5453	Sulphur	ISO20846
Water and Sediment	ASTM D2709	Water	ISO12937
Calcium + Magnesium	EN14538	Calcium + Magnesium	EN14538
Phosphorus	ASTM D4951	Phosphorus	EN14107
		Polyunsaturated esters *)	EN15779 *)
Potassium + Sodium	EN14538	Potassium + Sodium	EN14108/14109
Methanol	EN14110	Methanol	EN14110
		mono-, di-, tri-Glycerides	EN14105
Free + Total Glycerin	ASTM D6584	Free + Total Glycerol	EN14105
		FAME content *)	EN14103 *)
		Linolenic Acid *)	EN14103 *)
Distillation; 90% recovered	D1160		
Total Contamination	D7321	Total Contamination	EN12662
Cold Soak Filterability	ASTM D7501		

Table 10: requirements and test methods acc. to specifications ASTM D6751:15 and EN14214:12+A1:14/AC:14

It was explicitly requested to treat the samples as if they were routine samples. Therefore, each laboratory is advised to perform only those analyses that normally are done in daily routine (but the laboratories are allowed to do all analyses). Furthermore, it was requested to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to

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^{*)} not evaluated in this ASTM based round robin

report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

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According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM or IP reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other targets values were used. In some cases a reproducibility based on former its proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

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The z-scores were calculated according to:

 $z_{\text{(target)}} = \text{(test result - average of PT)} / \text{target standard deviation}$

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare.

The usual interpretation of z-scores is as follows:

```
|z| < 1 good

1 < |z| < 2 satisfactory

2 < |z| < 3 questionable

3 < |z| unsatisfactory
```

4 EVALUATION

In this proficiency test several problems were encountered during the execution.

For the regular Biodiesel PT (#17065): 3 participants reported test results after the final reporting date and 3 other participant did not report any test results at all.

For the Cetane Number PT (#17066): 1 participant reported test results after the final reporting date and 3 other participant did not report any test results at all.

For the Total Contamination PT: 2 participants reported the test result after the final reporting date and 3 participants did not report any test results at all.

For the Metals in Biodiesel PT: 1 participant reported the test results after the final reporting date and 4 participants did not report any test results at all.

For the Filter Blocking PT: 1 participant reported a result after the final reporting date and 6 participants did not report any test results at all.

Finally, in total 449 numerical test results were reported by the participants. Observed were 11 outlying results, which is 2.5%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER SAMPLE AND PER TEST

In this section, the reported test results are discussed per sample and per test. The specified test methods and requirements ASTM D6751:15 and additional tests (e.g. EN14214:12+A1:14/AC:14) which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the reported data. The abbreviations, used in these tables, are listed in appendix 3.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D6371) and an added designation for the year that the method was adopted or revised (e.g. D6371:05). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g.

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D6371:05(2010)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D6371:05 will be used.

For Biodiesel B100 sample #17065

Acid Number:

(ASTM)

This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM

D664:11ae1 (method B).

Cloud Point:

This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D2500:16b.

CFPP:

This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6371:16.

Carbon Residue on 100%: All reported results were near or below the applicable lower limit of D4530:15 (0.1%M/M). Therefore no significant conclusions were drawn.

Copper Strip Corrosion: No problems have been observed. All participants agreed on a test result of 1.

Density at 15°C

This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO12185:96.

Flash Point PMcc: This determination was not problematic. No statistical outliers were observed, but three test results were excluded from statistical calculations as the used test method is not comparable with ASTM D93. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D93C:15.

<u>Iodine Value:</u>

This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN14111:03.

Kin.Visco. at 40°C: The determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D445:17.

Oxidation Stability: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of EN15751:14.

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Sulphated Ash: All reported results were near or below the applicable lower limit of ASTM

D874:13a and/or ISO3987:10 (0.005% M/M). Therefore no significant

conclusions were drawn.

Sulphur: This determination was problematic at a low level of 1.6 mg/kg. No

statistical outliers were observed. However, the calculated

reproducibility is not in agreement with the requirements of ASTM

D5453:16e1.

Water: This determination was not problematic. Three statistical outliers were

observed. However the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM

D6304:16e1.

Water and Sediment: All reported results were near or below the applicable lower limit of

ASTM D2709:96(2011). Therefore no significant conclusions were drawn.

<u>Distillation:</u> Only three laboratories reported test results, therefore no significant

conclusions were drawn.

Methanol: This determination was problematic for a number of laboratories. Three

statistical outliers were observed. However, the calculated

reproducibility after rejection of the statistical outliers is in agreement

with the requirements of EN14110:03.

mono-Glycerides: This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in agreement with the

requirements of ASTM D6584:13.

<u>di-Glycerides:</u> This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in agreement with the

requirements of ASTM D6584:13.

tri-Glycerides: This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in agreement with the

requirements of ASTM D6584:13.

<u>Free Glycerine:</u> This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in agreement with the

requirements of ASTM D6584:13.

Total Glycerine: This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in agreement with the

requirements of ASTM D6584:13.

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For Biodiesel B100 sample #17066: Cetane Number

<u>Cetane Number:</u> This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in full agreement with the

requirements of ASTM D613:16a.

<u>Derived Cetane Number:</u> In total only three laboratories reported test results, therefore no significant conclusions were drawn.

For Biodiesel B100 sample #17067: Metals

Calcium and Magnesium: This determination was problematic. No statistical outliers were

observed. However, the calculated reproducibility is not in agreement with

the requirements of EN14538:06.

Phosphorus This determination was problematic. Two statistical outliers were

observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of EN14107:03. The samples were spiked with Phosphorus. The average recovery of Phosphorus (theoretical increment of 8.99 mg/kg) may be excellent: "less than 100%". The actual blank concentration for Phosphorus is unknown.

Potassium All reported results were near or below the applicable lower limit of

EN14214:14. Therefore no significant conclusions were drawn.

<u>Sodium</u> This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in agreement with the requirements of EN14214:12+A1:2014/AC:2014 and with the

requirements of EN14108:03.

The samples were spiked with Sodium. The average recovery of Sodium (theoretical increment of 5.99 mg/kg) may be satisfactory: "less

than 79%". The actual blank concentration for Sodium is unknown.

<u>Potassium and Sodium:</u> This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement

with the requirements of EN14538:06.

For Biodiesel B100 sample #17068: Particulate Contamination

Total Contamination: As this proficiency test was ASTM based, the intention was to

evaluate the Total contamination against ASTM D7321. Regretfully, the majority of the laboratories reported to have used EN12662 as test method. Therefore it was decided to evaluate the test results for ASTM D7321 and EN12662 separately, which have significant differences

and may produce different test results.

ASTM D7321: This determination was problematic. No statistical outliers were

observed. However, the calculated reproducibility is not in agreement

with the requirements of ASTM D7321:14.

EN12662:

This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of EN12662:98. The test results vary over a large range: <6 – 63.1 mg/kg.

For Biodiesel B100 sample #17069: CSFT&FBT

Filter Blocking Potential by Cold Soak test: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D7501:12a. The test results vary over a large range: 83 – 196 mg/kg.

Filter Blocking Tendency: This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D2068:14 (or IP387-B, which is identical). The low number of reported test results may partly explain the large variation.

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4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of laboratories that participated. The reproducibilities derived from literature standards (in casu the ASTM, EN, ISO and IP standards) and the calculated reproducibilities of the samples (see appendix 1) are compared in the next table.

Parameter	unit	n	average	R (Calc.)	R (lit)
Acid Number (D664-B)	mg KOH/g	23	0.37	0.11	0.12
Cloud Point	°C	28	-6.2	5.1	5
Cold Filter Plugging Point	°C	23	-21.4	4.2	4.7
Carbon Residue on 100% FAME	%M/M	15	<0.1	n.a.	n.a.
Copper Strip Corrosion		22	1	n.a.	na
Density at 15°C	kg/m ³	35	882.0	0.4	0.5
Flash Point (PMcc) ASTM	°C	27	157.9	15.2	14.7
lodine Value	g l ₂ /100g	18	111.6	6.8	5
Kin. Viscosity at 40°C	mm²/s	28	4.435	0.050	0.099
Oxidation Stability EN15751	hours	18	7.4	1.3	1.8
Sulphated Ash	%M/M	12	<0.005	n.a.	n.a.
Sulphur	mg/kg	21	1.6	1.3	0.8
Water	mg/kg	30	358	87	575
Water and Sediment	%V/V	11	<0.05	n.a.	n.a.
Distillation at 10mm Hg					
80% recovered	°C	3	(353.5)	(1.4)	(4.6)
90% recovered	°C	3	(355.2)	(1.9)	(4.6)
95% recovered	°C	3	(359.7)	(8.0)	(4.6)
Methanol	%M/M	12	0.029	0.005	0.009
mono-Glycerides	%M/M	14	0.270	0.159	0.204
di-Glycerides	%M/M	14	0.090	0.046	0.083
tri-Glycerides	%M/M	13	0.042	0.049	0.122
Free Glycerine	%M/M	11	0.003	0.004	0.005
Total Glycerine	%M/M	14	0.089	0.040	0.049

Table 11: comparison of the observed and target reproducibilities of Biodiesel B100 sample #17065 (results between brackets are for information only)

Parameter	unit	n	average	R (Calc.)	R (lit)
Cetane Number		4	51.6	4.3	4.3
DCN (D7668)		1	n.a.	n.a.	n.a.
DCN (D6890)		2	55.0	n.a.	n.a.

Table 12: comparison of the observed and target reproducibilities of Biodiesel B100 sample #17066

Parameter	unit	n	average	R (Calc.)	R (lit)
sum Calcium and Magnesium	mg/kg	12	12.6	5.8	3.1
Phosphorus	mg/kg	9	9.0	2.8	1.8
Potassium	mg/kg	9	<1	n.a.	n.a.
Sodium	mg/kg	11	4.7	2.5	3.4
Sum Potassium and Sodium	mg/kg	11	5.0	2.2	1.9

Table 13: comparison of the observed and target reproducibilities of Biodiesel B100 sample #17067

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Parameter	Unit	n	average	R (Calc.)	R (lit)
Particulate Contamination (D7321)	mg/L	4	25.4	11.2	7.4
Total Contamination (EN12662)	mg/kg	11	15.0	13.6	4.5

Table 14: comparison of the observed and target reproducibilities of Biodiesel B100 sample #17068

Parameter	Unit	n	average	R (Calc.)	R (lit)
Filter Blocking Potential by CST	S	7	138	104	50
Filter Blocking Tendency		5	5.2	2.5	1.7

Table 15: comparison of the observed and target reproducibilities of Biodiesel B100 sample #17069

Without further statistical calculations it can be concluded that for many tests there is a good compliance of the group of participating laboratories with the relevant standards. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2017 WITH PREVIOUS PTS

	May 2017	May 2016	October 2015	April 2015
Type of FAME	Rapeseed	Rapeseed	Rapeseed	Rapeseed
Number of reporting labs	38	54	54	60
Number of results reported	449	596	788	965
Number of statistical outliers	11	25	19	23
Percentage statistical outliers	2.5%	4.2%	2.4%	2.4%

Table 16: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given the following table:

Determination	May 2017	May 2016	October 2015	April 2015
Acid Number (D664-B)	+/-	+	+	++
Cloud Point	+/-		+	+
Cold Filter Plugging Point	+	-	++	+/-
Carbon Residue on 100% FAME	n.e.	n.e.	(-)	n.e.
Density at15°C	+	++	++	+/-
Flash Point PMcc ASTM	+/-	+/-	-	+
lodine Value	-	+/-	+	+/-
Kin. Viscosity at 40°C	++	++	+/-	-
Oxidation Stability	+	+	++	++
Sulphated Ash	n.e.	n.e.	n.e.	()
Sulphur	-	+	+	+
Water	++	++	+	++
Distillation (80, 90 and 95% rec.)	(+)	-	n.e.	n.e.
Methanol	++	-	-	-
mono-Glycerides	++	++	+	+
di-Glycerides	++	++	+/-	+/-
tri-Glycerides	++	++	+	++
Free Glycerine	+	++	++	+
Total Glycerine	+	+	+	+

Table 17: comparison of group performances against the standard requirements

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Determination	May 2017	May 2016	October 2015	April 2015
Cetane Number	+/-	n.e.	n.e.	n.e.
DCN	n.e.	n.e.	n.e.	n.e.
Sum of Calcium and Magnesium	-	+/-	-	-
Phosphorus	-			
Potassium	n.e.	++	++	++
Sodium	+	+	+	+
Sum of Potassium and Sodium	-		n.e.	n.e.
Particle contamination		n.e.	n.e.	n.e.
Total Contamination			-	-
Filter Blocking Potential by CST		+/-	n.e.	n.e.
Filter Blocking Tendency	-	-	n.e.	n.e.

Table 18: comparison of group performances against the standard requirements

The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

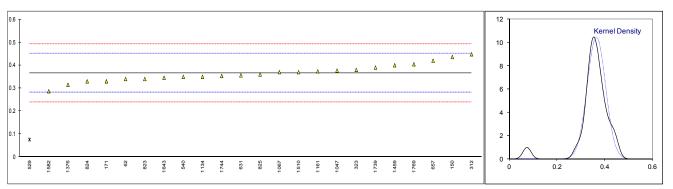
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- -- : group performed much worse than the standard
- n.e.: not evaluated

^{*} Signs between brackets are for assigned values below the application range of the respective reference test method and therefore should be used with due care

APPENDIX 1

Determination of Acid Number total conform ASTM spec. on sample #17065; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
62	D664-B	0.34		-0.62	
120					
150	D664-B	0.436		1.65	
171	D664-B	0.33		-0.86	
312	EN14104	0.448		1.93	
323	EN14104	0.38		0.32	
333					
335					
336					
337 338					
511					
529	D664	0.0757	R(0.01)	-6.87	
540	D664-B	0.35	11(0.01)	-0.39	
556	D00+ B				
558					
604					
621					
631	D974	0.356		-0.25	
657	D664-B	0.42		1.27	
823	D664-B	0.34		-0.62	
824	D664-B	0.33		-0.86	
825	D664-B	0.36		-0.15	
1033	EN144404				
1047	EN14104	0.377		0.25	
1067	EN14104	0.37		0.09	
1134 1161	D664-B D664-B	0.35 0.374		-0.39 0.18	
1199	D004-D	0.374		0.16	
1376	D664-B	0.315		-1.21	
1459	D664-B	0.40		0.80	
1494	200.0				
1510	D974	0.37		0.09	
1582	D664-B	0.2867		-1.88	
1634					
1643	D664-A	0.346		-0.48	
1739	EN14104	0.39		0.56	
1744	D664-B	0.3533		-0.31	
1769	D664-B	0.40440		0.90	
6069					
	normality	OK			
	n	23			
	outliers	1			
	mean (n)	0.36637			
	st.dev. (n)	0.038257			
	R(calc.)	0.10712			
	R(D664B:11ae1)	0.11845			



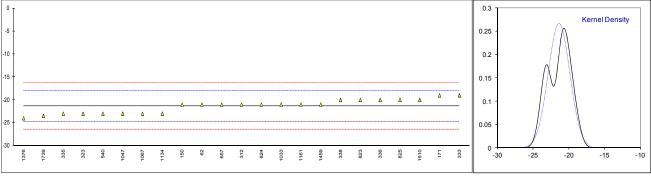
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Determination of Cloud Point on sample #17065; results in °C

lab	method	Value	mark	z(targ)	remarks
62	D2500	-5.0	С	0.64	First reported 0
120	D2300	-5.0	C	0.04	First reported 0
150	D5771	-7.0		-0.48	
171	D2500	-8		-1.04	
312	D2500	-7		-0.48	
323	EN23015	- <i>7</i> -5		0.64	
333	D2500	-8		-1.04	
335	D2500 D2500	-0 -7		-0.48	
336	D2500 D2500	-7 -7.5		-0.46	
337	D2500	-7.5		-0.70	
338	EN23015	-9		-1.60	
511	LIN23013			-1.00	
529	D2500	-7		-0.48	
540	D2500	-8		-1.04	
556	D2000				
558					
604	D2500	-9		-1.60	
621					
631	D2500	-7		-0.48	
657	D2500	-5		0.64	
823	D2500	-6		0.08	
824	D2500	-4		1.20	
825	D2500	-5		0.64	
1033	D7689	-2.2		2.21	
1047	ISO3015	-8		-1.04	
1067	EN23015	-4		1.20	
1134	IP219	-4.0		1.20	
1161	D2500	-5		0.64	
1199					
1376	D2500	-7		-0.48	
1459	EN23015	-8		-1.04	
1494					
1510	D2500	-5		0.64	
1582					
1634					
1643	D2500	-4		1.20	
1739	EN23015	-3.5		1.48	
1744	D2500	-7		-0.48	
1769					
6069					
	normality	OK			
	n	28			
	outliers	0			
	mean (n)	-6.15			
	st.dev. (n)	1.819			
	R(calc.)	5.09			Compare D/FN22045/ICO2045) = 6
	R(D2500:16b)	5			Compare R(EN23015/ISO3015) = 6
1,1					0.25
-1 +					Kernel Density
-3 -					0.2 -
-5					Δ Δ Δ Δ Δ
					0.15
-7 -		Δ Δ Δ	Δ Δ Δ	Δ	
-9 - A - A	Δ Δ Δ Δ Δ				
	-				0.1 -
-11 -					
-13					0.05 -
-15	540 171 171 047	335	150 529 376	744 823 62	0 -15 -10 -5 0 5
" "	540 1771 1771 1047	m m m	150 529 631 1376	47.1 823 823 62	2 12 12 15 16 17 18 18 18 18 18 18 18 18 18 18 18 18 18

Determination of Cold Filter Plugging Point on sample #17065; results in °C

lab	method	value	mark	z(targ)	remarks
62	D6371	-21.0	С	0.22	First reported -16
120					
150	D6371	-21.0		0.22	
171	D6371	-19		1.40	
312	D6371	-21		0.22	
323	EN116	-23		-0.97	
333	D6371	-19		1.40	
335	D6371	-23		-0.97	
336	EN116	-20		0.81	
337	LIVITO				
338	EN116	-20		0.81	
511	LIVITO	-20			
529					
540	D6371	-23		-0.97	
556	D037 I	-23		-0.97	
558 604					
621					
631	IDOGG				
657	IP309	-21		0.22	
823	D6371	-20		0.81	
824	D6371	-21		0.22	
825	D6371	-20		0.81	
1033	IP309	-21		0.22	
1047	EN116	-23		-0.97	
1067	EN116	-23		-0.97	
1134	EN116	-23.0		-0.97	
1161	EN116	-21		0.22	
1199					
1376	D6371	-24		-1.56	
1459	EN116	-21		0.22	
1494					
1510	IP309	-20		0.81	
1582					
1634					
1643					
1739	EN116	-23.5		-1.26	
1744					
1769					
6069					
0000					
	normality	OK			
	n	23			
	outliers	0			
	mean (n)	-21.37			
	st.dev. (n)	1.494			
	R(calc.)	4.18			
	R(D6371:16)	4.73			Compare R(EN14241:12) = 2.94
	11(000/1.10)	4.73			Compare MEN 1424 1.12) - 2.34



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Determination of Carbon Residue on 100% FAME on sample #17065; results in %M/M

lab	method	value	mark	z(targ)	remarks
62	D4530	<0.10		_(
120	D-1000				
150	D4530	<0.01			
171	D4530	<0.10			
312	D4000				
323	EN10370	<0.10			
333	LINTOSTO				
335					
336					
337					
338					
511					
529					
540					
556					
558					
604					
621					
631	D4530	0.0099		-0.03	
657	D4530	0.0033		-0.03	
823	D4530 D4530	0.01		0.17	
824	D4530 D4530	<0.1		0.17	
825	D4530 D4530	L0.05			
1033	D4330				
1033	EN10370	0.011		-0.01	
1047	ISO10370	< 0.10		-0.01	
1134	D4530	<0.10			
1161	D4530	0.02		0.17	
1199	D4330	0.02		0.17	
1376	D4530	0.00		-0.23	
1459	D4330	0.00		-0.23	
1494					
1510	D4530	0.01		-0.03	
1582	D4550			-0.03	
1634					
1643					
1739					
1744					
1769					
6069					
0003					
	normality	unknown			
	n	15			
	outliers	0			
	mean (n)	<0.1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D4530:15)	n.a.			Application range: 0.1 – 0.3%M/M
	(= .000.10)				

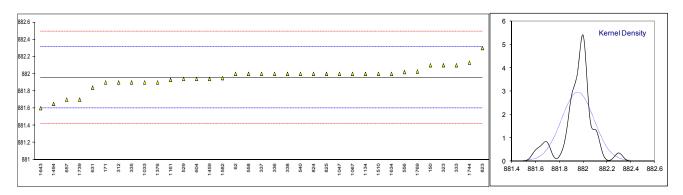
Determination of Copper Strip Corrosion 3 hrs/50°C on sample #17065

lab method value mark z(targ) remarks 62 D130 1b 120 150 D130 1a 171 D130 1a 312	
120 150 D130 1a 171 D130 1a 312 323 D130 1A	
150 D130 1a 171 D130 1a 312 323 D130 1A	
171 D130 1a 312 323 D130 1A	
312 323 D130 1A	
323 D130 1A	
335 D130 1b	
336 D130 1	
337	
338	
511	
529 D130 1A	
540 D130 1a	
556	
558	
604	
621	
631 D130 1A	
657 D130 1A	
823 D130 1a	
824 D130 1a	
825 D130 1A	
1033 IP154 1b	
1047 ISO2160 1a	
1067 ISO2160 1A	
1134 D130 1A	
1161 ISO2160 1a 1199	
1199 1376 D130 1b	
1459	
1494	
1510 IP154 1A	
1582	
1634 D130 1a	
1643	
1739 ISO2160 1a	
1744	
1769	
6069	
normality n.a.	
n 22	
outliers n.a.	
mean (n) 1	
st.dev. (n) n.a.	
R(calc.) n.a.	
R(lit) n.a.	

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Determination of Density at 15°C on sample #17065; results in kg/m³

lab	method	value	mark	z(targ)	remarks
62	D4052	882.0		0.25	
120	2.002				
150	D4052	882.1		0.81	
171	D4052	881.9		-0.31	
312	D4052	881.9		-0.31	
323	D4052	882.1		0.81	
333	D4052	882.1		0.81	
335	D4052	881.9		-0.31	
336	D4052	882.0		0.25	
337	D4052	882.0		0.25	
338	ISO12185	882.0		0.25	
511	10012103				
529	D4052	881.94		-0.09	
540	D4052 D4052	882.0	С	0.25	First reported 0.882 kg/m3
556	D4052 D4052	882.02	C	0.23	Tilst reported 0.002 kg/m3
558 604	D4052	882.0		0.25	
604	D4052	881.94		-0.09	
621	D40E2	001 04		0.65	
631	D4052	881.84		-0.65	
657	D4052	881.7		-1.43	
823	D4052	882.3		1.93	
824	D4052	882.0		0.25	
825	D4052	882.0		0.25	
1033	IP365	881.9		-0.31	
1047	ISO12185	882.0		0.25	
1067	ISO12185	882.0		0.25	
1134	IP365	882.0		0.25	
1161	ISO12185	881.93		-0.15	
1199					
1376	D4052	881.9		-0.31	
1459	ISO12185	881.94		-0.09	
1494	D4052	881.65		-1.71	
1510	IP365	882.0		0.25	
1582	D4052	881.95		-0.03	
1634	ISO12185	882.0		0.25	
1643	D4052	881.6		-1.99	
1739	ISO3675	881.7		-1.43	
1744	D4052	882.13		0.97	
1769	D4052	882.027		0.40	
6069					
	normality	suspect			
	n	35 35			
	outliers	0			
	mean (n)	881.956			
	st.dev. (n)	0.1358			
	R(calc.)	0.380			
	R(ISO12185:96)	0.500			
	13(130 12 103.90)	0.0			



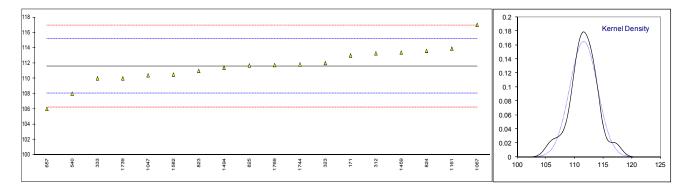
Determination of Flash Point (PMcc) conform ASTM spec. on sample #17065; results in °C

	ab	method	value	mark	z(targ)	remarks
	62	D93-C	154	IIIaik	-0.74	Telliains
	20	D93-C	154		-0.74	
	50	D93-C	150.0		-1.51	
	71	D93-C	165.6		1.47	
	12	D93-C	158.5		0.11	
	23	D93-C	160.0		0.40	
	33	D93-C	158.5		0.40	
	35	D93-C	162.0		0.78	
	36	D93-C	162.0		0.78	
	37	D00 0				
	38	ISO2719-C	167.0		1.73	
	11	D93-C	161.0		0.59	
	29	D93-C	160.5		0.49	
	40	D93-C	157.0		-0.17	
	56	NBR14598	146	С	-2.27	First reported 139
	58	D93-C	146.0	_	-2.27	
	04	D93-C	158		0.02	
	21					
	31	D93-A	154.0		-0.74	
	57	D93-C	164.0		1.16	
	23	D93-C	165.0		1.35	
	24	D93-C	158.0		0.02	
	25	D93-C	156.0		-0.36	
	33	ISO2719-A	158.0		0.02	
	47	ISO3679	169.6	ex	2.23	Result excluded, test method is not comparable with D93
	67	ISO3679	> 140			,
	34	D93-A	157.0		-0.17	
11	61	ISO3679	173.1	ex	2.89	Result excluded, test method is not comparable with D93
11	99					
13		D93-A	158.0		0.02	
	59	D93-C	162.0		0.78	
	94					
15		D93-A	150.0		-1.51	
	82					
	34	D93-C	160.8		0.55	
	43	D93-C	154.6		-0.63	
17		ISO3679	167.1	ex	1.75	Result excluded, test method is not comparable with D93
	44					
	69					
60	69					
		normality	OK			
		normality n	OK 27			
		outliers	0 (+3excl)			
		mean (n)	157.907			
		st.dev. (n)	5.423			
		R(calc.)	15.185			
		R(D93C:15)	14.7			Compare R(ISO2719C:16) = 14.7
		.(= = = = = : :)				
180 т						0.08
						Kernel Density
175 -						x 0.07 -
170 -						x 0.06 -
165 -						Δ Δ X 0.05 -
160 -				<u> </u>	^ ^ <u>^ </u>	0.04 -
155 -		Δ Δ Δ	Δ Δ Δ			0.03 -
1 1		Δ Δ				0.02
150		-				0.02
150 -	Δ Δ					
	Δ Δ					0.01 -
145 -	A A A 228	150 1510 62 631	825 540 134 604	376 312	32 3 33 3 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8	0.01 - 0.01 - 0.01 - 0.00 - 120 140 160 180 200

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Determination of Iodine Value conform EN spec. on sample #17065; results in g I₂/100g

lab	method	value	mark	z(targ)	remarks
62					
120					
150					
171	EN14111	113		0.79	
312	EN14111	113.28		0.94	
323	EN14111	112		0.23	
333	EN14111	110		-0.89	
335					
336					
337					
338					
511					
529					
540	EN14111	108		-2.01	
556					
558					
604					
621					
631					
657	EN14111	106		-3.13	
823	EN14111	111.0		-0.33	
824	EN14111	113.6		1.12	
825	EN14111	111.7		0.06	
1033					
1047	EN14111	110.4		-0.67	
1067	EN14111	117		3.03	
1134					
1161	EN14111	113.9		1.29	
1199					
1376					
1459	EN14111	113.4		1.01	
1494	EN14111	111.40		-0.11	
1510	= 11111111				
1582	EN14111	110.5		-0.61	
1634					
1643	EN14444	440			
1739	EN14111	110		-0.89	
1744	EN14111	111.831		0.13	
1769	EN14111	111.738		0.08	
6069					
	normality.	auanaat			
	normality n	suspect 18			
	outliers	0			
	mean (n)	111.60			
	st.dev. (n)	2.417			
	R(calc.)	6.76			
	R(EN14111:03)	5			
		-			



Determination of Kinematic Viscosity at 40°C on sample #17065; results in mm²/s

lab	method	value	mark	z(targ)	remarks
62	D445	4.424	mark	-0.32	Tentario
120	D440			-0.52	
150	D445	4.501	R(0.05)	1.85	
171	D445	4.424		-0.32	
312	D445	4.433	С	-0.07	First reported 4.578
323	ISO3104	4.424		-0.32	
333	D445	4.430		-0.15	
335	D445	4.431		-0.13	
336					
337					
338					
511	D445	4.463		0.78	
529	D.445	4.470			
540	D445	4.476		1.14	
556					
558 604	D445	4 4220		0.04	
604	D445	4.4339		-0.04	
621 631	D445	4.4231		-0.35	
657	D445 D445	4.424		-0.33	
823	D445 D445	4.433		-0.32 -0.07	
824	D445	4.420		-0.44	
825	D445	4.425		-0.29	
1033	2				
1047	ISO3104	4.434		-0.04	
1067	ISO3104	4.429		-0.18	
1134	IP71	4.439		0.10	
1161	ISO3104	4.454		0.52	
1199					
1376	D7042	4.4686		0.93	
1459	D7042	4.473		1.06	
1494	D445	4.4289		-0.18	
1510	D445	4.456		0.58	
1582	D445	4.4380		0.07	
1634	D445	4.424		-0.32	
1643	D445	4.4		-1.00	
1739	ISO3104	4.430		-0.15	
1744 1769	D445	4.4345		-0.03	
6069	D445	4.41938 		-0.45 	
0009					
	normality	OK			
	n	28			
	outliers	1			
	mean (n)	4.43544			
	st.dev. (n)	0.017795			
	R(calc.)	0.04983			
	R(D445:17)	0.09935			Compare R(EN14214:12) = 0.04457
4.6 T					35 1
4.55 -					Kernel Density
					30
4.5 -					х 25 -
4.45 -					
4.4 - A	<u> </u>	Δ Δ Δ Δ	Δ Δ Δ	Δ Δ Δ	
					15 -
4.35 -					
4.3 -					10 1
4.25 -					5 / /
4.2	624 631 171 62	634 634 494	739	312 823 604	0 4.35 4.4 4.45 4.5 4.55
1 + 1	- ~ ~ ~ ~ ~	2 2 2 4		., 3 9	= = = = = = = = = = = = = = = = = = =

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Determination of Oxidation Stability Induction period on sample #17065; results in hours

lab	method	value	r	nark	7/1	targ)	ror	narks						
62	metriou		•	Haik	2(101	Haiks	,					
120														
150	EN15751	7.81				0.68								
171	EN15751	6.4				-1.54								
312	EN15751	7.63				0.40								
323	EN15751	7.9				0.83								
333	EN15751	7.1			-	-0.43								
335	EN145754	 7.5												
336	EN15751	7.5 				0.20								
337 338														
511														
529														
540	EN14112	7.5				0.20								
556														
558														
604														
621														
631	EN45754	7.0				0.00								
657	EN15751	7.9				0.83								
823 824	EN15751	7.6				0.35								
825	LINIUIUI	7.0												
1033	EN15751	7.23				-0.23								
1047	EN15751	6.7				-1.06								
1067	EN14112	7.7				0.51								
1134														
1161	EN14112	7.2				-0.28								
1199	EN14112	6.62				-1.19								
1376	EN14112	7.69				0.50								
1459 1494	EN15751	7.78 				0.64								
1510	EN14112	7.1				-0.43								
1582	LIVITIIZ													
1634														
1643														
1739	EN14112	7.4				0.04								
1744														
1769														
6069														
	normality	OK												
	n	18												
	outliers	0												
	mean (n)	7.376												
	st.dev. (n)	0.4496												
	R(calc.) R(EN15751:14)	1.259					_							
	R(EN15751:14)	1.777					Co	mpare	e R(E	EN141	114:0	3) = 2	2.148	8
9.5 T														1 7
9 -														0.9 - Kernel Density
8.5 -														0.8 -
8 -												Δ	Δ	0.7 -
7.5 -				Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	-	4	0.6 -
7	Δ Δ	Δ Δ	Δ											0.5 -
	Δ Δ													0.4 -
6.5 + 🛕														0.3 -
6 +														0.2 - // \
5.5														0.1 -
5 - 5	8 7 8	2 %	g 9	9	98	24	2	92	37	20	150	323	22	
171	1199	1161	1033	940	336	824	312	1376	1067	1459	2	35	657	4 5 6 7 8 9

Determination of Sulphated Ash on sample #17065; results in %M/M

lab	method	value	mark	z(targ)	remarks
62					
120					
150	D874	< 0.005			
171	D874	<0.005			
312	D014				
323	ISO3987	0.006			
333	1000301				
335					
336					
337					
338					
511	D874	0.0021			
529	D014	0.0021			
	D074				
540 556	D874	<0.005 			
558					
604					
621 631	D874	0.0018			
657	D874	0.002			
823 824	D874	0.001			
825	D874 D874	< 0.005			
1033	D014	0.0005			
1033	ISO3987	<0,005			
1047	ISO3987	< 0.005			
1134	1303901	< 0.005 			
1161	ISO3987	<0,005			
1199	1000301				
1376					
1459					
1494					
1510					
1582					
1634					
1643					
1739	ISO3987	<0.01			
1744	1000001				
1769					
6069					
0000					
	normality	unknown			
	n	12			
	outliers	n.a.			
	mean (n)	<0.005			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D874:13a)	n.a.			
	(20:04)				

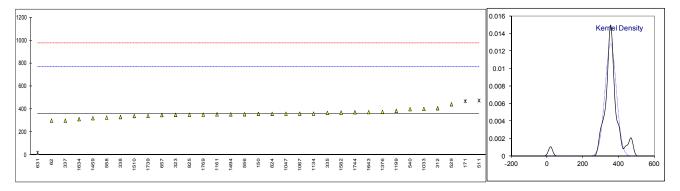
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Determination of Sulphur on sample #17065; results in mg/kg

lab	method	value	mark	z(targ)	remarks	
62	D5453	1.7		0.20		_
120						
150	D5453	1.33		-1.03		
171	D5453	1.9		0.87		
312	D5453	1.36		-0.93		
323	D5453	1.3		-1.13		
333	D5453	1.1		-1.80		
335	ISO20846	1.3		-1.13		
336	D5453	0.9		-2.46		
337	D5453	0.9		-2.46		
338	ISO20846	1.3		-1.13		
511	D5453	2.77		3.77		
529	D3433	2.77		J.77 		
	D5453					
540	D5453	2.2		1.87		
556						
558						
604						
621			_			
631	D4294	2.04	С	1.34	First reported 4.04	
657	D5453	1.9		0.87		
823	D5453	1.57		-0.23		
824	D5453	1.9		0.87		
825	D5453	2.2		1.87		
1033						
1047	ISO20846	1.4		-0.80		
1067	ISO20846	< 3.0				
1134	D5453	1.59		-0.16		
1161	ISO20846	1.80		0.54		
1199	ISO20884	<5.0				
1376	D5453	1.96		1.07		
1459	ISO8754	<5				
1494	1000704					
1510						
1582						
1634						
1643						
1739						
1744						
1769						
6069						
	normality	OK				
	n	21				
	outliers	0				
	mean (n)	1.639				
	st.dev. (n)	0.4675				
	R(calc.)	1.309				
	R(D5453:16e1)	0.840			Application range: 1 – 8000 mg	g/kg
compare						
	R(ISO20846:11)	1.304			Application range: 3 – 500 mg/	/kg
3 _T						0.9
					Δ	0.8 - Kernel Density
2.5 -						
					Δ Δ	0.7
2 -				Δ	<u>Δ</u> Δ Δ	0.6 -
				Δ		0.5 -
1.5		Δ Δ Δ	Δ Δ			
	Δ Δ Δ					0.4 -
1 + Δ Δ						0.3 -
0.5						0.2 -
0.5 +						

Determination of Water content by KF on sample #17065; results in mg/kg

lab	method	value	mark	z(targ)	remarks
62	D6304-A	300		-0.28	
120					
150	D6304-A	358		0.00	
171	D6304-A	469	R(0.05)	0.54	
312	ISO12937	408		0.24	
323	ISO12937	350		-0.04	
333					
335	ISO12937	367		0.04	
336					
337	D6304-A	300		-0.28	
338	ISO12937	330.71		-0.13	
511	D6304-A	475.04	R(0.05)	0.57	
529	D6304	442.91		0.41	
540	D6304-A	400		0.21	
556	D6304-A	355.6		-0.01	
558	D6304-A	326.43		-0.15	
604					
621					
631	D6304-B	19.7	R(0.01)	-1.65	
657	D6304-A	348		-0.05	
823					
824	D6304-A	359		0.01	
825	D6304-A	351		-0.03	
1033	IP438	402.49		0.22	
1047	ISO12937	360		0.01	
1067	ISO12937	360		0.01	
1134	IP438	360.67		0.01	
1161	ISO12937	354.541		-0.02	
1199	ISO12937	385		0.13	
1376	D6304-A	375.8		0.09	
1459	ISO12937	321		-0.18	
1494	E203	354.71		-0.01	
1510	IP438	340		-0.09	
1582 1634	ISO12937	370.0		0.06 -0.22	
1643	ISO12937 ISO6296	312.5 374		0.08	
1739	ISO12937	341		-0.08	
1739	E203	373		0.07	
1744	ISO12937	352.2050		-0.03	
6069	130 12931			-0.03	
0009					
	normality	suspect			
	n	30 30			
	outliers	3			
	mean (n)	357.79			
	st.dev. (n)	31.082			
	R(calc.)	87.03			
	R(D6304:16e1)	575.24			
	,	-			



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Determination of Water and sediment on sample #17065; results in %V/V

lab	method	value	mark	z(targ)	remarks
62	D2709	<0.05	Haik	2(tary)	TOTHIGHTO
120	D2108	<0.05 			
150					
171	D2709	<0.01			
312	D2109				
323	D2709	0.005			
333	D2109	0.005			
335					
336					
337					
338					
511					
529	D2709	0.005			
540	D2709	<0.05			
556	DZTOS				
558					
604					
621					
631	D2709	<0.01			
657	D2709	<0.01			
823	D2709	<0.05			
824	D2709	<0.01			
825	D2709	0			
1033	22.00				
1047					
1067					
1134					
1161					
1199					
1376	D2709	0			
1459					
1494					
1510					
1582					
1634					
1643					
1739					
1744					
1769					
6069					
	normality	n.a.			
	n	11			
	outliers	n.a.			
	mean (n)	< 0.05			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D2709:16)	n.a.			

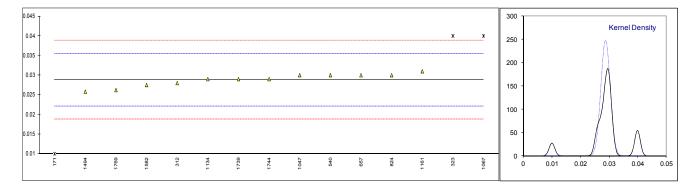
Determination of Distillation at 10 mm Hg, % recovered as AET on sample #17065; results in °C

62 120 150 150 151 171 D1160 353 353 353 333 335 335 336 337 338 338 338 339 338 339 339 339 339 339	lab	method	80% rec	mark z(targ)	90% rec	mark	z(targ)	95% rec	mark	z(targ)
120										
150										
171 D1160 353 355 358 312										
312		D1160	353		355			358		
223 333 336		255								
333 336 337 338 339 339 339 339 339 339 339 339 339										
335 336 337 338 339 338 339 339 339 339 339 339 339										
336 337 338 338 338 339 511 309 540 309 556 558 604 604 601 621 301 657 823 823 824 825 1033 1047 1067 1134 D1160 354 354 356 358 1199 1376 1494 1199 1376 1494 1199 1376 1494 1510 1582 1634 1643 1739 1744 1774 17744 1779 17744 1779 17744 1779 17744 1779 17744 1779 17744 1779 17744 1779 17744 17769 6069 100 100 100 100 100 100 100 100 100 1										
337 338 511 529 540 556 558 604 604 621 631 657 823 824 824 825 1033 1047 1067 1134 D1160 353.53 354.71 362.94 1199 1376 1459 1494 1510 1582 1634 1643 1739 1744 1774 1769 6069 normality unknown										
338 511 529 540 556 558 604 621 621 631										
511 529										
529										
540 556 558 604 604										
556 558 604 621										
558										
604 621 631 657										
621 631 657										
631										
657 823 824 824 1033 1047 1067 1134 D1160 354 1199 1376 1459 1494 1510 1582 1634 1643 1739 1739 1744 1769 6069 normality unknown	621									
657 823 824 824 1033 1047 1067 1134 D1160 354 1199 1376 1459 1510 1582 1634 1582 1634 1739 1739 1744 1769 6069 normality unknown	631									
823 824 825										
824 825 1047 1067 1134 D1160 354 356 358 1161 D1160 353.53 354.71 362.94 1199 1376 1459 1510 1582 1634 1643 1739 1769 6069 normality unknown unknown unknown										
825										
1033										
1047										
1067										
1134 D1160 354										
1161 D1160 353.53 354.71 362.94 1199 1376 1459 1510 1582 1634 1739 1744 1769 6069 normality unknown unknown unknown		D1160								
1199										
1376 1459		D1160								
1459 1494 1510 1582 1634 1643 1739 1744 1769 6069 normality unknown unknown unknown										
1494 1510 1582 1634 1643 1739 1744 1769 6069 normality unknown unknown unknown										
1510 1582										
1582 1634										
1634										
1643 1739 1744 1769	1582									
1739 1744 1769	1634									
1744	1643									
1769	1739									
6069 unknown unknown unknown	1744									
6069 unknown unknown unknown	1769									
normality unknown unknown unknown										
		normality	unknown		unknown			unknown		
II U I I I I I I I I I I I I I I I I I		•								
mean (n) (353.51) (355.24) (359.65)										
st.dev. (n) (0.500) (0.677) (2.852)			(0.500)							
R(calc.) (1.40) (1.89) (7.99)										
R(D1160:15) (4.64) (4.64)		K(D1160:15)	(4.64)		(4.64)			(4.64)		

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Determination of Methanol on sample #17065; results in %M/M

lab	method	value	mark	z(targ)	Remarks
62					
120					
150					
171	EN14110	0.01	G(0.01)	-5.62	
312	EN14110	0.028	- (/	-0.24	
323	EN14110	0.04	DG(0.01)	3.35	
333			- (/		
335					
336					
337					
338					
511					
529					
540	EN14110	0.03		0.36	
556					
558					
604					
621					
631					
657	EN14110	0.03		0.36	
823					
824	EN14110	0.03		0.36	
825					
1033					
1047	EN14110	0.03		0.36	
1067	EN14110	0.04	DG(0.01)	3.35	
1134	EN14110	0.029		0.06	
1161	EN14110	0.031		0.66	
1199					
1376					
1459	EN144440				
1494	EN14110	0.0258		-0.89	
1510	EN144440	0.0075		0.20	
1582 1634	EN14110	0.0275		-0.39 	
1643					
1739	EN14110	0.029		0.06	
1739	EN14110 EN14110	0.029		0.06	
1744	EN14110 EN14110	0.029		-0.78	
6069	LINI411U	0.0202		-0.76	
0009					
	normality	OK			
	n	12			
	outliers	3			
	mean (n)	0.0288			
	st.dev. (n)	0.00162			
	R(calc.)	0.0045			
	R(EN14110:03)	0.0094			
	(=)				



Determination of mono-Glycerides, total on sample #17065; results in %M/M

lab	method	valu	e	mark	z(targ)	remarks
62						
120 150						
171						
312	EN14105	0.25	5		-0.20	
323	EN14103	0.24			-0.41	
333 335	EN14105	0.32			0.69	
336						
337						
338						
511 529						
540	EN14105	0.31			0.55	
556	21114100					
558						
604						
621 631						
657	EN14105	0.38			1.51	
823						
824	D6584	0.25	6		-0.19	
825						
1033 1047	EN14105	0.27	2		0.03	
1067	EN14105	0.23			-0.47	
1134	EN14105	0.19	6		-1.01	
1161	EN14105	0.38		С	1.51	First reported 0.56
1199 1376						
1459						
1494	D6584	0.23	49		-0.48	
1510						
1582 1634						
1643						
1739	EN14105	0.23	29		-0.50	
1744	D6584	0.23			-0.48	
1769	D6584	0.22	80		-0.57	
6069						
	normality	susp	ect			
	n	14				
	outliers	0	00			
	mean (n) st.dev. (n)	0.269 0.050				
	R(calc.)	0.159				
	R(D6584:13)	0.20	41			Compare R(EN14105) = 0.1157
0.6 T						9]
						8 - Kernel Density
0.5						7 -
0.4						Δ Δ 6-
0.3			Δ Δ	Δ	Δ Δ	4-
0.2 - <u>A</u>	Δ Δ	Δ	Δ Δ	_		3 -
0.1 -						
0 7	95 93	4 .	\$ b	Ø	312	\$ \$ \$ \$ \$ \$ 0 0 0.1 0.2 0.3 0.4 0.5
261	1769	1744	1067	323	312	\$ \$ \frac{1}{2}\$ \$ \frac{1}{2}\$ \$ \frac{1}{2}\$ \$ \frac{1}{2}\$ \$ 0.1 \ 0.2 \ 0.3 \ 0.4 \ 0.5 \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \

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Determination of di-Glycerides, total on sample #17065; results in %M/M

lab	method	value	mark	z(targ)	remarks	
62				_(**** 9/		
120						
150						
171						
312	EN14105	0.056		-1.13		
323	EN14103	0.10		0.35		
333	EN14105	0.12		1.03		
335 336						
337						
338						
511						
529						
540	EN14105	0.08		-0.32		
556						
558						
604						
621 631						
657	EN14105	0.08		-0.32		
823	LIN 14 100	0.06		-0.32		
824	D6584	0.082		-0.25		
825						
1033						
1047	EN14105	0.097		0.25		
1067	EN14105	0.095		0.18		
1134	EN14105	0.071		-0.62		
1161	EN14105	0.11		0.69		
1199						
1376 1459						
1494	D6584	0.0836		-0.20		
1510	20001					
1582						
1634						
1643						
1739	EN14105	0.0885		-0.03		
1744	D6584	0.10503		0.52		
1769 6069	D6584	0.0853		-0.14		
0009						
	normality	OK				
	n	14				
	outliers	0				
	mean (n)	0.0895				
	st.dev. (n)	0.01651				
	R(calc.) R(D6584:13)	0.0462			0 0/51/4/405	
	R(D6584:13)	0.0831			Compare R(EN14105) = 0.0458	
0.2 T					30 7	
0.18					Kernel De	ensity
0.16					25 -	
0.14 -					20 -	
0.12					A	
0.1 -				. Δ	<u>A</u>	
0.08	Δ	Δ Δ	Δ Δ	Δ		
0.06	-				10 -	
0.04 -					5 -	
0.02 -						
0 0	4 F	δ 4	4 0	9 1	2 2 4 5 2 0	
312	1134	824	1494	1739	\$ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\	0.2

Determination of tri-Glycerides, total on sample #17065; results in %M/M

lab	method		value		mark	z(targ)	rema	rks					
62														
120 150														
171														
312	EN14105		0.010				-0.73							
323	EN14103		<0.10											
333	EN14105		0.03				-0.27							
335 336														
337														
338														
511														
529 540	EN14105		0.01											
556	LIVITIOS													
558														
604														
621														
631 657	EN14105		0.04				-0.05							
823														
824	D6584		0.050				0.18							
825														
1033 1047	EN14105		0.045				0.07							
1047	EN14105		0.043				0.41							
1134	EN14105		0.040				-0.05							
1161	EN14105		0.060				0.41							
1199														
1376 1459														
1494	D6584		0.0397				-0.05							
1510														
1582														
1634 1643														
1739	EN14105		0.05155	i			0.22							
1744	D6584		0.06890				0.62							
1769	D6584		0.0410				-0.02							
6069														
	normality		OK											
	n		13											
	outliers		0											
	mean (n)		0.0420											
	st.dev. (n) R(calc.)		0.01766 0.0494)										
	R(D6584:1	3)	0.1224					Com	pare R	(EN141	05) = 0	.0654		
	•	•								-	•			
0.2 T													30	
0.18 -														Kernel Density
0.16 +													25 -	\wedge
0.14 +													20 -	\bigwedge
0.12														// \
0.1													15 -	// \
0.08												Δ	10 -	// \
0.06							Δ	Δ	Δ	Δ	Δ] [$\mathcal{A} \setminus \mathcal{A} \setminus \mathcal{A}$
0.04		Δ	Δ Δ		-	-							5 -	// \
0	Δ												0	
312	940	333	1494		461	1769	1047	824	1739	1067	1161	1744	-0.05	0 0.05 0.1

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Determination of Free Glycerine on sample #17065; results in %M/M

lab	method	value	mark	z(targ)	remarks	
62						
120					•	
150						
171	EN14.440E					
312	EN14105	0.006		1.60		
323	EN14103	< 0.001		1.64		
333 335	EN14105	0.000		-1.64 		
336					•	
337					•	
338						
511						
529						
540	D6584	0.003		-0.02		
556					•	
558					•	
604					•	
621						
631 657	EN14105	0.004		0.52		
657 823	EN14105	0.004		0.52		
824	D6584	<0.005			•	
825	D0304				•	
1033						
1047	EN14105	0.003		-0.02		
1067	EN14105	< 0.010				
1134	EN14105	0.0031		0.04		
1161	EN14105	0.002		-0.56		
1199					•	
1376						
1459	D0504					
1494	D6584	0.0031		0.04		
1510 1582						
1634						
1643						
1739	EN14105	0.0043		0.68		
1744	D6584	0.0023	7	-0.36		
1769	D6584	0.0025		-0.29		
6069						
	normality	suspec	t			
	n	11				
	outliers	0				
	mean (n) st.dev. (n)	0.00303 0.00149				
	R(calc.)	0.0014				
	R(D6584:13)	0.00519			Compare R(EN14105) = 0.00666	
	11(10004.10)	0.0001			0.00000	
0.01					400 Kernel	Density
0.009 +					350 - Kemel	i Deligity
0.008 -					300 -	
0.007						
0.006 +					Δ 250	
0.005 -					200 -	
0.004 -					Δ Δ 150 - / / \	
0.003 -		Δ Δ	Δ	Δ Δ	 // \\	
0.002 -	Δ	Δ Δ			100 -	
0.001 -					50	
0	<u> </u>	4 0		k #		
8	1161	1744	92	1047	8	6 0.008 0.01

Determination of Total Glycerine on sample #17065; results in %M/M

lab	method		value		mark	z(tar	a)	remar	ks					
62														
120														
150														
171	EN4440E		0.070											
312 323	EN14105 EN14103		0.078 0.081			-0.6 -0.4								
333	EN14105		0.101			0.7								
335														
336														
337														
338 511														
529														
540	EN14105		0.09			0.0	8(
556														
558														
604 621														
631														
657	EN14105		0.115			1.5								
823														
824	D6584		0.086			-0.1								
825 1033														
1033	EN14105		0.087			-0.0								
1067	EN14105		0.080			-0.4								
1134	EN14105		0.0677			-1.1	18							
1161	EN14105		0.12		С	1.7		First re	ported	0.162				
1199 1376														
1459														
1494	D6584		0.0808			-0.4								
1510														
1582														
1634 1643														
1739	EN14105		0.0893			0.0								
1744	D6584		0.08603			-0.1								
1769	D6584		0.0780			-0.6	30							
6069														
	normality		suspect											
	n		14											
	outliers		0											
	mean (n)		0.0886											
	st.dev. (n)		0.01444											
	R(calc.) R(D6584:13)		0.0404 0.0494					Compa	ro D/F	-N1410	0.03	284		
	11(20004.10)		0.0404					Compe			,0, 0.0.	_0-		
0.16 _T													45	
													40 -	Kernel Density
0.14														\bigwedge
0.12 -											Δ	Δ	35 -	[]
0.1 -										Δ			30 -	14 11
0.08	Δ Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ					25 -	
0.06													20 -	
0.04 -													15 -	
													10 -	
0.02 +													5 -	
0 7	312	1067	1494	323	824	1744	1047	1739	940	333	657	1161	0 1	0.05 0.1 0.15
-	-	-	-			-	-	-				-		

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1376

Determination of Cetane Number on sample #17066

lab	method	value	mark	z(targ)	remarks	
120						
150						
171	D613	51.9		0.16		
323	D613	52.4		0.49		
657	D613	49.4		-1.46		
1067						
1107						
1134						
1161						
1376	D613	52.89		0.81		
	normality n outliers mean (n) st.dev. (n) R(calc.)	unknown 4 0 51.648 1.5519 4.345				
	R(D613:16a)	4.3				
57 —						
55 -	<u></u>					
53 -				Δ	Δ	

323

171

Determination of Derived Cetane Number (D7668) of sample #17066

lab	method	DCN	mark :	z(targ)	ID	mark	z(targ)	CD	mark	z(targ)	Wall Temp.
120											
150											
171											
323											
657											
1067											
1107	D7668	52.09			3.3330			4.5788			603.31
1134											
1161											
1376											
	normality	n.a.			n.a.			n.a.			
	n	n.a.			n.a.			n.a.			
	outliers	n.a.			n.a.			n.a.			
	mean (n)	n.a.			n.a.			n.a.			
	st.dev. (n)	n.a.			n.a.			n.a.			
	R(calc.)	n.a.			n.a.			n.a.			
	R(D7668:14a)	n.a.			n.a.			n.a.			

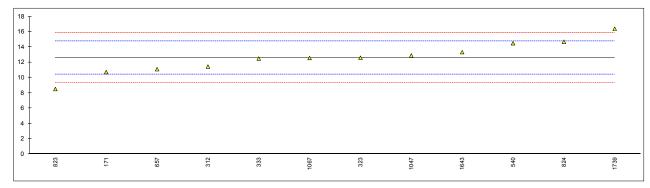
Determination of Derived Cetane Number (D6890) of sample #17066

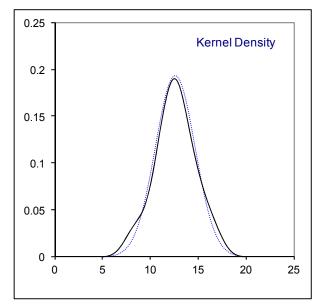
	-	•	 		<u> </u>			Wall
lab	method	DCN	mark z(targ)	ID	mark z(targ)	CD	mark z(targ)	Temp.
120								
150								
171								
323								
657								
1067	D6890	54.66		3.717				579.5
1107								
1134								
1161	ISO15195	55.32		3.669				585.1
1376								
	normality	n.a.		n.a.		n.a.		
	n	2		2		n.a.		
	outliers	n.a.		n.a.		n.a.		
	mean (n)	54.99		3.693		n.a.		
	st.dev. (n)	n.a.		n.a.		n.a.		
	R(calc.)	n.a.		n.a.		n.a.		
	R(D6890:16)	n.a.		n.a.		n.a.		

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Determination of sum of Calcium and Magnesium (as Ca + Mg) on sample #17067; results in mg/kg

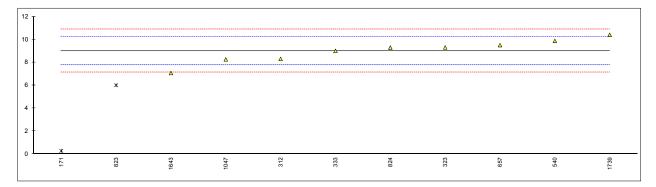
lab	method	value	mark	z(targ)	remarks
120					
150					
171	EN14538	10.72		-1.72	
312	EN14538	11.45		-1.06	
323	EN14538	12.6		-0.01	
333	EN14538	12.5		-0.10	
391					
540	EN14538	14.50		1.73	
657	EN14538	11.1		-1.38	
823	EN14538	8.5		-3.75	
824	EN14538	14.7		1.91	
1047	EN14538	12.895		0.26	
1067	EN14538	12.57		-0.03	
1134					
1643	D5185	13.3360		0.67	
1739	EN14538	16.4		3.47	
	normality	OK			
	n	12			
	outliers	0			
	mean (n)	12.606			
	st.dev. (n)	2.0674			
	R(calc.)	5.789			
	R(EN14538:06)	3.064			
	,				

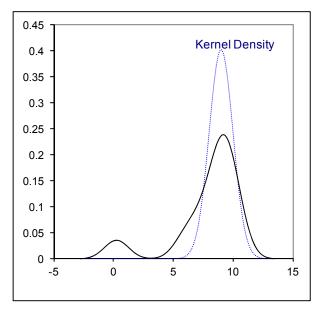




Determination of Phosphorus as P on sample #17067; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120					
150	D40E1	0.272	C(0.01)	12.04	
171 312	D4951 EN14107	0.272 8.3	G(0.01)	-13.94 -1.12	
323	EN14107	9.3		0.48	
333	EN14107	9.0		0.00	
391					
540	EN14107	9.87		1.39	
657 823	EN14107 D4951	9.5 6	G(0.05)	0.80 -4.79	
824	D4951 D4951	9.29	G(0.05)	0.47	
1047	EN14107	8.259		-1.18	
1067	EN14107	< 4.0			
1134	DE405	7.000			
1643 1739	D5185 EN14107	7.066 10.4		-3.09 2.24	
1739	EN 14 107	10.4		2.24	
	normality	OK			
	n	9			
	outliers	2	Spike:		D
	mean (n) st.dev. (n)	8.998 0.9950	8.99		Recovery <100%
	R(calc.)	2.786			
	R(EN14107:03)	1.753			





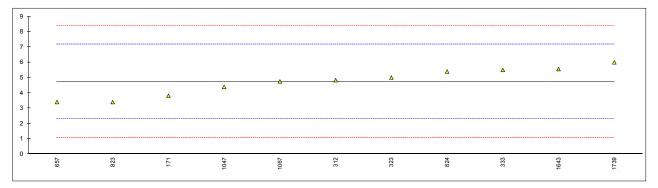
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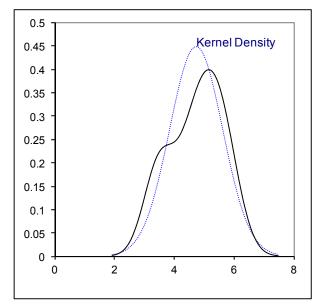
Determination of Potassium as K on sample #17067; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120					
150					
171	EN14538	1.46			
312	EN14538	<1			
323	EN14538	<1.0			
333	EN14538	<1.0			
391					
540					
657	EN14109	<0.5			
823	EN14538	0.1			
824	EN14538	<1			
1047	EN14538	<0,050			
1067	EN14538	< 1.0			
1134					
1643					
1739	EN14538	0.1			
	normality	n.a.			
	n	9			
	outliers	n.a.			
	mean (n)	<1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN14214:14)	n.a.			

Determination of Sodium as Na on sample #17067; results in mg/kg

lab	method	value	mark -		remarks
120			-		
150			-		
171	EN14538	3.81	-0	.75	
312	EN14538	4.835	0	.08	
323	EN14538	5.0	0	.22	
333	EN14538	5.5	0	.63	
391			-		
540			-		
657	EN14108	3.4	-1	.09	
823	EN14538	3.4		.09	
824	EN14538	5.4	0	.55	
1047	EN14538	4.401		.27	
1067	EN14538	4.75	0	.01	
1134					
1643	D5185	5.557	0	.67	
1739	EN14538	6.0	1	.04	
	normality	OK			
	n	11			
	outliers	0	<u>Spike</u>		
	mean (n)	4.732	5.99		Recovery <79%
	st.dev. (n)	0.8897	5.55		recovery 47070
	R(calc.)	2.491			
	R(EN14214:14)	3.423			Compare R(EN14108:03) = 2.600
	11(LIV17214.14)	0.420			Compare Main 14 100.00) - 2.000

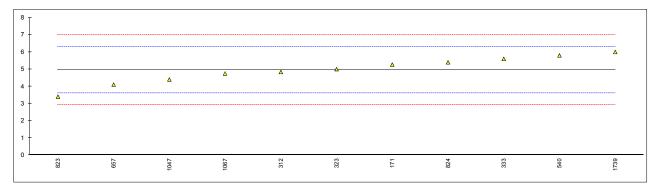


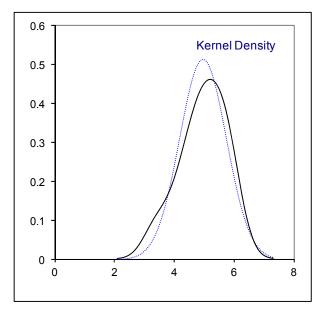


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Determination of sum of Potassium and Sodium (as K + Na) on sample #17067; results in mg/kg

lab	method	value	mark		remarks
120	•				
150					
171	EN14538	5.27		0.46	
312	EN14538	4.835		-0.18	
323	EN14538	5.0		0.06	
333	EN14538	5.6		0.95	
391					
540	EN14538	5.8		1.25	
657	EN14538	4.1		-1.27	
823	EN14538	3.4	E	-2.31	Calculation error?
824	EN14538	5.4		0.65	
1047	EN14538	4.401		-0.83	
1067	EN14538	4.75		-0.31	
1134					
1643					
1739	EN14538	6.0	E	1.54	Calculation error?
	normality	OK			
	n	11			
	outliers	0			
	mean (n)	4.960			
	st.dev. (n)	0.7768			
	R(calc.)	2.175			
	R(EN14538:06)	1.888			





Determination of Particulate Contamination on sample #17068; results in mg/L

lab	method	value	mark	z(targ)	Vol. filtered (ml)	No. of filtrations	remarks
150	D7321	27.7	С	0.86	400	1	First reported 43.75
171	D7321	20.0		-2.06	400	1	
312	D7321	24.9		-0.20	400		
323	D7321	29.1		1.40	380		
335							
337							
540							
657							
823							
1033							
1067							
1134							
1161							
1582							
1739							
1744							
	normality	unknown					
	n	4					
	outliers	0					
	mean (n)	25.425					
	st.dev. (n)	4.0161					
	R(calc.)	11.245					
	R(D7321:14)	7.374					
	,						
35 T							
20							

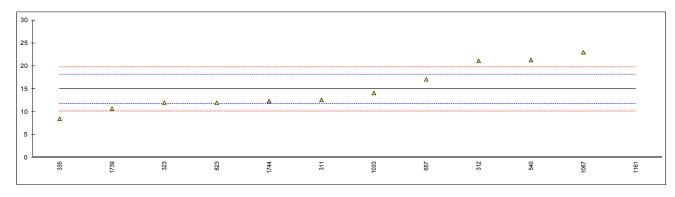


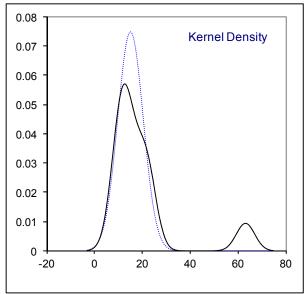
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Determination of Total Contamination (EN12662) on sample #17068; results in mg/kg

lab	method	value	mark	z(targ)	Vol. filtered (ml)	stopped after	remarks
150							
171	EN12662:2008	<6.0			220		False negative test result?
311	EN12662:2008	12.6		-1.48			
312	EN12662:1998	21.1		3.81			
323	EN12662:1998	12.0		-1.86	350	4	
335	EN12662:1998	8.5		-4.04			
337							
540	EN12662:1998	21.3		3.94	400	25	
657	EN12662:2014	17.1		1.32	300	1.0	see note below
823	EN12662:1998	12		-1.86	300		
1033	EN12662:2008	14.1		-0.55	298		
1067	EN12662:1998	23		5.00	355	2	
1134							
1161	EN12662:2014	63.1	G(0.01)	29.98			see note below
1582							
1739	EN12662:1998	10.74		-2.64			
1744	EN12662:2008	12.345		-1.64	800		
	normality	OK					
	n	11					
	outliers	1					
	mean (n)	14.98					
	st.dev. (n)	4.872					
	R(calc.)	13.64					
	R(EN12662:98)	4.49					
	,						

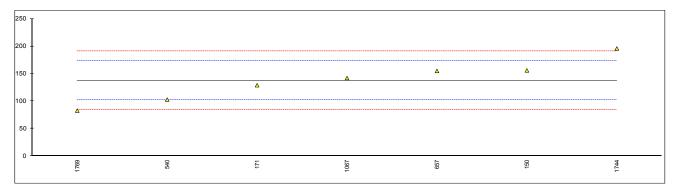
EN12662:2014 may not applicable to FAME (B100) according to CEN/TC 19; instead either method EN12662:1998 or EN12662:2008 is recommended to be used.





Determination of Filter Blocking Potential by Cold Soak Filterability Test (CSFT) on sample #17069; results in s

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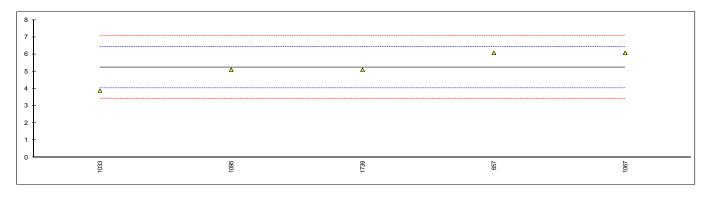


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Determination of Filter Blocking Tendency (FBT) on sample #17069

lab	method	value	mark	z(targ)	press. end test (kPa)	vol. pumped (ml)	Remarks
120							
150							
171							
323							
336							
540							
657	D2068-C	6.08		1.37		50	
823							
1033	IP387-B	3.88		-2.26	105		
1067	IP387-B	6.08		1.37	105	50	
1095	D2068-B	5.10		-0.24	105	60	
1134							
1739	IP387-B	5.10		-0.24	105	60	
1744							
1769							
6069							
	normality	unknown					
	n	5					
	outliers	0					
	mean (n)	5.248					
	st.dev. (n)	0.9083					
	R(calc.)	2.543					
	R(D2068:14)	1.697					

Lab 1033: also reported 1.20 (IP-PM-EA) as an FBT result



APPENDIX 2

Number of participants per country

- 1 lab in ARGENTINA
- 2 labs in BELGIUM
- 2 labs in BRAZIL
- 1 lab in CANADA
- 5 labs in COLOMBIA
- 6 labs in FRANCE
- 1 lab in HONG KONG
- 1 lab in INDONESIA
- 1 lab in ITALY
- 1 lab in MALAYSIA
- 1 lab in MEXICO
- 3 labs in NETHERLANDS
- 1 lab in PERU
- 1 lab in PHILIPPINES
- 1 lab in POLAND
- 3 labs in PORTUGAL
- 1 lab in SINGAPORE
- 3 labs in SOUTH KOREA
- 1 lab in SWEDEN
- 1 lab in TURKEY
- 3 labs in UNITED KINGDOM
- 3 labs in UNITED STATES OF AMERICA
- 1 lab in URUGUAY

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APPENDIX 3

Abbreviations:

C = final test result after checking of first reported suspect test result

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \\ DG(0.05) &= \text{straggler in Double Grubbs' outlier test} \\ \end{array}$

R(0.01) = outlier in Rosner's outlier test R(0.05) = straggler in Rosner's outlier test

ex = test result excluded from the statistical evaluation

E = probably an error in calculations

U = test result probably reported in different unit

n.a. = not applicablen.d. = not determinedfr. = first reported

W = test result withdrawn on request of the participant

n.e. = not evaluatedSDS = Safety Data Sheet

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
- 2 ASTM E178:02
- 3 ASTM E1301:03
- 4 ISO 5725:86
- 5 ISO 5725, parts 1-6, 1994
- 6 ISO 13528:05
- 7 M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367:84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 13 Analytical Methods Committee Technical Brief, No4 January 2001
- The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson.
- Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)