Results of Proficiency Test
Biodiesel 100% FAME (B100)
May 2018

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

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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. Since 2008 two PTs are organised for Biodiesel 100% FAME (B100). In the annual proficiency testing program of 2017/2018, it was decided to continue with the proficiency tests on Biodiesel B100 in accordance with the latest applicable version of ASTM D6751 and EN14214:2012 + A1:2014/AC:2014.
In this interlaboratory study in total 39 laboratories from 25 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 2018 Biodiesel B100 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 four different samples of Biodiesel B100, see table below.

Samples	Amount in L	Purpose	Spiked
#18065	1.5 L	For regular analysis	-
#18067	0.1 L	Analysis of metals	Sodium, Phosphorus, Potassium
#18068	1 L	Total Contamination	Arizona dust (fine)
#18069	0.5L	Cold Soak Test / Filter Blocking	-

Table 1: four different Biodiesel B100 samples used in iis18G02

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 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). This PT falls 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.

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 of approx. 400L of Biodiesel B100 (RME) was obtained from an European producer.

Biodiesel B100 #18065 - regular sample and Cold Soak Test / Filter Blocking Tendency sample #18069

After fit-for-use testing and homogenisation, 138 amber glass bottles of 1L and 138 amber glass bottles of 0.5L for the main round were filled and both labelled #18065. From the remaining batch used for the regular sample, 20 litres were used to fill 25 bottles of 0.5 litre amber glass bottles and labelled #18069. The homogeneity of the subsamples #18065 and #18069 was checked by the determination of Density in accordance with ISO 12185 on 8 stratified randomly selected samples:

	Density at 15°C in kg/m^3
sample -1	883.47
sample -2	883.46
sample -3	883.46
sample -4	883.46
sample -5	883.46
sample -6	883.46
sample -7	883.46
sample -8	883.46

Table 2: homogeneity test results of subsamples #18065 and #18069

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

Table 3: evaluation of the repeatability of subsamples #18065 and #18069

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the target method. Therefore, the homogeneity of the subsamples of #18065 and #18069 was assumed.

Biodiesel B100 #18067 - Metals

For metals in Biodiesel B100 a batch of approx. 3.2 kg was separated from the large batch and was spiked with Phosphorus (approx. 10 mg/kg), Sodium (approx. 10 mg/kg) and Potassium (approx. 10 mg/kg). After homogenisation, out of the batch 36 HDPE bottles of 0.1L were filled and labelled #18067.

The homogeneity of the subsamples of #18067 was checked by determination of Phosphorus and Sodium on 8 stratified randomly selected samples:

	Phosphorus in mg/kg	Sodium in mg/kg
sample #18067-1	9.1	6.9
sample #18067-2	9.3	7.0
sample #18067-3	9.2	7.1
sample #18067-4	9.4	7.0
sample #18067-5	9.4	6.9
sample #18067-6	9.3	6.9
sample #18067-7	9.1	7.0
sample #18067-8	9.2	6.9

Table 4: homogeneity test results of subsamples #18067

	Phosphorus in mg/kg	Sodium in mg/kg
r (observed)	0.33	0.21
reference test method	EN14107:03	EN14108:03
0.3 * R test method	0.54	0.96

Table 5: evaluation of repeatability of subsamples #18067

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, the homogeneity of the subsamples #18067 was assumed.

Biodiesel B100 #18068 – Total Contamination

Into 26 amber glass bottles, 1 ml of a freshly prepared and ultrasonically homogenized 18 g/kg Arizona Dust (fine) 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. The bottles were labelled #18068.

Depending on the registration of the participant, one 1 litre bottle and 0.5 litre bottle both labelled #18065, a 100mL bottle labelled #18067, a 1 litre bottle labelled #18068 and/or a 0.5 litre bottle labelled #18069, were dispatched to each of the participating laboratories on April 11, 2018. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of the Biodiesel B100, packed in the brown glass bottles and PE bottles, was checked. The material was 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 EN14214:12+A1:14 and/or ASTM D6751:15ce1.

Parameter	EN14214:12	Parameter	ASTM D6751:15ce1
Acid Value	EN14104	Acid Number	ASTM D664
Calorific Value	DIN51900		
		Carbon Residue on 100% FAME	ASTM D4530
CFPP	EN116		
Cloud Point	EN23015	Cloud Point	ASTM D2500
Copper Strip Corrosion	ISO2160	Copper Strip Corrosion	ASTM D130
Density at 15°C	ISO12185		
		Distillation	ASTM D1160
Flash Point (Recc)	ISO3679		
Flash Point (PMcc)	ISO2719	Flash Point	ASTM D93
Iodine Value	EN14111		
Kin. Visc. At 40°C	ISO3104	Kin. Visc. at 40°C	ASTM D445
Oxidation Stability	EN14112	Oxidation Stability	EN15751
Sulphated Ash	ISO3987	Sulphated Ash	ASTM D874
Sulphur	ISO20846	Sulphur	ASTM D5453
Water	ISO12937	Water and Sediment	ASTM D2709
Cetane Number	EN 5165	Cetane Number	ASTM D613
		Derived Cetane Number	ASTM D7668
Calcium + Magnesium	EN14538	Calcium + Magnesium	EN14538
Phosphorus	EN14107	Phosphorus	ASTM D4951
Potassium + Sodium	EN14108/14109	Potassium + Sodium	EN14538
Polyunsaturated esters	EN15779		
Methanol	EN14110	Methanol	EN14110
mono-, di-, tri-Glycerides	EN14105	Monoglyceride content	ASTM D6584
Free + Total Glycerol	EN14105	Free + Total Glycerol	ASTM D6584
Total ester content	EN14103		
Linolenic Acid	EN14103		
Total Contamination	EN12662		

Table 6: requirements and test methods acc. to specifications EN14214:12+A1:14 and/or ASTM D6751:15ce1.

It was explicitly requested to treat the samples as if they were routine samples and 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 report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

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 reanalyses). Additional or corrected test results are used for data analysis and original results are placed under 'Remarks' in the 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 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’ (iis-protocol, March 2017 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 judgment 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.

According to ISO 5725 the original test results per determination were submitted subsequently to Dixon’s and/or 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 the averages and the standard deviations.
For each assigned value, the uncertainty was determined in accordance with ISO 13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO 13528. In this PT, the criterion of ISO 13528, paragraph 9.2.1 was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need to be included in the PT report.

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 standard. Outliers and other data, which were excluded from the calculations, are represented as a “x”. Accepted data are represented as a triangle.
Furthermore, Kernel Density Graphs were made. The Kernel Density Graph 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 ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in 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 iis 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.

The z-scores were calculated in accordance with:

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores maybe as follows:

	$\lvert z \rvert$	< 1	good
1 <	$\lvert z \rvert$	< 2	satisfactory
2 <	$\lvert z \rvert$	< 3	questionable
3 <	$\lvert z \rvert$		unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered during the execution.
For the regular Biodiesel PT: all participants reported test results, however two participants reported test results after the final reporting date.
For the Metals in Biodiesel PT: one participant did not report any test results at all and one reported after the final reporting date.
For the Total Contamination PT: four participants did not report any test results at all and one reported after the final reporting date.
For Cold Soak Test/Filter Blocking Tendency PT: two participants did not report any test results at all one reported after the final reporting date.

Finally, in total 39 participants reported in total 563 numerical results. Observed were 22 outlying results, which is 3.9%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER SAMPLE AND PER TEST

In this section, the results are discussed per sample and per test. The test methods, which were used by the 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.

For Biodiesel B100 sample #18065

Acid Value (EN): This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN14104:03 and EN14214:12+A1:14

Acid Number (ASTM): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D664:17a (method B).

Cloud Point: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D2500:17a and EN14214:12+A1:14.

CFPP: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN116:15.

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

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

Density at 15°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ISO12185:96.

Flash Point PMcc: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D93-C:16a and ISO2719C:16.

Flash Point recc: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO3679:15.

Iodine Number: 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 problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ISO3104:94 and ASTM D7042:16e3 but in agreement with the requirements of ASTM D445:17.

Oxidation Stability: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN15751:14.

Pour Point: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO3016:94.

Sulphated Ash: All reported test results were near or below the application limit of ASTM D874-13a (2018)(0.005% M/M). Therefore, no significant conclusions were drawn.

Sulphur: This determination was not problematic. Two statistical outliers were observed and one other test excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ISO20846:11.

Water: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO12937:00.

Water and sediment: All reported test results were near or below the application limit of ASTM D2709:16 (0.05% V/V). Therefore, no significant conclusions were drawn.

Calorific Value: Only three participants submitted a result for Gross Calorific Value at constant volume, two participants for Net Calorific Value at constant volume and one participant for Net Calorific Value at constant pressure.
The determination on Gross Calorific Value was not problematic. The calculated reproducibility is in agreement with the requirements of DIN51900-1:00.

Distillation at 10mm Hg: This determination was not problematic for 80% recovered but problematic for 90% recovered and 95% recovered. No statistical outliers were observed. The calculated reproducibilities are in agreement with the requirements of ASTM D1160:15 for 80% recovered but not for 90% recovered and 95% recovered.

Methanol: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN14110:03.

mono-Glycerides This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN14105:11 and ASTM D6584:13.

di-Glycerides: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN14105:11 and ASTM D6584:13.

tri-Glycerides: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN14105:11 and ASTM D6584:13.

Free Glycerol: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN14105:11 and ASTM D6584:13.

Total Glycerol: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN14105:11 and ASTM D6584:13.

Total Ester content: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN14103:11.

Linolenic Acid Methyl Ester: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN14103:11.

Polyunsaturated Methyl Esters: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN15779:09+A1:13.

For Biodiesel B100 sample #18067 – Metals

Sum Ca + Mg: 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 EN141538:06.

Phosphorus: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN14107:03.
The samples were spiked with Phosphorus. The average recovery of Phosphorus (theoretical increment of 10 mg/kg) may be good: “less than 89%”. The actual blank concentration for Phosphorus is unknown.

Potassium: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN14109:03.
The samples were spiked with Potassium. The average recovery of Potassium (theoretical increment of 10 mg/kg) may be good: “less than 83%”. The actual blank concentration for Potassium is unknown.

Sodium: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN14108:03.
The samples were spiked with Sodium. The average recovery of Sodium (theoretical increment of 10 mg/kg) may be good: “less than 77%”. The actual blank concentration for Sodium is unknown.

Sum K + Na: This determination was problematic. Two statistical outliers were observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN14538:06.

For Biodiesel B100 sample #18068 – Particulate and Total Contamination

Some years ago there was some discussion about method EN12662 version 2014 for determining Total Contamination in Biodiesel (neat FAME or B100). The CEN/TC 19 working group published a letter in September 2015 (see lit. 17) about this issue. In short, for FAME blends (B100) either EN12662:1998 or EN12662:2008 should be used and not EN12662:14. Only one laboratory reported to use the 2014 version. Therefore, the test result of this laboratory was excluded from the statistical evaluation.

Particulate Contamination: Only three laboratories reported test results, therefore no significant conclusions were drawn.

Total Contamination: This determination was very problematic. No statistical outliers were observed and one test result was excluded. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of EN12662:98 and 08. The test results vary over a large range: <7.4 – 30.5 mg/kg.

For Biodiesel B100 sample #18069: CSFT&FBT

Filter Blocking Potential by Cold Soak test: This determination was very problematic. One statistical outlier was observed and one test result was excluded. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ASTM D7501:12a.

Filter Blocking Tendency: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D2068:17. The low number of reported test results may partly explain the large variation.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R (lit)
Acid Value (EN)	mg KOH/g	12	0.42	0.09	0.06
Acid Number (ASTM)	mg KOH/g	20	0.40	0.11	0.12
Cloud Point	°C	28	-6.3	3.3	5
Cold Filter Plugging Point	°C	23	-14.7	2.3	3
Carbon Residue (100% FAME)	%M/M	16	<0.1	n.a.	n.a.
Copper Strip Corrosion		21	1 (1A)	n.a.	n.a.
Density at 15°C	kg/m^3	35	883.5	0.3	0.5
Flash Point - PMcc	°C	19	156.1	18.8	14.7
Flash Point - recc (ISO3679)	°C	7	173.1	8.0	15
Iodine Value	g I_2/100g	20	114.8	6.1	5
Kinematic Viscosity at 40°C	mm^2/s	29	4.441	0.049	0.045
Oxidation Stability (EN15751)	hours	17	4.6	0.5	1.2
Pour Point	°C	19	-37.5	5.3	6.0
Sulphated Ash	%M/M	16	<0.005	n.a.	n.a.
Sulphur	mg/kg	17	1.5	0.9	1.3
Water	mg/kg	30	364	110	131
Water and sediment	%V/V	13	<0.05	n.a.	n.a.
Calorific Value, Gross	kJ/kg	3	39844	270	400
Distillation at 10mm Hg					
80% recovered, as AET	°C	8	353.1	3.8	4.6
90% recovered, as AET	°C	8	355.4	6.3	4.6
95% recovered, as AET	°C	8	359.4	9.6	4.6
Methanol	%M/M	17	0.038	0.017	0.011
mono-Glycerides	%M/M	15	0.324	0.087	0.126
di-Glycerides	%M/M	16	0.114	0.044	0.050
tri-Glycerides	%M/M	15	0.062	0.029	0.072
Free Glycerol	%M/M	13	0.002	0.004	0.007
Total Glycerol	%M/M	15	0.109	0.025	0.032
Total Ester Content	%M/M	20	97.9	2.9	4.2
Linolenic Acid Methyl Ester	%M/M	14	10.10	0.40	0.68
Polyunsat. Methyl Esters	%M/M	5	0.22	0.58	0.27

Table 7: comparison of the observed and target reproducibilities of Biodiesel B100 sample #18065

Parameter	unit	n	average	2.8 * sd	R (lit)
Sum Calcium & Magnesium	mg/kg	13	14.3	5.2	3.3
Phosphorus	mg/kg	12	8.9	2.9	1.7
Potassium	mg/kg	12	8.5	3.2	4.8
Sodium	mg/kg	13	7.9	4.0	3.4
Sum Potassium & Sodium	mg/kg	12	16.6	6.2	4.1

Table 8: comparison of the observed and target reproducibilities of Biodiesel B100 sample #18067

Parameter	unit	n	average	2.8 * sd	R (lit)
Particulate Contamination (D7321)	mg/L	3	n.a.	n.a.	n.a.
Total Contamination (EN12662)	mg/kg	13	18.2	20.4	7.1

Table 9: comparison of the observed and target reproducibilities of Biodiesel B100 sample #18068

Parameter	unit	n	average	2.8 * sd	R (lit)
Filter Blocking Potential by CST	s	13	123	118	42
Filter Blocking Tendency		5	5.8	8.3	2.1

Table 10: comparison of the observed and target reproducibilities of Biodiesel B100 sample #18069

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 reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2018 WITH PREVIOUS PTS

	May 2018	Oct 2017	May 2017	Oct 2016	May 2016
Type of FAME	Rapeseed	Offal-ME	Rapeseed	Rapeseed	Rapeseed
Number of reporting labs	39	70	38	79	54
Number of results reported	563	1054	449	1369	596
Number of statistical outliers	22	24	11	41	25
Percentage statistical outliers	3.9%	2.3%	2.5%	3.0%	4.2%

Table 11: 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 reference test methods. The conclusions are given the following table:

Parameter	May 2018	Oct 2017	May 2017	Oct 2016	May 2016
Acid Value (EN)	-	+/-	n.e.	+	n.e.
Acid Number (ASTM)	+	+	+/-	+	+
Cloud Point	+	++	+/-	+/-	--
Cold Filter Plugging Point	+	-	+	+/-	-
Carbon Residue (100% FAME)	n.e.	n.e.	n.e.	n.e.	n.e.
Density at 15°C	+	++	+	++	++
Flash Point - PMcc	-	-	+/-	+	+/-
Flash Point - recc.	++	++	n.e.	+/-	n.e.
Iodine Value	-	-	-	+/-	+/-

Parameter	May 2018	Oct 2017	May 2017	Oct 2016	May 2016
Kinematic Viscosity at 40°C	-	-	++	+	++
Oxidation Stability (EN15751)	++	--	+	+	+
Pour Point	+	+	n.e.	+	n.e.
Sulphated Ash	n.e.	n.e.	n.e.	n.e.	n.e.
Sulphur	+	+/-	-	+	+
Water	+	+	++	+	++
Calorific Value, Gross	+	+	n.e.	-	n.e.
Distillation at 10mm Hg	-	n.e.	(+)	n.e.	-
Methanol	-	n.e.	++	+/-	-
mono-Glycerides	+	n.e.	++	+/-	++
di-Glycerides	+	n.e.	++	+	++
tri-Glycerides	++	n.e.	++	+	++
Free Glycerol	+	+	+	+	++
Total Glycerol	+	--	+	+/-	+
Total Ester Content	+	-	n.e.	+	n.e.
Linolenic Acid Methyl Ester	+	+	n.e.	+	n.e.
Polyunsaturated Methyl Esters	-	+/-	n.e.	n.e.	n.e.
Sum of Calcium and Magnesium	-	--	-	-	+/-
Phosphorus	-	-	-	--	--
Potassium	+	-	n.e.	n.e.	++
Sodium	-	+	+	+	+
Sum of Potassium and Sodium	-	-	-	-	--
Particle Contamination (D7321)	n.e.	n.e.	--	n.e.	n.e.
Total Contamination ((EN12662)	--	--	--	--	--
Filter Blocking Potential by CST	--	n.e.	--	n.e.	+/-
Filter Blocking Tendency	--	n.e.	-	n.e.	-

Table 14: comparison of group performances against the standard requirements of all samples
* 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

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

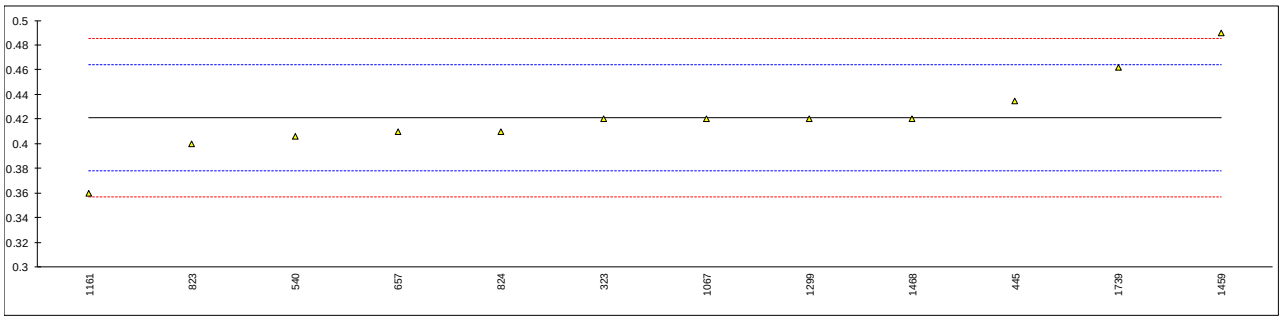
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test method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

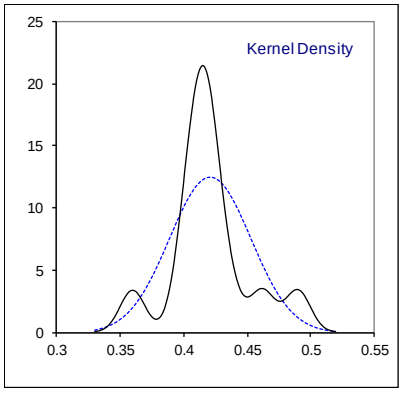
APPENDIX 1

Determination of Acid Value on sample #18065; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312		-----		-----	
323	EN14104	0.42		-0.05	
335		-----		-----	
336		-----		-----	
445	EN14104	0.435		0.65	
494		-----		-----	
511		-----		-----	
529		-----		-----	
540	EN14104	0.406		-0.70	
558		-----		-----	
631		-----		-----	
657	EN14104	0.41		-0.52	
823	EN14104	0.40		-0.98	
824	EN14104	0.41		-0.52	
1033		-----		-----	
1067	EN14104	0.42		-0.05	
1161	EN14104	0.36		-2.85	
1199		-----		-----	
1299	EN14104	0.42		-0.05	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459	EN14104	0.49		3.22	
1468	EN14104	0.42		-0.05	
1494		-----		-----	
1539		-----		-----	
1643		-----		-----	
1739	EN14104	0.462		1.91	
1744		-----		-----	
1769		-----		-----	
6054		-----		-----	
6069		-----		-----	
6179		-----		-----	

normality	suspect	
n	12	
outliers	0	
mean (n)	0.4211	
st.dev. (n)	0.03201	
R(calc.)	0.0896	
st.dev.(EN14104:03)	0.02143	
R(EN14104:03)	0.06	Compare R(EN14214:12+A1:14)= 0.06

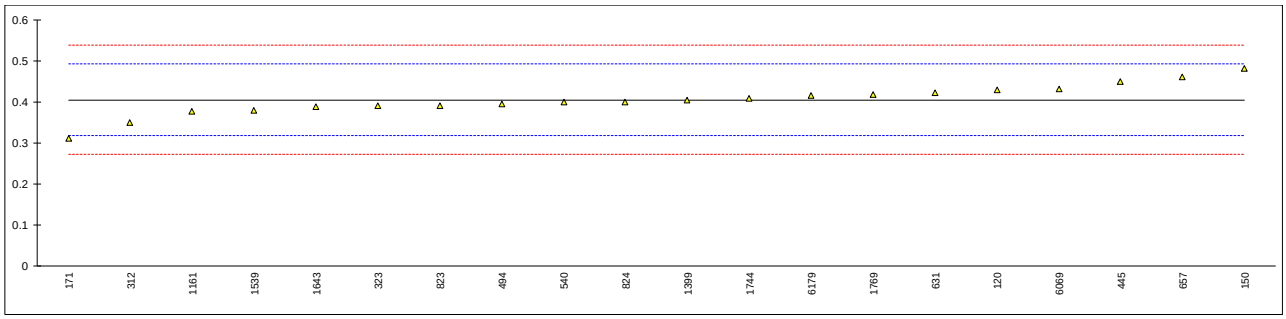


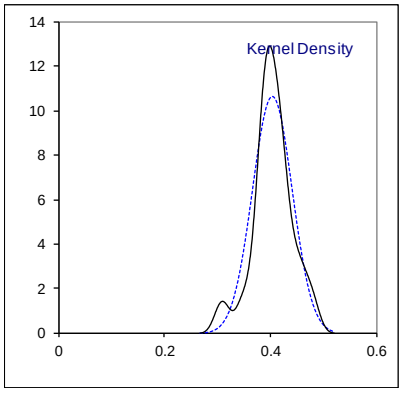


Determination of Acid Number, total on sample #18065; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
120	D664-B	0.429		0.55	
150	D664-B	0.48		1.71	
171	D664-B	0.31		-2.15	
240		-----		-----	
312	D974	0.35		-1.25	
323	D664-B	0.39		-0.34	
335		-----		-----	
336		-----		-----	
445	D664-B	0.449		1.00	
494	D664-B	0.395		-0.22	
511		-----		-----	
529		-----		-----	
540	D664-B	0.400		-0.11	
558		-----		-----	
631	D974	0.4218		0.38	
657	D664-B	0.46		1.25	
823	D664-B	0.39		-0.34	
824	D664-B	0.40		-0.11	
1033		-----		-----	
1067		-----		-----	
1161	D664-B	0.376		-0.66	
1199		-----		-----	
1299		-----		-----	
1399	D664-B	0.4050		0.00	
1406		-----		-----	
1407		-----		-----	
1459		-----		-----	
1468		-----		-----	
1494		-----		-----	
1539	ISO6618	0.38		-0.56	
1643	D664-B	0.389		-0.36	
1739		-----		-----	
1744	D664-B	0.408		0.07	
1769	D664-B	0.41765		0.29	
6054		-----		-----	
6069	D664-B	0.4309		0.59	
6179	D664-B	0.416		0.25	

normality	suspect
n	20
outliers	0
mean (n)	0.4049
st.dev. (n)	0.03754
R(calc.)	0.1051
st.dev.(D664-B:17a)	0.04403
R(D664-B:17a)	0.1233



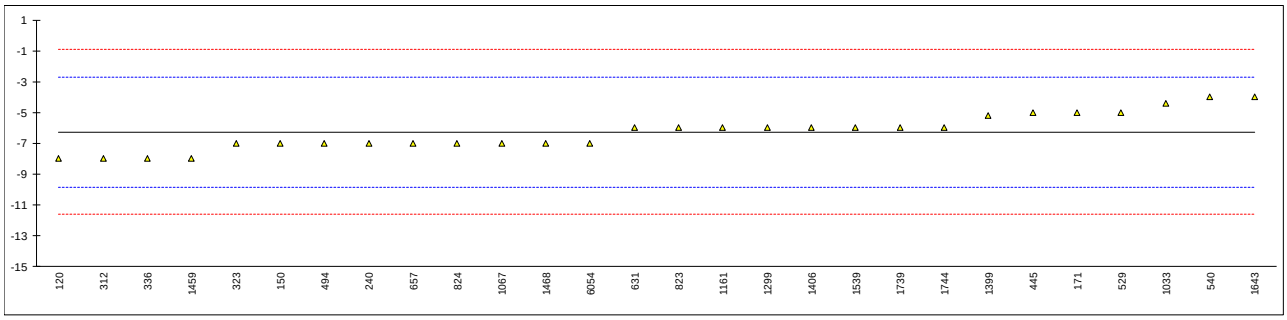


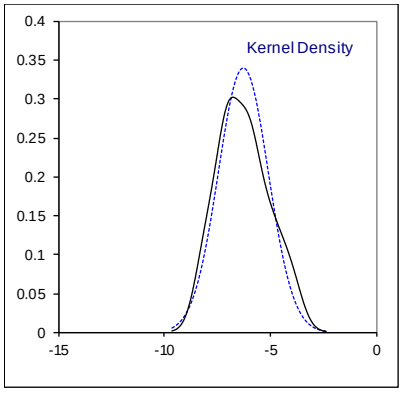
Determination of Cloud Point on sample #18065; results in °C

lab	method	value	mark	z(targ)	remarks
120	D2500	-8		-0.97	
150	D5771	-7		-0.41	
171	D2500	-5		0.71	
240	D2500	-7		-0.41	
312	D2500	-8		-0.97	
323	D2500	-7		-0.41	
335		-----		-----	
336	D2500	-8		-0.97	
445	D2500	-5		0.71	
494	ISO3015	-7		-0.41	
511		-----		-----	
529	D2500	-5		0.71	
540	D2500	-4.0		1.27	
558		-----		-----	
631	D2500	-6		0.15	
657	D2500	-7		-0.41	
823	D2500	-6		0.15	
824	D2500	-7		-0.41	
1033	D7689	-4.4		1.05	
1067	ISO3015	-7		-0.41	
1161	EN23015	-6		0.15	
1199		-----		-----	
1299	D2500	-6		0.15	
1399	D5773	-5.2		0.60	
1406	ISO3015	-6		0.15	
1407		-----		-----	
1459	EN23015	-8.0		-0.97	
1468	EN23015	-7		-0.41	
1494		-----		-----	
1539	ISO3015	-6		0.15	
1643	D2500	-4		1.27	
1739	EN23015	-6		0.15	
1744	D2500	-6		0.15	
1769		-----		-----	
6054	D2500	-7.0		-0.41	
6069		-----		-----	
6179		-----		-----	

normality	OK
n	28
outliers	0
mean (n)	-6.27
st.dev. (n)	1.173
R(calc.)	3.28
st.dev.(D2500:17a)	1.786
R(D2500:17a)	5

Compare R(EN14214:12+A1:14)= 4

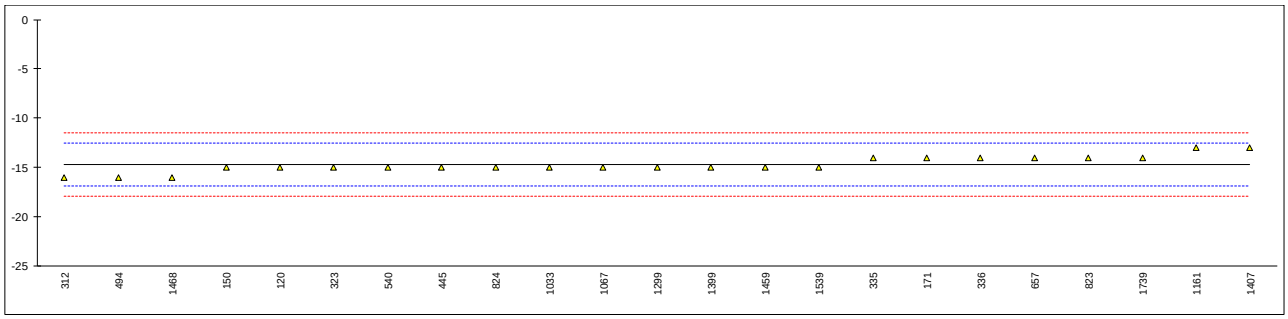


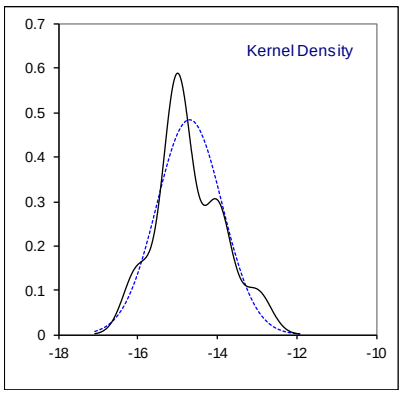


Determination of Cold Filter Plugging Point (CFPP) on sample #18065; results in °C

lab	method	value	mark	z(targ)	remarks
120	D6371	-15		-0.28	
150	EN116	-15		-0.28	
171	D6371	-14		0.65	
240		-----		-----	
312	EN116	-16		-1.22	
323	EN116	-15		-0.28	
335	EN116	-14		0.65	
336	EN116	-14		0.65	
445	IP309	-15		-0.28	
494	EN116	-16		-1.22	
511		-----		-----	
529		-----		-----	
540	D6371	-15.0		-0.28	
558		-----		-----	
631		-----		-----	
657	IP309	-14		0.65	
823	EN116	-14		0.65	
824	D6371	-15	C	-0.28	First reported -19
1033	IP309	-15		-0.28	
1067	EN116	-15		-0.28	
1161	EN116	-13		1.58	
1199		-----		-----	
1299	EN116	-15		-0.28	
1399	IP309	-15		-0.28	
1406		-----		-----	
1407	EN116	-13		1.58	
1459	EN116	-15.0		-0.28	
1468	EN116	-16		-1.22	
1494		-----		-----	
1539	EN116	-15		-0.28	
1643		-----		-----	
1739	EN116	-14		0.65	
1744		-----		-----	
1769		-----		-----	
6054		-----		-----	
6069		-----		-----	
6179		-----		-----	

normality	OK
n	23
outliers	0
mean (n)	-14.70
st.dev. (n)	0.822
R(calc.)	2.30
st.dev.(EN116:15)	1.071
R(EN116:15)	3





Determination of Carbon Residue on 100% FAME on sample #18065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D4530	0.015		-----	
150	D4530	<0.10		-----	
171	D4530	<0.10		-----	
240		-----		-----	
312		-----		-----	
323	D4530	<0.01		-----	
335		-----		-----	
336		-----		-----	
445	D4530	0.01		-----	
494	D4530	0.007		-----	
511	D189	0.0198		-----	
529		-----		-----	
540		-----		-----	
558		-----		-----	
631	D4530	0.1111		-----	
657	D4530	0.01		-----	
823	D4530	0.02		-----	
824	D4530	0.01		-----	
1033		-----		-----	
1067		-----		-----	
1161	ISO10370	0.02		-----	
1199		-----		-----	
1299	D4530	0.03		-----	
1399		-----		-----	
1406		-----		-----	
1407	EN10370	0.027		-----	
1459		-----		-----	
1468	ISO10370	0.01		-----	
1494		-----		-----	
1539	ISO10370	0.02		-----	
1643		-----		-----	
1739		-----		-----	
1744		-----		-----	
1769		-----		-----	
6054	D4530	0.00529		-----	
6069		-----		-----	
6179		-----		-----	
	n	16			
	mean (n)	<0.1			

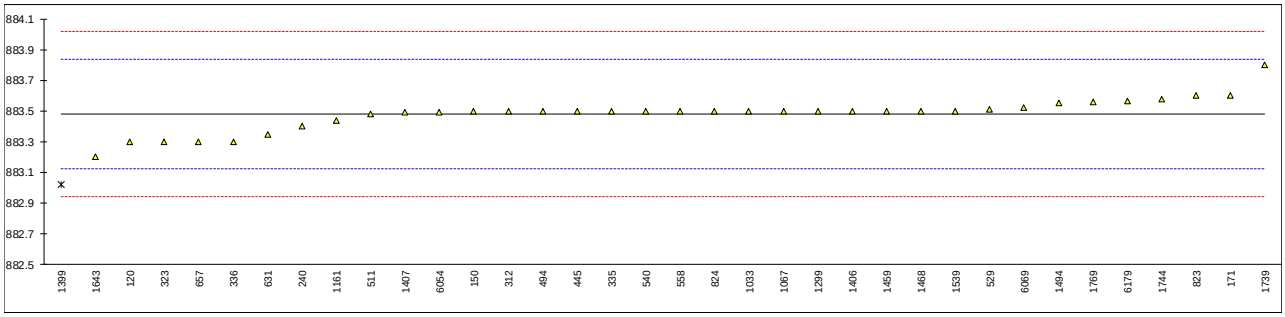
Determination of Copper Strip Corrosion 3 hrs/50°C on sample #18065

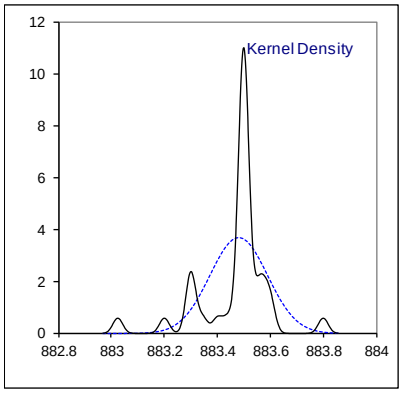
lab	method	value	mark	z(targ)	remarks
120	D130	1A		-----	
150	D130	1A		-----	
171	D130	1a		-----	
240		-----		-----	
312		-----		-----	
323	D130	cucor1a		-----	
335		-----		-----	
336		-----		-----	
445	IP154	1A		-----	
494	D130	1a		-----	
511	D130	1A		-----	
529	D130	1A		-----	
540	D130	1a		-----	
558		-----		-----	
631	D130	1a		-----	
657	D130	1a		-----	
823	D130	1a		-----	
824	D130	1a		-----	
1033	IP154	1a		-----	
1067	D130	1A		-----	
1161	ISO2160	1a		-----	
1199		-----		-----	
1299	D130	1A		-----	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459		-----		-----	
1468	ISO2160	1a		-----	
1494		-----		-----	
1539	ISO2160	1a		-----	
1643		-----		-----	
1739	ISO2160	1a		-----	
1744		-----		-----	
1769		-----		-----	
6054	D130	1a		-----	
6069		-----		-----	
6179		-----		-----	
	n	21			
	mean (n)	1A			

Determination of Density at 15°C on sample #18065; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
120	D4052	883.3		-1.01	
150	D4052	883.5		0.11	
171	D4052	883.6		0.67	
240	D4052	883.4		-0.45	
312	D4052	883.5		0.11	
323	ISO12185	883.3		-1.01	
335	ISO12185	883.5		0.11	
336	ISO12185	883.3		-1.01	
445	IP365	883.5		0.11	
494	ISO12185	883.5		0.11	
511	D4052	883.48		0.00	
529	D4052	883.51	C	0.16	First reported 833.51
540	D4052	883.50		0.11	
558	D4052	883.5		0.11	
631	D4052	883.345		-0.76	
657	D4052	883.3		-1.01	
823	ISO12185	883.6		0.67	
824	ISO12185	883.5		0.11	
1033	IP365	883.5		0.11	
1067	ISO12185	883.5		0.11	
1161	ISO12185	883.44		-0.23	
1199		-----		-----	
1299	D4052	883.5		0.11	
1399	D4052	883.024	R(0.01)	-2.56	
1406	ISO12185	883.50		0.11	
1407	ISO12185	883.49		0.05	
1459	ISO12185	883.50		0.11	
1468	ISO12185	883.5		0.11	
1494	D4052	883.55		0.39	
1539	ISO12185	883.5		0.11	
1643	D4052	883.2		-1.57	
1739	ISO3675	883.8	C	1.79	First reported 884.24
1744	D4052	883.58		0.56	
1769	D4052	883.562		0.45	
6054	D4052	883.49		0.05	
6069	D4052	883.520		0.22	
6179	D4052	883.563		0.46	

normality	not OK
n	35
outliers	1
mean (n)	883.48
st.dev. (n)	0.109
R(calc.)	0.30
st.dev.(ISO12185:96)	0.179
R(ISO12185:96)	0.5



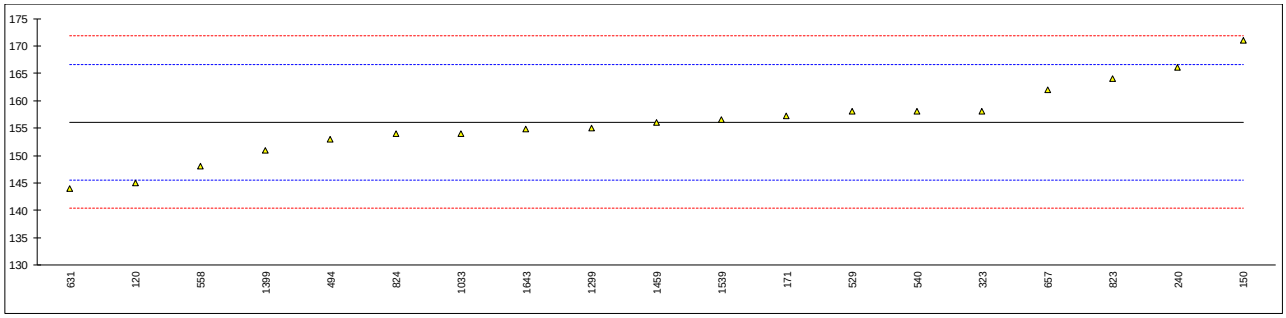


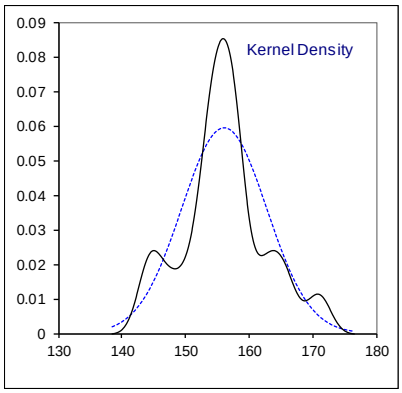
Determination of Flash Point, PMcc on sample #18065; results in °C

lab	method	value	mark	z(targ)	remarks
120	D93-C	145		-2.11	
150	D93-C	171.0		2.84	
171	D93-C	157.2		0.21	
240	D93-A	166		1.89	
312		-----		-----	
323	D93-C	158.0		0.37	
335		-----		-----	
336		-----		-----	
445		-----		-----	
494	D93-C	153.0		-0.59	
511		-----		-----	
529	D93-C	158.0		0.37	
540	D93-C	158.00		0.37	
558	D93-C	148		-1.54	
631	D93-A	144.0		-2.30	
657	D93-C	162		1.13	
823	D93-C	164.0		1.51	
824	D93-C	154.0		-0.40	
1033	D93-A	154.0		-0.40	
1067		-----		-----	
1161		-----		-----	
1199		-----		-----	
1299	D93-A	155.0		-0.21	
1399	D93-C	151		-0.97	
1406		-----		-----	
1407		-----		-----	
1459	ISO2719-A	156.0		-0.01	
1468		-----		-----	
1494		-----		-----	
1539	ISO2719-C	156.5		0.08	
1643	D93-C	154.78		-0.25	
1739		-----		-----	
1744		-----		-----	
1769		-----		-----	
6054		-----		-----	
6069		-----		-----	
6179		-----		-----	

	normality	OK
	n	19
	outliers	0
	mean (n)	156.08
	st.dev. (n)	6.717
	R(calc.)	18.807
	st.dev.(D93-C:16a)	5.250
	R(D93-C:16a)	14.7

Compare R(ISO2719-C:16)=14.7

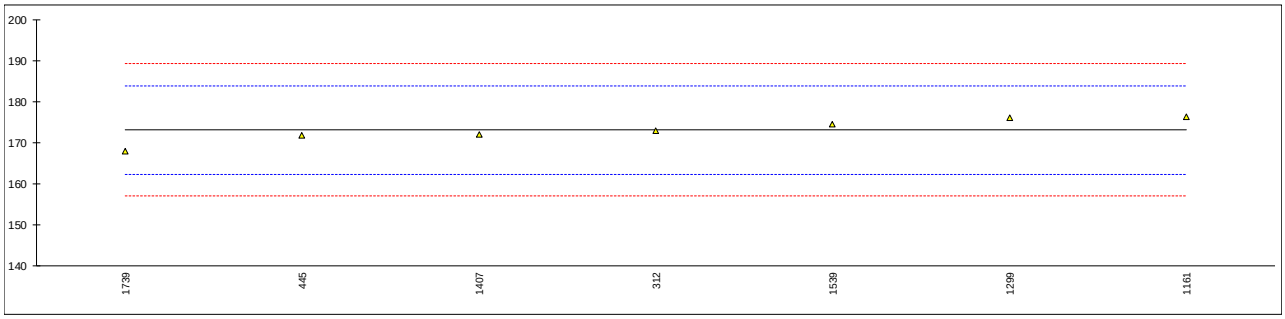


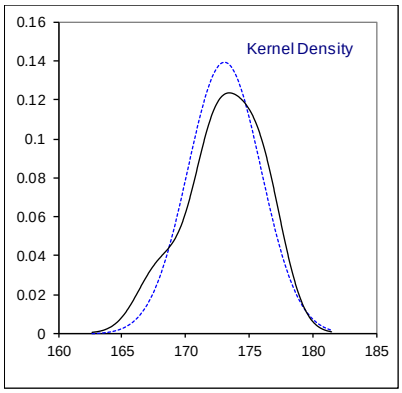


Determination of Flash Point, recc on sample #18065; results in °C

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	ISO3679	173.0		-0.01	
323		-----		-----	
335		-----		-----	
336		-----		-----	
445	ISO3679	171.8		-0.24	
494		-----		-----	
511		-----		-----	
529		-----		-----	
540		-----		-----	
558		-----		-----	
631		-----		-----	
657		-----		-----	
823		-----		-----	
824		-----		-----	
1033		-----		-----	
1067		-----		-----	
1161	ISO3679	176.2		0.59	
1199		-----		-----	
1299	ISO3679	176.0		0.55	
1399		-----		-----	
1406		-----		-----	
1407	ISO3679	172		-0.20	
1459		-----		-----	
1468		-----		-----	
1494		-----		-----	
1539	ISO3679	174.5		0.27	
1643		-----		-----	
1739	ISO3679	167.96		-0.95	
1744		-----		-----	
1769		-----		-----	
6054		-----		-----	
6069		-----		-----	
6179		-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	173.07
st.dev. (n)	2.867
R(calc.)	8.026
st.dev.(ISO3679:15)	5.357
R(ISO3679:15)	15

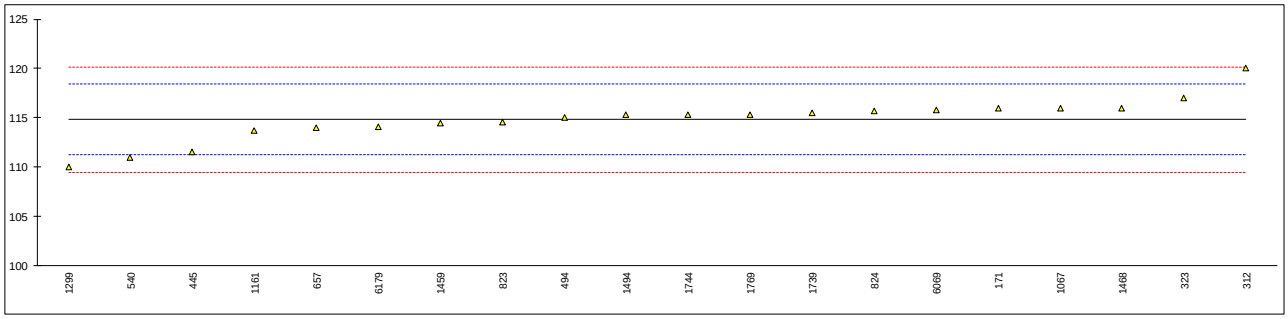


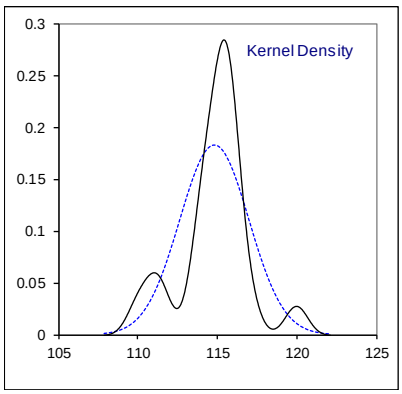


Determination of Iodine Value conform on sample #18065; results in g I_2/100g

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171	EN14111	116		0.66	
240		-----		-----	
312	EN14111	120		2.90	
323	EN14111	117		1.22	
335		-----		-----	
336		-----		-----	
445	EN14111	111.5		-1.86	
494	EN14111	115		0.10	
511		-----		-----	
529		-----		-----	
540	EN14111	111.0		-2.14	
558		-----		-----	
631		-----		-----	
657	EN14111	114		-0.46	
823	EN14111	114.6		-0.12	
824	EN14111	115.7		0.49	
1033		-----		-----	
1067	EN14111	116		0.66	
1161	EN14111	113.7	C	-0.63	First reported 137.713
1199		-----		-----	
1299	EN14111	110		-2.70	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459	EN16300	114.5		-0.18	
1468	EN14111	116		0.66	
1494	EN14111	115.29		0.26	
1539		-----		-----	
1643		-----		-----	
1739	EN14111	115.5		0.38	
1744	EN14111	115.30		0.27	
1769	EN14111	115.338		0.29	
6054		-----		-----	
6069	EN14111	115.810		0.56	
6179	EN14111	114.121		-0.39	

normality	suspect
n	20
outliers	0
mean (n)	114.82
st.dev. (n)	2.178
R(calc.)	6.099
st.dev.(EN14111:03)	1.786
R(EN14111:03)	5



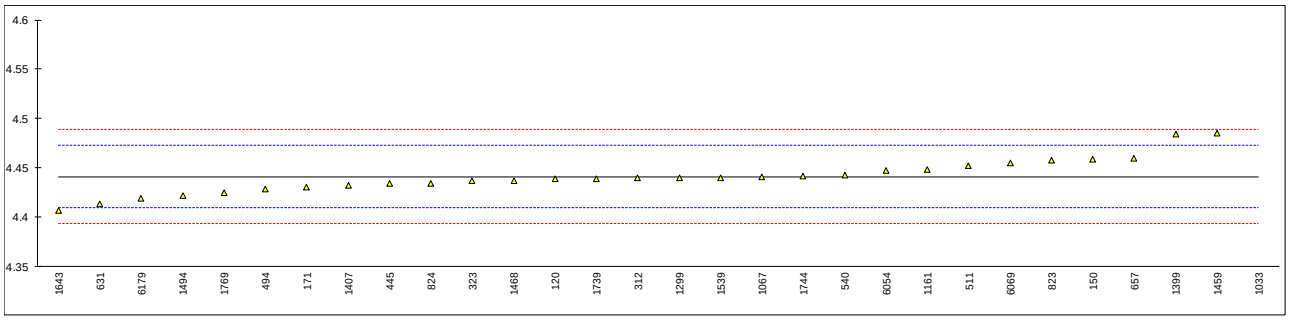


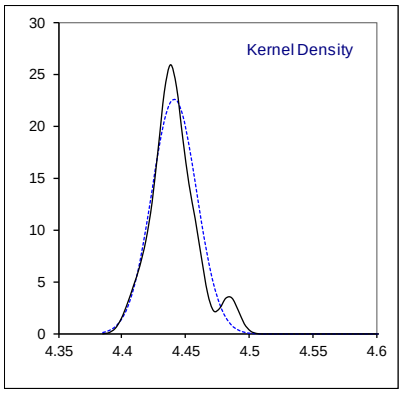
Determination of Kinematic Viscosity at 40°C on sample #18065; results in mm²/s

lab	method	value	mark	z(targ)	remarks
120	D445	4.4387		-0.15	
150	D445	4.459		1.12	
171	D445	4.430		-0.70	
240		-----		-----	
312	D445	4.440		-0.07	
323	ISO3104	4.437		-0.26	
335		-----		-----	
336		-----		-----	
445	IP71	4.434		-0.45	
494	ISO3104	4.4289		-0.77	
511	D445	4.4525		0.71	
529		-----		-----	
540	D445	4.4430		0.12	
558		-----		-----	
631	D445	4.4131		-1.76	
657	D445	4.460		1.19	
823	D445	4.458		1.06	
824	D445	4.434		-0.45	
1033	IP71	5.597	D(0.01)	72.54	
1067	ISO3104	4.441		-0.01	
1161	ISO3104	4.448		0.43	
1199		-----		-----	
1299	D445	4.440		-0.07	
1399	D7042	4.4839		2.68	
1406		-----		-----	
1407	In house	4.4321		-0.57	
1459	D7042	4.4848		2.74	
1468	ISO3104	4.437		-0.26	
1494	D445	4.4222		-1.19	
1539	ISO3104	4.440		-0.07	
1643	D445	4.407		-2.14	
1739	ISO3104	4.4387		-0.15	
1744	D445	4.4422		0.07	
1769	D445	4.425	C	-1.01	First reported 4.387
6054	D445	4.447653		0.41	
6069	D445	4.4552		0.88	
6179	D445	4.4194		-1.36	

normality	suspect
n	29
outliers	1
mean (n)	4.4411
st.dev. (n)	0.01759
R(calc.)	0.0493
st.dev.(ISO3104:94)	0.01593
R(ISO3104:94)	0.0446

Compare R(D7042:16e3)=0.0426, R(D445:17)=0.0995

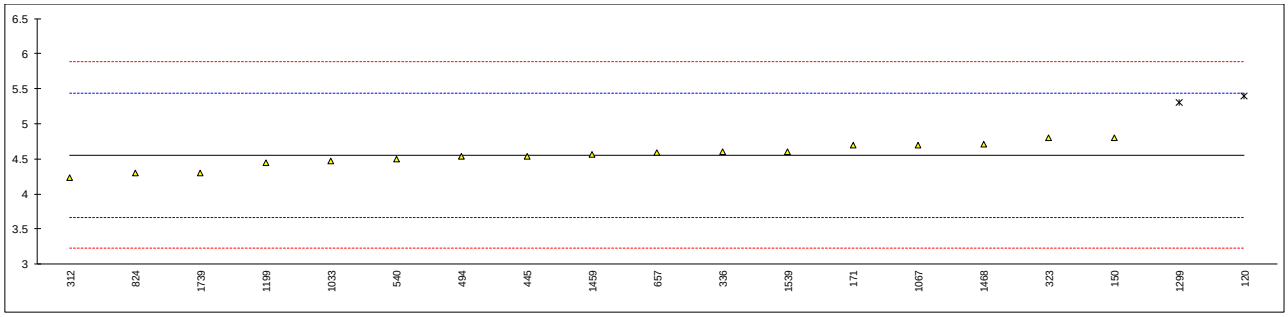


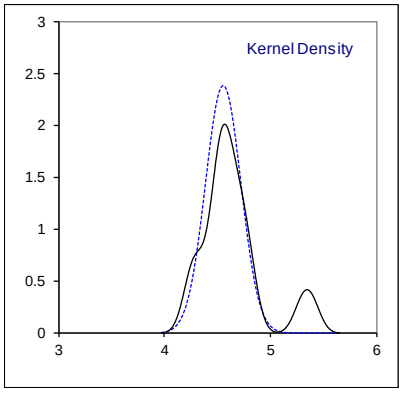


Determination of Oxidation Stability Induction period on sample #18065; results in hours

lab	method	value	mark	z(targ)	remarks
120	EN15751	5.39	DG(0.01)	1.89	
150	EN15751	4.8		0.56	
171	EN15751	4.7		0.33	
240		-----		-----	
312	EN15751	4.23		-0.73	
323	EN15751	4.8		0.56	
335		-----		-----	
336	EN15751	4.6		0.11	
445		4.54		-0.03	
494	EN15751	4.53		-0.05	
511		-----		-----	
529		-----		-----	
540	EN14112	4.50		-0.12	
558		-----		-----	
631		-----		-----	
657	EN15751	4.59		0.09	
823		-----		-----	
824	EN15751	4.3		-0.57	
1033	EN14112	4.475		-0.17	
1067	EN14112	4.7		0.33	
1161		-----		-----	
1199	EN14112	4.45		-0.23	
1299	EN15751	5.3	DG(0.01)	1.69	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459	EN15751	4.56		0.02	
1468	EN15751	4.71		0.36	
1494		-----		-----	
1539	EN15751	4.6		0.11	
1643		-----		-----	
1739	EN14112	4.30		-0.57	
1744		-----		-----	
1769		-----		-----	
6054		-----		-----	
6069		-----		-----	
6179		-----		-----	

normality	OK
n	17
outliers	2
mean (n)	4.552
st.dev. (n)	0.1674
R(calc.)	0.469
st.dev.(EN15751:14)	0.4426
R(EN15751:14)	1.239

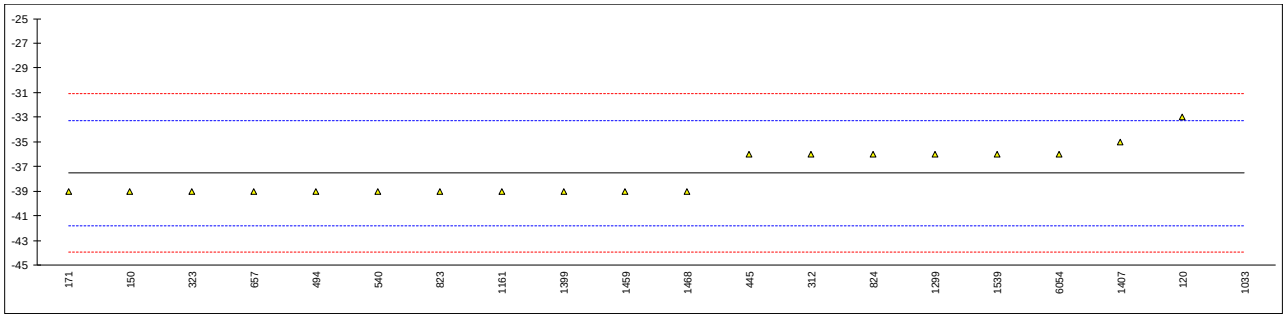


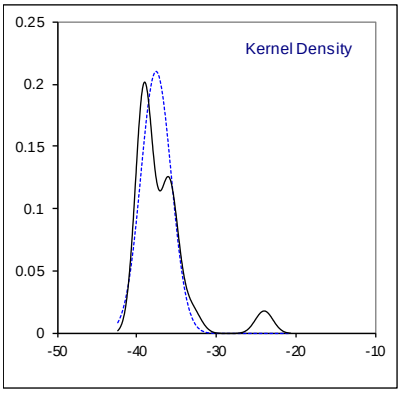


Determination of Pour Point on sample #18065; results in °C

lab	method	value	mark	z(targ)	remarks
120	D97	-33		2.11	
150	D5950	-39		-0.69	
171	D5950	-39.0		-0.69	
240	D97	< -33		-----	
312	D5950	-36		0.71	
323	ISO3016	-39		-0.69	
335		-----		-----	
336		-----		-----	
445	D5950	-36		0.71	
494	ISO3016	-39		-0.69	
511		-----		-----	
529		-----		-----	
540	D5950	-39		-0.69	
558		-----		-----	
631	D97	<-36		-----	
657	D97	-39		-0.69	
823	ISO3016	-39		-0.69	
824	D97	-36		0.71	
1033	D7346	-24.0	D(0.01)	6.31	
1067	ISO3016	< -39		-----	
1161	ISO3016	-39		-0.69	
1199		-----		-----	
1299	D97	-36		0.71	
1399	ISO3016	-39		-0.69	
1406	ISO3016	<-35		-----	
1407	ISO3016	-35		1.18	
1459	ISO3016	-39.0		-0.69	
1468	ISO3016	-39		-0.69	
1494		-----		-----	
1539	ISO3016	-36		0.71	
1643		-----		-----	
1739		-----		-----	
1744		-----		-----	
1769	D5950	<39.0		-----	
6054	D97	-36.0		0.71	
6069		-----		-----	
6179	D97	<-21		-----	

normality	OK
n	19
outliers	1
mean (n)	-37.5
st.dev. (n)	1.90
R(calc.)	5.3
st.dev.(ISO3016:94)	2.14
R(ISO3016:94)	6.0





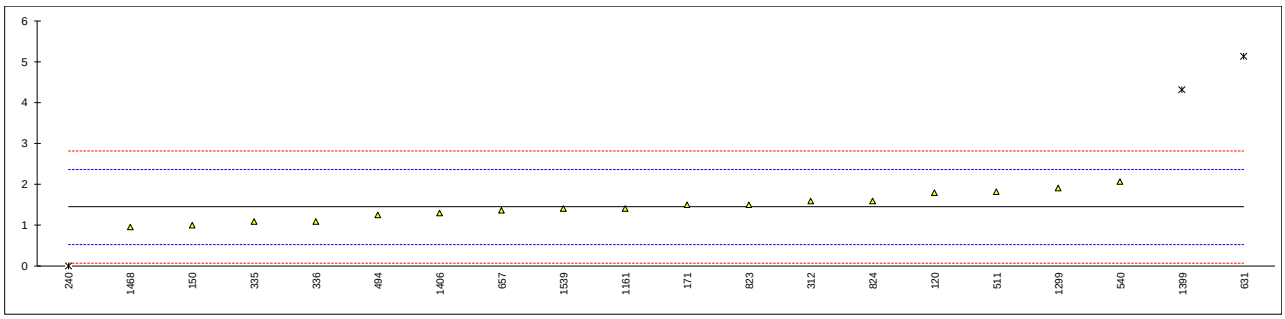
Determination of Sulphated Ash on sample #18065; results in %M/M

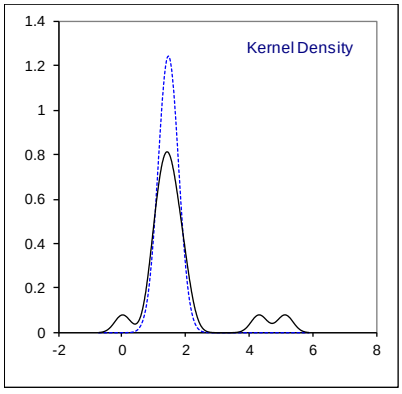
lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171	D874	<0.005		-----	
240		-----		-----	
312		-----		-----	
323	ISO3987	<0.001		-----	
335		-----		-----	
336		-----		-----	
445	D874	<0.005		-----	
494	D874	0.0		-----	
511	D874	0.0010		-----	
529		-----		-----	
540	ISO3987	<0.005		-----	
558		-----		-----	
631	D874	<0.001		-----	
657	D874	0.002	C	-----	First reported 0.07
823	D874	<0.005		-----	
824	D874	0.0006		-----	
1033		-----		-----	
1067	ISO3987	< 0.005		-----	
1161	ISO3987	0.0042		-----	
1199		-----		-----	
1299	ISO3987	<0.005		-----	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459	In house	0.00093		-----	
1468		-----		-----	
1494		-----		-----	
1539	ISO3987	0.0014		-----	
1643		-----		-----	
1739	ISO3987	0.0011	C	-----	First reported 0.011
1744		-----		-----	
1769		-----		-----	
6054		-----		-----	
6069		-----		-----	
6179		-----		-----	
	n	16			
	mean (n)	<0.005			

Determination of Sulphur on sample #18065; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	D5453	1.8		0.76	
150	D5453	1.0		-0.98	
171	D5453	1.5		0.11	
240	D4294	0.0149	ex	-3.13	ex: reported presumable in a different unit
312	D5453	1.6		0.33	
323	ISO20846	<3.0		-----	
335	ISO20846	1.1		-0.77	
336	ISO20846	1.1		-0.77	
445	D2622	<3		-----	
494	ISO20846	1.25		-0.44	
511	D5453	1.82		0.81	
529		-----		-----	
540	D5453	2.06		1.33	
558		-----		-----	
631	D4294	5.117	D(0.01)	8.00	
657	D5453	1.354		-0.21	
823	ISO20846	1.5		0.11	
824	D5453	1.6		0.33	
1033		-----		-----	
1067	ISO20846	< 3.0		-----	
1161	ISO20846	1.42		-0.07	
1199	ISO20884	<5.0		-----	
1299	ISO20884	1.9		0.98	
1399	D4294	4.3	D(0.01)	6.22	
1406	ISO20846	1.30		-0.33	
1407		-----		-----	
1459	In house	<5		-----	
1468	ISO20846	0.96		-1.07	
1494		-----		-----	
1539	ISO20846	1.4		-0.11	
1643		-----		-----	
1739	ISO13032	<3.3 [LQ]		-----	
1744		-----		-----	
1769		-----		-----	
6054		-----		-----	
6069		-----		-----	
6179		-----		-----	

normality	OK	
n	17	
outliers	2 (+1ex)	
mean (n)	1.451	
st.dev. (n)	0.3208	
R(calc.)	0.898	
st.dev.(ISO20846:11)	0.4580	
R(ISO20846:11)	1.282	Compare R(D5453:16e1)=0.7345

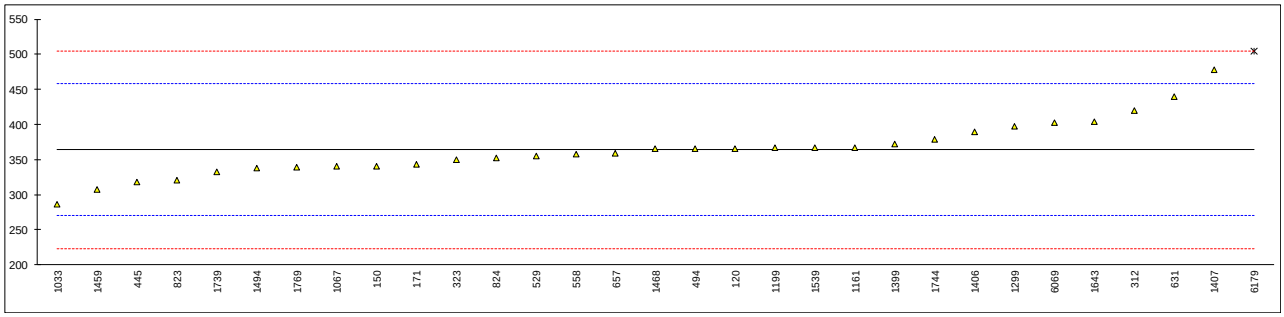


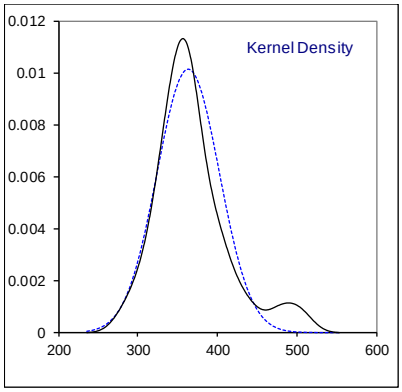


Determination of Water content by KF on sample #18065; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	D6304-A	366		0.04	
150	D6304-A	340		-0.51	
171	D6304-A	343		-0.45	
240		-----		-----	
312	ISO12937	420	C	1.20	First reported 70
323	ISO12937	350		-0.30	
335		-----		-----	
336		-----		-----	
445	D6304-B	318		-0.98	
494	ISO12937	365.5		0.03	
511		-----		-----	
529	D6304-A	354.32	C	-0.20	First reported 0.035
540		-----		-----	
558	D6304-A	358.2		-0.12	
631	D6304-B	440	C	1.62	First reported 0.044%
657	D6304-A	359.3		-0.10	
823	ISO12937	320		-0.94	
824	D6304-A	352		-0.25	
1033	IP438	286.825		-1.64	
1067	ISO12937	340		-0.51	
1161	ISO12937	367.005		0.07	
1199	ISO12937	367		0.07	
1299	ISO12937	397		0.71	
1399	IP438	372		0.17	
1406	ISO12937	390		0.56	
1407	ISO12937	478.3		2.44	
1459	ISO12937	307		-1.21	
1468	ISO12937	365		0.02	
1494	ISO12937	337.49		-0.56	
1539	ISO12937	367		0.07	
1643	ISO6296	404	C	0.86	First reported 0.0404
1739	ISO12937	332.1		-0.68	
1744	E203	379		0.32	
1769	ISO12937	338.817		-0.54	
6054		-----		-----	
6069	E203	402.0		0.81	
6179	ISO12937	504	G(0.05)	2.99	

normality	suspect
n	30
outliers	1
mean (n)	363.90
st.dev. (n)	39.305
R(calc.)	110.06
st.dev.(ISO12937:00)	46.852
R(ISO12937:00)	131.19





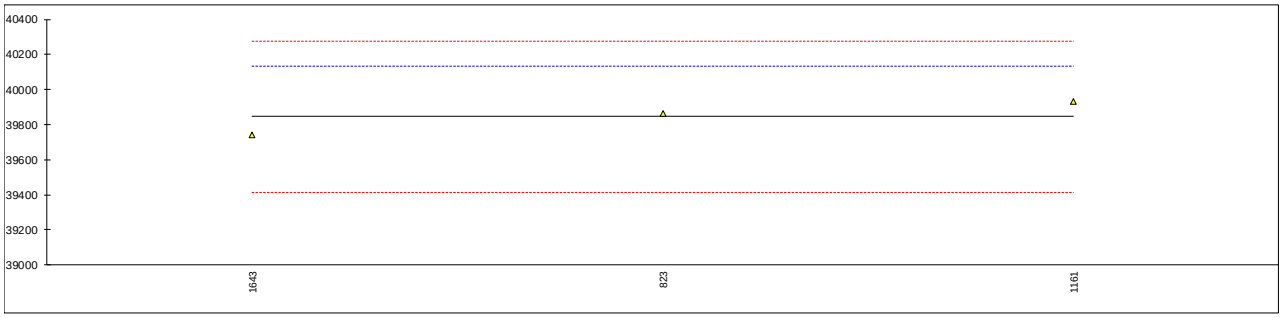
Determination of Water and Sediment on sample #18065; results in %V/V

lab	method	value	mark	z(targ)	remarks
120	D2709	0		-----	
150		-----		-----	
171	D2709	<0.01		-----	
240		-----		-----	
312		-----		-----	
323	D2709	<0.05		-----	
335		-----		-----	
336		-----		-----	
445	D2709	<0.01		-----	
494	D2709	< 0,01		-----	
511	D2709	0.00		-----	
529	D2709	0.0025		-----	
540	D2709	<0.05		-----	
558		-----		-----	
631	D2709	<0.01		-----	
657	D2709	0.01		-----	
823	D2709	0.000		-----	
824	D2709	<0.01		-----	
1033		-----		-----	
1067		-----		-----	
1161		-----		-----	
1199		-----		-----	
1299	D2709	<0.01		-----	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459		-----		-----	
1468		-----		-----	
1494		-----		-----	
1539		-----		-----	
1643		-----		-----	
1739		-----		-----	
1744		-----		-----	
1769		-----		-----	
6054		-----		-----	
6069		-----		-----	
6179		-----		-----	
	n	13			
	mean (n)	<0.05			

Determination of Calorific Value on sample #18065; results in kJ/kg

lab	method	Gross at constant vol.	mark	z(targ)	Net at constant vol.	Net at constant press
120		-----		-----	-----	-----
150		-----		-----	-----	-----
171		-----		-----	-----	-----
240		-----		-----	-----	-----
312		-----		-----	-----	-----
323		-----		-----	-----	-----
335		-----		-----	-----	-----
336		-----		-----	-----	-----
445		-----		-----	-----	-----
494		-----		-----	-----	-----
511		-----		-----	-----	-----
529		-----		-----	-----	-----
540		-----		-----	-----	-----
558		-----		-----	-----	-----
631		-----		-----	-----	-----
657		-----		-----	-----	-----
823	D240	39864		0.14	37318	-----
824		-----		-----	-----	-----
1033		-----		-----	-----	-----
1067		-----		-----	-----	-----
1161	DIN51900-1	39930		0.60	37280	-----
1199		-----		-----	-----	-----
1299		-----		-----	-----	-----
1399		-----		-----	-----	-----
1406		-----		-----	-----	-----
1407		-----		-----	-----	-----
1459		-----		-----	-----	-----
1468		-----		-----	-----	-----
1494		-----		-----	-----	-----
1539		-----		-----	-----	-----
1643	D240	39740	C	-0.73	-----	37.17
1739		-----		-----	-----	-----
1744		-----		-----	-----	-----
1769		-----		-----	-----	-----
6054		-----		-----	-----	-----
6069		-----		-----	-----	-----
6179		-----		-----	-----	-----
	normality	unknown			unknown	unknown
	n	3			2	1
	outliers	0			n.a.	n.a.
	mean (n)	39844.7			n.a.	n.a.
	st.dev. (n)	96.46			n.a.	n.a.
	R(calc.)	270.1			n.a.	n.a.
	st.dev.(DIN51900-1:00)	142.86			n.a.	n.a.
	R(DIN51900-1:00)	400			n.a.	n.a.

Lab 1643 reported 39.74. Probably unit error MJ/kg?



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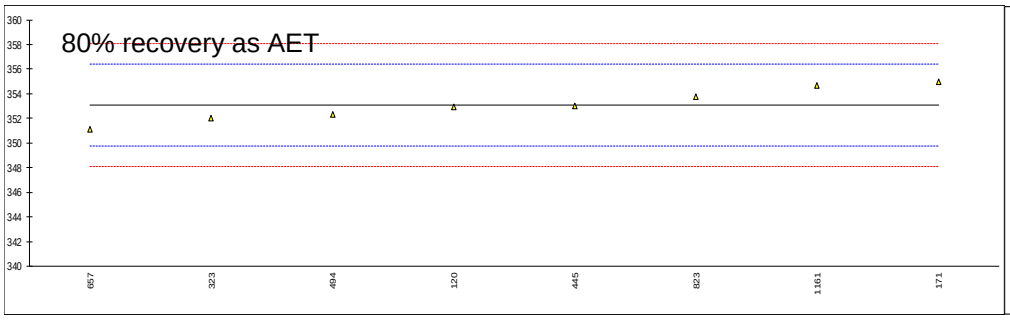
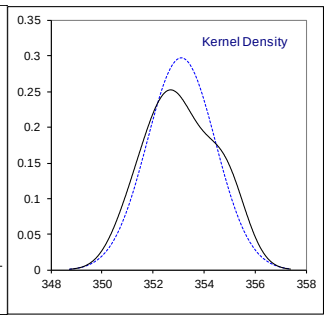
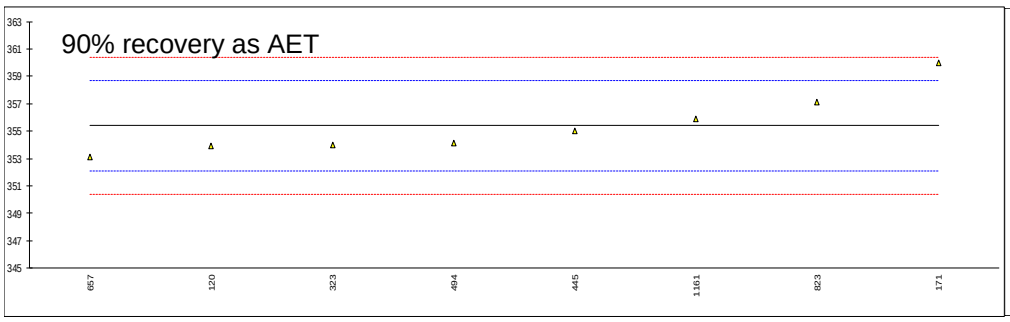
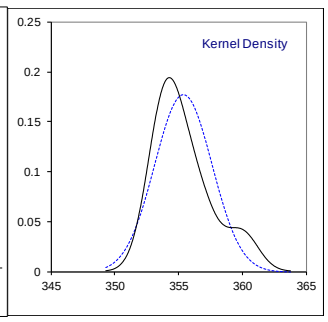
Determination of Distillation at 10 mm Hg, % recovered as AET on sample #18065; results in °C

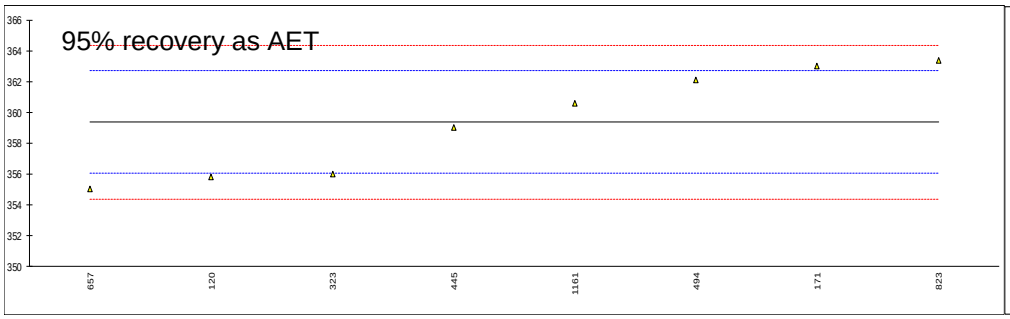
lab	method	80%rec	mark	z(targ)	90%rec	mark	z(targ)	95%rec	mark	z(targ)
120	D1160	352.9		-0.12	353.9		-0.90	355.8		-2.15
150	D1160	-----		-----	-----		-----	-----		-----
171	D1160	355		1.15	360		2.78	363		2.20
240		-----		-----	-----		-----	-----		-----
312		-----		-----	-----		-----	-----		-----
323	D1160	352		-0.66	354		-0.84	356		-2.03
335		-----		-----	-----		-----	-----		-----
336		-----		-----	-----		-----	-----		-----
445	D1160	353		-0.06	355		-0.23	359		-0.22
494	D1160	352.3		-0.48	354.1		-0.78	362.1		1.65
511		-----		-----	-----		-----	-----		-----
529		-----		-----	-----		-----	-----		-----
540		-----		-----	-----		-----	-----		-----
558		-----		-----	-----		-----	-----		-----
631		-----		-----	-----		-----	-----		-----
657	D1160	351.1		-1.21	353.1		-1.38	355.0		-2.63
823		353.8		0.42	357.1		1.03	363.4		2.44
824		-----		-----	-----		-----	-----		-----
1033		-----		-----	-----		-----	-----		-----
1067		-----		-----	-----		-----	-----		-----
1161	D1160	354.7		0.97	355.9		0.31	360.6		0.75
1199		-----		-----	-----		-----	-----		-----
1299		-----		-----	-----		-----	-----		-----
1399		-----		-----	-----		-----	-----		-----
1406		-----		-----	-----		-----	-----		-----
1407		-----		-----	-----		-----	-----		-----
1459		-----		-----	-----		-----	-----		-----
1468		-----		-----	-----		-----	-----		-----
1494		-----		-----	-----		-----	-----		-----
1539		-----		-----	-----		-----	-----		-----
1643		-----		-----	-----		-----	-----		-----
1739		-----		-----	-----		-----	-----		-----
1744		-----		-----	-----		-----	-----		-----
1769		-----		-----	-----		-----	-----		-----
6054		-----		-----	-----		-----	-----		-----
6069		-----		-----	-----		-----	-----		-----
6179		-----		-----	-----		-----	-----		-----
	normality	unknown			unknown			unknown		
	n	8			8			8		
	outliers	0			0			0		
	mean (n)	353.10			355.39			359.36		
	st.dev. (n)	1.340			2.257			3.417		
	R(calc.)	3.75			6.32			9.57		
	st.dev.(D1160:15)	1.657			1.657			1.657		
	R(D1160:15)	4.64			4.64			4.64		

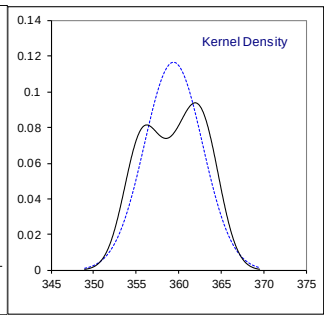

80% recovery as AET
360
358
356
354
352
350
348
346
344
342
340
657
323
494
120
445
823
1161
171


Kernel Density
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
348
350
352
354
356
358


90% recovery as AET
363
361
359
357
355
353
351
349
347
345
657
120
323
494
445
1161
823
171


Kernel Density
0.25
0.2
0.15
0.1
0.05
0
345
350
355
360
365

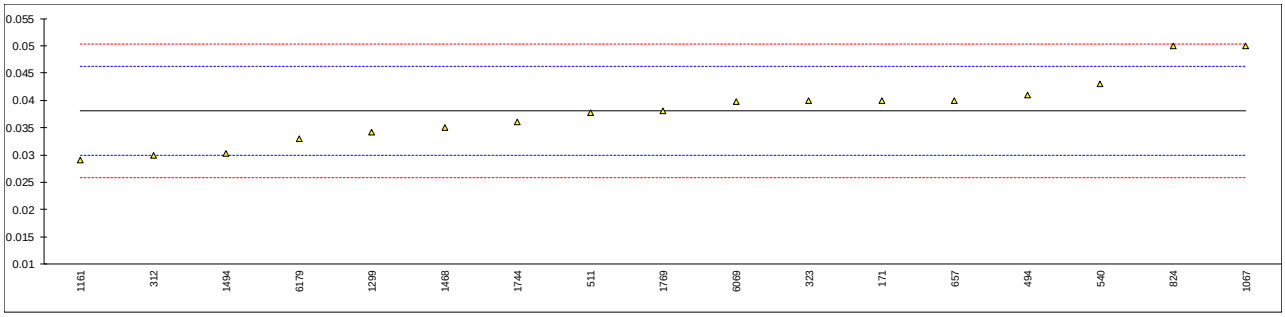

95% recovery as AET
366
364
362
360
358
356
354
352
350
657
120
323
445
1161
494
171
823

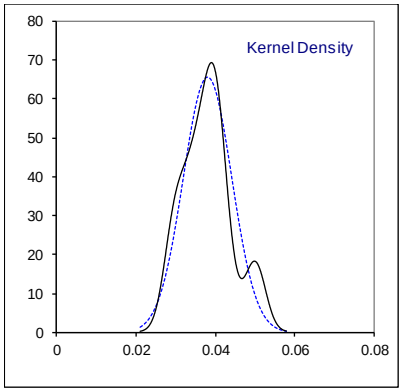

Kernel Density
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
345
350
355
360
365
370
375

Determination of Methanol on sample #18065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171	EN14110	0.04		0.47	
240		-----		-----	
312	EN14110	0.03		-1.98	
323	EN14110	0.04		0.47	
335		-----		-----	
336		-----		-----	
445		-----		-----	
494	EN14110	0.041		0.72	
511	EN14110	0.0378		-0.07	
529		-----		-----	
540	EN14110	0.043		1.21	
558		-----		-----	
631		-----		-----	
657	EN14110	0.04		0.47	
823		-----		-----	
824	EN14110	0.05		2.93	
1033		-----		-----	
1067	EN14110	0.05		2.93	
1161	EN14110	0.029		-2.23	
1199		-----		-----	
1299	EN14110	0.0342		-0.95	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459		-----		-----	
1468	EN14110	0.035		-0.75	
1494	EN14110	0.0303		-1.91	
1539		-----		-----	
1643		-----		-----	
1739		-----		-----	
1744	EN14110	0.036		-0.51	
1769	EN14110	0.0381		0.01	
6054		-----		-----	
6069	EN14110	0.0398		0.42	
6179	EN14110	0.033		-1.24	

normality	OK
n	17
outliers	0
mean (n)	0.0381
st.dev. (n)	0.00609
R(calc.)	0.0171
st.dev.(EN14110:03)	0.00408
R(EN14110:03)	0.0114



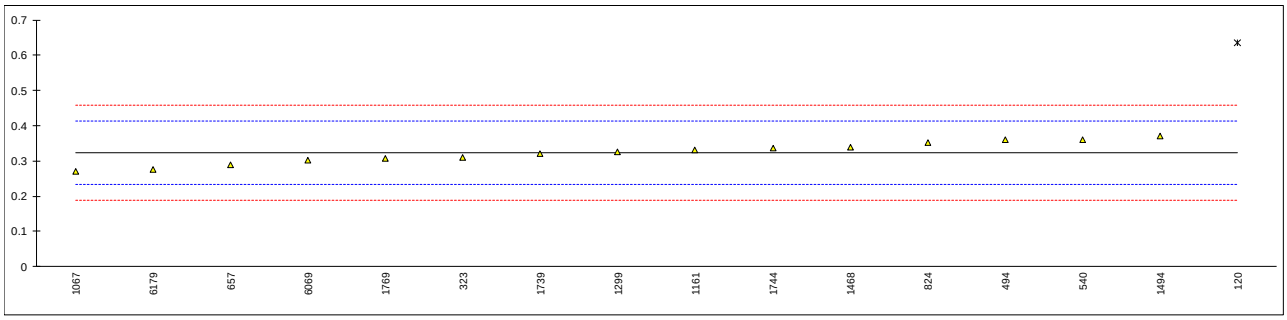


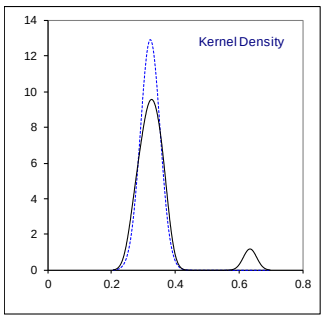
Determination of mono-Glycerides on sample #18065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D6584	0.636	D(0.01)	6.95	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312		-----		-----	
323	EN14105	0.31		-0.30	
335		-----		-----	
336		-----		-----	
445		-----		-----	
494	EN14105	0.360		0.81	
511		-----		-----	
529		-----		-----	
540	EN14105	0.360		0.81	
558		-----		-----	
631		-----		-----	
657	EN14105	0.29		-0.75	
823		-----		-----	
824	EN14105	0.352		0.63	
1033		-----		-----	
1067	EN14105	0.27		-1.19	
1161	EN14105	0.33		0.14	
1199		-----		-----	
1299	EN14105	0.326		0.06	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459		-----		-----	
1468	EN14105	0.34		0.37	
1494	D6584	0.3720		1.08	
1539		-----		-----	
1643		-----		-----	
1739	EN14105	0.3206		-0.06	
1744	D6584	0.3355		0.27	
1769	D6584	0.3076		-0.35	
6054		-----		-----	
6069	D6584	0.3024		-0.47	
6179	D6584	0.2767	C	-1.04	First reported 0.0021

normality	OK
n	15
outliers	1
mean (n)	0.3235
st.dev. (n)	0.03089
R(calc.)	0.0865
st.dev.(EN14105:11)	0.04493
R(EN14105:11)	0.1258

Compare R(D6584:13)=0.2285

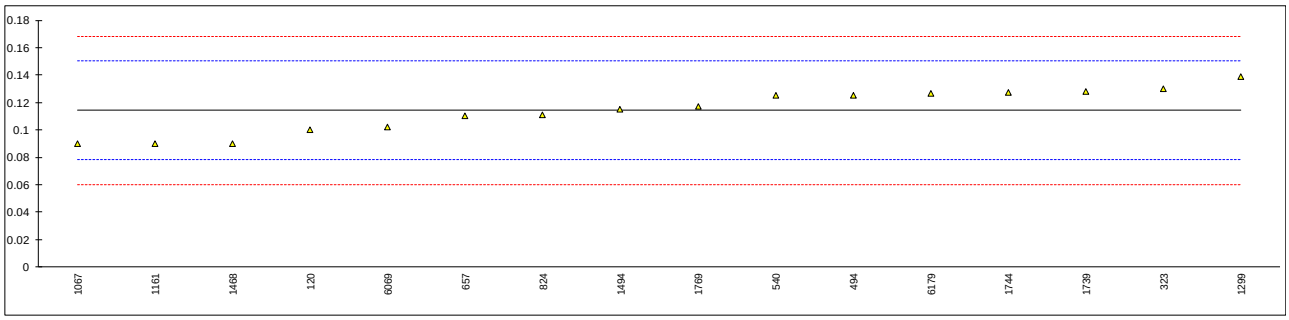


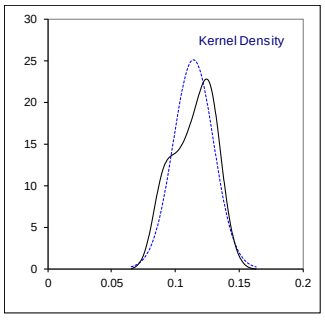


Determination of di-Glycerides on sample #18065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D6584	0.100		-0.79	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312		-----		-----	
323	EN14105	0.13		0.88	
335		-----		-----	
336		-----		-----	
445		-----		-----	
494	EN14105	0.125		0.60	
511		-----		-----	
529		-----		-----	
540	EN14105	0.125		0.60	
558		-----		-----	
631		-----		-----	
657	EN14105	0.11		-0.23	
823		-----		-----	
824	EN14105	0.111		-0.18	
1033		-----		-----	
1067	EN14105	0.09		-1.34	
1161	EN14105	0.090		-1.34	
1199		-----		-----	
1299	EN14105	0.139		1.38	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459		-----		-----	
1468	EN14105	0.09		-1.34	
1494	D6584	0.1151		0.05	
1539		-----		-----	
1643		-----		-----	
1739	EN14105	0.1282		0.78	
1744	D6584	0.1276		0.74	
1769	D6584	0.1174		0.18	
6054		-----		-----	
6069	D6584	0.1023		-0.66	
6179	D6584	0.1269	C	0.70	First reported 0.2753

normality	OK	
n	16	
outliers	0	
mean (n)	0.1142	
st.dev. (n)	0.01586	
R(calc.)	0.0444	
st.dev.(EN14105:11)	0.01801	
R(EN14105:11)	0.0504	Compare R(D6584:13)=0.1042



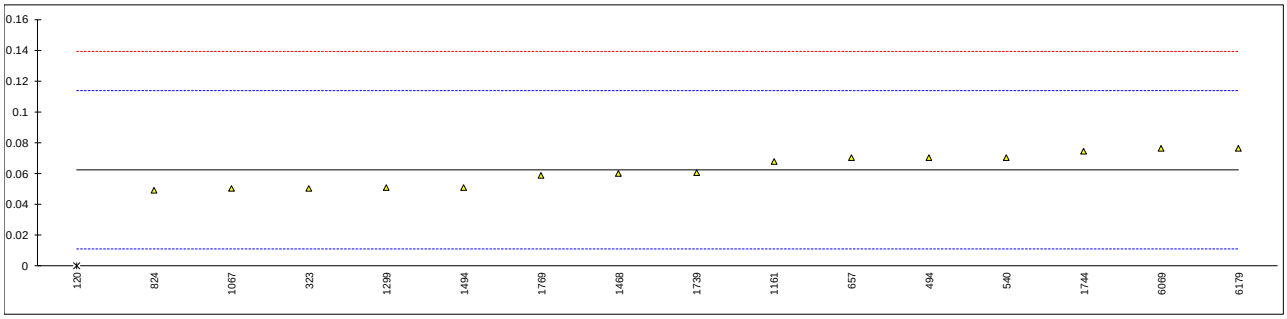


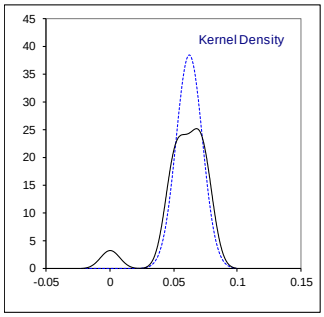
Determination of tri-Glycerides on sample #18065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D6584	0	D(0.01)	-2.43	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312		-----		-----	
323	EN14105	0.05		-0.48	
335		-----		-----	
336		-----		-----	
445		-----		-----	
494	EN14105	0.07		0.30	
511		-----		-----	
529		-----		-----	
540	EN14105	0.070		0.30	
558		-----		-----	
631		-----		-----	
657	EN14105	0.07		0.30	
823		-----		-----	
824	EN14105	0.049		-0.52	
1033		-----		-----	
1067	EN14105	0.05		-0.48	
1161	EN14105	0.068		0.22	
1199		-----		-----	
1299	EN14105	0.051		-0.44	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459		-----		-----	
1468	EN14105	0.06		-0.09	
1494	D6584	0.0511		-0.44	
1539		-----		-----	
1643		-----		-----	
1739	EN14105	0.0605		-0.07	
1744	D6584	0.0746		0.48	
1769	D6584	0.0586		-0.15	
6054		-----		-----	
6069	D6584	0.0762		0.54	
6179	D6584	0.0762	C	0.54	First reported 0.1280

	normality	OK
	n	15
	outliers	1
	mean (n)	0.0623
	st.dev. (n)	0.01036
	R(calc.)	0.0290
	st.dev.(EN14105:11)	0.02565
	R(EN14105:11)	0.0718

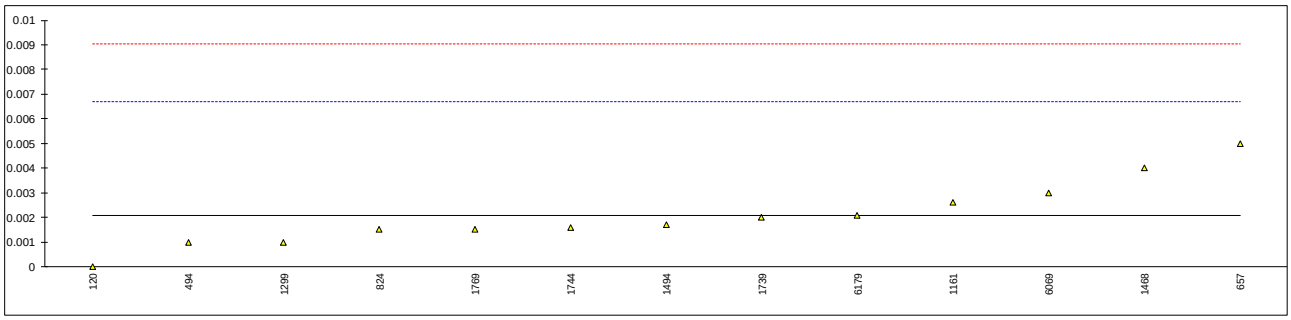
Compare R(D6584:13)=0.1605

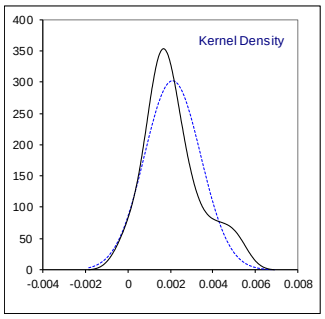




Determination of Free Glycerol on sample #18065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D6584	0		-0.90	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312		-----		-----	
323	EN14105	<0.001		-----	
335		-----		-----	
336		-----		-----	
445		-----		-----	
494	EN14105	0.001		-0.47	
511		-----		-----	
529		-----		-----	
540	EN14105	<0.01		-----	
558		-----		-----	
631		-----		-----	
657	EN14105	0.005		1.26	
823		-----		-----	
824	EN14105	0.0015		-0.25	
1033		-----		-----	
1067	EN14105	< 0.010		-----	
1161	EN14105	0.0026		0.22	
1199		-----		-----	
1299	EN14105	0.001		-0.47	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459		-----		-----	
1468	EN14105	0.004		0.83	
1494	D6584	0.0017		-0.16	
1539		-----		-----	
1643		-----		-----	
1739	EN14105	0.0020		-0.03	
1744	D6584	0.0016		-0.21	
1769	D6584	0.00153		-0.24	
6054		-----		-----	
6069	D6584	0.0030		0.40	
6179	D6584	0.0021	C	0.01	First reported 0.0779
	normality	OK			
	n	13			
	outliers	0			
	mean (n)	0.0021			
	st.dev. (n)	0.00132			
	R(calc.)	0.0037			
	st.dev.(EN14105:11)	0.00231			
	R(EN14105:11)	0.0065			Compare R(D6584:13)=0.0047



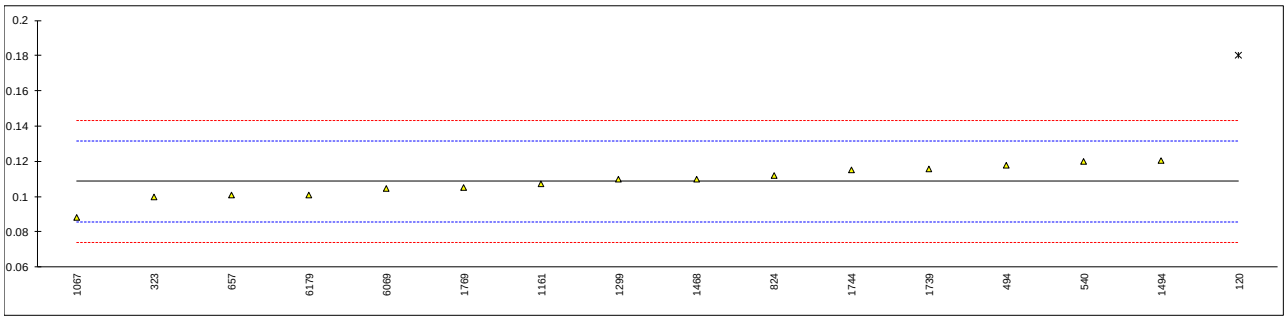


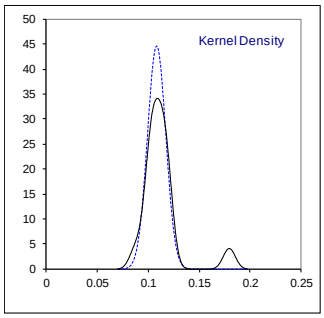
Determination of Total Glycerol on sample #18065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D6584	0.180	D(0.01)	6.23	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312		-----		-----	
323	EN14105	0.100		-0.74	
335		-----		-----	
336		-----		-----	
445		-----		-----	
494	EN14105	0.118		0.83	
511		-----		-----	
529		-----		-----	
540	EN14105	0.120		1.00	
558		-----		-----	
631		-----		-----	
657	EN14105	0.101		-0.66	
823		-----		-----	
824	EN14105	0.112		0.30	
1033		-----		-----	
1067	EN14105	0.088		-1.79	
1161	EN14105	0.107		-0.13	
1199		-----		-----	
1299	EN14105	0.110		0.13	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459		-----		-----	
1468	EN14105	0.11		0.13	
1494	D6584	0.1206		1.05	
1539		-----		-----	
1643		-----		-----	
1739	EN14105	0.1158		0.63	
1744	D6584	0.1153		0.59	
1769	D6584	0.1048		-0.32	
6054		-----		-----	
6069	D6584	0.1044		-0.36	
6179	D6584	0.1010	C	-0.66	First reported 0.1006

normality	OK
n	15
outliers	1
mean (n)	0.1085
st.dev. (n)	0.00896
R(calc.)	0.0251
st.dev.(EN14105:11)	0.01148
R(EN14105:11)	0.0321

Compare R(D6584:13)=0.0573

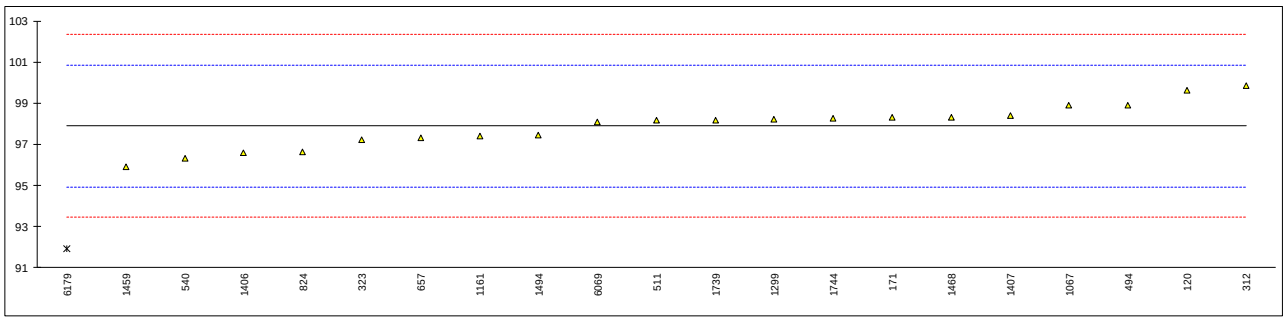


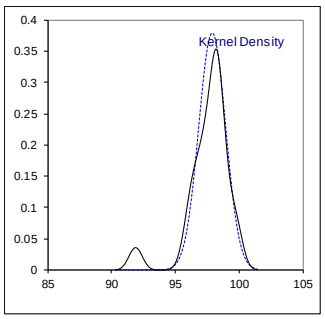


Determination of Total Ester content (FAME) on sample #18065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	EN14103	99.64		1.17	
150		-----		-----	
171	EN14103	98.3		0.27	
240		-----		-----	
312	EN14103	99.84		1.31	
323	EN14103	97.2		-0.47	
335		-----		-----	
336		-----		-----	
445		-----		-----	
494	EN14103	98.9		0.68	
511	EN14103	98.16		0.18	
529		-----		-----	
540	EN14103	96.30		-1.07	
558		-----		-----	
631		-----		-----	
657	EN14103	97.3		-0.40	
823		-----		-----	
824	EN14103	96.61		-0.86	
1033		-----		-----	
1067	EN14103	98.9		0.68	
1161	EN14103	97.4		-0.33	
1199		-----		-----	
1299	EN14103	98.2		0.21	
1399		-----		-----	
1406	EN14078	96.57		-0.89	
1407	EN14103	98.4		0.34	
1459	EN14103	95.9		-1.34	
1468	EN14103	98.3		0.27	
1494	EN14103	97.4618		-0.29	
1539		-----		-----	
1643		-----		-----	
1739	EN14103	98.16		0.18	
1744	EN14103	98.26		0.25	
1769		-----		-----	
6054		-----		-----	
6069	EN14103	98.10		0.14	
6179	EN14103	91.900	D(0.01)	-4.04	

normality	OK
n	20
outliers	1
mean (n)	97.895
st.dev. (n)	1.0523
R(calc.)	2.947
st.dev.(EN14103:11)	1.4857
R(EN14103:11)	4.160

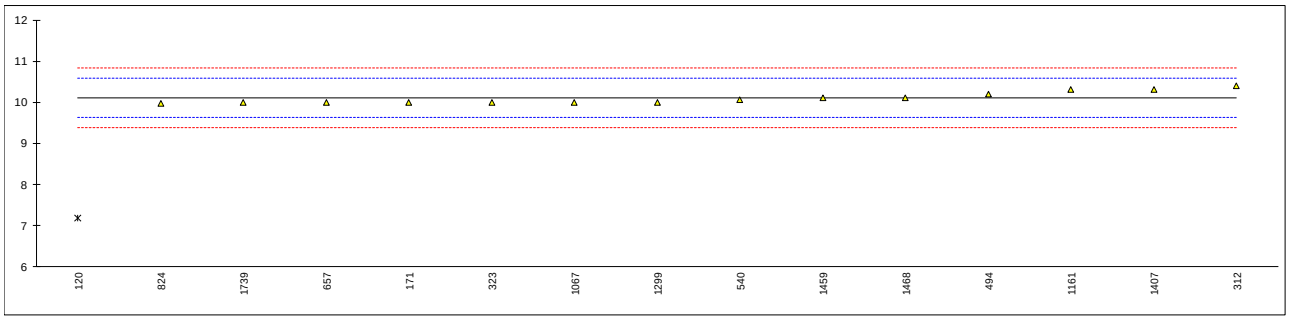


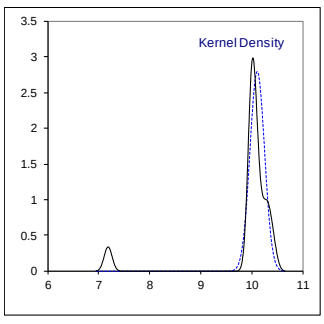


Determination of Linolenic Acid Methyl Ester content on sample #18065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	EN14103	7.19	D(0.01)	-12.08	
150		-----		-----	
171	EN14103	10.0		-0.43	
240		-----		-----	
312	EN14103	10.41		1.27	
323	EN14103	10.0		-0.43	
335		-----		-----	
336		-----		-----	
445		-----		-----	
494	EN14103	10.2		0.40	
511		-----		-----	
529		-----		-----	
540	EN14103	10.06		-0.18	
558		-----		-----	
631		-----		-----	
657	EN14103	10.0		-0.43	
823		-----		-----	
824	EN14103	9.98		-0.51	
1033		-----		-----	
1067	EN14103	10.0		-0.43	
1161	EN14103	10.3		0.82	
1199		-----		-----	
1299	EN14103	10.0		-0.43	
1399		-----		-----	
1406		-----		-----	
1407	EN14103	10.3		0.82	
1459	EN14103	10.1		-0.01	
1468	EN14103	10.1		-0.01	
1494		-----		-----	
1539		-----		-----	
1643		-----		-----	
1739	EN14103	9.99		-0.47	
1744		-----		-----	
1769		-----		-----	
6054		-----		-----	
6069		-----		-----	
6179		-----		-----	

normality	suspect
n	14
outliers	1
mean (n)	10.103
st.dev. (n)	0.1425
R(calc.)	0.399
st.dev.(EN14103:11)	0.2411
R(EN14103:11)	0.675



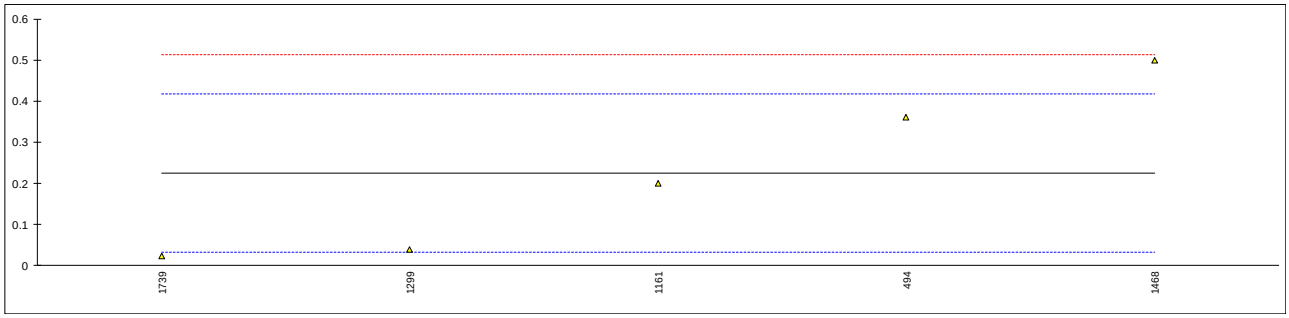


Determination of Polyunsaturated Methyl Esters content on sample #18065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	EN15779	<0.6		-----	
323	EN15779	<0.6		-----	
335		-----		-----	
336		-----		-----	
445		-----		-----	
494	EN15779	0.36		1.41	
511		-----		-----	
529		-----		-----	
540		-----		-----	
558		-----		-----	
631		-----		-----	
657	EN15779	<0.6		-----	
823		-----		-----	
824		-----		-----	
1033		-----		-----	
1067	EN15779	< 0.10		-----	
1161	EN15779	0.2		-0.25	
1199		-----		-----	
1299	EN15779	0.04		-1.91	
1399		-----		-----	
1406		-----		-----	
1407		-----		-----	
1459		-----		-----	
1468	EN15779	0.50		2.86	
1494		-----		-----	
1539		-----		-----	
1643		-----		-----	
1739	EN15779	0.022		-2.10	
1744		-----		-----	
1769		-----		-----	
6054		-----		-----	
6069		-----		-----	
6179		-----		-----	

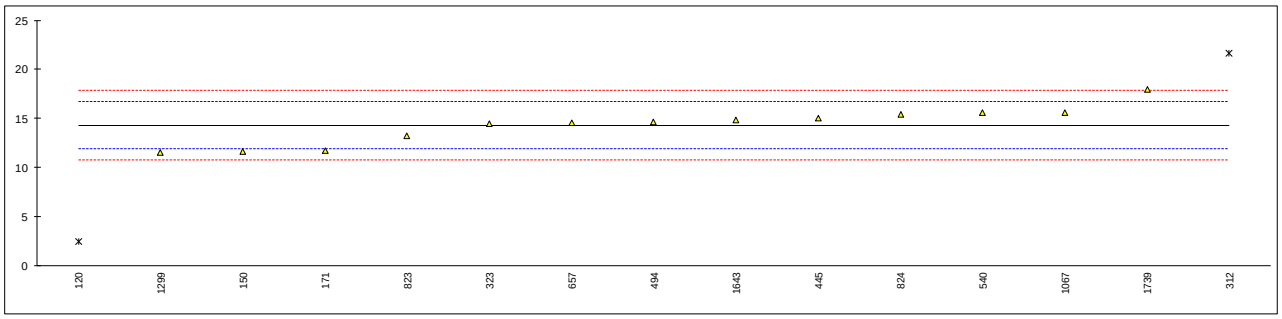
normality	unknown
n	5
outliers	0
mean (n)	0.224
st.dev. (n)	0.2061
R(calc.)	0.577
st.dev.(EN15779:09+A1(2013))	0.0964
R(EN15779:09+A1(2013))	0.27

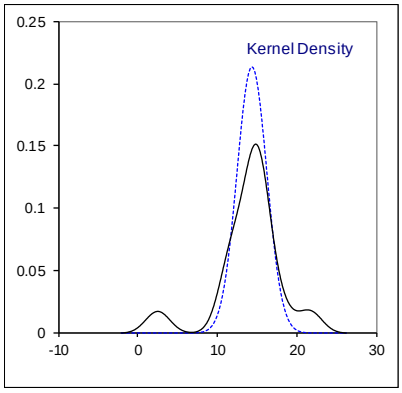
Application range 0.1 – 1.5 %M/M



Determination of sum of Calcium and Magnesium as Ca + Mg on sample #18067; results in mg/kg

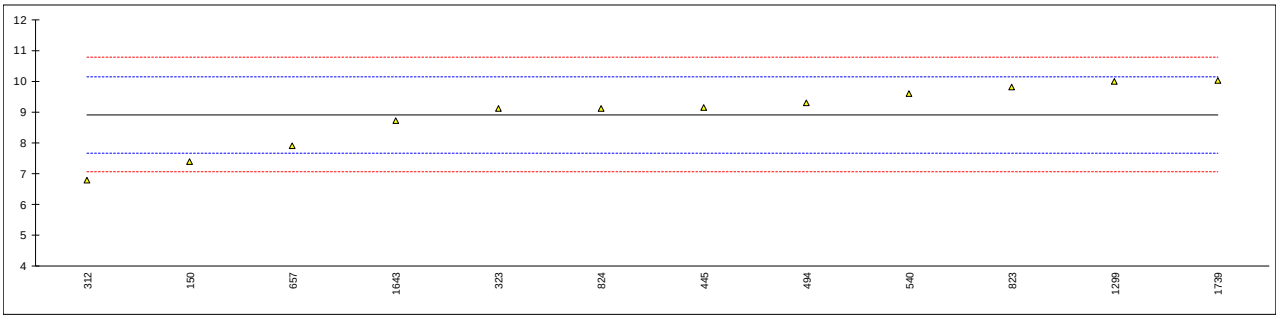
lab	method	value	mark	z(targ)	remarks
120	EN14538	2.486	D(0.01)	-9.98	
150	EN14538	11.6		-2.29	
171	EN14538	11.7		-2.21	
312	EN14538	21.6	C,D(0.05)	6.14	First reported 18.7
323	EN14538	14.5		0.15	
445	EN14538	15.03		0.60	
494	EN14538	14.7		0.32	
540	EN14538	15.6		1.08	
657	EN14538	14.6		0.24	
823	EN14538	13.2		-0.95	
824	EN14538	15.4		0.91	
1067	EN14538	15.6		1.08	
1161		-----		-----	
1299	EN14538	11.5		-2.38	
1468		-----		-----	
1643	D5185	14.808		0.41	
1739	EN14538	17.93		3.04	
	normality	OK			
	n	13			
	outliers	2			
	mean (n)	14.321			
	st.dev. (n)	1.8695			
	R(calc.)	5.235			
	st.dev.(EN14538:06)	1.1856			
	R(EN14538:06)	3.320			Application range 1-10 mg/kg

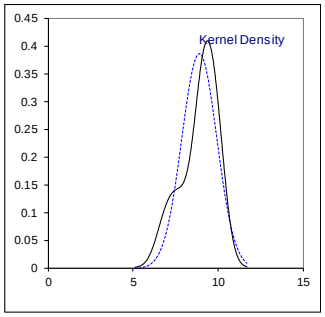




Determination of Phosphorus as P on sample #18067; results in mg/kg

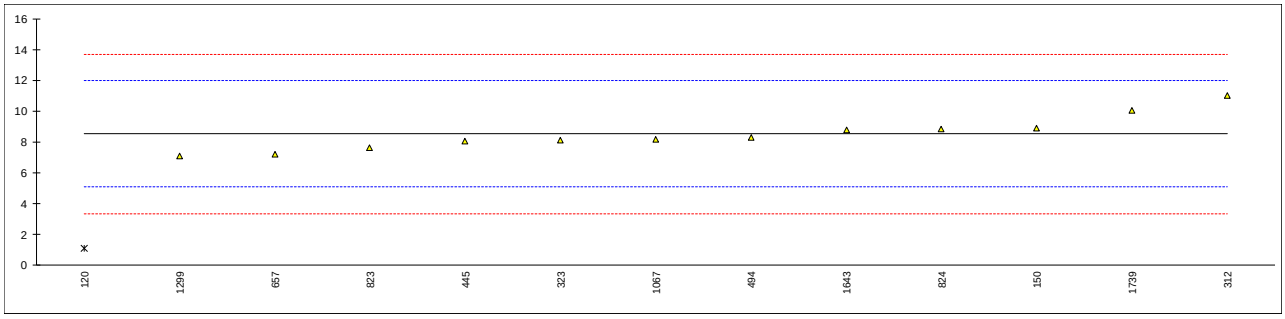
lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150	EN14107	7.4		-2.43	
171	D4951	<0.5		<-13.57	False negative test result?
312	EN14107	6.8		-3.40	
323	EN14107	9.1		0.31	
445	EN14107	9.13		0.36	
494	EN14107	9.3		0.64	
540	EN14107	9.6		1.12	
657	EN14107	7.9		-1.62	
823	D7111	9.8		1.44	
824	EN14107	9.1		0.31	
1067	EN14107	< 4.0		<-7.92	False negative test result?
1161		-----		-----	
1299	EN14107	10.0		1.77	
1468		-----		-----	
1643	D5185	8.716		-0.31	
1739	EN14107	10.02		1.80	
	normality	OK			
	n	12			
	outliers	0	Spike		
	mean (n)	8.905	10.00	Recovery < 89%	
	st.dev. (n)	1.0336			
	R(calc.)	2.894			
	st.dev.(EN14107:03)	0.6196			
	R(EN14107:03)	1.735			Application range 4-20 mg/kg

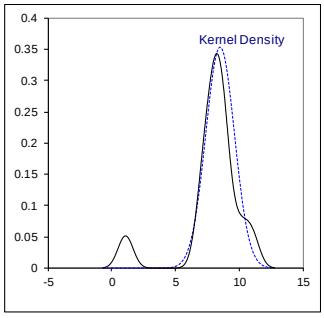




Determination of Potassium as K on sample #18067; results in mg/kg

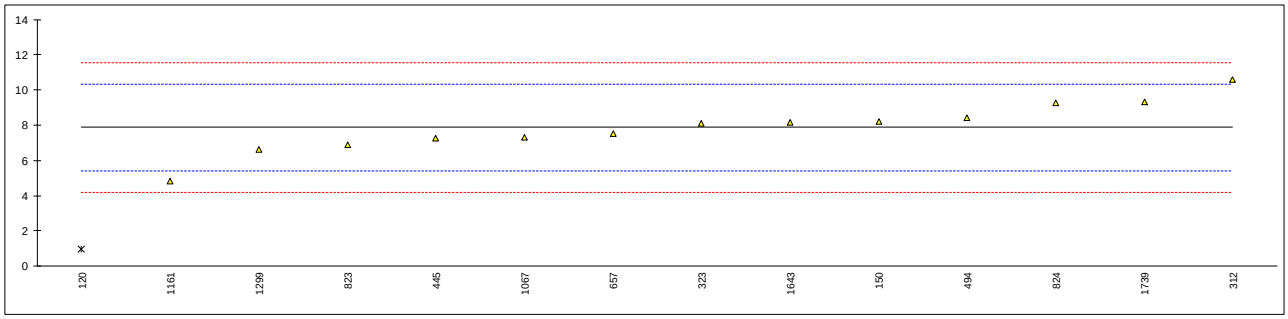
lab	method	value	mark	z(targ)	remarks
120	EN14538	1.095	D(0.01)	-4.31	
150	EN14538	8.9		0.22	
171		-----		-----	
312	EN14538	11.0		1.44	
323	EN14109	8.1		-0.24	
445	EN14538	8.06		-0.26	
494	EN14538	8.3		-0.12	
540		-----		-----	
657	EN14538	7.2		-0.76	
823	EN14538	7.6		-0.53	
824	EN14538	8.86		0.20	
1067	EN14538	8.2		-0.18	
1161	EN14109	<1		<-4.36	False negative test result?
1299	EN14538	7.1		-0.82	
1468		-----		-----	
1643	D5185	8.783		0.16	
1739	EN14538	10.06		0.90	
	normality	suspect			
	n	12			
	outliers	1	Spike		
	mean (n)	8.514	10.28	Recovery <83%	
	st.dev. (n)	1.1279			
	R(calc.)	3.158			
	st.dev.(EN14109:03)	1.7219			
	R(EN14109:03)	4.821			

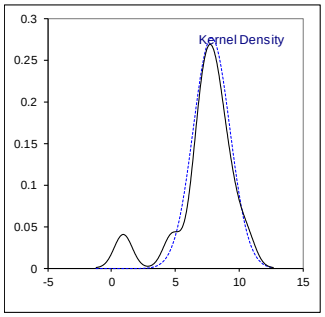




Determination of Sodium as Na on sample #18067; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	EN14538	0.944	D(0.01)	-5.67	
150	EN14538	8.2		0.26	
171		-----		-----	
312	EN14538	10.6		2.22	
323	EN14108	8.1		0.18	
445	EN14538	7.27		-0.50	
494	EN14538	8.4		0.43	
540		-----		-----	
657	EN14538	7.5		-0.31	
823	EN14538	6.9		-0.80	
824	EN14538	9.25		1.12	
1067	EN14538	7.3		-0.47	
1161	EN14108	4.8		-2.52	
1299	EN14538	6.6		-1.05	
1468		-----		-----	
1643	D5185	8.171		0.24	
1739	EN14538	9.34		1.19	
	normality	suspect			
	n	13			
	outliers	1	Spike		
	mean (n)	7.879	10.28	Recovery <77%	
	st.dev. (n)	1.4391			
	R(calc.)	4.029			
	st.dev.(EN14108:03)	1.2240			
	R(EN14108:03)	3.427			

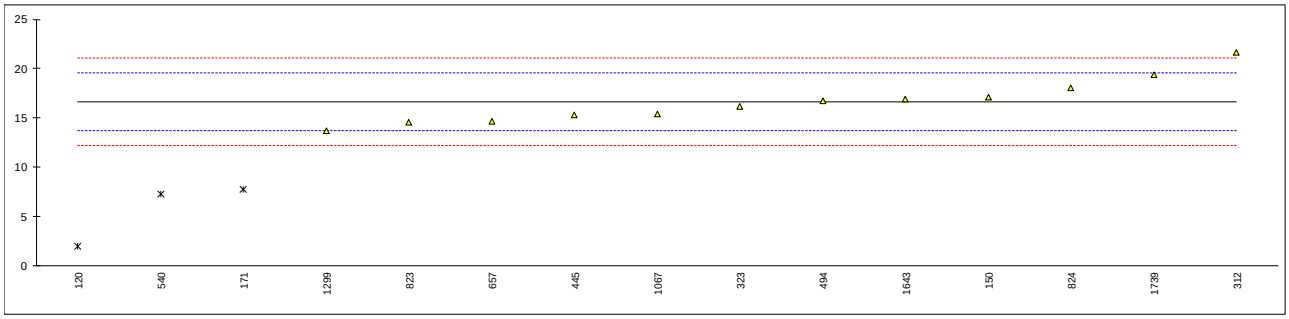


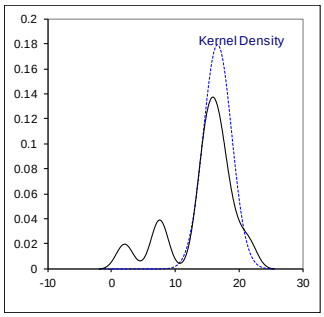


Determination of Sum of Potassium and Sodium as K + Na on sample #18067; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	EN14538	2.039	ex	-9.93	Outliers in K and Na determination
150	EN14538	17.1		0.31	
171	EN14538	7.8	C,D(0.01)	-6.01	First reported 3.9
312	EN14538	21.6		3.36	
323	EN14538	16.2		-0.30	
445	EN14538	15.33		-0.90	
494	EN14538	16.7		0.03	
540	EN14538	7.3	D(0.01)	-6.35	
657	EN14538	14.7		-1.32	
823	EN14538	14.6		-1.39	
824	EN14538	18.1		0.99	
1067	EN14538	15.4		-0.85	
1161		-----		-----	
1299	EN14538	13.7		-2.00	
1468		-----		-----	
1643	D5185	16.954		0.21	
1739	EN14538	19.40		1.87	

normality	OK
n	12
outliers	2 (+1ex)
mean (n)	16.649
st.dev. (n)	2.2295
R(calc.)	6.243
st.dev.(EN14538:06)	1.4717
R(EN14538:06)	4.121





Determination of Particulate Contamination on sample #18068; results in mg/L

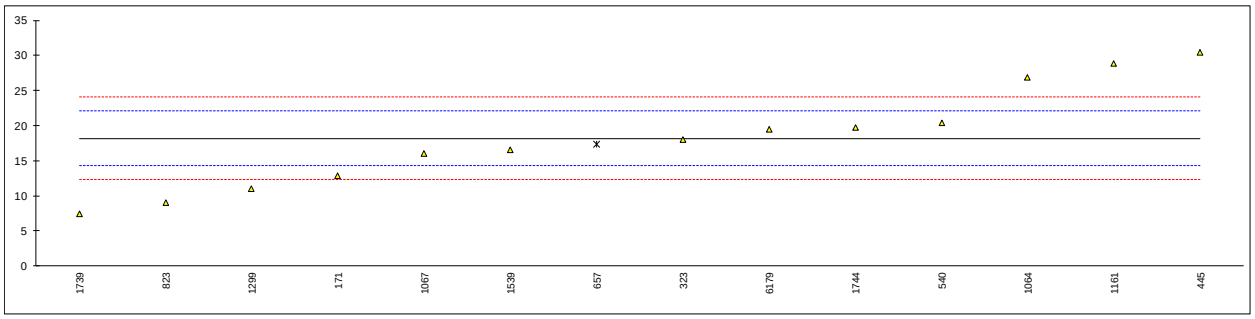
lab	method	value	mark	z(targ)	Vol. filtered	Number of filtrations	remarks
120	D7321	13.6	C	-----	500	1	First reported 0.0136
150		-----		-----	-----	-----	
171	D7321	14.8		-----	400	1	
323		-----		-----	-----	-----	
335		-----		-----	-----	-----	
445	D7321	59.4	False +?	-----	450	2	
494		-----		-----	-----	-----	
540		-----		-----	-----	-----	
657		-----		-----	-----	-----	
823		-----		-----	-----	-----	
1033		-----		-----	-----	-----	
1064		-----		-----	-----	-----	
1067		-----		-----	-----	-----	
1161		-----		-----	-----	-----	
1299		-----		-----	-----	-----	
1539		-----		-----	-----	-----	
1739		-----		-----	-----	-----	
1744		-----		-----	-----	-----	
6179		-----		-----	-----	-----	
	n	3					
	mean (n)	n.a.					

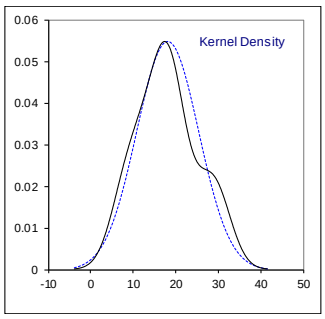
Determination of Total Contamination (EN12662) on sample #18068; results in mg/kg

lab	method	value	mark	z(targ)	Incomplete	Vol. filtered	stopped	
120		-----		-----	-----	500	180	
150		-----		-----	-----	-----	-----	
171	EN12662:2008	12.8		-2.77	-----	300	-----	
323	EN12662:1998	18		-0.10	-----	350	-----	
335		-----		-----	-----	-----	-----	
445	EN12662:2008	30.5		6.31	-----	-----	-----	
494		-----		-----	-----	-----	-----	
540	EN12662:1998	20.35		1.10	-----	400	15	
657	EN12662:2014	17.3	ex	-0.46	-----	286	3	*) see §4.1
823	EN12662:1998	9.0		-4.72	NO	300	1	
1033		-----		-----	-----	-----	-----	
1064	EN12662:2008	26.92		4.47	-----	400	-----	
1067	EN12662:1998	16		-1.13	NO	-----	-----	
1161	EN12662:2008	28.8		5.43	-----	-----	-----	
1299	EN12662:1998	11.0		-3.69	-----	300	-----	
1539	EN12662:2008	16.6		-0.82	-----	-----	-----	
1739	EN12662:1998	7.4		-5.54	-----	-----	-----	
1744	EN12662:2008	19.78		0.81	-----	800	137	
6179	EN12662:2008	19.49		0.66	-----	-----	-----	

				Only 1998	Only 2008
normality	OK			OK	OK
n	13			6	7
outliers	0 (+1ex)	Spike	recovery	0	0
mean (n)	18.203	18.4	<99%	13.625	22.127
st.dev. (n)	7.2926			5.2352	6.6785
R(calc.)	20.419			14.6587	18.6997
st.dev.(EN12662:98 or 08)	1.9503			2.5370	2.5370
R(EN12662:98 or 08)	5.461			6.351	7.749
Compare					
R(EN12662:14)	7.104				

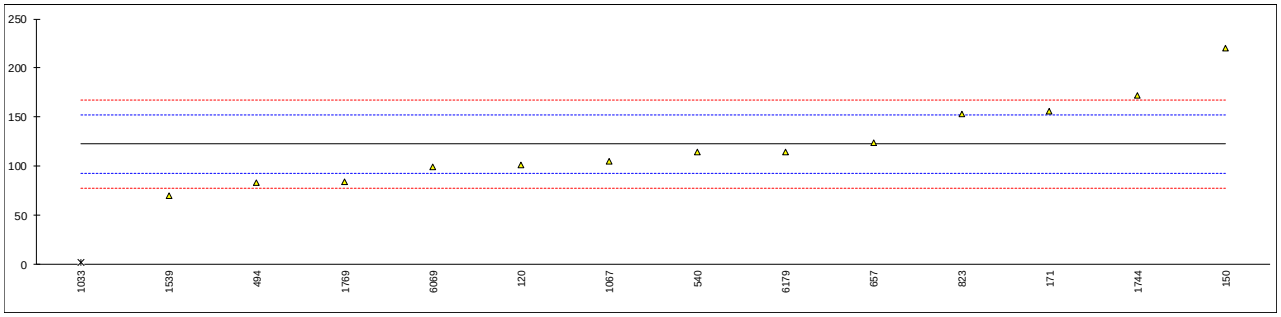
*) EN12662:2014 is not applicable to FAME (B100) according to CEN/TC 19 Committee, instead either method EN12662:1998 or 12662:2008 should be used.

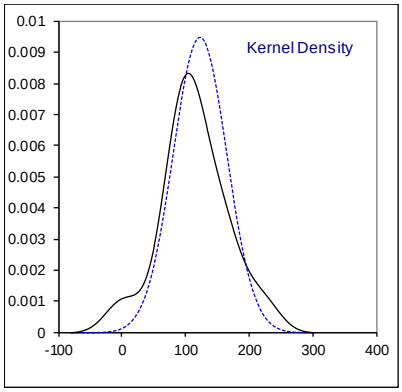




Determination of Filter Blocking Potential by Cold Soak Filterability Test (CSFT) on sample #18069; results in s

lab	method	value	mark	z(targ)	Vol. at time >720sec	remarks
120	D7501	101		-1.45	-----	
150	D7501	220	C	6.50	-----	First reported 269
171	D7501	156		2.22	-----	
323		-----		-----	-----	
494	D7501	83		-2.66	-----	
540	D7501	114.0		-0.58	0	
657	D7501	124		0.08	-----	
823	D7501	153		2.02	300	
1033	IP PM-EA	2.36	ex	-8.05	-----	Reported as FBT result
1067	D7501	105		-1.19	-----	
1161		-----		-----	-----	
1402		-----		-----	-----	
1539	D7501	70		-3.53	-----	
1739		-----		-----	-----	
1744	D7501	172		3.29	-----	
1769	D7501	84.22		-2.58	-----	
6069	D7501	99.21		-1.57	-----	
6179	D7501	114.13		-0.58	-----	
	normality	suspect				
	n	13				
	outliers	0 (+1ex)				
	mean (n)	122.74				
	st.dev. (n)	42.077				
	R(calc.)	117.82				
	st.dev.(D7501:12a)	14.956				
	R(D7501:12a)	41.88				



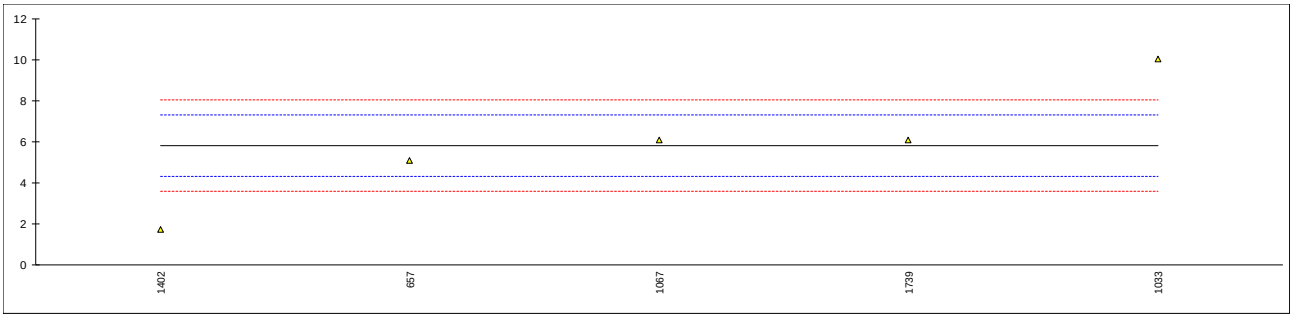


Determination of Filter Blocking Tendency (FBT) on sample #18069

lab	method	value	mark	z(targ)	press. end test (kPa)	vol. pumped	remarks
120		-----		-----	23	-----	
150		-----		-----	-----	-----	
171		-----		-----	-----	-----	
323		-----		-----	-----	-----	
494		-----		-----	-----	-----	
540		-----		-----	-----	-----	
657	IP387-B	5.10		-0.95	105	60	
823		-----		-----	-----	-----	
1033	IP387-B	10.05		5.72	105	-----	
1067	IP387-B	6.08		0.37	105	50	
1161		-----		-----	-----	-----	
1402	IP387-B	1.73		-5.50	105	229	
1539		-----		-----	-----	-----	
1739	IP387-B	6.08		0.37	105	20	
1744		-----		-----	-----	-----	
1769		-----		-----	-----	-----	
6069		-----		-----	-----	-----	
6179		-----		-----	-----	-----	

normality	unknown
n	5
outliers	0
mean (n)	5.81
st.dev. (n)	2.970
R(calc.)	8.31
st.dev.(D2068:17)	0.742
R(D2068:17)	2.08

Lab 1033 also reported 2.36 (IP-PM-EA) as an FBT result



APPENDIX 2

Number of participants per country for sample #18065

1 lab in	ARGENTINA
1 lab in	AUSTRIA
1 lab in	BELGIUM
1 lab in	BRAZIL
5 labs in	COLOMBIA
1 lab in	COTE D'IVOIRE
1 lab in	EQUATORIAL GUINEA
3 labs in	FRANCE
1 lab in	GERMANY
2 labs in	GREECE
1 lab in	HONG KONG
1 lab in	MEXICO
2 labs in	NETHERLANDS
1 lab in	PERU
1 lab in	PHILIPPINES
1 lab in	POLAND
1 lab in	PORTUGAL
1 lab in	SINGAPORE
1 lab in	SOUTH AFRICA
2 labs in	SOUTH KOREA
1 lab in	SPAIN
1 lab in	SWEDEN
1 lab in	TURKEY
2 labs in	UNITED KINGDOM
3 labs in	UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C = final test result after checking of first reported suspect test result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
E = probably an error in calculations
U = test result probably reported in a different unit
W = test result withdrawn on request of participant
ex = test result excluded from statistical evaluation
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported
SDS = Safety Data Sheet

Literature:

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11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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13 Analytical Methods Committee Technical Brief, No 4 January 2001
14 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst 2002, 127, 1359-1364, (2002)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), 165-172, (1983)
16 Horwitz, W and Albert, R, J. AOAC Int, 79, 3, 589, (1996)
17 Letter of CEN: CEN/TC 19 explanation on total contamination test result and applicability for FAME, dated 16-9-2015 and issued by Ortwin Costenoble on behalf of Liesbeth Jansen (CEN/TC 19 Chairman) and Nigel Elliot (CEN/TC 19/WG 24 Convenor)