Results of Proficiency Test
Biodiesel 100% FAME (B100)
May 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Correctors: ing. A.S. Noordman - de Neef & ing. G.A. Oosterlaken - Buijs
Report no.: iis19G02

July 2019

CONTENTS

Appendices:

1 INTRODUCTION

Since 1991, the Institute for Interlaboratory Studies (iis) organizes every year proficiency tests (PT) for Fatty Acid Methyl Esters (FAME) used as Biodiesel B100. Since 2008 the proficiency test Biodiesel B100 is organized twice per year. During the annual proficiency testing program of 2018/2019, it was decided to continue with the proficiency tests on Biodiesel B100 in accordance with the latest applicable version of ASTM D6751 and EN14214:12+A2:2019.
The number of participants per proficiency test of Biodiesel B100 are: 46 laboratories in 23 countries for the main round (iis19G02), 24 laboratories in 13 countries for the round on Metals (iis19G02M), 30 laboratories in 17 countries for the Total Contamination determination (iis19G02TC) and 21 laboratories in 10 countries for the Cold Soak Test (iis19G02CST).
In this interlaboratory study in total 48 laboratories from 24 different countries registered for participation. See appendix 2 for the number of participants per country for sample #19065 (main sample). In this report, the results of the first 2019 Biodiesel B100 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). In this proficiency test on Biodiesel B100, a sample of Rapeseed Methyl Ester was used. Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 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
#19065	1.5	For regular analyses
#19066	0.1	Analysis of metals
#19067	1	Total Contamination
#19068	0.5	Cold Soak Test

Table 1: four different Biodiesel B100 samples used in iis19G02

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/IEC17043:2010 (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 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 June 2018 (iis-protocol, version 3.5). 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 approximately 200L of Biodiesel B100 (RME) was obtained from a European producer.

Sample #19065 - main sample and sample #19068 – Cold Soak Test

From the above batch, after homogenization, 54 amber glass bottles of 1L and 54 amber glass bottles of 0.5L for the main round were filled and labelled #19065. Also, 25 amber glass bottles of 0.5L for the Cold Soak Test round were filled and labelled #19068. The homogeneity of the subsamples #19065 and #19068 were 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 1	883.23
sample 2	883.23
sample 3	883.22
sample 4	883.23
sample 5	883.22
sample 6	883.22
sample 7	883.22
sample 8	883.22

Table 2: homogeneity test results of subsamples #19065 and #19068

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility 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 * R (ref. test method)	0.15

Table 3: evaluation of the repeatability of subsamples #19065 and #19068

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, the homogeneities of the subsamples of #19065 and #19068 were assumed.

Biodiesel B100 #19066 - Metals

From the remaining batch approximately 4.4 kg was separated and Phosphorus, Sodium and Potassium were added. After homogenization, out of the batch 43 HDPE bottles of 0.1L were filled and labelled #19066.
The homogeneity of the subsamples of #19066 was checked by determination of Sodium in accordance with EN14538 on 8 stratified randomly selected samples:

	Sodium in mg/kg
sample #19066-1	9.6
sample #19066-2	9.3
sample #19066-3	9.3
sample #19066-4	9.3
sample #19066-5	9.1
sample #19066-6	9.6
sample #19066-7	9.3
sample #19066-8	9.2

Table 4: homogeneity test results of subsamples #19066

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the reference test method, in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Sodium in mg/kg
r (observed)	0.5
reference test method	EN14108:03
0.3 * R (reference method)	1.1

Table 5: evaluation of repeatability of subsamples #19066

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #19066 was assumed.

Biodiesel B100 #19067 – Total Contamination

Into 36 amber glass bottles, 1 ml of a freshly prepared and homogenized stock solution of approximately 21 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. Each bottle was filled with 1 liter Biodiesel B100. The bottles were labelled #19067.

Depending on the registration of the participant, one 1 liter bottle and one 0.5 liter bottle both labelled #19065, one 100mL bottle labelled #19066, one 1 liter bottle labelled #19067 and/or one 0.5 liter bottle, labelled #19068, were sent to each of the participating laboratories on April 10, 2019. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of the Biodiesel B100 packed in the amber glass bottles and HDPE bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The tests methods in accordance with the requirements of EN14214:12+A2:19 and/or ASTM D6751:18 are listed in table below and used for the scope of this PT.

Parameter	EN14214:12+A2:19	Parameter	ASTM D6751:18
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		
Kinematic Viscosity at 40°C	ISO3104	Kinematic Viscosity at 40°C	ASTM D445
Oxidation Stability	EN14112	Oxidation Stability	EN15751
Sulfated Ash	ISO3987	Sulfated Ash	ASTM D874
Sulfur	ISO20846	Sulfur	ASTM D5453
Water	ISO12937	Water and Sediment	ASTM D2709
Cetane Number	EN5165	Cetane Number	ASTM D613
		Derived Cetane Number	ASTM D7668
Calcium + Magnesium	EN14538	Calcium + Magnesium	EN14538
Phosphorus	EN14107	Phosphorus	ASTM D4951
Potassium	EN14109	Sum of Potassium + Sodium	EN14538
Sodium	EN14108		
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+A2:19 and/or ASTM D6751:18.

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 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 appropriate 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 reanalyzes). Additional or corrected test results are used for data analysis and original test 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 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 June 2018 (iis-protocol, version 3.5).

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.

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. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not 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 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, EN or ISO 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 target 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 according to:

$z_{(target)}$ = (test result - average of PT) / 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 no problems were encountered during the dispatch of the samples.
For the main round: one participant reported test results after the final reporting date and three other participants did not report any test results at all.
For the Metals in Biodiesel PT: one participant reported the test results after the final reporting date and three other participants did not report any test results at all.
For the Total Contamination PT: none of the participants reported the test results after the final reporting date, but five participants did not report any test results at all.
For the Cold Soak Test in Biodiesel PT: one participant reported the test results after the final reporting date and one other participant did not report any test results at all.
Finally, in total 45 participants reported in total 753 numerical results. Observed were 30 outlying results, which is 4.0%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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.

4.1 EVALUATION PER SAMPLE AND PER TEST

In this section, the reported test results are discussed per sample and per test. The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

In the iis PT reports, ASTM test methods are referred to with a number e.g. D874 and an added designation for the year that the test method was adopted or revised e.g. D874:13a. If applicable, a designation in the parentheses is added to designate the year of reapproval e.g. D874:13a(2018). In the test results tables of appendix 1 only the test method number and year of adoption will be used.

Sample #19065

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+A2:19.

Acid Number, total (ASTM): This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D664-B:18e2.

Cloud 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 ASTM D2500:17a and EN14214:12+A2:19.

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 and ISO10370:14 (0.1 %M/M). Therefore, no z-scores were calculated.

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

Density at 15°C: 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 ISO12185:96.

Flash Point, PMcc: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D93-C:18 and ISO2719-C:16.

Flash Point, recc: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO3679:15.

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

Kin.Visco. at 40°C: The determination was problematic depending on the test method used. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ISO3104:94, but is in agreement with the requirements of ASTM D445:17.

Oxidation Stability: This determination was not problematic. Two statistical outliers were observed. However, 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. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO3016:94.

Sulfated Ash: All reported test results were near or below the application limit of 0.005%M/M of ASTM D874:13a(2018) and ISO3987:10. Therefore, no z-scores were calculated.

Sulfur: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO20846:11 and ASTM D5453:19.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility 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 z-scores were calculated.

Calorific Value: Five participants submitted a test result for Gross Calorific Value at constant volume. The determination on Gross Calorific Value may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of DIN51900-1:00.

Distillation at 10mm Hg: This determination was not problematic for 80% and 90% recovered. In total three statistical outliers were observed. The calculated reproducibilities for 80% and 90% recovered after rejection of the statistical outliers are in agreement with the requirements of ASTM D1160:18. The calculated reproducibility for 95% recovered is not in agreement with the requirements of ASTM D1160:18.

Methanol: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier 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 EN14105:11 and ASTM D6584:17.

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 D6584:17.

tri-Glycerides: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN14105:11 and ASTM D6584:17.

Free Glycerol: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN14105:11 and ASTM D6584:17.

Total 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 D6584:17.

Total Ester content (FAME): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN14103:11.

Linolenic Acid Methyl Ester: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN14103:11.

Polyunsaturated Methyl Esters: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN15779:09+A1:13.

Sample #19066

Sum Calcium + Magnesium: 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 EN141538:06.

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.

Potassium: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN14109:03.

Sodium: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN14108:03.

Sum Potassium + Sodium: 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 EN14538:06.

Sample #19067

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. Also, the latest version of EN14214:12+A2:19 (February 2019) states that EN12662 version 2008 should be used or EN12662:1998 as alternative. The method EN12662:14 is not mentioned anymore in the specification (see also iis Memo 1903, lit 18). It was therefore decided to exclude the test results which were determined according EN12662:14.

Particulate Contamination: No laboratory reported a test result.

Total Contamination: This determination was problematic. Five test results were excluded from statistical evaluation and two statistical outliers were observed. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN12662:08 or EN12662:98.

Sample #19068

Filter Blocking Potential by Cold Soak test: This determination was very problematic. The reported test results appear to be bimodally divided. Therefore, no z-scores were calculated

Filter Blocking Tendency: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D2068-B: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 participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (e.g. ASTM, EN and ISO test methods) are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acid Value (EN)	mg KOH/g	20	0.38	0.08	0.06
Acid Number, total (ASTM)	mg KOH/g	26	0.36	0.08	0.12
Cloud Point	°C	31	-6.7	2.2	5
Cold Filter Plugging Point (CFPP)	°C	32	-16.2	2.2	3
Carbon Residue (100% FAME)	%M/M	19	<0.1	n.a.	n.a.
Copper Corrosion, 3hrs at 50°C		28	1 (1A)	n.a.	n.a.
Density at 15°C	kg/m^3	41	883.3	0.5	0.5
Flash Point, PMcc	°C	22	149.6	15.3	14.7
Flash Point, recc (ISO3679)	°C	8	168.0	8.7	15
Iodine Value	g I_2/100g	28	113.5	4.3	5
Kinematic Viscosity at 40°C	mm^2/s	36	4.458	0.056	0.045
Oxidation Stability (EN15751)	hours	18	0.97	0.49	0.56

Parameter	unit	n	average	2.8 * sd	R (lit)
Pour Point	°C	24	-38	5	6
Sulfated Ash	%M/M	21	<0.005	n.a.	n.a.
Sulfur	mg/kg	31	2.9	1.3	1.4
Water	mg/kg	42	504	104	154
Water and Sediment	%V/V	12	<0.05	n.a.	n.a.
Calorific Value, Gross	kJ/kg	5	39956	779	400
80% recovered, as AET	°C	5	352.6	1.5	4.6
90% recovered, as AET	°C	5	354.4	4.1	4.6
95% recovered, as AET	°C	4	360.4	5.4	4.6
Methanol	%M/M	21	0.052	0.014	0.015
mono-Glycerides	%M/M	23	0.34	0.11	0.13
di-Glycerides	%M/M	22	0.10	0.05	0.05
tri-Glycerides	%M/M	21	0.04	0.03	0.06
Free Glycerol	%M/M	19	0.002	0.004	0.006
Total Glycerol	%M/M	23	0.11	0.03	0.03
Total Ester Content	%M/M	26	97.70	3.11	4.16
Linolenic Acid Methyl Ester	%M/M	21	9.21	0.53	0.65
Polyunsaturated Methyl Esters	%M/M	8	0.12	0.22	0.27

Table 7: reproducibilities of tests on sample #19065

Parameter	unit	n	average	2.8 * sd	R (lit)
Sum Calcium and Magnesium	mg/kg	18	18.4	6.8	3.9
Phosphorus	mg/kg	17	11.7	3.0	2.3
Potassium	mg/kg	17	10.4	7.4	5.7
Sodium	mg/kg	17	9.4	8.3	3.8
Sum Potassium and Sodium	mg/kg	17	18.8	10.3	4.5

Table 8: reproducibilities of tests on sample #19066

Parameter	unit	n	average	2.8 * sd	R (lit)
Total Contamination (EN12662)	mg/kg	18	19.9	10.0	6.0

Table 9: reproducibility of tests on sample #19067

Parameter	unit	n	average	2.8 * sd	R (lit)
Filter Blocking Potential (CSFT)	s	13	216	380	(92)
Filter Blocking Tendency (FBT)		6	5.53	2.17	1.88

Table 10: reproducibilities of tests on sample #19068, results between brackets: no z-scores were calculated

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 2019 WITH PREVIOUS PTS

	May 2019	October 2018	May 2018	October 2017	May 2017
Type of FAME	Rapeseed	Rapeseed	Rapeseed	Offal-ME	Rapeseed
Number of reporting labs	45	83	39	70	38
Number of test results	753	1332	563	1054	449
Number of statistical outliers	30	33	22	24	11
Percentage outliers	4.0%	2.5%	3.9%	2.3%	2.5%

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 in the following table.

Parameter	May 2019	October 2018	May 2018	October 2017	May 2017
Acid Value (EN)	-	-	-	+/-	n.e.
Acid Number, total (ASTM)	+	+	+	+	+/-
Cloud Point	++	+	+	++	+/-
Cold Filter Plugging Point (CFPP)	+	-	+	-	+
Carbon Residue (100% FAME)	n.e.	n.e.	n.e.	n.e.	n.e.
Density at 15°C	+/-	++	+	++	+
Flash Point, PMcc	+/-	-	-	-	+/-
Flash Point, recc.(ISO3679)	+	+	++	++	n.e.
Iodine Value	+	+/-	-	-	-
Kinematic Viscosity at 40°C	-	-	-	-	++
Oxidation Stability (EN15751)	+	+	++	--	+
Pour Point	+	+	+	+	n.e.
Sulfated Ash	n.e.	n.e.	n.e.	n.e.	n.e.
Sulfur	+/-	+	+	+/-	-
Water	+	+	+	+	++
Calorific Value, Gross	--	+	+	+	n.e.
Distillation	+	-	-	n.e.	(+)
Methanol	+/-	-	-	n.e.	++
mono-Glycerides	+	-	+	n.e.	++
di-Glycerides	+/-	+	+	n.e.	++
tri-Glycerides	++	++	++	n.e.	++
Free Glycerol	+	++	+	+	+
Total Glycerol	+/-	-	+	--	+
Total Ester Content (FAME)	+	+	+	-	n.e.
Linolenic Acid Methyl Ester	+	+	+	+	n.e.
Polyunsaturated Methyl Esters	+	+/-	-	+/-	n.e.
Sum of Calcium and Magnesium	--	-	-	--	-
Phosphorus	-	-	-	-	-
Potassium	-	+/-	+	-	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 (CSFT)	(--)	--	n.e.	--	n.e.
Filter Blocking Tendency (FBT)	-	--	n.e.	-	n.e.

Table 12: comparison determinations against the reference test methods. Data between brackets: no z-scores were calculated

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:

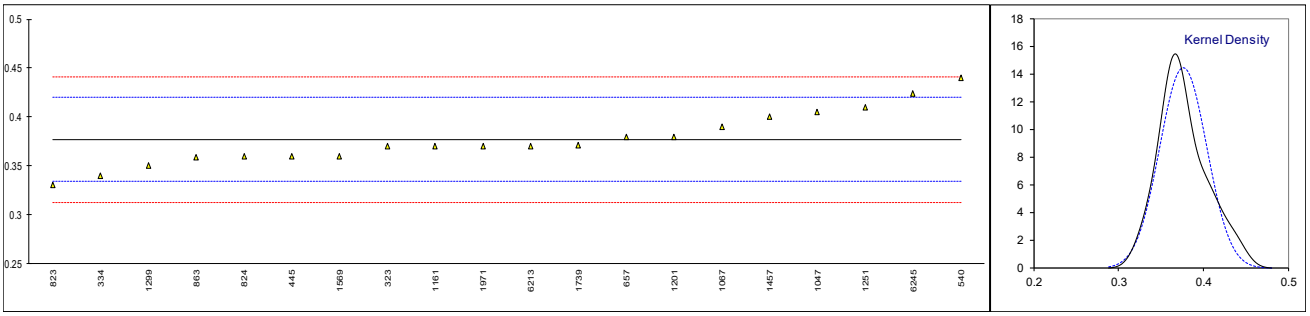
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Acid Value (EN) on sample #19065; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312		-----		-----	
323	EN14104	0.37		-0.32	
334	EN14104	0.34		-1.72	
335		-----		-----	
336		-----		-----	
445	EN14104	0.36		-0.79	
448		-----		-----	
511		-----		-----	
540	EN14104	0.440		2.94	
551		-----		-----	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657	EN14104	0.38		0.14	
823	EN14104	0.33		-2.19	
824	EN14104	0.36		-0.79	
863	EN14104	0.359		-0.84	
1047	EN14104	0.405		1.31	
1067	EN14104	0.39		0.61	
1134		-----		-----	
1161	EN14104	0.370		-0.32	
1199		-----		-----	
1201	EN14104	0.38		0.14	
1227		-----		-----	
1251	EN14104	0.410		1.54	
1299	EN14104	0.35		-1.26	
1457	EN14104	0.400		1.08	
1459		-----		-----	
1494		-----		-----	
1539		-----		-----	
1569	EN14104	0.36		-0.79	
1643		-----		-----	
1739	EN14104	0.371		-0.28	
1769		-----		-----	
1971	EN14104	0.37		-0.32	
1984		-----		-----	
6069		-----		-----	
6170		-----		-----	
6179		-----		-----	
6201		-----		-----	
6213	EN14104	0.37		-0.32	
6245	EN14104	0.424		2.20	

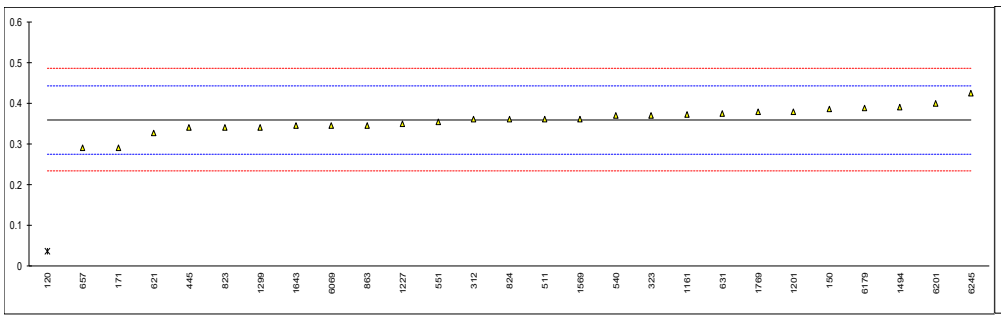
	normality	OK
	n	20
	outliers	0
	mean (n)	0.3769
	st.dev. (n)	0.02762
	R(calc.)	0.0773
	st.dev.(EN14104:03)	0.02143
	R(EN14104:03)	0.06
Compare		
	R(EN14214:12+A2:19)	0.06

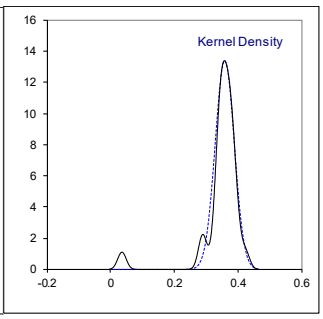


Determination of Acid Number, total (ASTM) on sample #19065; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
120	D664-B	0.037	R(0.01)	-7.68	
150	D664-B	0.386	C	0.64	First reported 0.498
171	D664-B	0.29		-1.65	
240		-----		-----	
312	D974	0.36		0.02	
323	D664-B	0.37		0.26	
334		-----		-----	
335		-----		-----	
336		-----		-----	
445	D664-B	0.34		-0.46	
448		-----		-----	
511	D974	0.36		0.02	
540	D664-B	0.370		0.26	
551	D664-B	0.353		-0.15	
558		-----		-----	
621	D664-B	0.327		-0.77	
631	D974	0.374		0.35	
657	D664-B	0.29		-1.65	
823	D664-B	0.34		-0.46	
824	D664-B	0.36		0.02	
863	D664-B	0.346		-0.31	
1047		-----		-----	
1067		-----		-----	
1134		-----		-----	
1161	D664-B	0.372		0.31	
1199		-----		-----	
1201	D664-B	0.38		0.50	
1227	D664-B	0.35		-0.22	
1251		-----		-----	
1299	D664-B	0.34		-0.46	
1457		-----		-----	
1459		-----		-----	
1494	D664-B	0.3894		0.72	
1539		-----		-----	
1569	D664-B	0.36		0.02	
1643	D664-A	0.344		-0.36	
1739		-----		-----	
1769	D664-B	0.37839		0.46	
1971		-----		-----	
1984		-----		-----	
6069	D664-B	0.3453		-0.33	
6170		-----		-----	
6179	D664-B	0.3885		0.70	
6201	D664-B	0.40		0.97	
6213		-----		-----	
6245	EN14104	0.424		1.55	

normality	suspect
n	26
outliers	1
mean (n)	0.3591
st.dev. (n)	0.02981
R(calc.)	0.0835
st.dev.(D664-B:18e2)	0.04197
R(D664-B:18e2)	0.1175

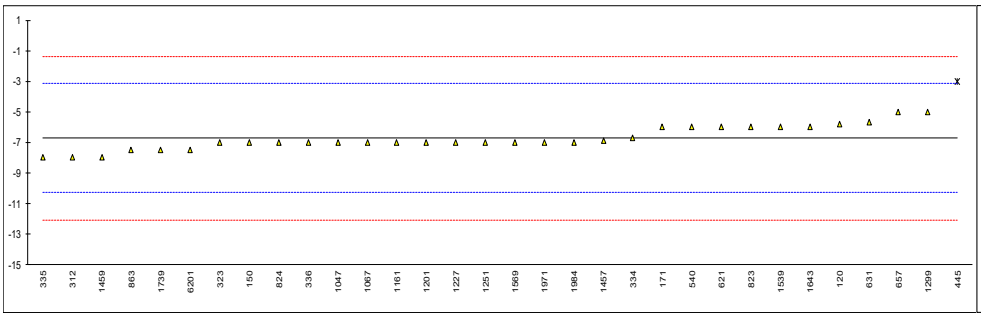


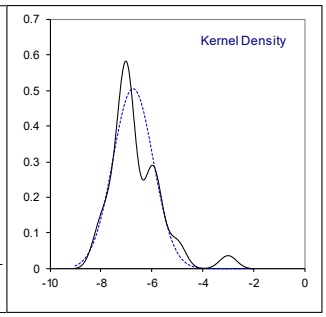


Determination of Cloud Point on sample #19065; results in °C

lab	method	value	mark	z(targ)	remarks
120	D5771	-5.8		0.52	
150	D2500	-7		-0.15	
171	D2500	-6		0.41	
240		-----		-----	
312	D2500	-8		-0.71	
323	D2500	-7		-0.15	
334	D2500	-6.7		0.02	
335	EN23015	-8		-0.71	
336	D2500	-7		-0.15	
445	D2500	-3	R(0.01)	2.09	
448		-----		-----	
511		-----		-----	
540	D2500	-6.0		0.41	
551		-----		-----	
558		-----		-----	
621	D2500	-6.0		0.41	
631	D5772	-5.7		0.58	
657	D2500	-5		0.97	
823	D2500	-6		0.41	
824	D2500	-7		-0.15	
863	EN23015	-7.5		-0.43	
1047	ISO3015	-7		-0.15	
1067	D2500	-7		-0.15	
1134		-----		-----	
1161	D2500	-7		-0.15	
1199		-----		-----	
1201	D2500	-7		-0.15	
1227	D2500	-7		-0.15	
1251	D2500	-7		-0.15	
1299	D2500	-5		0.97	
1457	D2500	-6.9		-0.10	
1459	EN23015	-8.0		-0.71	
1494		-----		-----	
1539	ISO3015	-6		0.41	
1569	EN23015	-7		-0.15	
1643	D2500	-6		0.41	
1739	EN23015	-7.5		-0.43	
1769		-----		-----	
1971	ISO3015	-7		-0.15	
1984	EN23015	-7		-0.15	
6069		-----		-----	
6170		-----		-----	
6179		-----		-----	
6201	EN23015	-7.5		-0.43	
6213		-----		-----	
6245		-----		-----	

	normality	OK
	n	31
	outliers	1
	mean (n)	-6.73
	st.dev. (n)	0.790
	R(calc.)	2.21
	st.dev.(D2500:17a)	1.786
	R(D2500:17a)	5
Compare		
	R(EN14214:12+A2:19)	4

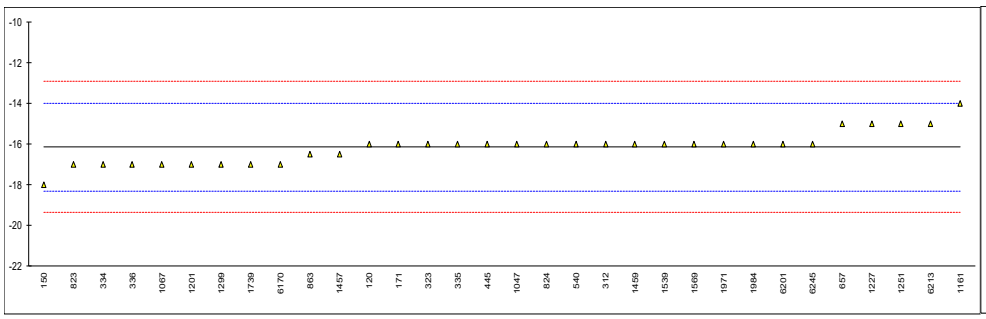


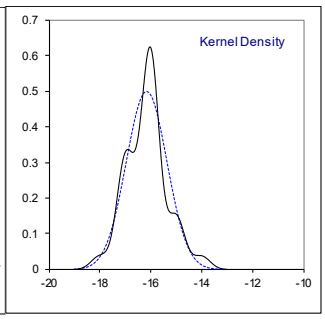


Determination of Cold Filter Plugging Point (CFPP) on sample #19065; results in °C

lab	method	value	mark	z(targ)	remarks
120	D6371	-16.0		0.15	
150	EN116	-18		-1.72	
171	D6371	-16		0.15	
240		-----		-----	
312	D6371	-16		0.15	
323	EN116	-16		0.15	
334	EN116	-17		-0.79	
335	EN116	-16		0.15	
336	EN116	-17		-0.79	
445	EN116	-16		0.15	
448		-----		-----	
511		-----		-----	
540	D6371	-16.0		0.15	
551		-----		-----	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657	IP309	-15		1.08	
823	EN116	-17		-0.79	
824	EN116	-16		0.15	
863	EN116	-16.5		-0.32	
1047	EN116	-16		0.15	
1067	EN116	-17		-0.79	
1134		-----		-----	
1161	EN116	-14		2.01	
1199		-----		-----	
1201	EN116	-17		-0.79	
1227	EN116	-15		1.08	
1251	EN116	-15		1.08	
1299	EN116	-17	C	-0.79	First reported -7
1457	EN116	-16.5		-0.32	
1459	EN116	-16.0		0.15	
1494		-----		-----	
1539	EN116	-16		0.15	
1569	EN116	-16		0.15	
1643		-----		-----	
1739	EN116	-17.0		-0.79	
1769		-----		-----	
1971	EN116	-16		0.15	
1984	EN116	-16		0.15	
6069		-----		-----	
6170	EN116	-17.0		-0.79	
6179		-----		-----	
6201	EN116	-16		0.15	
6213	EN116	-15		1.08	
6245	EN116	-16		0.15	

normality	OK
n	32
outliers	0
mean (n)	-16.16
st.dev. (n)	0.798
R(calc.)	2.23
st.dev.(EN116:15)	1.071
R(EN116:15)	3





Determination of Carbon Residue on 100% FAME on sample #19065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D4530	0.01		-----	
150	D4530	0.0001		-----	
171		-----		-----	
240		-----		-----	
312		-----		-----	
323		-----		-----	
334	ISO10370	0.01	C	-----	First reported 0.22
335		-----		-----	
336		-----		-----	
445	D4530	0.02		-----	
448		-----		-----	
511	D189	0.0118		-----	
540	D4530	<0.10		-----	
551	D4530	< 0.01		-----	
558		-----		-----	
621	D189	< 0.1		-----	
631	D4530	<0.1		-----	
657	D4530	<0.10		-----	
823	D4530	<0.1		-----	
824	D4530	0.01		-----	
863	ISO10370	<0.10		-----	
1047		-----		-----	
1067		-----		-----	
1134		-----		-----	
1161		-----		-----	
1199		-----		-----	
1201	D4530	0.000		-----	
1227	D4530	0		-----	
1251		-----		-----	
1299		-----		-----	
1457	D4530	0.013		-----	
1459		-----		-----	
1494		-----		-----	
1539	EN10370	0.01		-----	
1569		-----		-----	
1643		-----		-----	
1739		-----		-----	
1769		-----		-----	
1971	ISO10370	<0,10		-----	
1984		-----		-----	
6069		-----		-----	
6170		-----		-----	
6179		-----		-----	
6201	D4530	0.015		-----	
6213		-----		-----	
6245		-----		-----	
	n	19			
	mean (n)	<0.1			

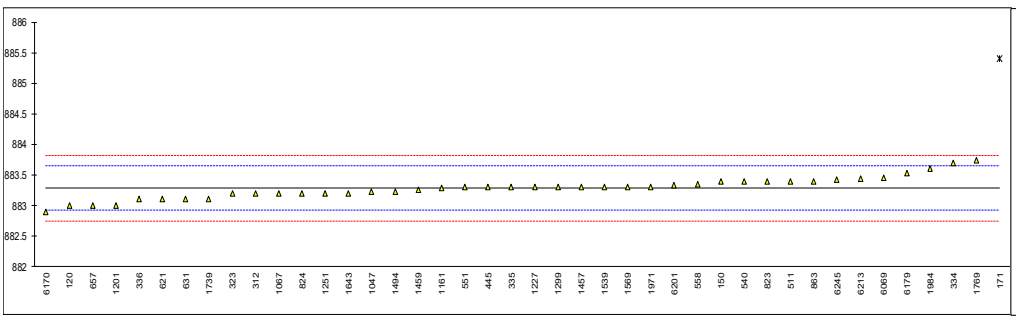
Determination of Copper Corrosion 3 hrs at 50°C on sample #19065

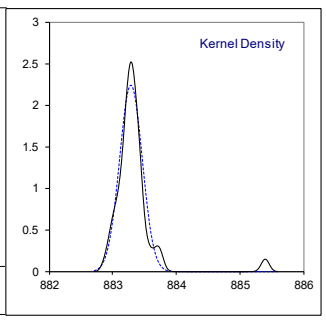
lab	method	value	mark	z(targ)	remarks
120	D130	1A		-----	
150	D130	1A		-----	
171	D130	1a		-----	
240		-----		-----	
312		-----		-----	
323		-----		-----	
334	D130	1A		-----	
335		-----		-----	
336	D130	1		-----	
445	D130	1A		-----	
448		-----		-----	
511	D130	1a		-----	
540	D130	1A		-----	
551	D130	1A		-----	
558		-----		-----	
621	D130	1A		-----	
631	D130	1a		-----	
657	D130	1a		-----	
823	D130	1a		-----	
824	D130	1a		-----	
863	ISO2160	1a		-----	
1047	ISO2160	1		-----	
1067	ISO2160	1A		-----	
1134		-----		-----	
1161	ISO2160	1a		-----	
1199		-----		-----	
1201	D130	1A		-----	
1227	D130	1A		-----	
1251	ISO2160	1a		-----	
1299		1A		-----	
1457	D130	1A		-----	
1459		-----		-----	
1494		-----		-----	
1539	ISO2160	1A		-----	
1569	ISO2160	1a		-----	
1643		-----		-----	
1739	ISO2160	1a		-----	
1769		-----		-----	
1971	ISO2160	1a		-----	
1984		-----		-----	
6069		-----		-----	
6170		-----		-----	
6179		-----		-----	
6201	D130	1A		-----	
6213		-----		-----	
6245		-----		-----	
	n	28			
	mean (n)	1(1A)			

Determination of Density at 15°C on sample #19065; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
120	D4052	883.0		-1.60	
150	D4052	883.4		0.64	
171	D4052	885.4	R(0.01)	11.84	
240		-----		-----	
312	ISO12185	883.2		-0.48	
323	ISO12185	883.2		-0.48	
334	ISO12185	883.7		2.32	
335	ISO12185	883.3		0.08	
336	ISO12185	883.1		-1.04	
445	ISO12185	883.3		0.08	
448		-----		-----	
511	D4052	883.4		0.64	
540	D4052	883.40		0.64	
551	D4052	883.3		0.08	
558	D4052	883.35		0.36	
621	D4052	883.1		-1.04	
631	D4052	883.1		-1.04	
657	D4052	883.0		-1.60	
823	ISO12185	883.4		0.64	
824	ISO12185	883.2		-0.48	
863	ISO12185	883.40		0.64	
1047	ISO12185	883.22		-0.37	
1067	ISO12185	883.2		-0.48	
1134		-----		-----	
1161	ISO12185	883.29		0.02	
1199		-----		-----	
1201	ISO12185	883.0	C	-1.60	First reported 833.0
1227	D4052	883.3	C	0.08	First reported 888.3
1251	ISO12185	883.2		-0.48	
1299	ISO12185	883.3		0.08	
1457	ISO12185	883.3		0.08	
1459	ISO12185	883.25		-0.20	
1494	D4052	883.2215		-0.36	
1539	ISO12185	883.3		0.08	
1569	ISO12185	883.3		0.08	
1643	D4052	883.2		-0.48	
1739	ISO3675	883.10		-1.04	
1769	D4052	883.748		2.58	
1971	D4052	883.3		0.08	
1984	ISO12185	883.6		1.76	
6069	D4052	883.45		0.92	
6170	ISO3675	882.9		-2.16	
6179	D4052	883.524		1.33	
6201	ISO12185	883.33		0.24	
6213	ISO12185	883.44		0.86	
6245	ISO12185	883.42		0.75	

normality	OK
n	41
outliers	1
mean (n)	883.29
st.dev. (n)	0.178
R(calc.)	0.50
st.dev.(ISO12185:96)	0.179
R(ISO12185:96)	0.5

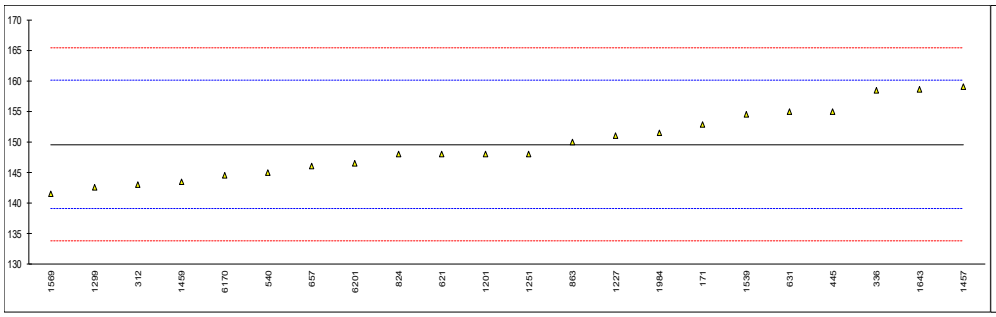


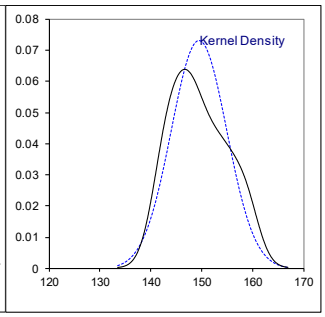


Determination of Flash Point, PMcc on sample #19065; results in °C

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		152.8		0.62	
240		-----		-----	
312	D93-C	143.0		-1.25	
323		-----		-----	
334		-----		-----	
335		-----		-----	
336	ISO2719-C	158.5		1.70	
445	D93-C	155.0		1.04	
448		-----		-----	
511		-----		-----	
540	D93-C	145.00		-0.87	
551		-----		-----	
558		-----		-----	
621	D93-A	148.0		-0.30	
631	D93-A	155.0		1.04	
657	D93-C	146.0		-0.68	
823		-----		-----	
824	D93-C	148.0		-0.30	
863	ISO2719-C	150.0		0.08	
1047		-----		-----	
1067		-----		-----	
1134		-----		-----	
1161		-----		-----	
1199		-----		-----	
1201	D93-C	148.0		-0.30	
1227	D93-C	151		0.27	
1251	D93-C	148.0		-0.30	
1299	D93-C	142.5		-1.35	
1457	D93-C	159.1		1.82	
1459	ISO2719-A	143.5		-1.16	
1494		-----		-----	
1539	ISO2719-C	154.5		0.94	
1569	ISO2719-C	141.5		-1.54	
1643	D93-C	158.55		1.71	
1739		-----		-----	
1769		-----		-----	
1971		-----		-----	
1984	ISO2719-C	151.5		0.37	
6069		-----		-----	
6170	ISO2719-C	144.5		-0.96	
6179		-----		-----	
6201	ISO2719-A	146.5		-0.58	
6213		-----		-----	
6245		-----		-----	

	normality	OK
	n	22
	outliers	0
	mean (n)	149.57
	st.dev. (n)	5.454
	R(calc.)	15.27
	st.dev.(D93-C:18)	5.250
	R(D93-C:18)	14.7
Compare		
	R(ISO2719-C:16)	14.7

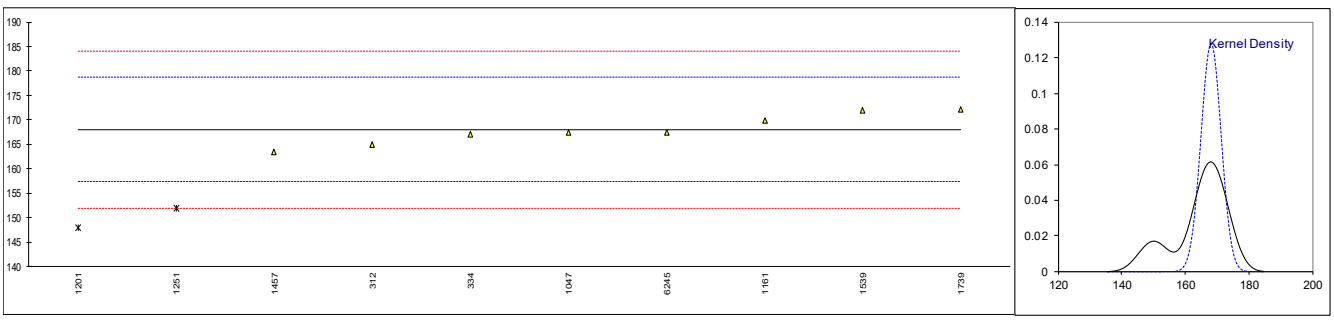




Determination of Flash Point, recc on sample #19065; results in °C

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	ISO3679	165.0	C	-0.57	First reported 146.0
323		-----		-----	
334	ISO3679	167.0		-0.19	
335		-----		-----	
336		-----		-----	
445		-----		-----	
448		-----		-----	
511		-----		-----	
540		-----		-----	
551		-----		-----	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657		-----		-----	
823		-----		-----	
824		-----		-----	
863		-----		-----	
1047	ISO3679	167.35		-0.13	
1067		-----		-----	
1134		-----		-----	
1161	ISO3679	169.9		0.35	
1199		-----		-----	
1201	ISO3679	148	DG(0.01)	-3.74	
1227		-----		-----	
1251	ISO3679	152.0	DG(0.01)	-2.99	
1299		-----		-----	
1457	ISO3679	163.5		-0.85	
1459		-----		-----	
1494		-----		-----	
1539	ISO3679	172.0		0.74	
1569		-----		-----	
1643		-----		-----	
1739	ISO3679	172.16		0.77	
1769		-----		-----	
1971		-----		-----	
1984		-----		-----	
6069		-----		-----	
6170		-----		-----	
6179		-----		-----	
6201		-----		-----	
6213		-----		-----	
6245	ISO3679	167.4		-0.12	

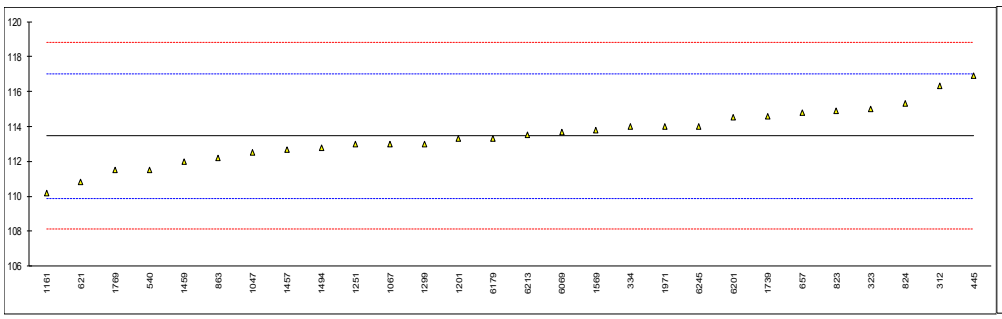
normality	OK
n	8
outliers	2
mean (n)	168.04
st.dev. (n)	3.115
R(calc.)	8.72
st.dev.(ISO3679:15)	5.357
R(ISO3679:15)	15

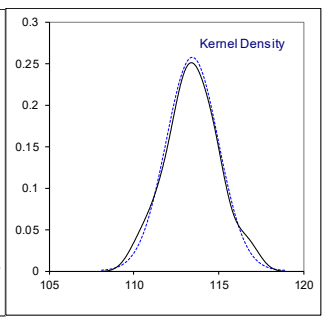


Determination of Iodine Value conform on sample #19065; results in g I_2/100g

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	EN14111	116.3		1.59	
323	EN14111	115		0.86	
334	EN14111	114		0.30	
335		-----		-----	
336		-----		-----	
445	EN14111	116.9		1.92	
448		-----		-----	
511		-----		-----	
540	EN14111	111.5		-1.10	
551		-----		-----	
558		-----		-----	
621	EN14111	110.8		-1.49	
631		-----		-----	
657	EN14111	114.8		0.75	
823	EN14111	114.9		0.80	
824	EN14111	115.3		1.03	
863	EN14111	112.2		-0.71	
1047	EN14111	112.5		-0.54	
1067	EN14111	113		-0.26	
1134		-----		-----	
1161	EN14111	110.2		-1.83	
1199		-----		-----	
1201	EN16300	113.3		-0.09	
1227		-----		-----	
1251	EN16300	112.97		-0.28	
1299	EN14111	113		-0.26	
1457	EN14111	112.7		-0.43	
1459	EN16300	112.0		-0.82	
1494	EN14111	112.77		-0.39	
1539		-----		-----	
1569	EN16300	113.8		0.19	
1643		-----		-----	
1739	EN14111	114.6		0.64	
1769	EN14111	111.497		-1.10	
1971	EN14111	114.0		0.30	
1984		-----		-----	
6069	EN14111	113.68		0.12	
6170		-----		-----	
6179	EN14111	113.33		-0.08	
6201	EN14111	114.5		0.58	
6213	EN14111	113.5		0.02	
6245	EN14111	114.0		0.30	

normality	OK
n	28
outliers	0
mean (n)	113.47
st.dev. (n)	1.549
R(calc.)	4.34
st.dev.(EN14111:03)	1.786
R(EN14111:03)	5

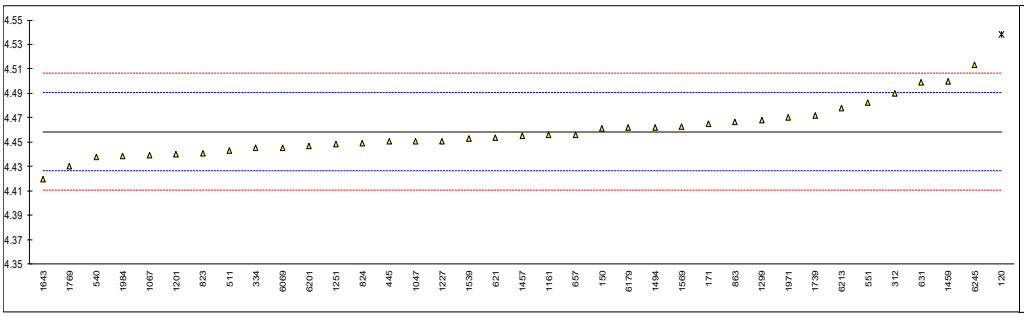


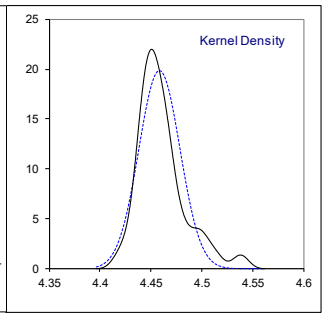


Determination of Kinematic Viscosity at 40°C on sample #19065; results in mm²/s

lab	method	value	mark	z(targ)	remarks
120	D445	4.538	R(0.01)	4.98	
150	D445	4.4612		0.18	
171		4.465		0.41	
240		-----		-----	
312	D445	4.490	C	1.98	First reported 6.095 (at 20°C)
323		-----		-----	
334	ISO3104	4.445		-0.84	
335		-----		-----	
336		-----		-----	
445	IP71	4.4504		-0.50	
448		-----		-----	
511	D445	4.4433		-0.94	
540	D445	4.4375		-1.31	
551	D445	4.482		1.48	
558		-----		-----	
621	D445	4.454		-0.27	
631	D445	4.49896		2.54	
657	D445	4.456		-0.15	
823	ISO3104	4.441		-1.09	
824	ISO3104	4.449		-0.59	
863	ISO3104	4.4665		0.51	
1047	ISO3104	4.451		-0.46	
1067	ISO3104	4.439		-1.21	
1134		-----		-----	
1161	ISO3104	4.4558		-0.16	
1199		-----		-----	
1201	ISO3104	4.440		-1.15	
1227	D445	4.451	C	-0.46	First reported 4.5807
1251	ISO3104	4.448		-0.65	
1299	D445	4.468		0.60	
1457	ISO3104	4.455		-0.21	
1459	D7042	4.50		2.60	
1494	D445	4.4622		0.24	
1539	ISO3104	4.453		-0.34	
1569	ISO3104	4.463		0.29	
1643	D445	4.420		-2.40	
1739	ISO3104	4.4718		0.84	
1769	D445	4.4300		-1.78	
1971	ISO3104	4.470		0.73	
1984	ISO3104	4.43875	C	-1.23	First reported 4.3875
6069	D445	4.4450		-0.84	
6170		-----		-----	
6179	D445	4.462		0.23	
6201	ISO3104	4.447		-0.71	
6213	ISO3104	4.4782		1.24	
6245	ISO3104	4.513		3.42	

	normality	OK
	n	36
	outliers	1
	mean (n)	4.4584
	st.dev. (n)	0.02011
	R(calc.)	0.0563
	st.dev.(ISO3104:94)	0.01599
	R(ISO3104:94)	0.0448
Compare		
	R(D445:17)	0.0999

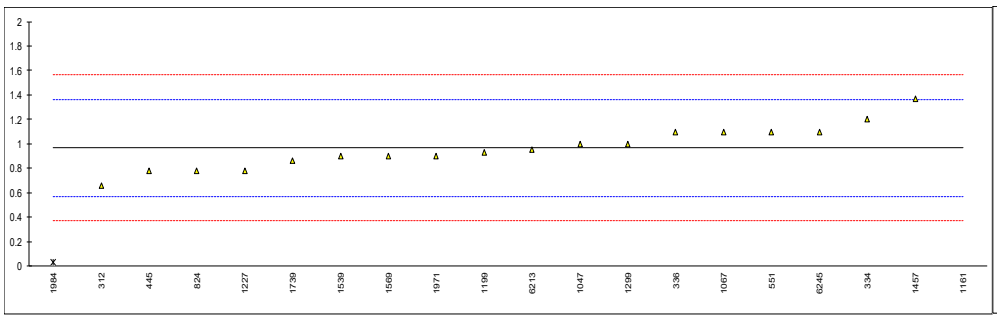


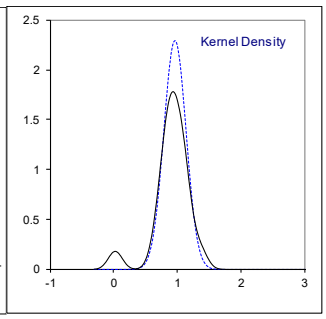


Determination of Oxidation Stability Induction period on sample #19065; results in hours

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	EN15751	0.66		-1.54	
323		-----		-----	
334	EN15751	1.2		1.17	
335		-----		-----	
336	EN15751	1.1		0.67	
445	EN14112	0.78		-0.94	
448		-----		-----	
511		-----		-----	
540		-----		-----	
551	EN14112	1.10		0.67	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657	EN15751	<0.1		<-4.36	Possibly a false negative test result?
823		-----		-----	
824	EN15751	0.78	C	-0.94	First reported 0.04
863		-----		-----	
1047	EN15751	1.0		0.16	
1067	EN14112	1.1		0.67	
1134		-----		-----	
1161	EN14112	6	C,R(0.01)	25.31	First reported 10
1199	EN14112	0.93		-0.19	
1201		-----		-----	
1227	EN14112	0.78	C	-0.94	First reported 0.03
1251		-----		-----	
1299	EN15751	1.0		0.16	
1457	EN14112	1.37	C	2.03	First reported 2.37
1459		-----	W	-----	Test result withdrawn, reported 0.05
1494		-----		-----	
1539	EN14112	0.9		-0.34	
1569	EN15751	0.9		-0.34	
1643		-----		-----	
1739	EN14112	0.86		-0.54	
1769		-----		-----	
1971	EN14112	0.9		-0.34	
1984	EN15751	0.03	R(0.01)	-4.71	
6069		-----		-----	
6170		-----		-----	
6179		-----		-----	
6201		-----		-----	
6213	EN14112	0.95		-0.09	
6245	EN14112	1.10		0.67	

normality	OK
n	18
outliers	2
mean (n)	0.967
st.dev. (n)	0.1741
R(calc.)	0.488
st.dev.(EN15751:14)	0.1989
R(EN15751:14)	0.557

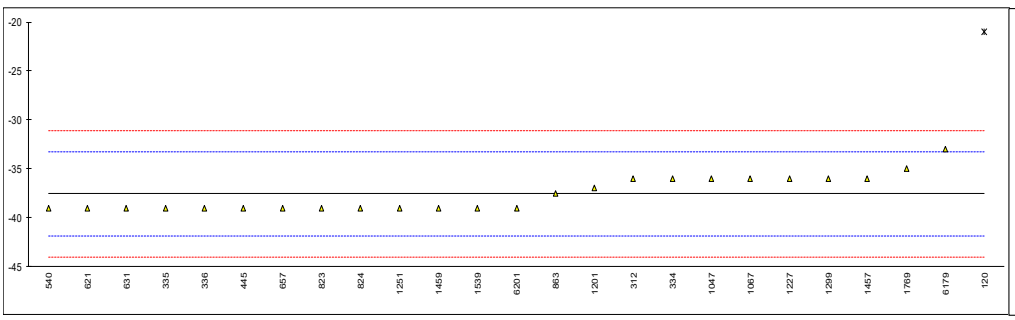


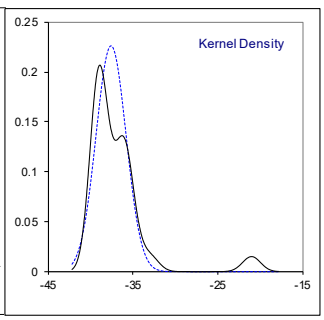


Determination of Pour Point on sample #19065; results in °C

lab	method	value	mark	z(targ)	remarks
120	D5949	-21	R(0.01)	7.73	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	D5950	-36		0.73	
323		-----		-----	
334	ISO3016	-36		0.73	
335	ISO3016	-39		-0.67	
336	ISO3016	-39		-0.67	
445	D97	-39		-0.67	
448		-----		-----	
511		-----		-----	
540	D5950	-39.0		-0.67	
551		-----		-----	
558		-----		-----	
621	D97	-39.0		-0.67	
631	D5950	-39		-0.67	
657	D97	-39		-0.67	
823	ISO3016	-39		-0.67	
824	ISO3016	-39		-0.67	
863	ISO3016	-37.5		0.03	
1047	ISO3016	-36		0.73	
1067	ISO3016	-36		0.73	
1134		-----		-----	
1161		-----		-----	
1199		-----		-----	
1201	ISO3016	-37		0.26	
1227	D97	-36		0.73	
1251	ISO3016	-39		-0.67	
1299	D97	-36.0		0.73	
1457	ISO3016	-36		0.73	
1459		-39.0		-0.67	
1494		-----		-----	
1539	ISO3016	-39		-0.67	
1569	D97	<-39		-----	
1643		-----		-----	
1739		-----		-----	
1769	D5950	-35		1.20	
1971		-----		-----	
1984		-----		-----	
6069		-----		-----	
6170		-----		-----	
6179	D97	-33		2.13	
6201	ISO3016	-39		-0.67	
6213		-----		-----	
6245		-----		-----	

normality	OK
n	24
outliers	1
mean (n)	-37.6
st.dev. (n)	1.77
R(calc.)	4.9
st.dev.(ISO3016:94)	2.14
R(ISO3016:94)	6





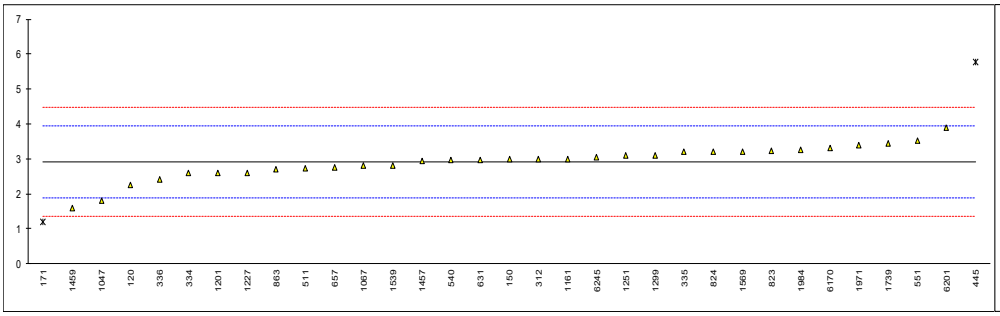
Determination of Sulfated Ash on sample #19065; results in %M/M

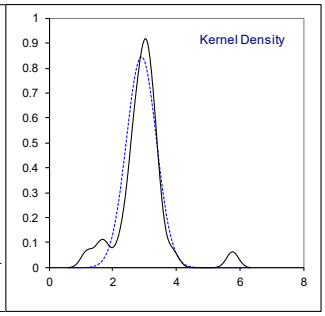
lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150	D874	<0.005		-----	
171	D874	<0.005		-----	
240		-----		-----	
312		-----		-----	
323		-----		-----	
334	ISO3987	0.002		-----	
335		-----		-----	
336		-----		-----	
445	D874	<0.005		-----	
448		-----		-----	
511	D874	0		-----	
540	ISO3987	<0.005		-----	
551	D874	< 0.001		-----	
558		-----		-----	
621	D874	< 0.005		-----	
631	D874	0.00247		-----	
657	D874	0.005		-----	
823	D874	<0.005		-----	
824	D874	0.001		-----	
863	ISO3987	<0.005		-----	
1047	ISO3987	<0,005		-----	
1067	ISO3987	0.000		-----	
1134		-----		-----	
1161	ISO3987	0.006		-----	
1199		-----		-----	
1201	D874	0		-----	
1227		-----		-----	
1251	ISO3987	0		-----	
1299	ISO3987	0.0008		-----	
1457		-----		-----	
1459	ISO3987	0.000		-----	
1494		-----		-----	
1539		-----		-----	
1569	D874	0.010		-----	
1643		-----		-----	
1739	ISO3987	0,000		-----	
1769		-----		-----	
1971	ISO3987	<0,005		-----	
1984		-----		-----	
6069		-----		-----	
6170		-----		-----	
6179		-----		-----	
6201		-----		-----	
6213		-----		-----	
6245		-----		-----	
	n	21			
	mean (n)	<0.005			

Determination of Sulfur on sample #19065; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	D2622	2.24		-1.31	
150	ISO20846	2.9814		0.13	
171	D5453	1.2	R(0.05)	-3.32	
240		-----		-----	
312	ISO20846	3.0		0.16	
323		-----		-----	
334	ISO20846	2.6		-0.61	
335	ISO20846	3.2		0.55	
336	ISO20846	2.4		-1.00	
445	ISO20846	5.78	R(0.01)	5.54	
448		-----		-----	
511	D5453	2.74		-0.34	
540	D5453	2.97		0.10	
551	D5453	3.51		1.15	
558		-----		-----	
621	D4294	< 20		-----	
631	D4294	2.977	C	0.12	First reported 5.977
657	D5453	2.762		-0.30	
823	ISO20846	3.24		0.63	
824	ISO20846	3.2		0.55	
863	ISO20846	2.7		-0.42	
1047	ISO20846	1.8		-2.16	
1067	ISO20846	2.8		-0.23	
1134		-----		-----	
1161	ISO20846	3.00		0.16	
1199	ISO20884	<5.0		-----	
1201	ISO20846	2.6		-0.61	
1227	D5453	2.6		-0.61	
1251	ISO20846	3.1		0.36	
1299	ISO20846	3.1		0.36	
1457	ISO20846	2.95		0.06	
1459	ISO20884	1.6		-2.55	
1494		-----		-----	
1539	ISO20846	2.8		-0.23	
1569	ISO20846	3.2		0.55	
1643		-----		-----	
1739	ISO13032	3.45		1.03	
1769		-----		-----	
1971	ISO20846	3.4		0.94	
1984	ISO20846	3.25		0.65	
6069		-----		-----	
6170	ISO20846	3.3		0.74	
6179		-----		-----	
6201	ISO20846	3.9		1.90	
6213		-----		-----	
6245	ISO20846	3.04		0.24	

	normality	suspect
	n	31
	outliers	2
	mean (n)	2.916
	st.dev. (n)	0.4720
	R(calc.)	1.322
	st.dev.(ISO20846:11)	0.5167
	R(ISO20846:11)	1.447
Compare		
	R(D5453:19)	1.294

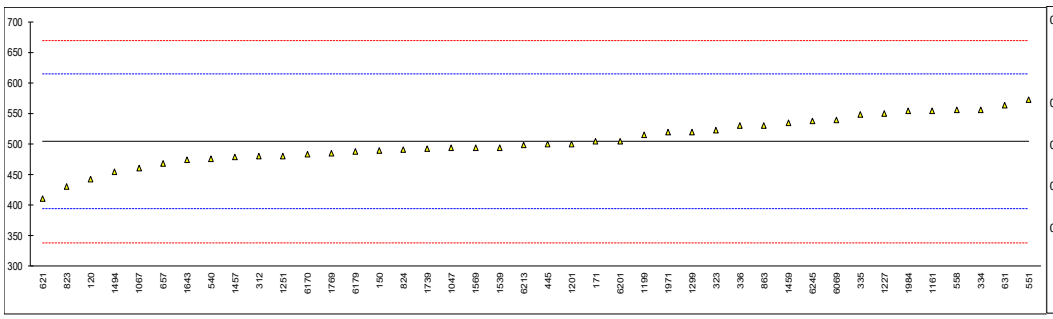


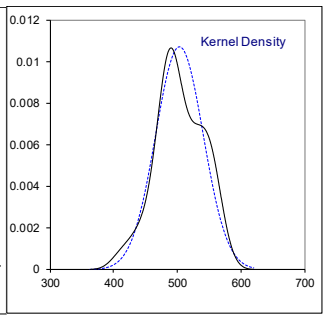


Determination of Water on sample #19065; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
120	E203	441.7		-1.13	
150	D6304-A	488.9	C	-0.27	First reported 0.04889 mg/kg
171	D6304-A	504		0.00	
240		-----		-----	
312	ISO12937	480		-0.43	
323	ISO12937	523		0.35	
334	ISO12937	556		0.94	
335	ISO12937	548		0.80	
336	ISO12937	530		0.47	
445	ISO12937	500		-0.07	
448		-----		-----	
511		-----		-----	
540	ISO12937	475.0		-0.53	
551	D6304-A	573		1.25	
558	D6304	555		0.93	
621	ISO12937	411		-1.69	
631	D6304-B	562.7		1.07	
657	D6304-A	468		-0.65	
823	ISO12937	431		-1.32	
824	ISO12937	490		-0.25	
863	ISO12937	530		0.47	
1047	ISO12937	493		-0.20	
1067	ISO12937	460		-0.80	
1134		-----		-----	
1161	ISO12937	553.738		0.90	
1199	ISO12937	515		0.20	
1201	ISO12937	500		-0.07	
1227		549		0.82	
1251	ISO12937	480	C	-0.43	First reported 297
1299	ISO12937	520		0.29	
1457	ISO12937	478		-0.47	
1459	ISO12937	534	C	0.54	First reported 650
1494	ISO12937	454.71		-0.89	
1539	ISO12937	494		-0.18	
1569	D6304-C	493		-0.20	
1643	ISO6296	474		-0.54	
1739	ISO12937	492.52		-0.21	
1769	ISO12937	483.950		-0.36	
1971	ISO12937	519.7		0.29	
1984	ISO12937	553.5		0.90	
6069	E203	538.5		0.63	
6170	ISO12937	483.9		-0.36	
6179	ISO12937	488.2		-0.29	
6201	ISO12937	504.1		0.00	
6213	ISO12937	498.4		-0.10	
6245	ISO12937	538		0.62	

normality	OK
n	42
outliers	0
mean (n)	503.96
st.dev. (n)	37.314
R(calc.)	104.48
st.dev.(ISO12937:00)	55.137
R(ISO12937:00)	154.38





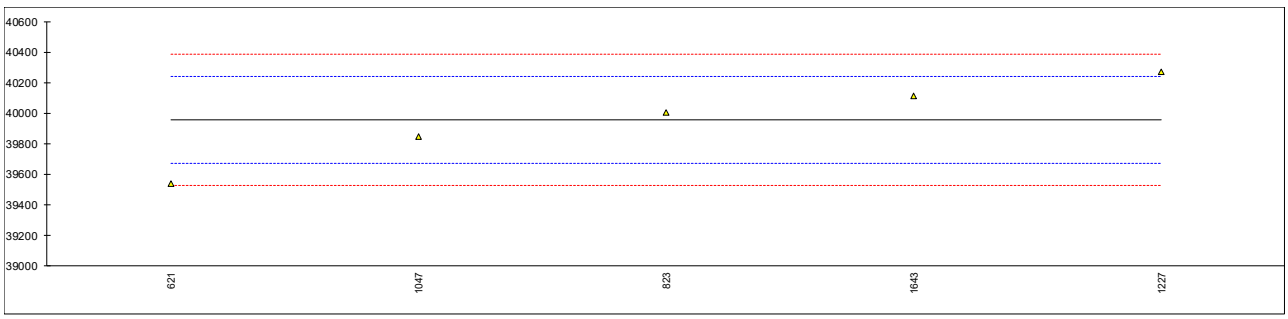
Determination of Water and Sediment on sample #19065; results in %V/V

lab	method	value	mark	z(targ)	remarks
120	D2709	0		-----	
150		-----		-----	
171	D2709	<0.01		-----	
240		-----		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
335		-----		-----	
336		-----		-----	
445		-----		-----	
448		-----		-----	
511	D2709	0		-----	
540	D2709	<0.05		-----	
551	D2709	< 0.05		-----	
558		-----		-----	
621	D2709	< 0.01		-----	
631	D2709	0.025		-----	
657	D2709	<0.005		-----	
823	D2709	0.00		-----	
824	D2709	0		-----	
863		-----		-----	
1047		-----		-----	
1067		-----		-----	
1134		-----		-----	
1161		-----		-----	
1199		-----		-----	
1201	D2709	0		-----	
1227		-----		-----	
1251		-----		-----	
1299		<0.01		-----	
1457		-----		-----	
1459		-----		-----	
1494		-----		-----	
1539		-----		-----	
1569		-----		-----	
1643		-----		-----	
1739		-----		-----	
1769		-----		-----	
1971		-----		-----	
1984		-----		-----	
6069		-----		-----	
6170		-----		-----	
6179		-----		-----	
6201		-----		-----	
6213		-----		-----	
6245		-----		-----	
	n	12			
	mean (n)	<0.05			

Determination of Calorific Value on sample #19065; results in kJ/kg

lab	method	Gross at const. vol.	mark	z(targ)	Net at const. vol.	Net at const. press
120		-----		-----	-----	-----
150		-----		-----	-----	-----
171		-----		-----	-----	-----
240		-----		-----	-----	-----
312		-----		-----	-----	-----
323		-----		-----	-----	-----
334		-----		-----	-----	-----
335		-----		-----	-----	-----
336		-----		-----	-----	-----
445		-----		-----	-----	-----
448		-----		-----	-----	-----
511		-----		-----	-----	-----
540		-----		-----	-----	-----
551		-----		-----	-----	-----
558		-----		-----	-----	-----
621	D240	39539		-2.92	-----	38473
631		-----		-----	-----	-----
657		-----		-----	-----	-----
823	D240	40007		0.36	37429	-----
824		-----		-----	-----	-----
863		-----		-----	-----	-----
1047	PN-C-04062	39850		-0.74	-----	-----
1067		-----		-----	-----	-----
1134		-----		-----	-----	-----
1161		-----		-----	-----	-----
1199		-----		-----	-----	-----
1201		-----		-----	-----	-----
1227	DIN51900-2	40268		2.19	-----	-----
1251		-----		-----	-----	-----
1299		-----		-----	-----	-----
1457		-----		-----	-----	-----
1459		-----		-----	-----	-----
1494		-----		-----	-----	-----
1539		-----		-----	-----	-----
1569		-----		-----	-----	-----
1643	D240	40113.6		1.11	37588.5	-----
1739		-----		-----	-----	-----
1769		-----		-----	-----	-----
1971		-----		-----	-----	-----
1984		-----		-----	-----	-----
6069		-----		-----	-----	-----
6170		-----		-----	-----	-----
6179		-----		-----	-----	-----
6201		-----		-----	-----	-----
6213		-----		-----	-----	-----
6245		-----		-----	-----	-----

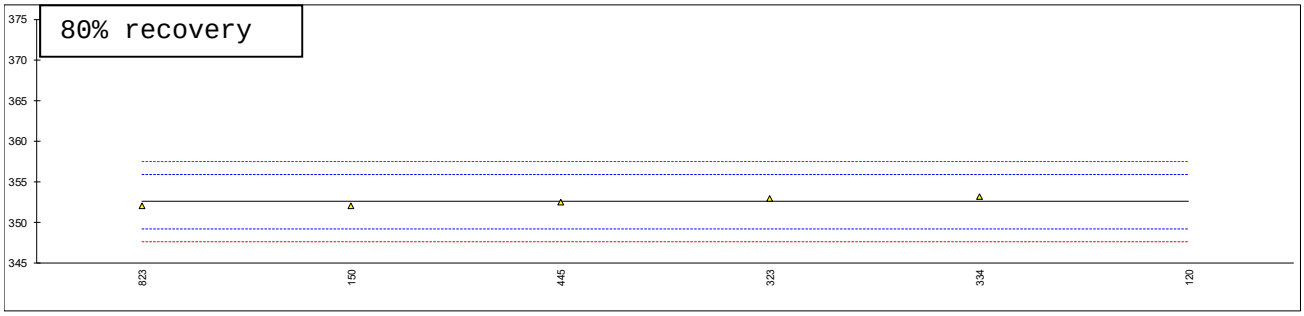
normality	unknown
n	5
outliers	0
mean (n)	39955.5
st.dev. (n)	278.35
R(calc.)	779.4
st.dev.(DIN51900-1:00)	142.86
R(DIN51900-1:00)	400

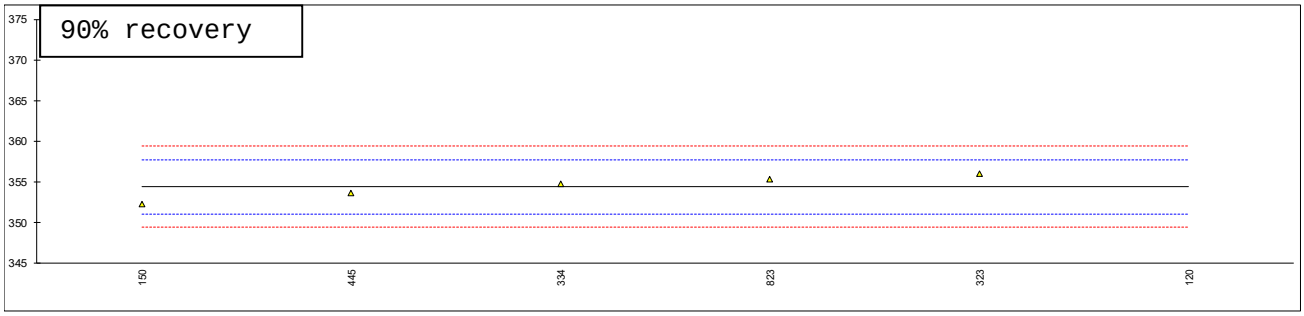


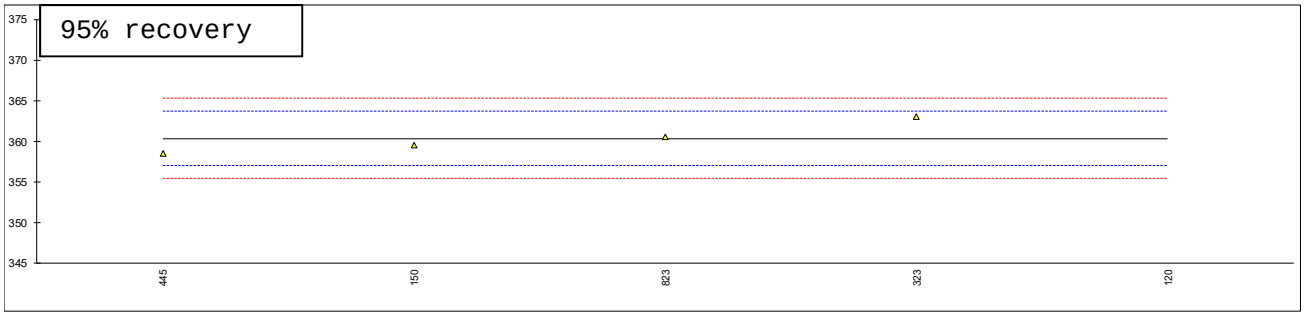
Determination of Distillation at 10 mm Hg, % recovered as AET on sample #19065; results in °C

lab	method	80%rec.	mark	z(targ)	90%rec.	mark	z(targ)	95%rec.	mark	z(targ)
120	D1160	667.5	G(0.01)	190.05	670.6	G(0.01)	190.81	674.3	G(0.01)	189.44
150	D1160	352.1		-0.28	352.3		-1.27	359.5		-0.53
171		-----		-----	-----		-----	-----		-----
240		-----		-----	-----		-----	-----		-----
312		-----		-----	-----		-----	-----		-----
323	D1160	353		0.27	356		0.97	363		1.58
334		353.2		0.39	354.8		0.24	-----		-----
335		-----		-----	-----		-----	-----		-----
336		-----		-----	-----		-----	-----		-----
445	D1160	352.5		-0.04	353.6		-0.48	358.5		-1.13
448		-----		-----	-----		-----	-----		-----
511		-----		-----	-----		-----	-----		-----
540		-----		-----	-----		-----	-----		-----
551		-----		-----	-----		-----	-----		-----
558		-----		-----	-----		-----	-----		-----
621		-----		-----	-----		-----	-----		-----
631		-----		-----	-----		-----	-----		-----
657		-----		-----	-----		-----	-----		-----
823	D1160	352.0		-0.34	355.3		0.54	360.5		0.08
824		-----		-----	-----		-----	-----		-----
863		-----		-----	-----		-----	-----		-----
1047		-----		-----	-----		-----	-----		-----
1067		-----		-----	-----		-----	-----		-----
1134		-----		-----	-----		-----	-----		-----
1161		-----		-----	-----		-----	-----		-----
1199		-----		-----	-----		-----	-----		-----
1201		-----		-----	-----		-----	-----		-----
1227		-----		-----	-----		-----	-----		-----
1251		-----		-----	-----		-----	-----		-----
1299		-----		-----	-----		-----	-----		-----
1457		-----		-----	-----		-----	-----		-----
1459		-----		-----	-----		-----	-----		-----
1494		-----		-----	-----		-----	-----		-----
1539		-----		-----	-----		-----	-----		-----
1569		-----		-----	-----		-----	-----		-----
1643		-----		-----	-----		-----	-----		-----
1739		-----		-----	-----		-----	-----		-----
1769		-----		-----	-----		-----	-----		-----
1971		-----		-----	-----		-----	-----		-----
1984		-----		-----	-----		-----	-----		-----
6069		-----		-----	-----		-----	-----		-----
6170		-----		-----	-----		-----	-----		-----
6179		-----		-----	-----		-----	-----		-----
6201		-----		-----	-----		-----	-----		-----
6213		-----		-----	-----		-----	-----		-----
6245		-----		-----	-----		-----	-----		-----

normality	unknown	unknown	unknown
n	5	5	4
outliers	1	1	1
mean (n)	352.56	354.40	360.38
st.dev. (n)	0.532	1.465	1.931
R(calc.)	1.49	4.10	5.41
st.dev.(D1160:18)	1.657	1.657	1.657
R(D1160:18)	4.64	4.64	4.64


80% recovery
375
370
365
360
355
350
345
823
150
445
323
334
120

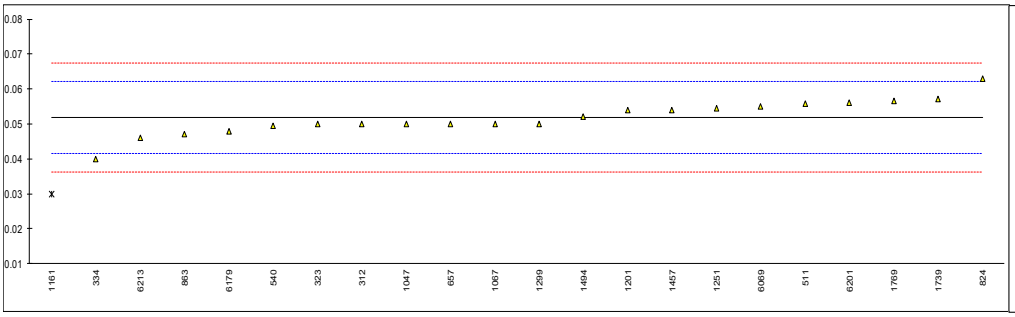

90% recovery
375
370
365
360
355
350
345
150
445
334
823
323
120

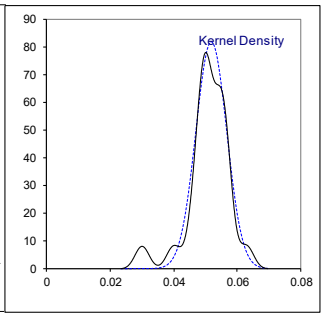

95% recovery
375
370
365
360
355
350
345
445
150
823
323
120

Determination of Methanol on sample #19065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	EN14110	0.05		-0.36	
323	EN14110	0.05		-0.36	
334	EN14110	0.04	C	-2.29	First reported 0.07
335		-----		-----	
336		-----		-----	
445		-----		-----	
448		-----		-----	
511	EN14110	0.0558		0.77	
540	EN14110	0.0495		-0.45	
551		-----		-----	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657	EN14110	0.05		-0.36	
823		-----		-----	
824	EN14110	0.063		2.16	
863	EN14110	0.047		-0.94	
1047	EN14110	0.05		-0.36	
1067	EN14110	0.05		-0.36	
1134		-----		-----	
1161	EN14110	0.030	C,R(0.01)	-4.23	First reported 0.036
1199		-----		-----	
1201	EN14110	0.054		0.42	
1227		-----		-----	
1251	EN14110	0.0545		0.52	
1299	EN14110	0.05		-0.36	
1457	EN14110	0.054		0.42	
1459		-----		-----	
1494	EN14110	0.0520		0.03	
1539		-----		-----	
1569		-----		-----	
1643		-----		-----	
1739	EN14110	0.057		1.00	
1769	EN14110	0.0567		0.94	
1971		-----		-----	
1984		-----		-----	
6069	EN14110	0.0551		0.63	
6170		-----		-----	
6179	EN14110	0.048		-0.74	
6201	EN14110	0.056		0.81	
6213	EN14110	0.046		-1.13	
6245		-----		-----	

normality	suspect
n	21
outliers	1
mean (n)	0.0518
st.dev. (n)	0.00486
R(calc.)	0.0136
st.dev.(EN14110:03)	0.00516
R(EN14110:03)	0.0145

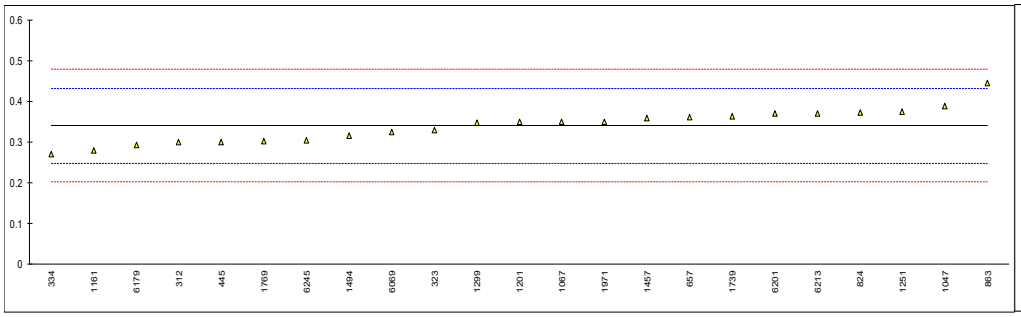


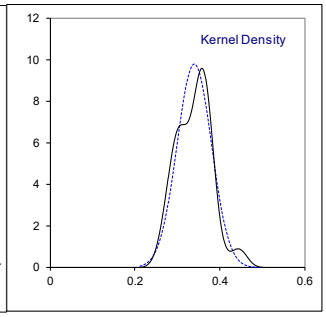


Determination of mono-Glycerides on sample #19065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	EN14105	0.30		-0.87	
323	EN14105	0.33		-0.22	
334	EN14105	0.27	C	-1.52	First reported 0.31
335		-----		-----	
336		-----		-----	
445	EN14105	0.30		-0.87	
448		-----		-----	
511		-----		-----	
540	EN14105	<0.25		-----	
551		-----		-----	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657	EN14105	0.36		0.43	
823		-----		-----	
824	D6584	0.372		0.69	
863	EN14105	0.445		2.28	
1047	EN14105	0.389		1.06	
1067	EN14105	0.35		0.22	
1134		-----		-----	
1161	EN14105	0.28		-1.30	
1199		-----		-----	
1201	EN14105	0.350		0.22	
1227		-----		-----	
1251	EN14105	0.374		0.74	
1299	EN14105	0.347		0.15	
1457	EN14105	0.359		0.41	
1459		-----		-----	
1494	D6584	0.3165		-0.51	
1539		-----		-----	
1569		-----		-----	
1643		-----		-----	
1739	EN14105	0.363		0.50	
1769	D6584	0.3019		-0.83	
1971	EN14105	0.35		0.22	
1984		-----		-----	
6069	D6584	0.3250		-0.33	
6170		-----		-----	
6179	D6584	0.2935		-1.01	
6201	EN14105	0.37		0.65	
6213	EN14105	0.37		0.65	
6245	EN14105	0.305		-0.76	

	normality	OK
	n	23
	outliers	0
	mean (n)	0.3400
	st.dev. (n)	0.04071
	R(calc.)	0.1140
	st.dev.(EN14105:11)	0.04603
	R(EN14105:11)	0.1289
Compare		
	R(D6584:17)	0.2357

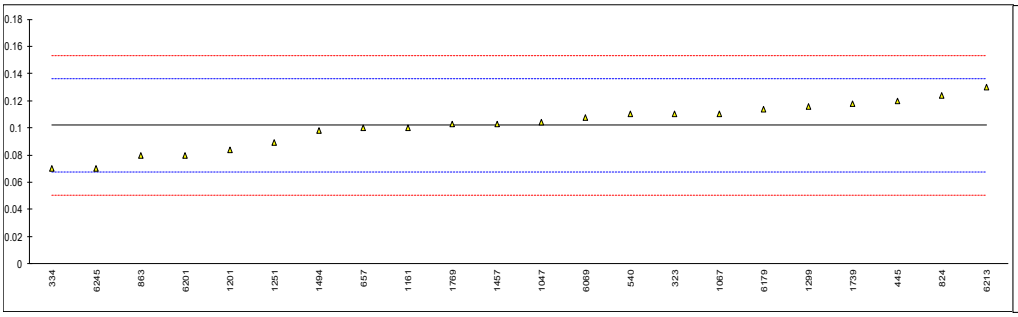


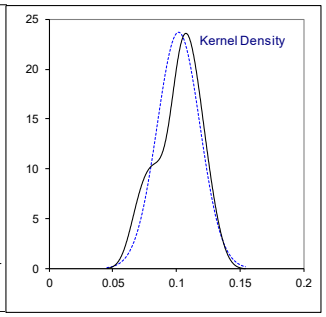


Determination of di-Glycerides on sample #19065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	EN14105	<0.10		-----	
323	EN14105	0.11		0.47	
334	EN14105	0.07		-1.85	
335		-----		-----	
336		-----		-----	
445	EN14105	0.12		1.06	
448		-----		-----	
511		-----		-----	
540	EN14105	0.110		0.47	
551		-----		-----	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657	EN14105	0.10		-0.11	
823		-----		-----	
824	D6584	0.124		1.29	
863	EN14105	0.08		-1.27	
1047	EN14105	0.104		0.12	
1067	EN14105	0.11		0.47	
1134		-----		-----	
1161	EN14105	0.1		-0.11	
1199		-----		-----	
1201	EN14105	0.084		-1.04	
1227		-----		-----	
1251	EN14105	0.089	C	-0.75	First reported 0.024
1299	EN14105	0.116		0.82	
1457	EN14105	0.103		0.07	
1459		-----		-----	
1494	D6584	0.0983		-0.21	
1539		-----		-----	
1569		-----		-----	
1643		-----		-----	
1739	EN14105	0.118		0.94	
1769	D6584	0.1028		0.05	
1971	EN14105	<0,10		-----	
1984		-----		-----	
6069	D6584	0.1079		0.35	
6170		-----		-----	
6179	D6584	0.114		0.71	
6201	EN14105	0.08		-1.27	
6213	EN14105	0.13		1.64	
6245	EN14105	0.070		-1.85	

	normality	OK
	n	22
	outliers	0
	mean (n)	0.1019
	st.dev. (n)	0.01683
	R(calc.)	0.0471
	st.dev.(EN14105:11)	0.01718
	R(EN14105:11)	0.0481
Compare		
	R(D6584:17)	0.0937

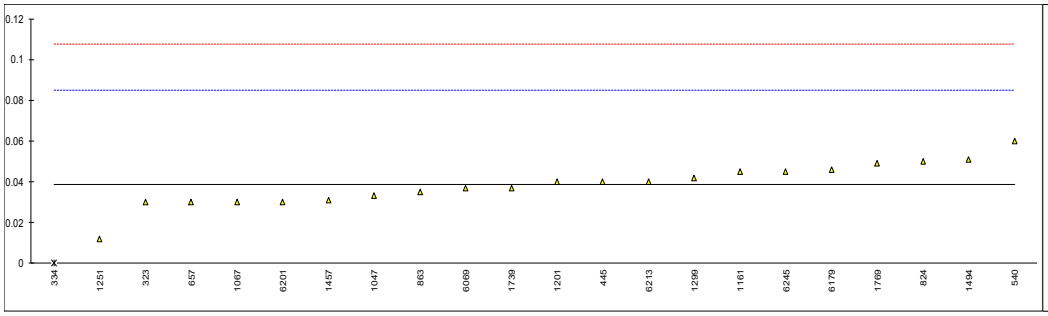


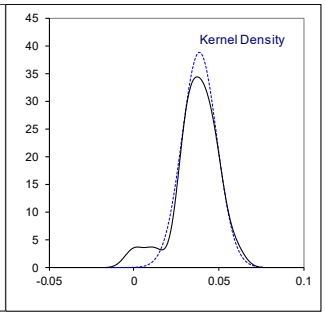


Determination of tri-Glycerides on sample #19065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	EN14105	<0.10		-----	
323	EN14105	0.03		-0.38	
334	EN14105	0.00	C,R(0.05)	-1.69	First reported 0.02
335		-----		-----	
336		-----		-----	
445	EN14105	0.04		0.06	
448		-----		-----	
511		-----		-----	
540	EN14105	0.060		0.93	
551		-----		-----	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657	EN14105	0.03		-0.38	
823		-----		-----	
824	D6584	0.050		0.49	
863	EN14105	0.035		-0.16	
1047	EN14105	0.033		-0.25	
1067	EN14105	0.03		-0.38	
1134		-----		-----	
1161	EN14105	0.045		0.27	
1199		-----		-----	
1201	EN14105	0.040		0.06	
1227		-----		-----	
1251	EN14105	0.012		-1.16	
1299	EN14105	0.042		0.14	
1457	EN14105	0.031		-0.34	
1459		-----		-----	
1494	D6584	0.0510		0.54	
1539		-----		-----	
1569		-----		-----	
1643		-----		-----	
1739	EN14105	0.037		-0.07	
1769	D6584	0.0490		0.45	
1971	EN14105	<0,10		-----	
1984		-----		-----	
6069	D6584	0.0369		-0.08	
6170		-----		-----	
6179	D6584	0.046		0.32	
6201	EN14105	0.03		-0.38	
6213	EN14105	0.04		0.06	
6245	EN14105	0.045		0.27	

	normality	suspect
	n	21
	outliers	1
	mean (n)	0.0387
	st.dev. (n)	0.01028
	R(calc.)	0.0288
	st.dev.(EN14105:11)	0.02297
	R(EN14105:11)	0.0643
Compare		
	R(D6584:17)	0.1157

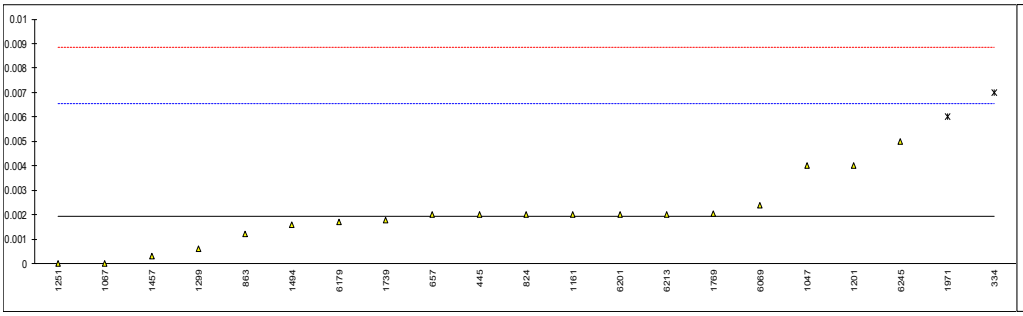


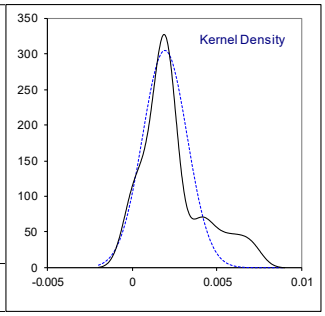


Determination of Free Glycerol on sample #19065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	EN14105	<0.001		-----	
323	EN14105	<0.001		-----	
334	EN14105	0.007	C,DG(0.05)	2.20	First reported 0.147
335		-----		-----	
336		-----		-----	
445	EN14105	0.002		0.03	
448		-----		-----	
511		-----		-----	
540	EN14105	<0.01		-----	
551		-----		-----	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657	EN14105	0.002		0.03	
823		-----		-----	
824	D6584	0.002		0.03	
863	EN14105	0.0012		-0.32	
1047	EN14105	0.004		0.90	
1067	EN14105	0.00		-0.84	
1134		-----		-----	
1161	EN14105	0.002		0.03	
1199		-----		-----	
1201	EN14105	0.004		0.90	
1227		-----		-----	
1251	EN14105	0		-0.84	
1299	EN14105	0.0006		-0.58	
1457	EN14105	0.0003		-0.71	
1459		-----		-----	
1494	D6584	0.0016		-0.14	
1539		-----		-----	
1569		-----		-----	
1643		-----		-----	
1739	EN14105	0.0018		-0.06	
1769	D6584	0.00203		0.04	
1971	EN14105	0.006	DG(0.05)	1.77	
1984		-----		-----	
6069	D6584	0.0024		0.20	
6170		-----		-----	
6179	D6584	0.0017		-0.10	
6201	EN14105	0.002		0.03	
6213	EN14105	0.002		0.03	
6245	EN14105	0.005		1.33	

	normality	OK
	n	19
	outliers	2
	mean (n)	0.00193
	st.dev. (n)	0.001309
	R(calc.)	0.00367
	st.dev.(EN14105:11)	0.002305
	R(EN14105:11)	0.00645
Compare		
	R(D6584:17)	0.00461

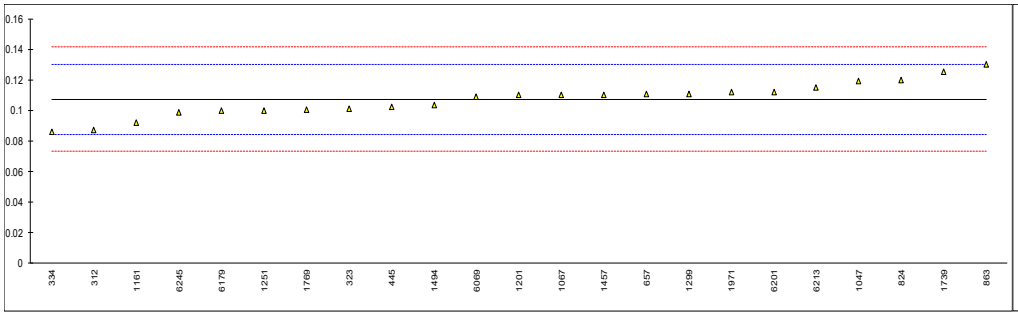


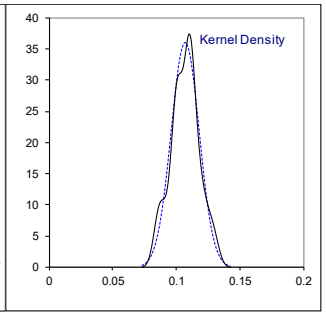


Determination of Total Glycerol on sample #19065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	EN14105	0.087		-1.77	
323	EN14105	0.101		-0.54	
334	EN14105	0.086	C	-1.86	First reported 0.239
335		-----		-----	
336		-----		-----	
445	EN14105	0.102		-0.45	
448		-----		-----	
511		-----		-----	
540	EN14105	<0.08		-----	
551		-----		-----	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657	EN14105	0.111		0.34	
823		-----		-----	
824	D6584	0.120		1.13	
863	EN14105	0.130		2.01	
1047	EN14105	0.119		1.04	
1067	EN14105	0.110		0.25	
1134		-----		-----	
1161	EN14105	0.092		-1.33	
1199		-----		-----	
1201	EN14105	0.110		0.25	
1227		-----		-----	
1251	EN14105	0.100		-0.63	
1299	EN14105	0.111		0.34	
1457	EN14105	0.110		0.25	
1459		-----		-----	
1494	D6584	0.1034		-0.33	
1539		-----		-----	
1569		-----		-----	
1643		-----		-----	
1739	EN14105	0.1250		1.57	
1769	D6584	0.1006		-0.58	
1971	EN14105	0.112		0.43	
1984		-----		-----	
6069	D6584	0.1089		0.15	
6170		-----		-----	
6179	D6584	0.0996		-0.66	
6201	EN14105	0.112		0.43	
6213	EN14105	0.115		0.69	
6245	EN14105	0.0989		-0.72	

	normality	OK
	n	23
	outliers	0
	mean (n)	0.1071
	st.dev. (n)	0.01106
	R(calc.)	0.0310
	st.dev.(EN14105:11)	0.01139
	R(EN14105:11)	0.0319
Compare		
	R(D6584:17)	0.0568

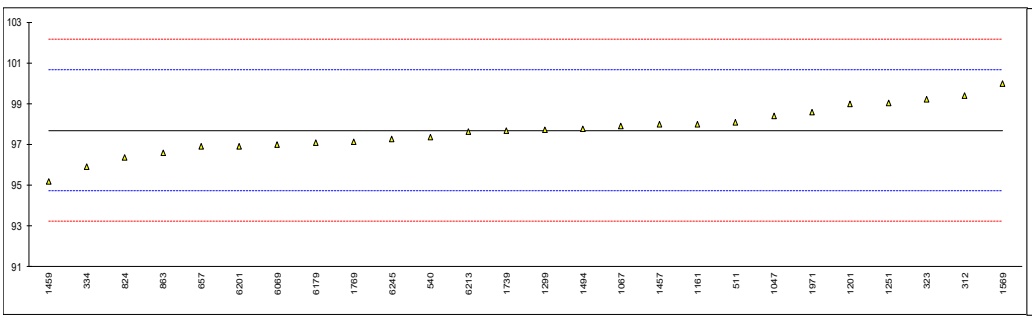


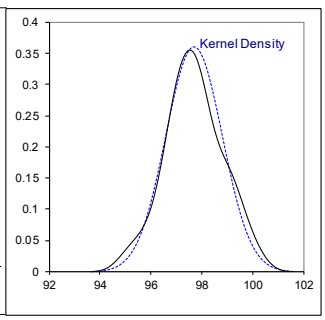


Determination of Total Ester content (FAME) on sample #19065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	EN14103	99.4		1.15	
323	EN14103	99.2		1.01	
334	EN14103	95.9		-1.21	
335		-----		-----	
336		-----		-----	
445		-----		-----	
448		-----		-----	
511	EN14103	98.07		0.25	
540	EN14103	97.35		-0.23	
551		-----		-----	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657	EN14103	96.9		-0.53	
823		-----		-----	
824	EN14103	96.38		-0.88	
863	EN14103	96.60		-0.74	
1047	EN14103	98.4		0.47	
1067	EN14103	97.9		0.14	
1134		-----		-----	
1161	EN14103	98.0		0.21	
1199		-----		-----	
1201	EN14103	98.98		0.87	
1227		-----		-----	
1251	EN14103	99.05		0.91	
1299	EN14103	97.7		0.00	
1457	EN14103	97.98		0.19	
1459	EN14103	95.2		-1.68	
1494	EN14103	97.7698		0.05	
1539		-----		-----	
1569	EN14103	100.00		1.55	
1643		-----		-----	
1739	EN14103	97.68		-0.01	
1769	EN14103	97.15		-0.37	
1971	EN14103	98.6		0.61	
1984		-----		-----	
6069	EN14103	96.99		-0.47	
6170		-----		-----	
6179	EN14103	97.10		-0.40	
6201	EN14103	96.9		-0.53	
6213	EN14103	97.61		-0.06	
6245	EN14103	97.25		-0.30	

normality	OK
n	26
outliers	0
mean (n)	97.695
st.dev. (n)	1.1093
R(calc.)	3.106
st.dev.(EN14103:11)	1.4857
R(EN14103:11)	4.160

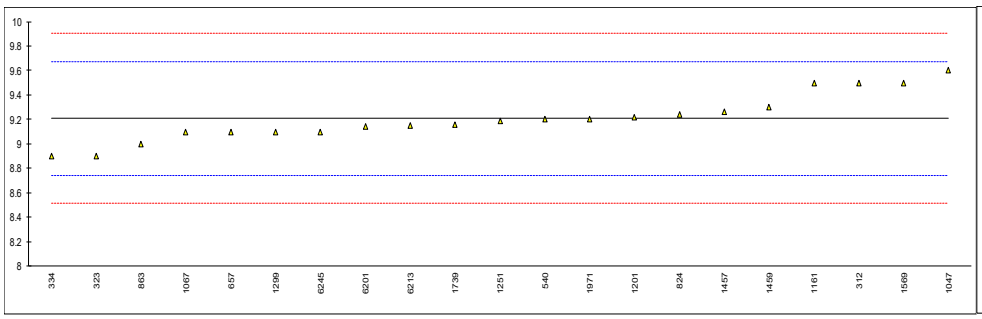


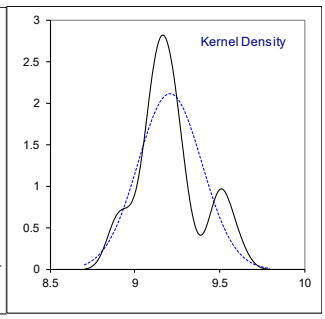


Determination of Linolenic Acid Methyl Ester content on sample #19065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	EN14103	9.5		1.26	
323	EN14103	8.9		-1.33	
334	EN14103	8.9		-1.33	
335		-----		-----	
336		-----		-----	
445		-----		-----	
448		-----		-----	
511		-----		-----	
540	EN14103	9.20		-0.03	
551		-----		-----	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657	EN14103	9.1		-0.46	
823		-----		-----	
824	EN14103	9.24		0.14	
863	EN14103	9.0		-0.89	
1047	EN14103	9.6		1.69	
1067	EN14103	9.1		-0.46	
1134		-----		-----	
1161	EN14103	9.5		1.26	
1199		-----		-----	
1201	EN14103	9.22		0.05	
1227		-----		-----	
1251	EN14103	9.19		-0.08	
1299	EN14103	9.1		-0.46	
1457	EN14103	9.26		0.23	
1459	EN14103	9.3		0.40	
1494		-----		-----	
1539		-----		-----	
1569	EN14103	9.5		1.26	
1643		-----		-----	
1739	EN14103	9.16		-0.21	
1769		-----		-----	
1971	EN14103	9.2		-0.03	
1984		-----		-----	
6069		-----		-----	
6170		-----		-----	
6179		-----		-----	
6201		9.14		-0.29	
6213	EN14103	9.15		-0.25	
6245	EN14103	9.10		-0.46	

normality	OK
n	21
outliers	0
mean (n)	9.2076
st.dev. (n)	0.18870
R(calc.)	0.5284
st.dev.(EN14103:11)	0.23201
R(EN14103:11)	0.6496

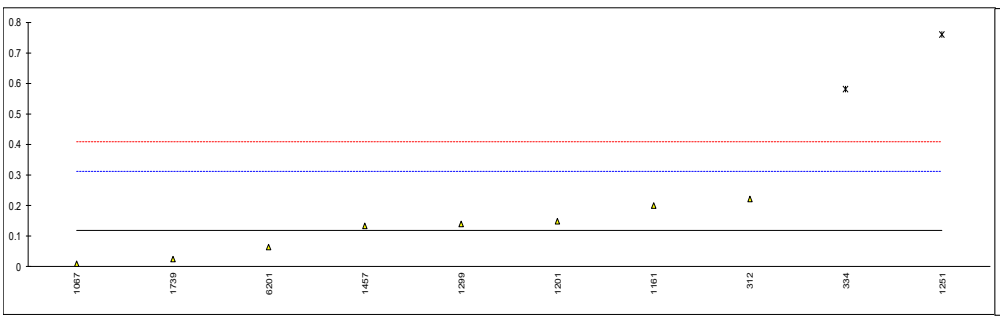


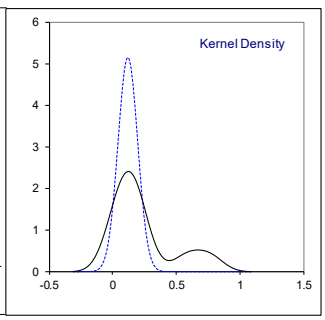


Determination of Polyunsaturated Methyl Esters content on sample #19065; results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171		-----		-----	
240		-----		-----	
312	EN15779	0.22		1.06	
323	EN15779	<0.6		-----	
334	EN15779	0.58	C,DG(0.01)	4.79	First reported 0.72
335		-----		-----	
336		-----		-----	
445		-----		-----	
448		-----		-----	
511		-----		-----	
540		-----		-----	
551		-----		-----	
558		-----		-----	
621		-----		-----	
631		-----		-----	
657	EN15779	<0.6		-----	
823		-----		-----	
824		-----		-----	
863	EN15779	<0.60		-----	
1047	EN15779	<0,60		-----	
1067	EN15779	0.01		-1.12	
1134		-----		-----	
1161	EN15779	0.2		0.85	
1199		-----		-----	
1201	EN15779	0.15		0.33	
1227		-----		-----	
1251	EN15779	0.76	C,DG(0.01)	6.66	First reported 0.65
1299	EN15779	0.14		0.23	
1457	EN15779	0.133		0.16	
1459		-----		-----	
1494		-----		-----	
1539		-----		-----	
1569		-----		-----	
1643		-----		-----	
1739	EN15779	0.026		-0.95	
1769		-----		-----	
1971	EN15779	<0,60		-----	
1984		-----		-----	
6069		-----		-----	
6170		-----		-----	
6179		-----		-----	
6201		0.064		-0.56	
6213		-----		-----	
6245		-----		-----	

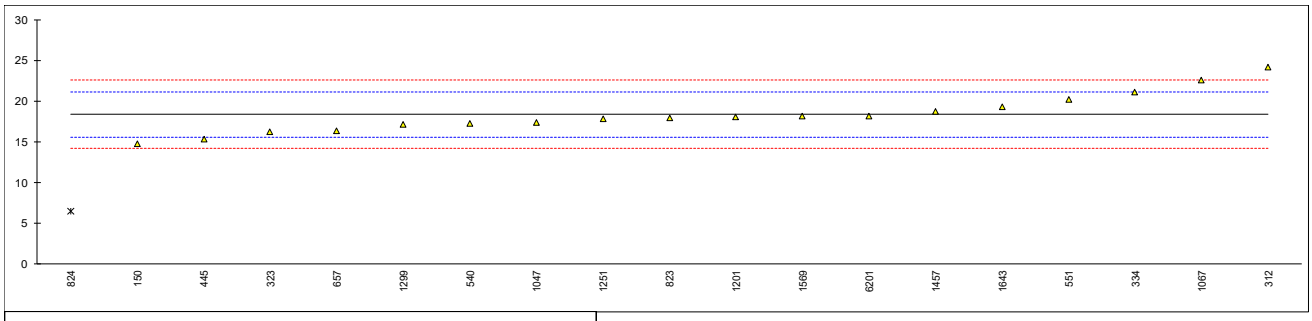
normality	OK
n	8
outliers	2
mean (n)	0.1179
st.dev. (n)	0.07737
R(calc.)	0.2166
st.dev.(EN15779:09+A1:13)	0.09643
R(EN15779:09+A1:13)	0.27

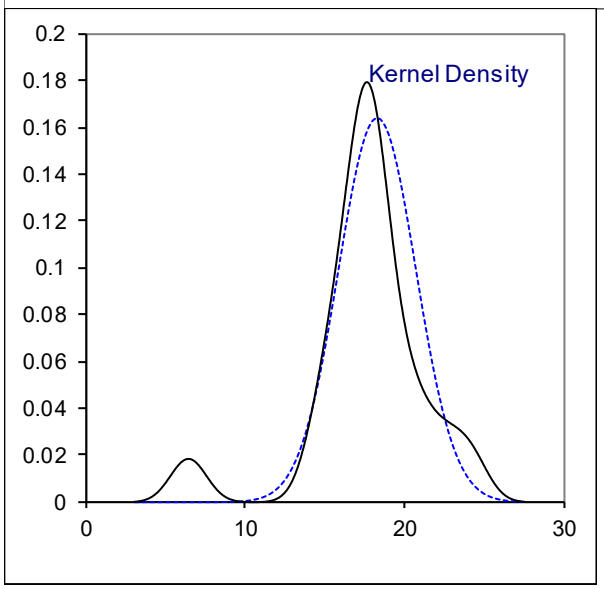




Determination of sum of Calcium and Magnesium as Ca + Mg on sample #19066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150	EN14538	14.716		-2.60	
171		-----		-----	
312	EN14538	24.2	C	4.18	First reported 30.8
323	EN14538	16.2		-1.54	
334	EN14538	21.1		1.96	
445	EN14538	15.3		-2.18	
540	EN14538	17.25		-0.79	
551	UOP389	20.2		1.32	
657	EN14538	16.3		-1.47	
823	EN14538	17.9		-0.32	
824	EN14538	6.5	G(0.01)	-8.46	
1047	EN14538	17.34		-0.72	
1067	EN14538	22.6		3.03	
1134		-----		-----	
1161		-----		-----	
1201	EN14538	18		-0.25	
1251	EN14538	17.8		-0.39	
1299	EN14538	17.1		-0.89	
1457	EN14538	18.7		0.25	
1539		-----		-----	
1569	In house	18.1		-0.18	
1643	D5185	19.34		0.71	
6201	EN14538	18.2		-0.11	
	normality	OK			
	n	18			
	outliers	1			
	mean (n)	18.353			
	st.dev. (n)	2.4279			
	R(calc.)	6.798			
	st.dev.(EN14538:06)	1.4002			
	R(EN14538:06)	3.921			Application range: 1 – 10 mg/kg



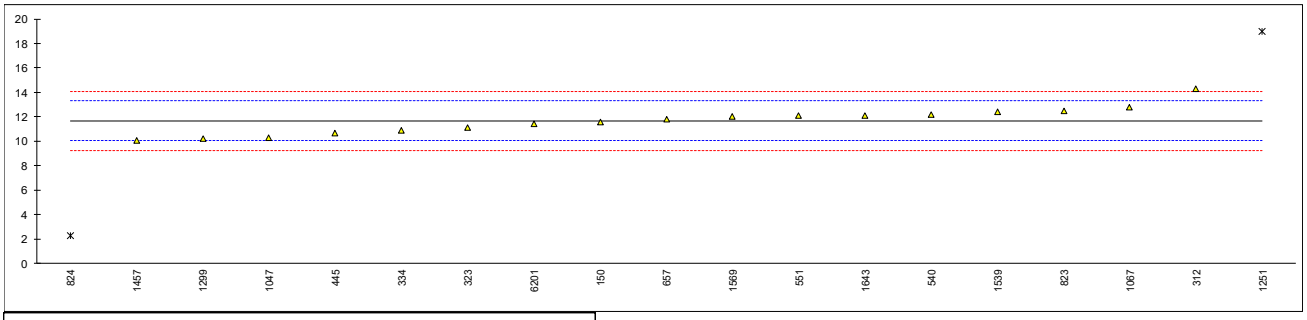


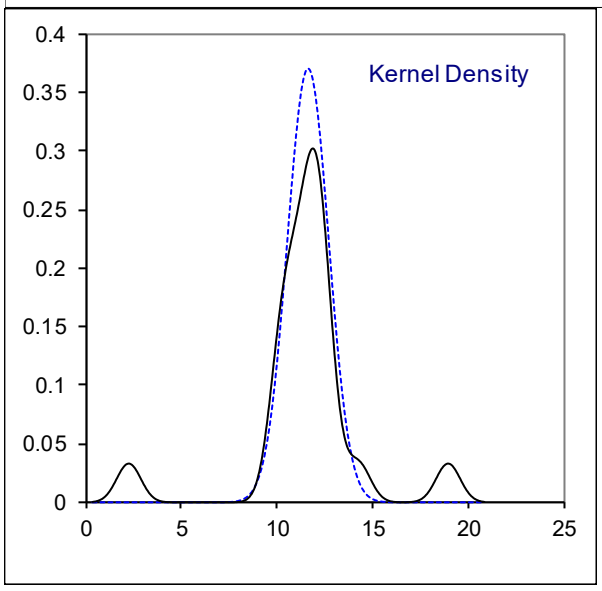
Determination of Phosphorus as P on sample #19066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150	EN14107	11.58		-0.12	
171		-----		-----	
312	EN14107	14.3		3.24	
323	EN14107	11.1		-0.71	
334	EN14107	10.9	C	-0.96	First reported 0.1
445	EN14107	10.7		-1.20	
540	EN14107	12.17		0.61	
551	UOP389	12.1		0.53	
657	EN14107	11.8		0.16	
823	D7111	12.49		1.01	
824	EN14107	2.3	G(0.01)	-11.58	
1047	EN14107	10.30		-1.70	
1067	EN14107	12.8		1.39	
1134		-----		-----	
1161		-----		-----	
1201		-----	W	-----	Test result withdrawn, reported 18
1251	EN14107	19.0	G(0.01)	9.05	
1299	EN14107	10.2	C	-1.82	First reported 5.3
1457	EN14107	10.1		-1.94	
1539	EN14107	12.4		0.90	
1569	EN14107	12.0		0.40	
1643	D5185	12.12		0.55	
6201	EN14107	11.4		-0.34	

normality	OK
n	17
outliers	2
mean (n)	11.674
st.dev. (n)	1.0783
R(calc.)	3.019
st.dev.(EN14107:03)	0.8094
R(EN14107:03)	2.266

Application range: 4 – 20 mg/kg

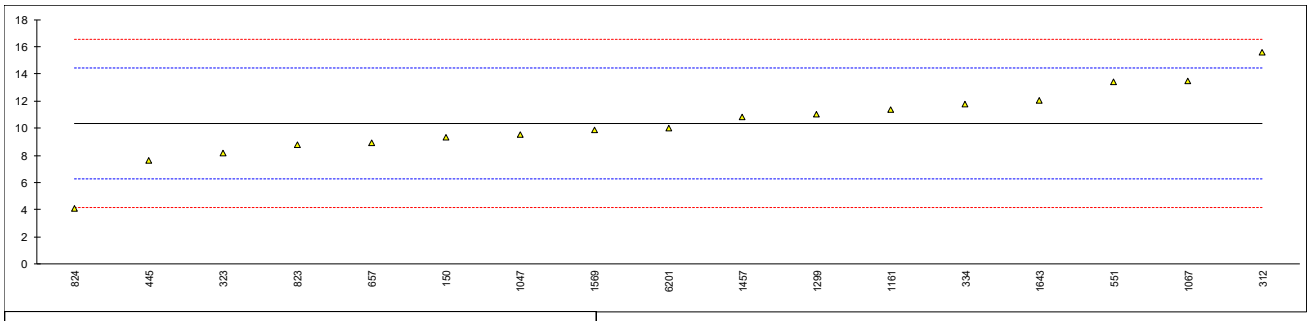


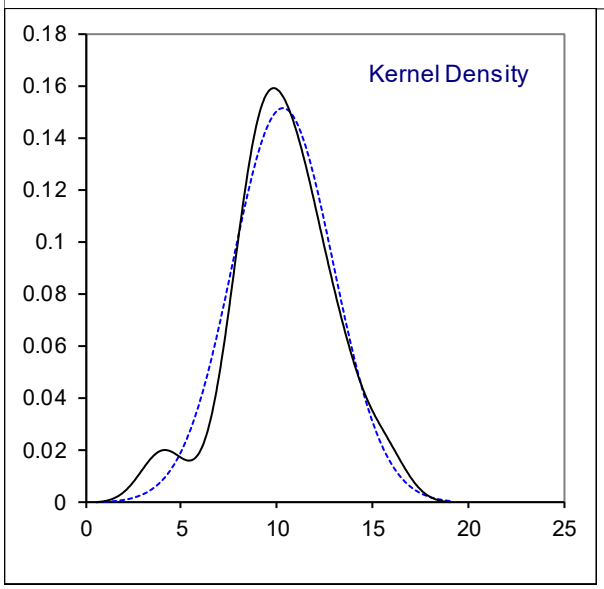


Determination of Potassium as K on sample #19066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150	EN14538	9.313		-0.50	
171		-----		-----	
312	EN14538	15.6		2.56	
323	EN14109	8.2		-1.05	
334	EN14538	11.8		0.71	
445	EN14109	7.6		-1.34	
540		-----		-----	
551	UOP389	13.4		1.49	
657	EN14538	8.9		-0.71	
823	D7111	8.80		-0.75	
824	EN14538	4.1		-3.04	
1047	EN14538	9.55		-0.39	
1067	EN14538	13.5		1.53	
1134		-----		-----	
1161	EN14109	11.4		0.51	
1201		-----		-----	
1251		-----		-----	
1299	EN14538	11.0		0.32	
1457	EN14538	10.8		0.22	
1539		-----		-----	
1569	In house	9.9		-0.22	
1643	D5185	12.08		0.84	
6201	EN14538	10.0		-0.17	

normality	suspect
n	17
outliers	0
mean (n)	10.350
st.dev. (n)	2.6387
R(calc.)	7.388
st.dev.(EN14109:03)	2.0531
R(EN14109:03)	5.749

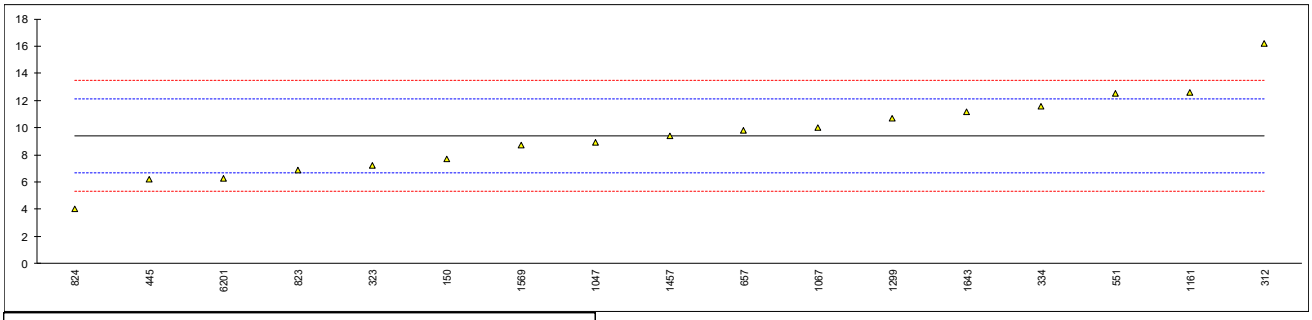


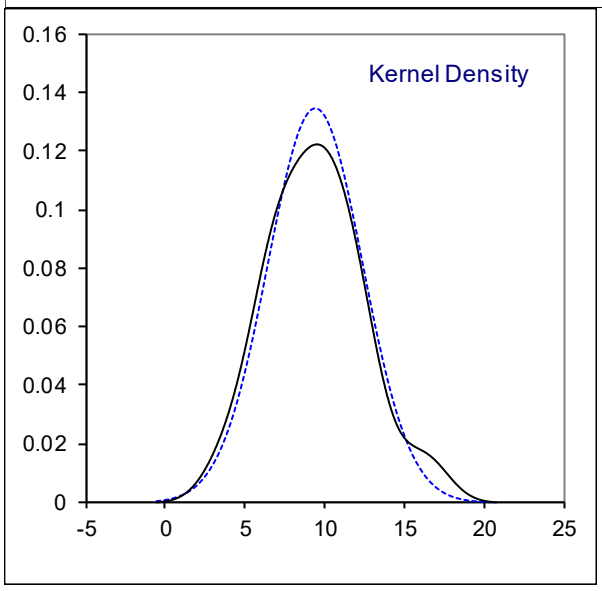


Determination of Sodium as Na on sample #19066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150	EN14538	7.678		-1.26	
171		-----		-----	
312	EN14538	16.2		4.97	
323	EN14108	7.2		-1.61	
334	EN14538	11.6		1.61	
445	EN14108	6.2		-2.34	
540		-----		-----	
551	UOP389	12.5		2.27	
657	EN14538	9.8		0.29	
823	D7111	6.85		-1.87	
824	EN14538	4.0		-3.95	
1047	EN14538	8.94		-0.34	
1067	EN14538	10.0		0.44	
1134		-----		-----	
1161	EN14108	12.6		2.34	
1201		-----		-----	
1251		-----		-----	
1299	EN14538	10.7		0.95	
1457	EN14538	9.4		0.00	
1539		-----		-----	
1569	In house	8.7		-0.51	
1643	D5185	11.15		1.28	
6201	EN14538	6.3		-2.27	

normality	OK
n	17
outliers	0
mean (n)	9.401
st.dev. (n)	2.9604
R(calc.)	8.289
st.dev.(EN14108:03)	1.3670
R(EN14108:03)	3.827

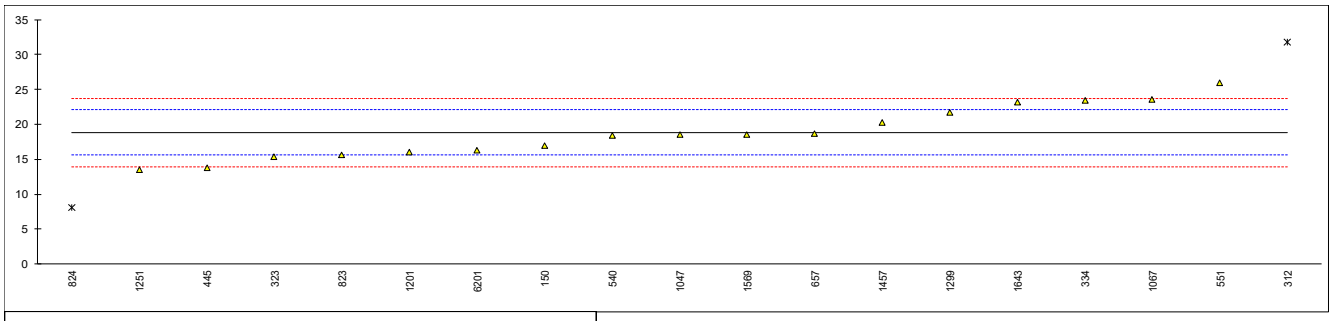


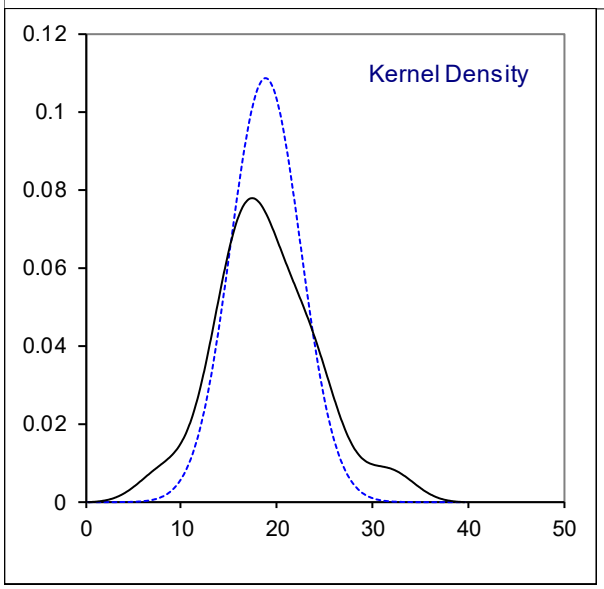


Determination of Sum of Potassium and Sodium as K + Na on sample #19066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150	EN14538	16.991		-1.12	
171		-----		-----	
312	EN14538	31.8	DG(0,05)	8.02	
323	EN14538	15.4		-2.11	
334	EN14538	23.4		2.83	
445	EN14538	13.8		-3.09	
540	EN14538	18.42		-0.24	
551	UOP389	25.9		4.38	
657	EN14538	18.7		-0.07	
823	D7111	15.65		-1.95	
824	EN14538	8.1	DG(0,05)	-6.61	
1047	EN14538	18.49		-0.20	
1067	EN14538	23.5		2.90	
1134		-----		-----	
1161		-----		-----	
1201	EN14538	16		-1.74	
1251	EN14538	13.5		-3.28	
1299	EN14538	21.7		1.78	
1457	EN14538	20.2		0.86	
1539		-----		-----	
1569	In house	18.6		-0.13	
1643	D5185	23.23		2.73	
6201	EN14538	16.3		-1.55	

normality	OK
n	17
outliers	2
mean (n)	18.811
st.dev. (n)	3.6732
R(calc.)	10.285
st.dev.(EN14538:06)	1.6192
R(EN14538:06)	4.534



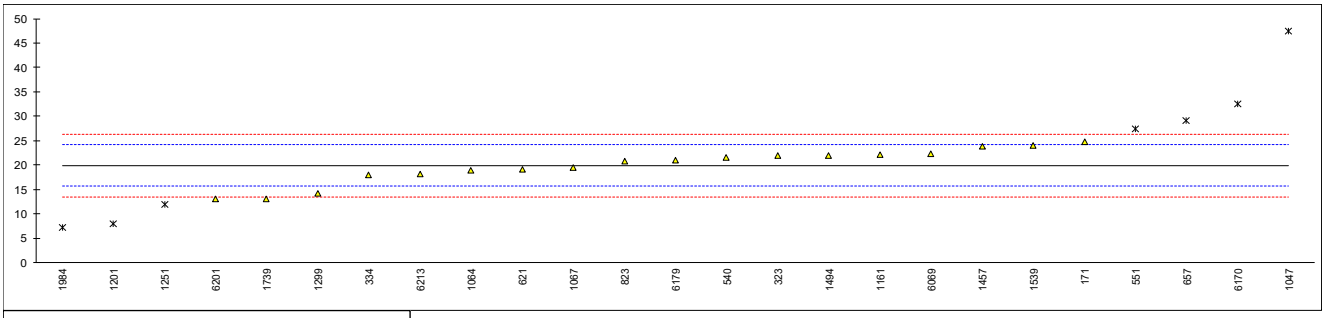


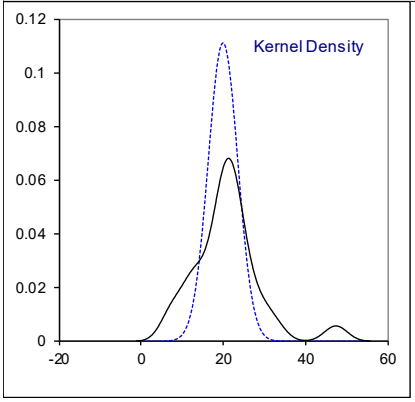
Determination of Total Contamination (EN12662) on sample #19067; results in mg/kg

lab	method	value	mark	z(targ)	Incomplete	Vol.filtered in mL	filtration stopped after minutes
120		-----		-----		-----	-----
150		-----		-----	YES	-----	-----
171	EN12662:2008	24.8		2.28	NO	-----	-----
323	EN12662:1998	22		0.97		-----	-----
334	EN12662:1998	18		-0.90	NO	-----	-----
335		-----		-----		-----	-----
445		-----		-----		-----	-----
540	EN12662:1998	21.65		0.81	NO	0	12
551	EN12662:2014	27.4	ex	3.50	NO	300	10
621	EN12662:2008	19.13		-0.37	NO	250	30
657	EN12662:2014	29.2	ex	4.34		590	465
823	EN12662:1998	20.8	C	0.41	NO	300	1.5
1047	EN12662:2008	47.4	G(0.01)	12.87	NO	300	-----
1064	EN12662:1998	18.93		-0.47	NO	-----	-----
1067	EN12662:1998	19.4		-0.25	NO	295	1
1134		-----		-----		-----	-----
1161	EN12662:2008	22.2		1.06		-----	-----
1201	EN12662:1998	8.0	G(0.05)	-5.59	NO	380	-----
1251	EN12662:2014	11.95	ex	-3.74	NO	-----	-----
1299	EN12662:1998	14.2		-2.68	NO	300	-----
1457	EN12662:1998	23.9		1.86	NO	357	1
1494	EN12662:2008	22.00		0.97	YES	-----	-----
1539	EN12662:2008	24.1		1.95		-----	-----
1739	EN12662:1998	13.01		-3.24	NO	-----	-----
1984	EN12662:2014	7.25	ex	-5.94	NO	-----	-----
6069	EN12662:2008	22.40		1.16		-----	-----
6170	EN12662:2014	32.497	ex	5.89		-----	-----
6179	EN12662:2008	20.96		0.48		-----	-----
6201	EN12662:1998	13		-3.24		-----	-----
6213	EN12662:2008	18.21		-0.80		-----	-----

	normality	OK
	n	18
	outliers	2 (+5ex)
	mean (n)	19.927
	st.dev. (n)	3.5877
	R(calc.)	10.0455
	st.dev.(EN12662:08)	2.1351
	R(EN12662:08)	5.978
Compare		
	R(EN12622:98)	5.978

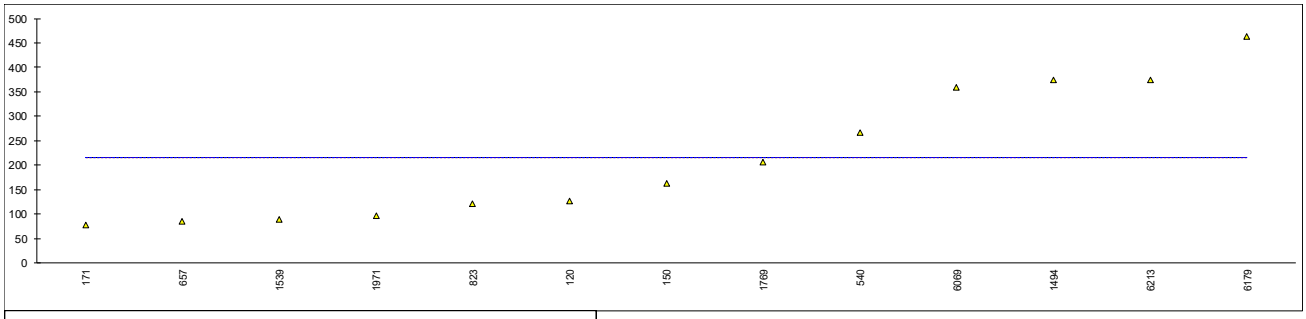
Lab 823: first reported 42.8
ex = test result excluded as EN12662:2014 may not be used, see §4.1 and lit.17+18

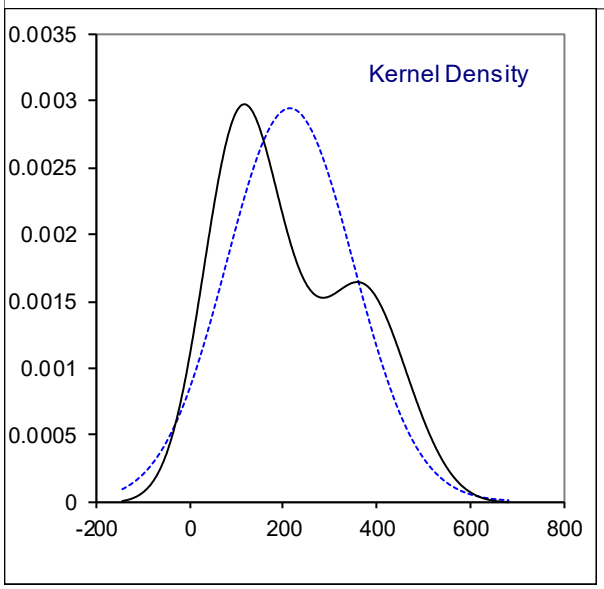




Determination of Filter Blocking Potential by Cold Soak Filterability Test (CSFT) on sample #19068; results in s

lab	method	value	mark	z(targ)	Vol. (mL) at time >720sec	Remarks
120	D7501	127.08		-----	-----	
150	D7501	162		-----	300	
171	D7501	77		-----	-----	
323		-----		-----	-----	
540	D7501	267		-----	0	
657	D7501	85		-----	300	
823	D7501	122		-----	300	
1067		-----		-----	-----	
1134		-----		-----	-----	
1201		-----		-----	-----	
1251		-----		-----	-----	
1402		-----		-----	-----	
1494	D7501	374		-----	-----	
1539	D7501	90		-----	-----	
1739		-----		-----	-----	
1769	D7501	206		-----	-----	
1971	D7501	96		-----	-----	
6069	D7501	360		-----	-----	
6179	D7501	463.22		-----	-----	
6201		-----		-----	-----	
6213	D7501	375		-----	-----	
	normality	OK				
	n	13				
	outliers	0				
	mean (n)	215.7				
	st.dev. (n)	135.77				
	R(calc.)	380.2				
	st.dev.(D7501:18a)	(32.84)				
	R(D7501:18a)	(92.0)				

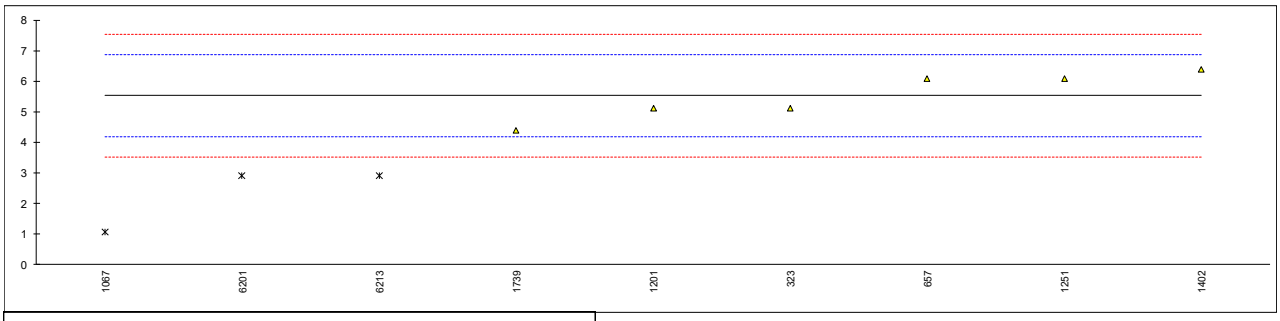


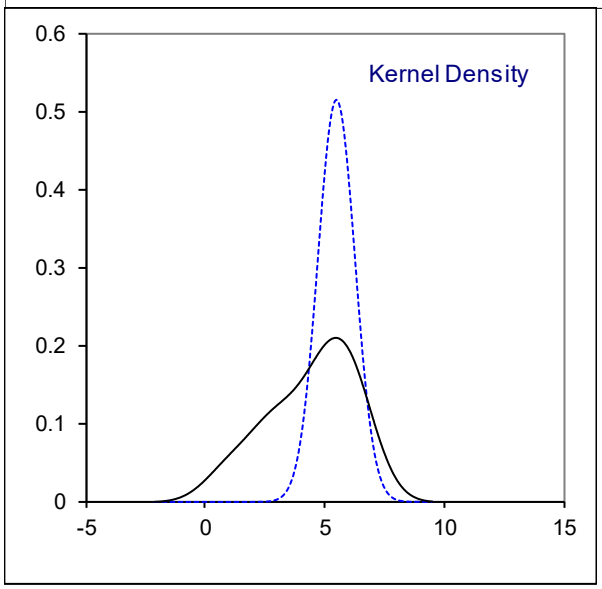


Determination of Filter Blocking Tendency (FBT) on sample #19068

lab	method	value	mark	z(targ)	press. end test (kPa)	vol. pumped mL	remarks
120		-----		-----	-----	-----	
150		-----		-----	-----	-----	
171		-----		-----	-----	-----	
323	IP387-B	5.10		-0.63	105	60	
540		-----		-----	-----	-----	
657	IP387-B	6.08		0.83	105	50	
823		-----		-----	-----	-----	
1067	IP387-B	1.07	G(0.05)	-6.63	40	300	
1134		-----		-----	-----	-----	
1201	IP387-B	5.10		-0.63	105	60	
1251	IP387-B	6.08		0.83	105	50	
1402	IP387-B	6.39		1.29	105.2	47.5	
1494		-----		-----	-----	-----	
1539		-----		-----	-----	-----	
1739	IP387-B	4.40		-1.68	105	70	
1769		-----		-----	-----	-----	
1971		-----		-----	-----	-----	
6069		-----		-----	-----	-----	
6179		-----		-----	-----	-----	
6201	IP387-B	2.90	DG(0.05)	-3.91	105	110	
6213	D2068-A	2.90	DG(0.05)	-3.91	105	110	

normality	OK
n	6
outliers	3
mean (n)	5.525
st.dev. (n)	0.7735
R(calc.)	2.166
st.dev.(D2068-B:17)	0.6716
R(D2068-B:17)	1.880





APPENDIX 2

Number of participants per country for sample #19065

1 lab in	ARGENTINA
1 lab in	BELGIUM
2 labs in	BRAZIL
1 lab in	CHINA, People's Republic
4 labs in	COLOMBIA
1 lab in	CROATIA
1 lab in	EQUATORIAL GUINEA
5 labs in	FRANCE
1 lab in	GREECE
2 labs in	HONG KONG
1 lab in	INDONESIA
6 labs in	NETHERLANDS
1 lab in	PERU
1 lab in	PHILIPPINES
4 labs in	POLAND
1 lab in	PORTUGAL
1 lab in	SINGAPORE
2 labs in	SOUTH KOREA
2 labs in	SPAIN
1 lab in	SWEDEN
1 lab in	TURKEY
3 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	= possibly an error in calculations
U	= test result possibly 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:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ASTM E178:16
3 ASTM E1301:95(2003)
4 ISO 5725:86 (1994)
5 ISO 5725, parts 1-6, 1994
6 ISO 13528:05
7 M. Thompson and R. Wood, J. AOAC Int, 76, 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, 118, 455, (1993)
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)
18 iis Memo 1903, Biodiesel B100 (100% FAME) for Total Contamination EN12662.