

**Results of Proficiency Test  
Biodiesel 100% FAME (B100)  
May 2016**

**Organised by:** Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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## SUMMARY OF CHANGES

This revised report replaces the original report iis16G02ASTM of July 2016.

iis discovered that the reproducibility of ASTM D2500:2011 was used instead of ASTM D2500:2016 in the calculation and evaluation of the Cloud point determination.

In ASTM D2500:2016 a new reproducibility for Biodiesel is present and this has a large effect on the calculations and statistical evaluation of the Cloud point determination.

Therefore, in this revised report the table and graph of the Cloud point determination in appendix 1, the summary of the Cloud point in table 9 and 14 as well as the evaluation in paragraph 4.1 were changed in this revised report.

Therefore the following pages in this report have been revised:

- Paragraph 4.1: Cloud point evaluation page 12 (page 10 in the original report)
- Paragraph 4.2 (table 9): comparison of Cloud point determination page 16 (page 14 in the original report)
- Paragraph 4.3 (table 14): comparison of Cloud point determination page 17 (page 15 in the original report)
- Appendix 1 page 20 (page 18 in the original report)
- Page numbering was changed, due to the addition of this page

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## 1 INTRODUCTION

Since 1991, the Institute for Interlaboratory Studies organises every year proficiency tests (PT) for Fatty Acid Methyl Esters (FAME) used as Biodiesel B100. In the annual proficiency testing program of 2015/2016, it was decided to continue the May proficiency tests on Biodiesel B100 in accordance with the latest applicable version of ASTM D6751:15ce1 specification and additional tests (e.g. EN14214:2012+A1:2014/AC:2014).

In this interlaboratory study, in total, 54 laboratories from 31 different countries have participated. See appendix 2 for a list of number of participants per country. In this report the results of the 2016 Biodiesel B100 ASTM proficiency test are presented and discussed. This report is also electronically available through the iis website site [www.iisnl.com](http://www.iisnl.com).

## 2 SET UP

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

Samples	Amount in L	Purpose	Spiked
#16065	1.5	For regular analysis	--
#16066	0.1	Analysis of Phosphorus, Potassium, Sodium and Calcium & Magnesium	Phosphorus, Sodium, Calcium
#16067	1	Total Contamination test	--
#16068	0.5	Cold Soak Test / Filter Blocking	--

Table 1: four different Biodiesel B100 samples used in iis16G02ASTM

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

### 2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkensisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010 (R007). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Also customer's satisfaction is measured on regular basis by the distribution of questionnaires.

### 2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol is electronically available through the iis website site [www.iisnl.com](http://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, approximately 220 L of Biodiesel (100% RME) for this PT was obtained from an European producer.

### **Biodiesel B100 #16065**

After fit-for-use testing and homogenisation, 86 amber glass bottles of 1L and 86 amber glass bottles of 0.5L, were filled, closed with inner and outer caps and labelled #16065. The homogeneity of the subsamples #16065 was checked by the determination of Density in accordance with ASTM D4052 on 8 stratified randomly selected samples:

	Density at 15°C in kg/m <sup>3</sup>
sample 1 #16065-1	883.01
sample 2 #16065-2	882.98
sample 3 #16065-3	883.01
sample 4 #16065-4	882.99
sample 5 #16065-5	883.01
sample 6 #16065-6	883.01
sample 7 #16065-7	883.03
sample 8 #16065-8	882.98

Table 2: homogeneity test of subsamples #16065

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

	Density at 15°C in kg/m <sup>3</sup>
r (observed)	0.05
reference test method	ISO12185:96
0.3 x R (ref. test method)	0.15

Table 3: repeatability of subsamples #16065

**Metals in Biodiesel B100 #16066**

For subsample #16066, for metals in Biodiesel only, a batch of approx. 9.0 kg RME was spiked with Calcium (approx. 4 mg/kg), Phosphorus (approx. 6 mg/kg) and Sodium (approx. 6 mg/kg). After homogenisation, the material was subsequently divided over 96 HDPE bottles of 0.1L and labelled #16066. The homogeneity of the subsamples #16066 was checked by determination of Phosphorus in accordance with EN14107 on 8 stratified randomly selected samples:

	Phosphorus in mg/kg
sample 1 #16066-1	6.6
sample 2 #16066-2	6.6
sample 3 #16066-3	6.4
sample 4 #16066-4	6.4
sample 5 #16066-5	6.3
sample 6 #16066-6	6.4
sample 7 #16066-7	6.4
sample 8 #16066-8	6.3

Table 4: homogeneity test of subsamples #16066

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

	Phosphorus in mg/kg
r (observed)	0.33
reference test method	EN14107:03
0.3 x R (ref. test method)	0.37

Table 5: repeatability of subsamples #16066

**Total Contamination #16067**

For Total Contamination, out of the same batch of regular Biodiesel B100 and filled together with #16065, another 62 amber glass bottles of 1 litre with inner and outer caps were filled and labelled #16067. For homogeneity see table 2 and 3.

**Cold Soak Test / Filter Blocking Tendency #16068**

Several days later, from the remaining batch of Biodiesel, after renewed homogenisation, 32 bottles of 0.5 litre amber glass bottles were filled and labelled #16068. The homogeneity of the subsamples was checked by the determination of density in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Density at 15°C in kg/m <sup>3</sup>
sample 1 #16068-1	882.95
sample 2 #16068-2	882.95
sample 3 #16068-3	882.96
sample 4 #16068-4	882.96
sample 5 #16068-5	882.96
sample 6 #16068-6	882.96
sample 7 #16068-7	882.96
sample 8 #16068-8	882.96

Table 6: homogeneity test of subsamples #16068

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

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

Table 7: repeatability of subsamples #16068

The calculated repeatabilities for sample #16065, #16066, #16067 and #16068 were less than 0.3 times the corresponding reproducibility of the respective reference method. Therefore, homogeneity of the subsamples was assumed.

Depending on the registration of the participant, one 1 litre bottle and 0.5 litre bottle both labelled #16065, and/or one 100mL bottle labelled #16066, and/or 1 litre bottle labelled #16067 and/or 0.5 litre bottle labelled #16068, were dispatched to each of the participating laboratories on April 13, 2016.

## 2.5 STABILITY OF THE SAMPLES

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



## 2.6 ANALYSES

The tests methods to be used by the participating laboratories should be in accordance with the requirements of ASTM D6751:15ce1 and additional tests (e.g. EN14214:12+A1:14/AC:14):

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

Table 8: requirements and test methods acc. to specifications ASTM D6751:15ce1 and EN14214:12+A1:14/AC:14

\*) not evaluated in this ASTM based round robin

To get comparable test results a detailed report form, on which the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made available on the data entry portal [www.kpmd.co.uk/sgs-iis/](http://www.kpmd.co.uk/sgs-iis/). A SDS and a form to confirm receipt of the samples were added to the sample package.

## 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/](http://www.kpmd.co.uk/sgs-iis/). The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these

suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1.

Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

### 3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the test results 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

### 3.2 GRAPHICS

In order to visualise 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 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.

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:

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

The  $z_{(\text{target})}$  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 some problems were encountered during the execution.

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

For the Total Contamination PT: 1 participant reported the test result after the final reporting date and 3 participants did not report any test results at all.

For the Metals in Biodiesel PT: 1 participant reported the test results after the final reporting date and 2 participants did not report any test results at all.

For the Filter Blocking PT: none of the participants reported a result after the final reporting date, however 7 participants did not report any test result at all.

Finally, 596 numerical test results were reported by the participants. Observed were 25 outlying results, which is 4.2%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

#### 4.1 EVALUATION PER TEST

In this section, the results are discussed per sample and per test. The specified test methods and requirements ASTM D6751:15ce1 and additional tests (e.g. EN14214:12+A1:14/AC:14) were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the reported data. The abbreviations, used in the tables of Appendix 1, are listed in appendix 3.

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

In the iis PT reports, ASTM methods are referred to with a number (e.g. D6371) and an added designation for the year that the method was adopted or revised (e.g. D6371:05). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D6371:05(2010)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D6371:05 will be used.

##### **For Biodiesel B100 sample #16065**

Acid Number: (ASTM) This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D664:11a (method B).

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

CFPP: 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 ASTM D6371:05(2010).

Carbon Residue on 100%: All reported results were near or below the applicable lower limit of D4530:11 (0.1%M/M). Therefore no significant conclusions were drawn.

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

Density at 15°C This determination was problematic for a number of laboratories. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96.

Note: API /ASTM tables do not apply to FAME that falls within EN14214:12+A1:2014. See Annex B of EN14214:12+A1:2014 for calculation of conversion factor.

- 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 D93C:15.
- Iodine Value: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of EN14111:03.
- Kin.Visco. at 40°C: The determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D445:15a.
- Oxidation Stability: 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 EN15751:14.
- Sulphated Ash: All reported results were near or below the applicable lower limit of ASTM D874:13a and/or ISO3987:10 (0.005% M/M). Therefore no significant conclusions were drawn.
- Sulphur: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5453:16.
- Water: This determination was not problematic. One statistical outlier was observed. However the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D6304:07.
- Water and Sediment: All reported results were near or below the applicable lower limit of ASTM D2709:96(2011). Therefore no significant conclusions were drawn.
- Distillation: This determination was problematic. In total six statistical outliers are observed, caused by only two laboratories. The calculated reproducibility for 80% recovered is in agreement with the requirements of ASTM D1160:15. The calculated reproducibilities for 90 and 95% recovered are not in agreement with the requirements.
- Methanol: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN14110:03.
- mono-Glycerides: This determination was not problematic. No statistical outliers were observed. However, the calculated reproducibility is in agreement with the requirements of D6584:13.

- di-Glycerides: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements ASTM D6584:13.
- tri-Glycerides: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6584:13.
- Free Glycerine: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of statistical outlier is in agreement with the requirements of ASTM D6584:13.
- Total Glycerine: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6584:13.

### **For Biodiesel B100 sample #16066**

- Calcium and Magnesium: This determination was problematic for a number of laboratories. Two statistical outliers were observed. However, the calculated reproducibility, after rejection of the statistical outliers is in full agreement with the requirements of EN14538:06. The samples were spiked with Calcium (theoretical increment of 4 mg/kg), but since a summation with Magnesium is used, a recovery cannot be calculated. The actual blank concentration for the sum of Calcium and Magnesium is not known.
- Phosphorus 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 EN14107:03 or ASTM D4951:14. The samples were spiked with Phosphorus. The average recovery of Phosphorus (theoretical increment of 5.94 mg/kg) may be satisfactory: "less than 106%". The actual blank concentration for Phosphorus is unknown.
- Potassium This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN14214:12+A1:2014/AC:2014.
- Sodium This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN14214:12+A1:2014/AC:2014, but not in agreement with EN14108:03. The samples were spiked with Sodium. The average recovery of Sodium (theoretical increment of 5.99 mg/kg) may be satisfactory: "less than 86%". The actual blank concentration for Sodium is unknown.

Potassium and Sodium: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN14538:06.

#### **For Biodiesel B100 sample #16067**

Total Contamination: As this proficiency test was ASTM based, the intention was to evaluate the Total contamination against ASTM D7321. Regretfully, the majority of the laboratories reported to have used EN12662 as test method. Therefore it was decided to evaluate the test results for ASTM D7321, EN12662:2014 and EN12662:1998 (and 2008) separately, which have significant differences and may produce different test results.

ASTM D7321: Regretfully only two very different test results were reported according ASTM D7321. Therefore, no significant conclusions were drawn.

EN12662:2014: 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 EN12662:14. The test results vary over a large range.

EN12662:1998 (+2008): This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of EN12662:98. The test results vary over a large range.

#### **For Biodiesel B100 sample #16068**

Filter Blocking Potential by Cold Soak test: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D7501:12a

Filter Blocking Tendency: This determination was problematic. One statistical outlier was observed and one test result was excluded as this result was probably calculated by measured pressure. The calculated reproducibility, after rejection of the suspect data, is not in agreement with the requirements of ASTM D2068:14 (or IP387-B, which is identical). The low number of reported test results may partly explain the large spread.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	R (Calc.)	R (lit)
Acid Number (D664-B)	mg KOH/g	28	0.26	0.07	0.10
Cloud Point	°C	34	-6.7	4.9	5.0
Cold Filter Plugging Point	°C	22	-20.8	5.3	4.7
Carbon Residue on 100% FAME	%M/M	17	<0.1	n.a.	n.a.
Copper Strip Corrosion		18	1	n.a.	n.a.
Density at 15°C	kg/m <sup>3</sup>	41	883.0	0.3	0.5
Flash Point (PMcc) ASTM	°C	33	163.8	14.2	14.7
Iodine Value	g I <sub>2</sub> /100g	23	111.4	5.4	5.0
Kin. Viscosity at 40°C	mm <sup>2</sup> /s	38	4.498	0.055	0.101
Oxidation Stability EN15751	hours	23	8.2	1.7	1.9
Sulphated Ash	%M/M	17	<0.005	n.a.	n.a.
Sulphur	mg/kg	26	2.8	0.9	1.3
Water	mg/kg	37	450	60	660
Water and Sediment	%V/V	13	<0.05	n.a.	n.a.
Distillation at 10mm Hg					
80% recovered	°C	5	353.3	2.4	4.6
90% recovered	°C	5	355.9	5.9	4.6
95% recovered	°C	6	364.3	19.9	4.6
Methanol	%M/M	19	0.031	0.013	0.010
mono-Glycerides	%M/M	21	0.511	0.179	0.303
di-Glycerides	%M/M	20	0.099	0.064	0.091
tri-Glycerides	%M/M	20	0.047	0.061	0.133
Free Glycerine	%M/M	15	0.0021	0.0030	0.0047
Total Glycerine	%M/M	20	0.154	0.062	0.074

Table 9: comparison of the observed and target reproducibilities of Biodiesel B100 sample #16065

Parameter	unit	n	average	R (Calc.)	R (lit)
Calcium and Magnesium	mg/kg	16	8.98	2.71	2.52
Phosphorus	mg/kg	14	6.29	2.19	1.23
Potassium	mg/kg	10	0.32	0.78	2.08
Sodium	mg/kg	16	5.13	3.18	3.55
Sum Potassium and Sodium	mg/kg	13	5.44	3.39	1.98

Table 10: comparison of the observed and target reproducibilities of Biodiesel B100 sample #16066

Parameter	unit	n	average	R (Calc.)	R (lit)
Total Contamination (D7321)	mg/L	2	3.0 - 20	n.a.	n.a.
Total Contamination (EN12662:14)	mg/kg	8	16.5	10.0	6.8
Total Contamination (EN12662:98)	mg/kg	10	9.8	10.2	2.9

Table 11: comparison of the observed and target reproducibilities of Biodiesel B100 sample #16067



Parameter	unit	n	average	R (Calc.)	R (lit)
Filter Blocking Potential by CST	s	8	166.8	65.9	65.6
Filter Blocking Tendency		4	6.3	4.0	2.5

Table 12: comparison of the observed and target reproducibilities of Biodiesel B100 sample #16068

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

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2016 WITH PREVIOUS PTS

	May 2016	October 2015	April 2015	September 2014
Type of FAME	Rapeseed	Rapeseed	Rapeseed	Rapeseed
Number of reporting labs	54	54	60	54
Number of results reported	596	788	965	836
Number of statistical outliers	25	19	23	35
Percentage statistical outliers	4.2	2.4%	2.4%	4.2%

Table 13: comparison with previous proficiency tests

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

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

Determination	May 2016	October 2015	April 2015	September 2014
Acid Number (D664-B)	+	+	++	-
Cloud Point	+/-	+	+	-
Cold Filter Plugging Point	-	++	+/-	++
Carbon Residue on 100% FAME	n.e.	(-)	n.e.	(++)
Density at 15°C	++	++	+/-	++
Flash Point PMcc ASTM	+/-	-	+	+
Iodine Value	+/-	+	+/-	-
Kin. Viscosity at 40°C	++	+/-	-	+
Oxidation Stability	+	++	++	+
Sulphated Ash	n.e.	n.e.	(--)	(--)
Sulphur	+	+	+	+
Water	++	+	++	+
Distillation (80, 90 and 95% rec.)	-	n.e.	n.e.	n.e.
Methanol	-	-	-	+/-
mono-Glycerides	++	+	+	+
di-Glycerides	++	+/-	+/-	+/-
tri-Glycerides	++	+	++	+
Free Glycerol	++	++	+	+/-
Total Glycerol	+	+	+	+

Table 14 : comparison of group performances against the standard requirements

Determination	May 2016	October 2015	April 2015	September 2014
Sum of Calcium and Magnesium	+/-	-	-	-
Phosphorus	--	--	--	--
Potassium	++	++	++	(++)
Sodium	+	+	+	-
Sum of Potassium and Sodium	--	n.e.	n.e.	n.e.
Total Contamination	--	-	-	--
Filter Blocking Potential by CST	+/-	n.e.	n.e.	n.e.
Filter Blocking Tendency	-	n.e.	n.e.	n.e.

Table 15 : comparison of group performances against the standard requirements

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

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

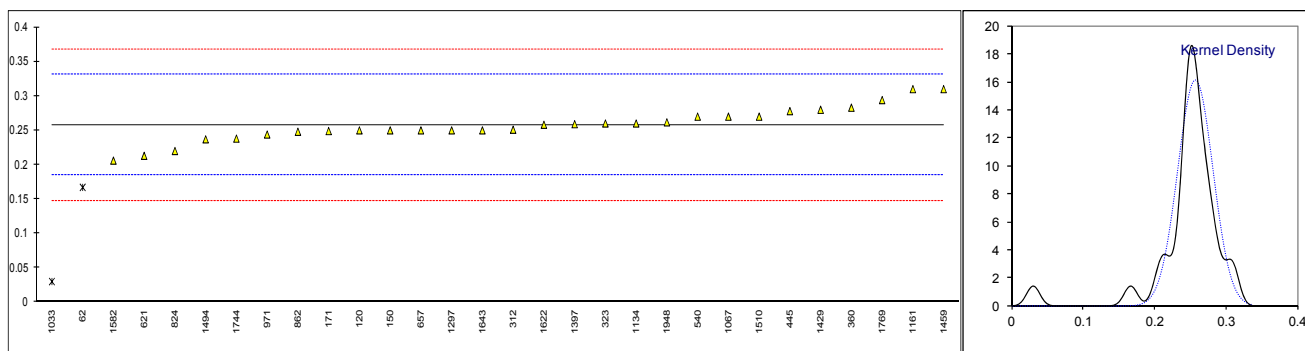
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.e.: not evaluated

## **APPENDIX 1**

Determination of Acid Number total conform ASTM spec. on sample #16065; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
62	D664	0.167	R(0.05)	-2.47	
120	D664	0.25		-0.21	
150	D664	0.25		-0.21	
171	D664	0.249		-0.24	
225		----		----	
228		----		----	
253		----		----	
312	EN14104	0.251		-0.19	
323	EN14104	0.26		0.06	
333		----		----	
334		----		----	
335		----		----	
336		----		----	
338		----		----	
360	D664	0.283		0.68	
391		----		----	
445	D664	0.278		0.55	
511		----		----	
529		----		----	
540	D664	0.27		0.33	
556		----		----	
558		----		----	
621	D664	0.213		-1.22	
657	D664	0.25		-0.21	
823		----		----	
824	D664	0.22		-1.03	
862	D664	0.248		-0.27	
970		----		----	
971	D664	0.244		-0.38	
1033	D664	0.03	R(0.01)	-6.20	
1067	EN14104	0.27		0.33	
1134	D664	0.26		0.06	
1161	D664	0.31		1.42	
1199		----		----	
1286		----		----	
1297	D664	0.25		-0.21	
1397	EN14104	0.259		0.03	
1429	EN14104	0.28		0.60	
1459	D664	0.31		1.42	
1494	D664	0.237		-0.57	
1510	D974	0.27		0.33	
1582	D664	0.206		-1.41	
1622	D664	0.2582		0.01	
1634		----		----	
1643	D664	0.250		-0.21	
1744	D664	0.238		-0.54	
1769	D664	0.29410		0.99	
1948	D664	0.2617		0.10	
6033		----		----	

normality OK  
n 28  
outliers 2  
mean (n) 0.2579  
st.dev. (n) 0.02467  
R(calc.) 0.0691  
R(D664B:11a) 0.1029

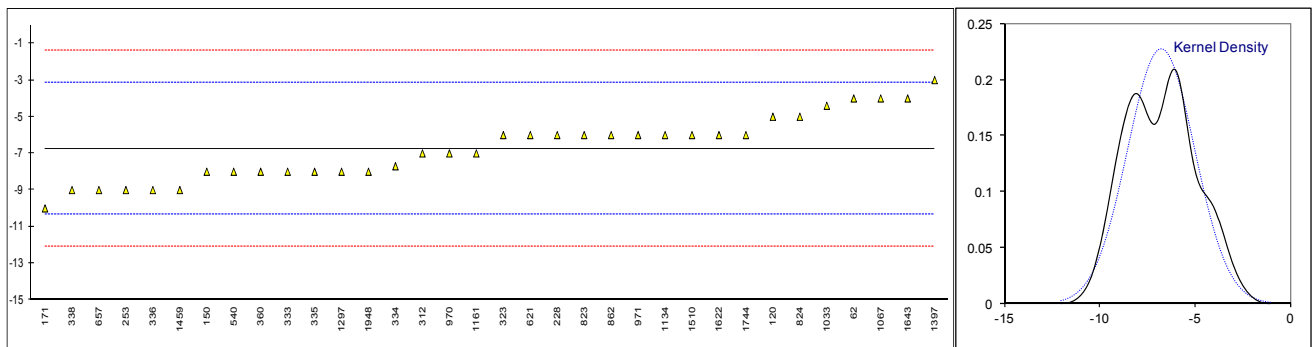


Determination of Cloud Point on sample #16065; results in °C

lab	method	value	mark	z(targ)	remarks
62	D2500	-4		1.53	
120	D2500	-5		0.97	
150	D2500	-8		-0.71	
171	D2500	-10.0		-1.83	
225		----		----	
228	D2500	-6		0.41	
253	D2500	-9.0		-1.27	
312	D2500	-7		-0.15	
323	EN23015	-6		0.41	
333	D2500	-8		-0.71	
334	D2500	-7.7		-0.54	
335	EN23015	-8		-0.71	
336	D2500	-9		-1.27	
338	EN23015	-9		-1.27	
360	D2500	-8		-0.71	
391		----		----	
445	IP219	N/A		----	
511		----		----	
529		----		----	
540	D2500	-8		-0.71	
556		----		----	
558		----		----	
621	D2500	-6.0		0.41	
657	D2500	-9		-1.27	
823	D2500	-6		0.41	
824	D2500	-5		0.97	
862	D2500	-6		0.41	
970	D2500	-7		-0.15	
971	D2500	-6		0.41	
1033	D5772	-4.4		1.31	
1067	D5771	-4		1.53	
1134	D2500	-6		0.41	
1161	D2500	-7		-0.15	
1199		----		----	
1286		----		----	
1297	D5771	-8.0		-0.71	
1397	D2500	-3		2.09	
1429		----		----	
1459	EN23015	-9		-1.27	
1494		----		----	
1510	D2500	-6		0.41	
1582		----		----	
1622	D2500	-6		0.41	
1634		----		----	
1643	D2500	-4		1.53	
1744	D2500	-6		0.41	
1769		----		----	
1948	D2500	-8		-0.71	
6033		----		----	

normality OK  
n 34  
outliers 0  
mean (n) -6.74  
st.dev. (n) 1.757  
R(calc.) 4.92  
R(D2500:11) 5.00

Compare R(EN23015/ISO3015) = 6.00

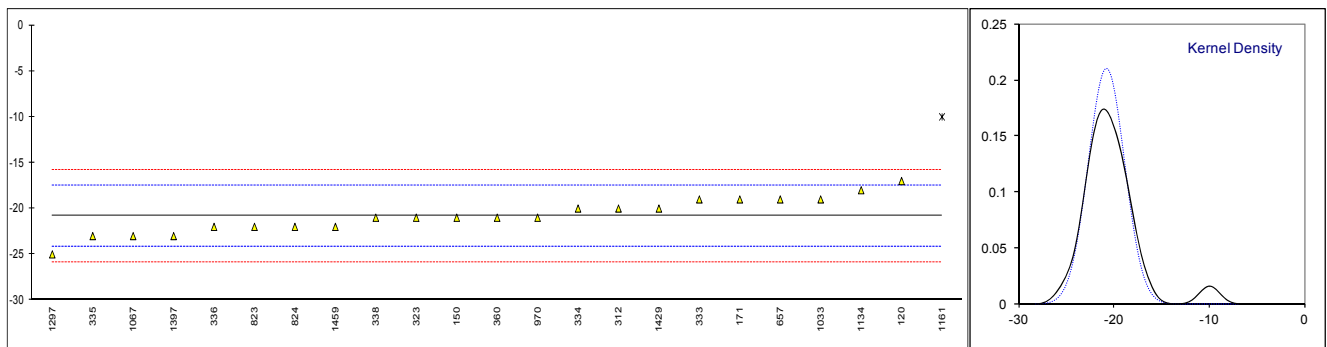


Determination of Cold Filter Plugging Point on sample #16065; results in °C

lab	method	value	mark	z(targ)	remarks
62		----		----	
120	D6371	-17		2.29	
150	D6371	-21		-0.11	
171	D6371	-19.0		1.09	
225		----		----	
228		----		----	
253		----		----	
312	EN116	-20		0.49	
323	EN116	-21		-0.11	
333	D6371	-19		1.09	
334	D6371	-20		0.49	
335	EN116	-23		-1.31	
336	D6371	-22		-0.71	
338	EN116	-21		-0.11	
360	D6371	-21		-0.11	
391		----		----	
445	IP309	N/A		----	
511		----		----	
529		----		----	
540	D6371	<-20		----	
556		----		----	
558		----		----	
621		----		----	
657	IP309	-19		1.09	
823	D6371	-22		-0.71	
824	D6371	-22		-0.71	
862		----		----	
970	IP309	-21.0		-0.11	
971		----		----	
1033	IP309	-19		1.09	
1067	IP309	-23		-1.31	
1134	IP309	-18		1.69	
1161	EN116	-10	C,R(0.01)	6.48	First reported -26
1199		----		----	
1286		----		----	
1297	D6371	-25.0		-2.51	
1397	EN116	-23		-1.31	
1429	IP309	-20		0.49	
1459	EN116	-22		-0.71	
1494		----		----	
1510		----		----	
1582		----		----	
1622		----		----	
1634		----		----	
1643		----		----	
1744		----		----	
1769		----		----	
1948		----		----	
6033		----		----	

normality OK  
n 22  
outliers 1  
mean (n) -20.82  
st.dev. (n) 1.893  
R(calc.) 5.30  
R(D6371:05) 4.67

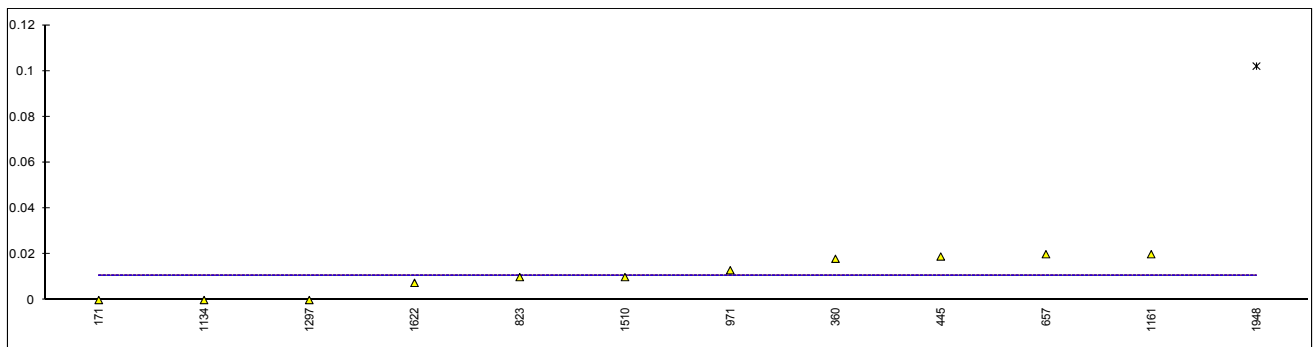
Compare (EN14214:12) = 4.72



Determination of Carbon Residue on 100% FAME on sample #16065; results in %M/M

lab	method	value	mark	z(targ)	remarks
62		----		----	
120	D4530	<0.1		----	
150	D4530	<0.1		----	
171	D189	0		----	
225		----		----	
228		----		----	
253		----		----	
312		----		----	
323		----		----	
333		----		----	
334		----		----	
335		----		----	
336		----		----	
338		----		----	
360	D4530	0.018		----	
391		----		----	
445	EN10370	0.019		----	
511		----		----	
529		----		----	
540		----		----	
556		----		----	
558		----		----	
621	D189	<0.01		----	
657	D4530	0.02		----	
823	D4530	0.01		----	
824	D4530	<0.10		----	
862	D4530	<0.01		----	
970		----		----	
971	D4530	0.013		----	
1033		----		----	
1067		----		----	
1134	D4530	0		----	
1161	D4530	0.02		----	
1199		----		----	
1286		----		----	
1297	D4530	0.000		----	
1397	D4530	<0.01		----	
1429		----		----	
1459		----		----	
1494		----		----	
1510	D4530	0.01		----	
1582		----		----	
1622	D4530	0.0075	C	----	First reported 0.0433
1634		----		----	
1643		----		----	
1744		----		----	
1769		----		----	
1948	EN10370	0.1021	G(0.01)	----	
6033		----		----	
	normality	n.a.			
	n	17			
	outliers	1			
	mean (n)	<0.1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D4530:11)	n.a.			

Application range: 0.10 – 0.30 %M/M



Determination of Copper Strip Corrosion 3 hrs/50°C on sample #16065

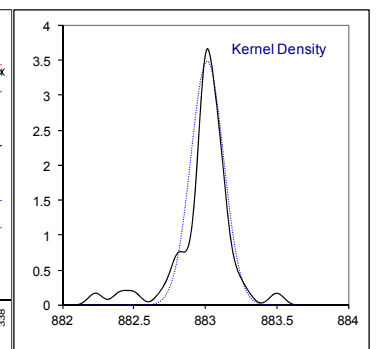
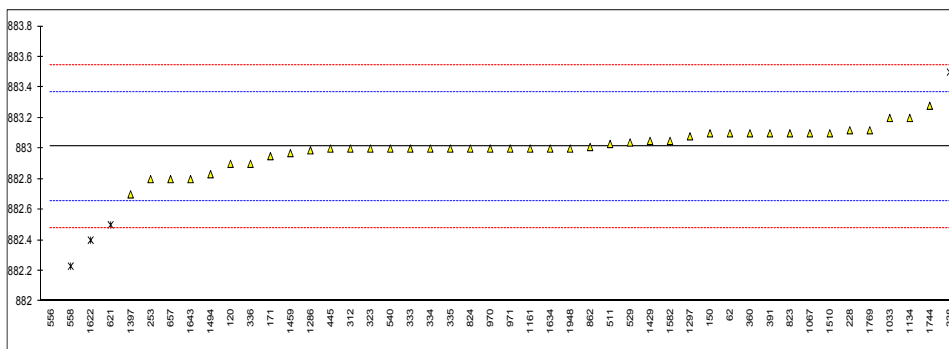
lab	method	value	mark	z(targ)	remarks
62	D130	1b		----	
120	D130	1A		----	
150	D130	1a		----	
171	D130	1a		----	
225		----		----	
228		----		----	
253	D130	1a		----	
312	ISO2160	1A		----	
323	D130	1A		----	
333		----		----	
334		----		----	
335	D130	1b		----	
336	D130	1		----	
338		----		----	
360	D130	1A		----	
391		----		----	
445	IP154	1A		----	
511		----		----	
529	D130	1A		----	
540	D130	1A		----	
556		----		----	
558		----		----	
621	D130	1A		----	
657	D130	1a		----	
823	D130	1a		----	
824	D130	1a		----	
862	D130	1a		----	
970	D130	1a		----	
971	D130	1a		----	
1033	IP154	1a		----	
1067	D130	1a		----	
1134	D130	1a		----	
1161	ISO2160	1A		----	
1199		----		----	
1286		----		----	
1297	D130	1A		----	
1397	D130	1		----	
1429	D130	1A		----	
1459		----		----	
1494		----		----	
1510	D130	1A		----	
1582		----		----	
1622	D130	1a		----	
1634	D130	1a		----	
1643		----		----	
1744		----		----	
1769		----		----	
1948	ISO2160	1A		----	
6033		----		----	
	normality	n.a.			
	n	18			
	outliers	n.a.			
	mean (n)	1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			



Determination of Density at 15°C on sample #16065; results in kg/m<sup>3</sup>

lab	method	value	mark	z(targ)	remarks
62	D4052	883.1		0.49	
120	ISO12185	882.9		-0.63	
150	D4052	883.1		0.49	
171	D4052	882.95	C	-0.35	First reported 0.88295
225		----		----	
228	D4052	883.12		0.60	
253	D4052	882.8		-1.19	
312	ISO12185	883.0		-0.07	
323	ISO12185	883.0		-0.07	
333	ISO12185	883.0		-0.07	
334	ISO12185	883.0		-0.07	
335	ISO12185	883.0		-0.07	
336	ISO12185	882.9		-0.63	
338	ISO12185	883.5	R(0.01)	2.73	
360	ISO12185	883.1		0.49	
391	ISO12185	883.1		0.49	
445	IP365	883.0		-0.07	
511	D4052	883.03		0.10	
529	D4052	883.04		0.15	
540	ISO12185	883.0		-0.07	
556	D4052	880.01	R(0.01)	-16.81	
558	D4052	882.23	R(0.01)	-4.38	
621	D4052	882.5	R(0.01)	-2.87	
657	D4052	882.8		-1.19	
823	ISO12185	883.1		0.49	
824	ISO12185	883.0		-0.07	
862	ISO12185	883.01		-0.01	
970	D4052	883.0		-0.07	
971	D4052	883.0		-0.07	
1033	IP365	883.2		1.05	
1067	ISO12185	883.1		0.49	
1134	IP365	883.2		1.05	
1161	ISO12185	883.0		-0.07	
1199		----		----	
1286	ISO12185	882.9885		-0.13	
1297	D4052	883.08		0.38	
1397	D4052	882.7		-1.75	
1429	ISO12185	883.05		0.21	
1459	ISO12185	882.97		-0.24	
1494	D4052	882.832	C	-1.01	First reported 882.47
1510	IP365	883.1		0.49	
1582	D4052	883.05		0.21	
1622	D4052	882.4	C,R(0.01)	-3.43	First reported 0.8737
1634	ISO12185	883.0		-0.07	
1643	ISO12185	882.8		-1.19	
1744	D4052	883.28		1.50	
1769	D4052	883.120		0.60	
1948	ISO12185	883.0		-0.07	
6033		----		----	

normality suspect  
n 41  
outliers 5  
mean (n) 883.013  
st.dev. (n) 0.1147  
R(calc.) 0.321  
R(ISO12185:96) 0.500

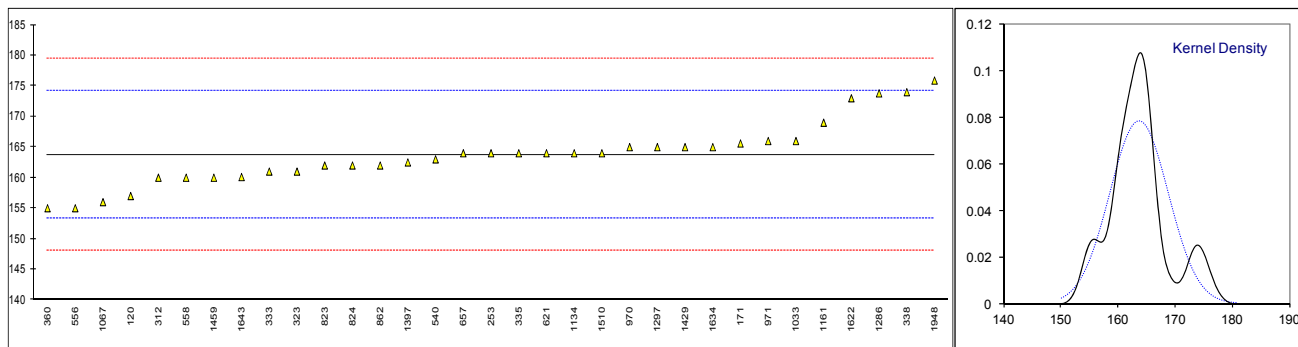


Determination of Flash Point (PMcc) conform ASTM spec. on sample #16065; results in °C

lab	method	value	mark	z(targ)	remarks
62		----		----	
120	D93-C	157		-1.29	
150	D93-C	>130.0		----	
171	D93-C	165.6		0.35	
225		----		----	
228		----		----	
253	D93-B	164.0		0.05	
312	ISO3679	160.0		-0.72	
323	ISO2719-A	161.0		-0.52	
333	D93-C	161.0		-0.52	
334		----		----	
335	D93-C	164		0.05	
336		----		----	
338	D93-C	174.0		1.95	
360	D93-C	155.0		-1.67	
391		----		----	
445	D93-C	N/A		----	
511		----		----	
529		----		----	
540	D93-C	163.0		-0.14	
556	NBR14598	155.0		-1.67	
558	D93-C	160		-0.72	
621	D93-A	164.0		0.05	
657	D93-C	164.0		0.05	
823	D93-C	162.0		-0.33	
824	D93-C	162.0		-0.33	
862	D93-C	162.0		-0.33	
970	D93-C	165.0		0.24	
971	D93-A	166.0		0.43	
1033	ISO2719-A	166.0		0.43	
1067	ISO2719-A	156.0		-1.48	
1134	D93-C	164.0		0.05	
1161	ISO3679	169		1.00	
1199		----		----	
1286		173.8		1.91	
1297	D93-C	165.0		0.24	
1397	D93-C	162.5		-0.24	
1429	D93-A	165.0		0.24	
1459	D93-C	160.0		-0.72	
1494		----		----	
1510	D93-A	164		0.05	
1582		----		----	
1622	D93-A	173.0		1.76	
1634	D93-C	165.0		0.24	
1643	D93-C	160.1		-0.70	
1744		----		----	
1769		----		----	
1948	D93-C	175.9		2.31	
6033		----		----	

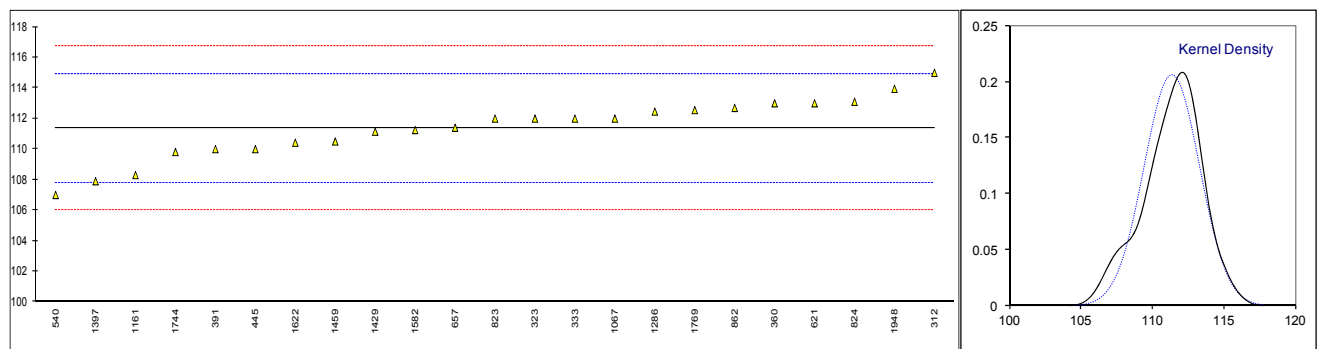
normality OK  
n 33  
outliers 0  
mean (n) 163.75  
st.dev. (n) 5.086  
R(calc.) 14.24  
R(D93C:15) 14.70

Compare R(ISO2719C:16) = 14.70



Determination of Iodine Value conform EN spec. on sample #16065; results in g I<sub>2</sub>/100g

lab	method	value	mark	z(targ)	remarks
62		----		----	
120		----		----	
150		----		----	
171		----		----	
225		----		----	
228		----		----	
253		----		----	
312	EN14111	115.0		2.03	
323	EN14111	112		0.35	
333	EN14111	112	C	0.35	First reported 105
334		----		----	
335		----		----	
336		----		----	
338		----		----	
360	EN14111	113.0		0.91	
391	EN14111	110		-0.77	
445	EN14111	110.0		-0.77	
511		----		----	
529		----		----	
540	EN14111	107		-2.45	
556		----		----	
558		----		----	
621	EN14111	113		0.91	
657	EN14111	111.4		0.02	
823	EN14111	112		0.35	
824	EN14111	113.1		0.97	
862	EN14111	112.7		0.75	
970		----		----	
971		----		----	
1033		----		----	
1067	EN14111	112		0.35	
1134		----		----	
1161	EN14111	108.3		-1.72	
1199		----		----	
1286	EN14111	112.46		0.61	
1297		----		----	
1397	EN16300	107.9		-1.94	
1429	EN14111	111.14		-0.13	
1459	EN14111	110.5		-0.49	
1494		----		----	
1510		----		----	
1582	EN14111	111.25		-0.07	
1622	INH-25	110.42	C	-0.53	First reported 49.9675
1634		----		----	
1643		----		----	
1744	EN14111	109.81		-0.87	
1769	EN14111	112.555		0.66	
1948	EN14111	113.95		1.45	
6033		----		----	
normality		OK			
n		23			
outliers		0			
mean (n)		111.37			
st.dev. (n)		1.937			
R(calc.)		5.42			
R(EN14111:03)		5.00			

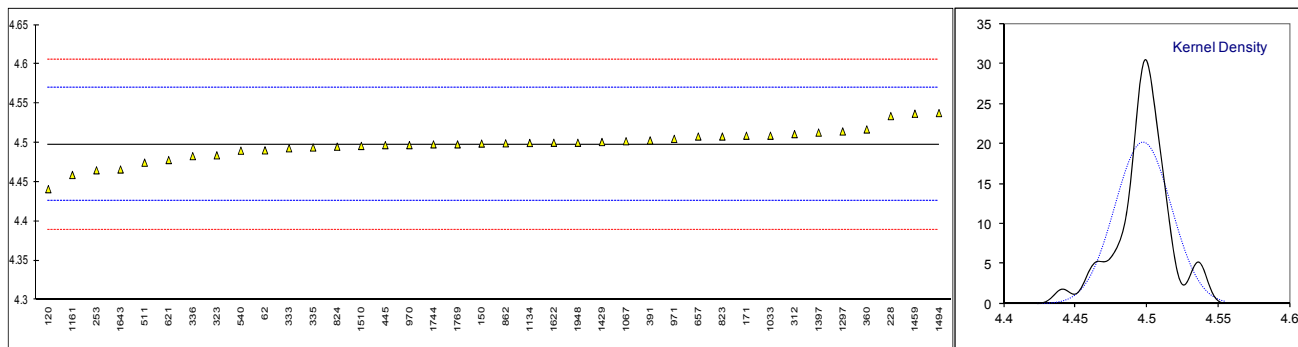


Determination of Kinematic Viscosity at 40°C on sample #16065; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
62	D445	4.4904		-0.20	
120	D445	4.441		-1.57	
150	D445	4.499		0.04	
171	D445	4.509		0.32	
225		----		----	
228	D445	4.534	C	1.01	First reported 4.556
253	D445	4.465		-0.90	
312	ISO3104	4.511	C	0.37	First reported 4.642
323	ISO3104	4.484		-0.38	
333	D445	4.493		-0.13	
334		----		----	
335	ISO3104	4.494		-0.10	
336	D445	4.483		-0.40	
338		----		----	
360	D445	4.5170		0.54	
391	D445	4.503		0.15	
445	IP71	4.497		-0.01	
511	D445	4.4747		-0.63	
529		----		----	
540	D445	4.490		-0.21	
556		----		----	
558		----		----	
621	D445	4.478		-0.54	
657	D445	4.508		0.29	
823	D445	4.508		0.29	
824	ISO3104	4.495		-0.07	
862	D445	4.4993		0.05	
970	D445	4.497		-0.01	
971	D445	4.505		0.21	
1033	IP71	4.509		0.32	
1067	D445	4.502		0.12	
1134	D445	4.4997119		0.06	
1161	ISO3104	4.459		-1.07	
1199		----		----	
1286		----		----	
1297	D7042	4.5144		0.47	
1397	D7042	4.513		0.43	
1429	D445	4.501		0.10	
1459	D7042	4.537		1.10	
1494	D445	4.5378		1.12	
1510	D445	4.496		-0.04	
1582		----		----	
1622	D445	4.500	C	0.07	First reported 4.557
1634		----		----	
1643	D445	4.466		-0.88	
1744	D445	4.498		0.01	
1769	D445	4.49800		0.01	
1948	ISO3104	4.500		0.07	
6033		----		----	

normality suspect  
n 38  
outliers 0  
mean (n) 4.4975  
st.dev. (n) 0.01973  
R(calc.) 0.0552  
R(D445:15a) 0.1007

Compare R(EN14214:12) = 0.0451

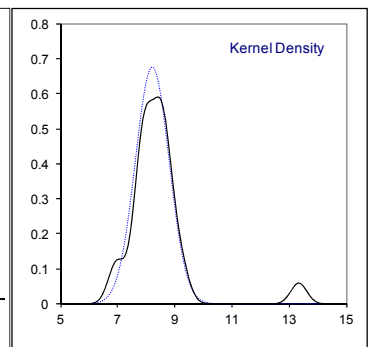
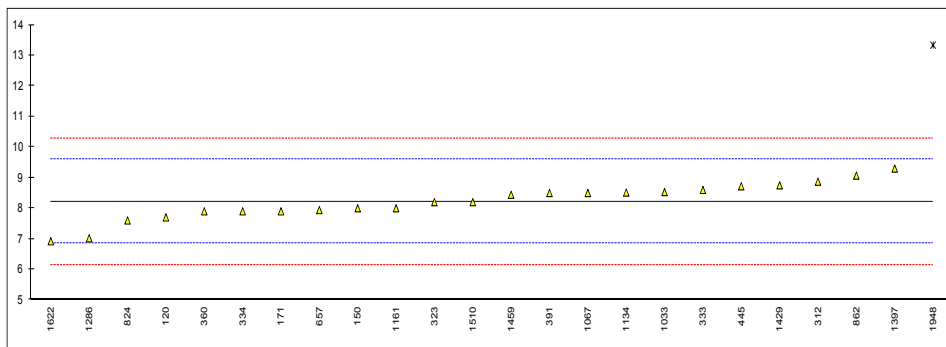


Determination of Oxidation Stability on sample #16065; results in hours

lab	method	value	mark	z(targ)	remarks
62		----		----	
120	EN15751	7.7		-0.75	
150	EN14112	8.0		-0.32	
171	EN15751	7.9		-0.46	
225		----		----	
228		----		----	
253		----		----	
312	EN14112	8.87		0.94	
323	EN15751	8.2		-0.03	
333	EN14112	8.6		0.55	
334	EN15751	7.9		-0.46	
335		----		----	
336		----		----	
338		----		----	
360	EN15751	7.90		-0.46	
391	EN14112	8.5		0.40	
445	EN15751	8.720		0.72	
511		----		----	
529		----		----	
540		----		----	
556		----		----	
558		----		----	
621		----		----	
657	EN15751	7.94		-0.41	
823		----		----	
824	EN15751	7.6		-0.90	
862	EN14112	9.07		1.23	
970		----		----	
971		----		----	
1033	EN15751	8.53		0.45	
1067	EN14112	8.5		0.40	
1134	EN15751	8.515		0.43	
1161	EN14112	8.0		-0.32	
1199		----		----	
1286	EN14112	7.02		-1.73	
1297		----		----	
1397	EN15751	9.3		1.56	
1429	EN14112	8.75		0.76	
1459	EN15751	8.44		0.32	
1494		----		----	
1510	EN14112	8.2		-0.03	
1582		----		----	
1622	EN15751	6.92		-1.88	
1634		----		----	
1643		----		----	
1744		----		----	
1769		----		----	
1948	EN14112	13.34	R(0.01)	7.40	
6033		----		----	

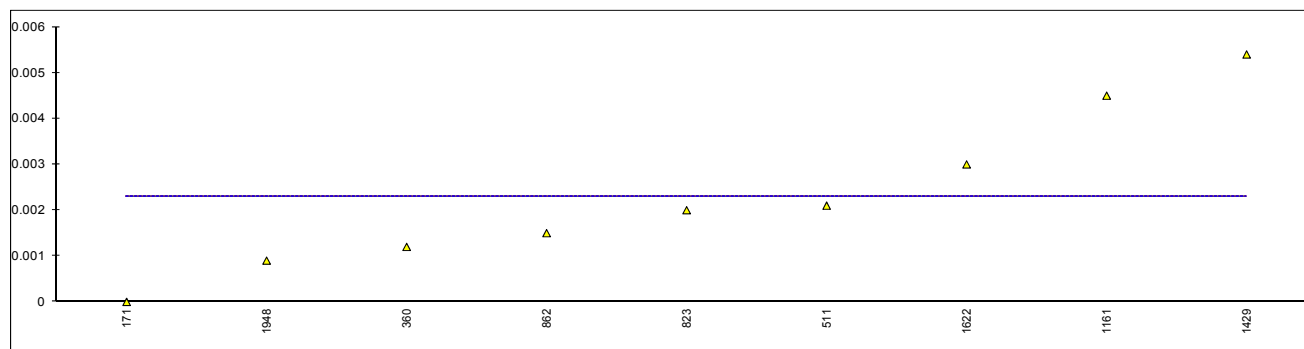
normality OK  
n 23  
outliers 1  
mean (n) 8.221  
st.dev. (n) 0.5894  
R(calc.) 1.650  
R(EN15751:14) 1.938

Compare R(EN14112:03) = 2.367



Determination of Sulphated Ash on sample #16065; results in %M/M

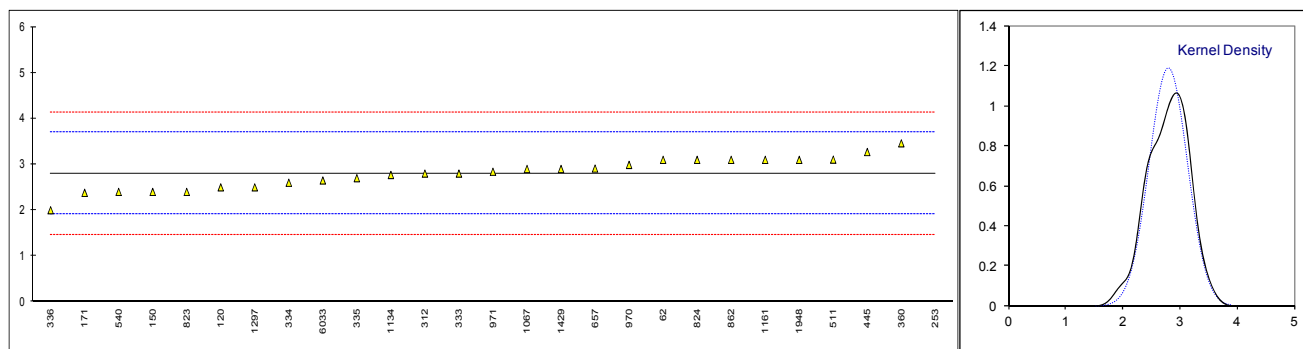
lab	method	value	mark	z(targ)	remarks
62		----		----	
120	D874	<0.001		----	
150	D874	<0.005		----	
171	D874	0		----	
225		----		----	
228		----		----	
253		----		----	
312		----		----	
323	ISO3987	< 0.005		----	
333	D874	<0.005		----	
334		----		----	
335		----		----	
336		----		----	
338		----		----	
360	D874	0.0012		----	
391		----		----	
445	D874	<0.001		----	
511	D874	0.0021		----	
529		----		----	
540	D874	<0.005		----	
556		----		----	
558		----		----	
621	D874	<0.005		----	
657		----		----	
823	D874	0.002		----	
824	D874	<0.005		----	
862	D874	0.0015		----	
970		----		----	
971		----		----	
1033		----		----	
1067		----		----	
1134		----		----	
1161	ISO3987	0.0045		----	
1199		----		----	
1286		----		----	
1297		----		----	
1397	ISO3987	<0.005		----	
1429	ISO3987	0.0054		----	
1459		----		----	
1494		----		----	
1510		----		----	
1582		----		----	
1622	D874	0.003		----	
1634		----		----	
1643		----		----	
1744		----		----	
1769		----		----	
1948	ISO3987	0.0009		----	
6033		----		----	
normality		n.a.			
n		17			
outliers		n.a.			
mean (n)		<0.005			
st.dev. (n)		n.a.			
R(calc.)		n.a.			
R(D874:13a)		n.a.			



Determination of Sulphur on sample #16065; results in mg/kg

lab	method	value	mark	z(targ)	remarks
62	D5453	3.1		0.67	
120	D5453	2.5		-0.67	
150	D5453	2.4		-0.89	
171	D2622	2.38		-0.94	
225		----		----	
228		----		----	
253	D4294	385	R(0.01)	853.08	
312	ISO20846	2.8		0.00	
323	ISO20846	< 3.0		----	
333	D5453	2.8		0.00	
334	D5453	2.6		-0.44	
335	ISO20846	2.7		-0.22	
336	D5453	2.0		-1.78	
338		----		----	
360	D5453	3.46		1.48	
391		----		----	
445	D5453	3.27		1.05	
511	D5453	3.105		0.68	
529		----		----	
540	D5453	2.4		-0.89	
556		----		----	
558		----		----	
621	D4294	< 20		----	
657	D5453	2.91		0.25	
823	D5453	2.4		-0.89	
824	D5453	3.1		0.67	
862	D5453	3.1		0.67	
970	D5453	2.990		0.43	
971	D5453	2.84		0.09	
1033		----		----	
1067	ISO20846	2.9		0.23	
1134	D5453	2.77		-0.06	
1161	ISO20846	3.1		0.67	
1199	ISO20884	<5.0		----	
1286		----		----	
1297	D5453	2.50		-0.67	
1397	ISO20846	<3		----	
1429	ISO20846	2.9		0.23	
1459		----		----	
1494		----		----	
1510		----		----	
1582		----		----	
1622	D5453	<17		----	
1634		----		----	
1643		----		----	
1744		----		----	
1769		----		----	
1948	ISO20846	3.10		0.67	
6033	D5453	2.65		-0.33	
	normality	OK			
	n	26			
	outliers	1			
	mean (n)	2.799			
	st.dev. (n)	0.3358			
	R(calc.)	0.940			
	R(D5453:16)	1.254			

Compare R(ISO20846:11) = 1.433

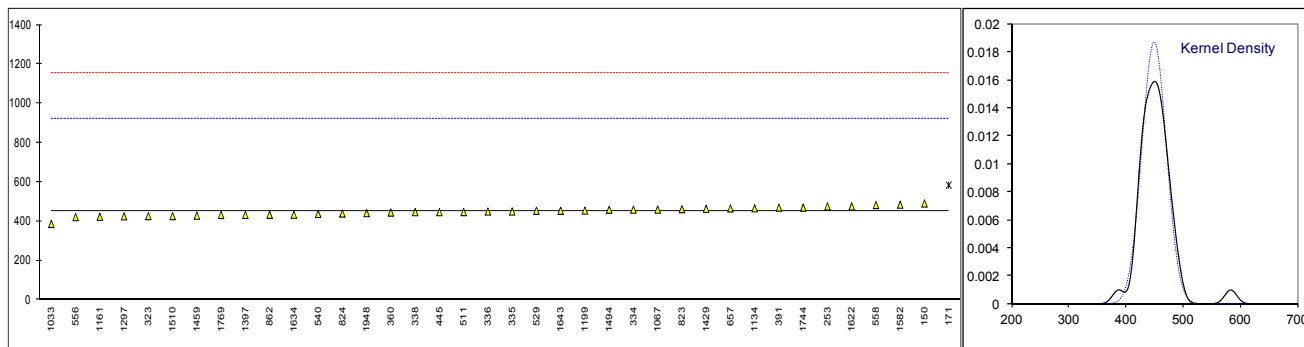


Determination of Water content by KF on sample #16065; results in mg/kg

lab	method	value	mark	z(targ)	remarks
62		----		----	
120		----		----	
150	D6304-A	491		0.18	
171	D6304-C	584.4	C,R(0.01)	0.57	First reported 584.4 %M/M
225		----		----	
228		----		----	
253	D6304-A	476		0.11	
312		----		----	
323	ISO12937	427		-0.10	
333		----		----	
334	ISO12937	460		0.04	
335	ISO12937	450		0.00	
336	ISO12937	450		0.00	
338	ISO12937	447		-0.01	
360	ISO12937	445		-0.02	
391	ISO12937	470		0.09	
445	D6304-A	447		-0.01	
511	D6304-A	447.13		-0.01	
529	D6304	454		0.02	
540	D6304-A	438		-0.05	
556	D6304-A	421.6		-0.12	
558	D6304	483		0.14	
621		----		----	
657	D6304-A	466		0.07	
823	D6304-A	462		0.05	
824	D6304-A	440		-0.04	
862	D6304-A	433.6		-0.07	
970		----		----	
971		----		----	
1033	IP438	387		-0.27	
1067	ISO12937	460		0.04	
1134	IP438	466.575		0.07	
1161	ISO12937	423.222		-0.11	
1199	ISO12937	455		0.02	
1286		----		----	
1297	D6304-A	425.6		-0.10	
1397	ISO12937	433		-0.07	
1429	ISO12937	464		0.06	
1459	ISO12937	429		-0.09	
1494	E203	459.44		0.04	
1510	IP438	427		-0.10	
1582	ISO12937	485.0		0.15	
1622	D6304-A	476.35		0.11	
1634	ISO12937	434		-0.07	
1643	ISO6296	454		0.02	
1744	E203	470		0.09	
1769	ISO12937	432.800		-0.07	
1948	ISO12937	442.02		-0.03	
6033		----		----	

normality OK  
n 37  
outliers 1  
mean (n) 449.52  
st.dev. (n) 21.363  
R(calc.) 59.82  
R(D6304:07) 659.67

Compare R(ISO12937:00) = 145.81





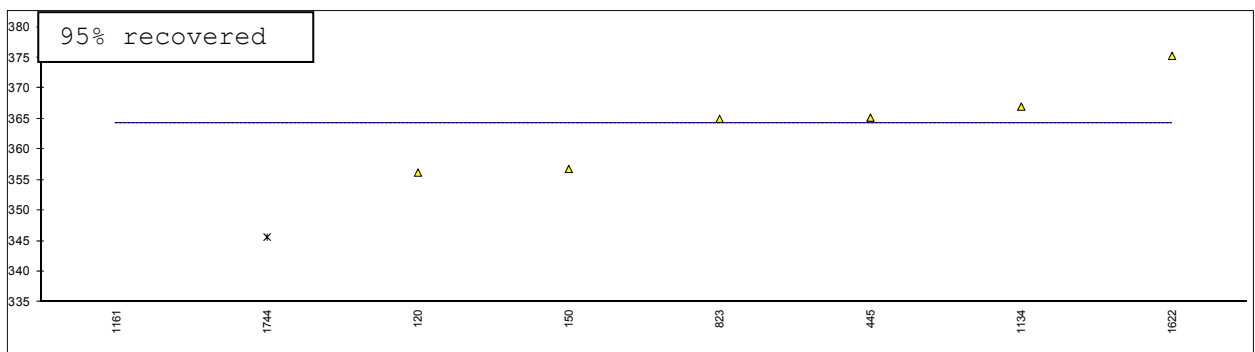
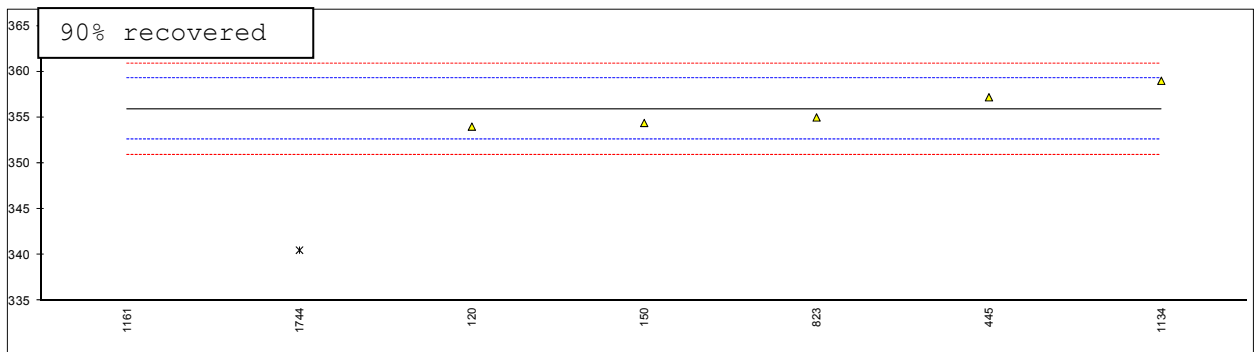
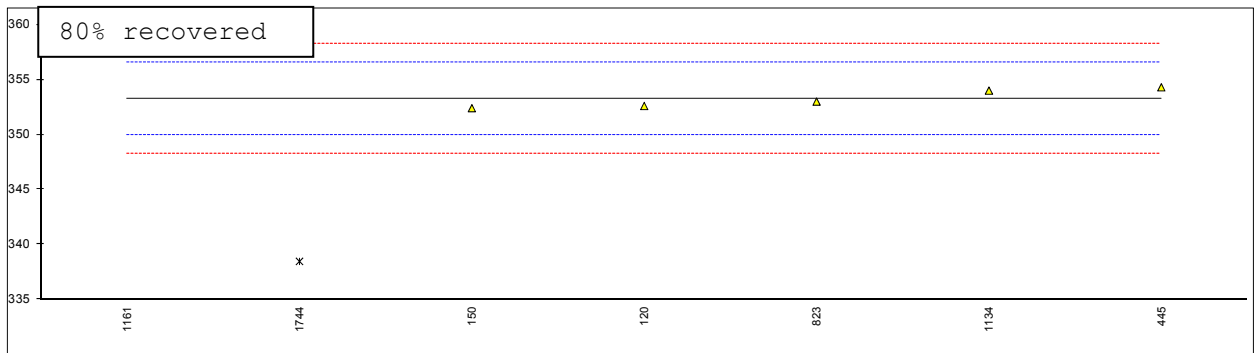
## Determination of Water and sediment on sample #16065; results in %V/V

lab	method	value	mark	z(targ)	remarks
62		----		----	
120	D2709	<0.05		----	
150	D2709	0.005		----	
171	D2709	0.025		----	
225		----		----	
228		----		----	
253		----		----	
312		----		----	
323	D2709	<0.05		----	
333		----		----	
334		----		----	
335		----		----	
336		----		----	
338		----		----	
360	D2709	0.010		----	
391		----		----	
445	D2709	N/A		----	
511	D2709	0.005		----	
529	D2709	0.05		----	
540	D2709	<0.05		----	
556		----		----	
558		----		----	
621	D2709	0.005		----	
657	D2709	0.005		----	
823	D2709	0		----	
824	D2709	0.050		----	
862		----		----	
970		----		----	
971		----		----	
1033		----		----	
1067		----		----	
1134		----		----	
1161		----		----	
1199		----		----	
1286		----		----	
1297		----		----	
1397		----		----	
1429		----		----	
1459		----		----	
1494		----		----	
1510		----		----	
1582		----		----	
1622	D2709	0		----	
1634		----		----	
1643		----		----	
1744		----		----	
1769		----		----	
1948		----		----	
6033		----		----	
	normality	n.a.			
	n	13			
	outliers	n.a.			
	mean (n)	<0.05			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D2709)	n.a.			

Determination of Distillation at 10 mm Hg, % recovered as AET on sample #16065; results in °C

lab	method	80% rec	mark	z(targ)	90% rec	mark	z(targ)	95% rec	mark	z(targ)
62		----		----	----		----	----		----
120	D1160	352.6		-0.40	354.0		-1.16	356.2		----
150	D1160	352.4		-0.52	354.4		-0.92	356.8		----
171		----		----	----		----	----		----
225		----		----	----		----	----		----
228		----		----	----		----	----		----
253		----		----	----		----	----		----
312		----		----	----		----	----		----
323		----		----	----		----	----		----
333		----		----	----		----	----		----
334		----		----	----		----	----		----
335		----		----	----		----	----		----
336		----		----	----		----	----		----
338		----		----	----		----	----		----
360		----		----	----		----	----		----
391		----		----	----		----	----		----
445	D1160	354.3		0.63	357.2		0.77	365.2		----
511		----		----	----		----	----		----
529		----		----	----		----	----		----
540		----		----	----		----	----		----
556		----		----	----		----	----		----
558		----		----	----		----	----		----
621		----		----	----		----	----		----
657		----		----	----		----	----		----
823	D1160	353		-0.16	355		-0.56	365		----
824		----		----	----		----	----		----
862		----		----	----		----	----		----
970		----		----	----		----	----		----
971		----		----	----		----	----		----
1033		----		----	----		----	----		----
1067		----		----	----		----	----		----
1134	D1160	354		0.45	359		1.86	367		----
1161	D1160	209.56	G(0.01)	-86.72	210.8	G(0.01)	-87.57	212.04	G(0.01)	----
1199		----		----	----		----	----		----
1286		----		----	----		----	----		----
1297		----		----	----		----	----		----
1397		----		----	----		----	----		----
1429		----		----	----		----	----		----
1459		----		----	----		----	----		----
1494		----		----	----		----	----		----
1510		----		----	----		----	----		----
1582		----		----	----		----	----		----
1622		----	W	----	----	W	----	375.3		----
1634		----		----	----		----	----		----
1643		----		----	----		----	----		----
1744	D86	338.47	G(0.01)	-8.93	340.52	G(0.05)	-9.29	345.64	G(0.05)	----
1769		----		----	----		----	----		----
1948		----		----	----		----	----		----
6033		----		----	----		----	----		----
	normality	unknown			unknown			unknown		
	n	5			5			6		
	outliers	2			2			2		
	mean (n)	353.26			355.92			364.25		
	st.dev. (n)	0.847			2.119			7.091		
	R(calc.)	2.37			5.93			19.86		
	R(D1160:15)	4.64			4.64			(4.64)		

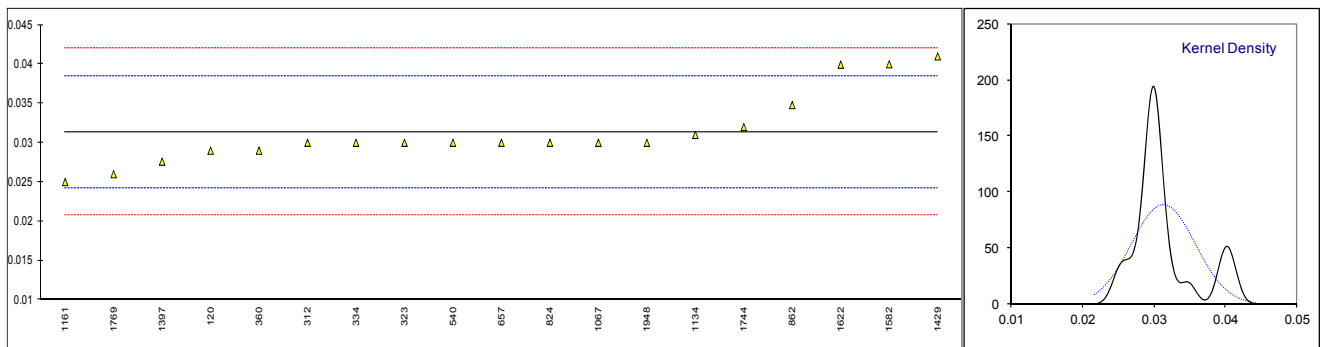
Lab 1622: results withdrawn, reported resp. 374.5, 374.8



Determination of Methanol on sample #16065; results in %M/M

lab	method	value	mark	z(targ)	remarks
62		----		----	
120	EN14110	0.029		-0.66	
150		----		----	
171		----		----	
225		----		----	
228		----		----	
253		----		----	
312	EN14110	0.03		-0.38	
323	EN14110	0.03		-0.38	
333		----		----	
334	EN14110	0.03		-0.38	
335		----		----	
336		----		----	
338		----		----	
360	EN14110	0.029		-0.66	
391		----		----	
445		----		----	
511		----		----	
529		----		----	
540	EN14110	0.03		-0.38	
556		----		----	
558		----		----	
621		----		----	
657	EN14110	0.03		-0.38	
823		----		----	
824	EN14110	0.03		-0.38	
862	EN14110	0.0348		0.98	
970		----		----	
971		----		----	
1033		----		----	
1067	EN14110	0.03		-0.38	
1134	EN14110	0.031		-0.09	
1161	EN14110	0.025		-1.79	
1199		----		----	
1286		----		----	
1297		----		----	
1397	EN14110	0.0276		-1.05	
1429	EN14110	0.041		2.73	
1459		----		----	
1494		----		----	
1510		----		----	
1582	EN14110	0.040		2.44	
1622	EN14110	0.03995		2.43	
1634		----		----	
1643		----		----	
1744	EN14110	0.032		0.19	
1769	EN14110	0.0260	C	-1.50	First reported 0.0200
1948	EN14110	0.03		-0.38	
6033		----		----	

normality suspect  
n 19  
outliers 0  
mean (n) 0.0313  
st.dev. (n) 0.00449  
R(calc.) 0.0126  
R(EN14110:03) 0.0099

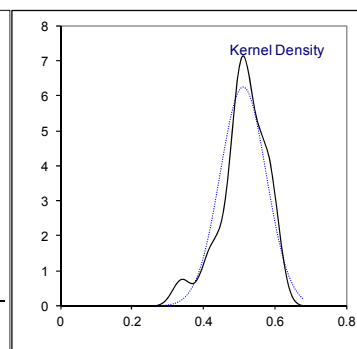
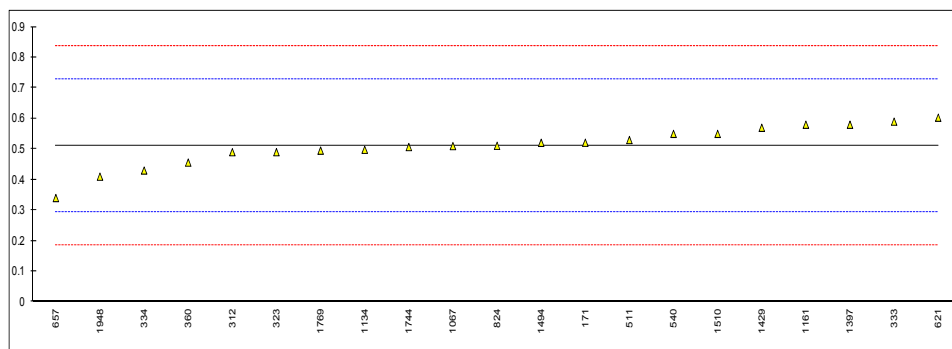


Determination of mono-Glycerides, total on sample #16065; results in %M/M

lab	method	value	mark	z(targ)	remarks
62		----		----	
120		----		----	
150		----		----	
171	D6584	0.521		0.09	
225		----		----	
228		----		----	
253		----		----	
312	EN14105	0.49		-0.19	
323	EN14105	0.49		-0.19	
333	EN14105	0.59		0.73	
334	EN14105	0.43		-0.75	
335		----		----	
336		----		----	
338		----		----	
360	EN14105	0.456		-0.51	
391		----		----	
445		----		----	
511	D6584	0.5298		0.17	
529		----		----	
540	EN14105	0.55		0.36	
556		----		----	
558		----		----	
621	D6584	0.603		0.85	
657	EN14105	0.34		-1.58	
823		----		----	
824	D6584	0.511		0.00	
862		----		----	
970		----		----	
971		----		----	
1033		----		----	
1067	EN14105	0.51		-0.01	
1134	EN14105	0.498		-0.12	
1161	EN14105	0.58		0.64	
1199		----		----	
1286		----		----	
1297		----		----	
1397	EN14105	0.580		0.64	
1429	EN14105	0.57		0.54	
1459		----		----	
1494	D6584	0.5208		0.09	
1510	EN14105	0.55		0.36	
1582		----		----	
1622		----		----	
1634		----		----	
1643		----		----	
1744	D6584	0.5068		-0.04	
1769	D6584	0.4951		-0.15	
1948	EN14105	0.41		-0.93	
6033		----		----	

normality suspect  
n 21  
outliers 0  
mean (n) 0.5110  
st.dev. (n) 0.06389  
R(calc.) 0.1789  
R(D6584:13) 0.3034

Compare R(EN14105) = 0.1608

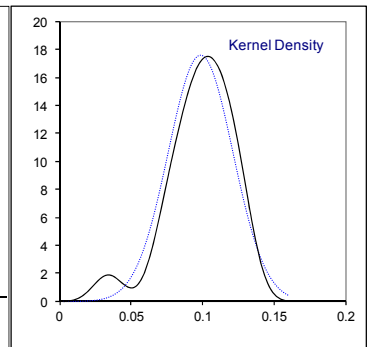
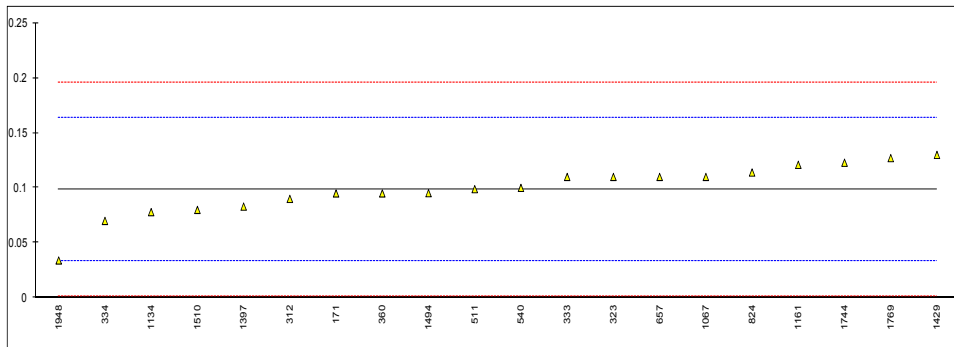


Determination of di-Glycerides, total on sample #16065; results in %M/M

lab	method	value	mark	z(targ)	remarks
62		----		----	
120		----		----	
150		----		----	
171	D6584	0.095		-0.11	
225		----		----	
228		----		----	
253		----		----	
312	EN14105	0.09		-0.27	
323	EN14105	0.11		0.35	
333	EN14105	0.11		0.35	
334	EN14105	0.07		-0.88	
335		----		----	
336		----		----	
338		----		----	
360	EN14105	0.095		-0.11	
391		----		----	
445		----		----	
511	D6584	0.0989		0.01	
529		----		----	
540	EN14105	0.10		0.04	
556		----		----	
558		----		----	
621		----		----	
657	EN14105	0.11		0.35	
823		----		----	
824	D6584	0.114		0.47	
862		----		----	
970		----		----	
971		----		----	
1033		----		----	
1067	EN14105	0.11		0.35	
1134	EN14105	0.078		-0.64	
1161	EN14105	0.121		0.69	
1199		----		----	
1286		----		----	
1297		----		----	
1397	EN14105	0.083		-0.48	
1429	EN14105	0.13		0.96	
1459		----		----	
1494	D6584	0.0952		-0.11	
1510	EN14105	0.08		-0.58	
1582		----		----	
1622		----		----	
1634		----		----	
1643		----		----	
1744	D6584	0.1229		0.74	
1769	D6584	0.1271		0.87	
1948	EN14105	0.034		-1.99	
6033		----		----	

normality not OK  
n 20  
outliers 0  
mean (n) 0.0987  
st.dev. (n) 0.02267  
R(calc.) 0.0635  
R(D6584:13) 0.0910

Compare R(EN14105) = 0.0475

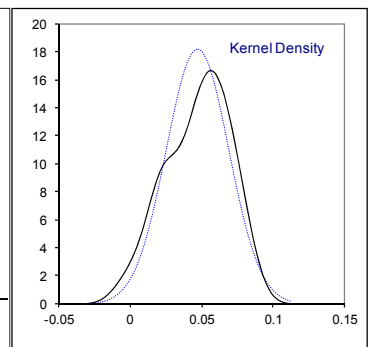
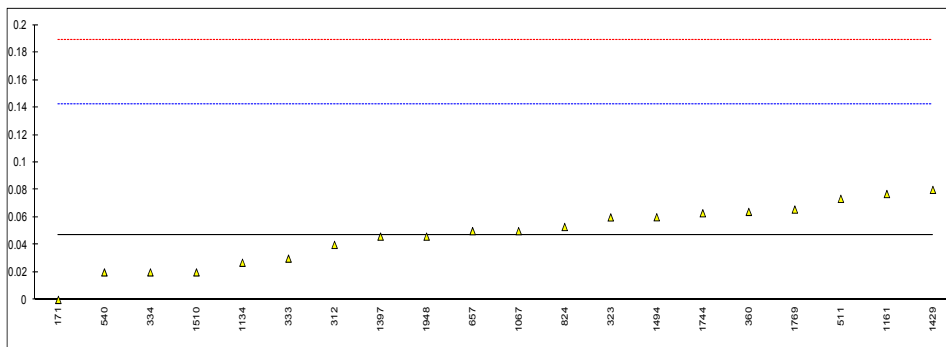


Determination of tri-Glycerides, total on sample #16065; results in %M/M

lab	method	value	mark	z(targ)	remarks
62		----		----	
120		----		----	
150		----		----	
171	D6584	0		-1.00	
225		----		----	
228		----		----	
253		----		----	
312	EN14105	0.04		-0.15	
323	EN14105	0.06		0.27	
333	EN14105	0.03		-0.36	
334	EN14105	0.02		-0.58	
335		----		----	
336		----		----	
338		----		----	
360	EN14105	0.064		0.35	
391		----		----	
445		----		----	
511	D6584	0.0735		0.55	
529		----		----	
540	EN14105	0.02		-0.58	
556		----		----	
558		----		----	
621		----		----	
657	EN14105	0.05		0.06	
823		----		----	
824	D6584	0.053		0.12	
862		----		----	
970		----		----	
971		----		----	
1033		----		----	
1067	EN14105	0.05		0.06	
1134	EN14105	0.027		-0.43	
1161	EN14105	0.077		0.63	
1199		----		----	
1286		----		----	
1297		----		----	
1397	EN14105	0.046		-0.03	
1429	EN14105	0.08		0.69	
1459		----		----	
1494	D6584	0.0601		0.27	
1510	EN14105	0.02		-0.58	
1582		----		----	
1622		----		----	
1634		----		----	
1643		----		----	
1744	D6584	0.063		0.33	
1769	D6584	0.0657		0.39	
1948	EN14105	0.046		-0.03	
6033		----		----	

normality OK  
n 20  
outliers 0  
mean (n) 0.0473  
st.dev. (n) 0.02190  
R(calc.) 0.0613  
R(D6584:13) 0.1327

Compare R(EN14105) = 0.0605

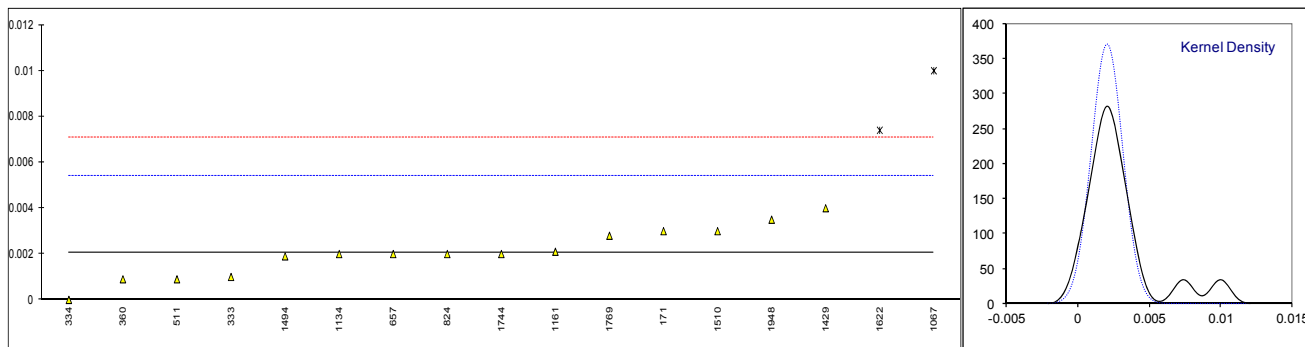


Determination of Free Glycerine on sample #16065; results in %M/M

lab	method	value	mark	z(targ)	remarks
62		----		----	
120		----		----	
150		----		----	
171	D6584	0.003		0.55	
225		----		----	
228		----		----	
253		----		----	
312	EN14105	<0.001		----	
323	EN14105	<0.005	C	----	First reported 0.057
333	EN14105	0.001		-0.64	
334	EN14105	0		-1.24	
335		----		----	
336		----		----	
338		----		----	
360	EN14105	0.0009		-0.70	
391		----		----	
445		----		----	
511	D6584	0.0009		-0.70	
529		----		----	
540		----		----	
556		----		----	
558		----		----	
621	D6584	<0.005		----	
657	EN14105	0.002		-0.04	
823		----		----	
824	D6584	0.002		-0.04	
862		----		----	
970		----		----	
971		----		----	
1033		----		----	
1067	EN14105	0.01	G(0.01)	4.72	
1134	EN14105	0.0020		-0.04	
1161	EN14105	0.0021		0.02	
1199		----		----	
1286		----		----	
1297		----		----	
1397	EN14105	<0.005		----	
1429	EN14105	0.004	C	1.15	First reported 0.017
1459		----		----	
1494	D6584	0.0019		-0.10	
1510	EN14105	0.003		0.55	
1582		----		----	
1622	D6584	0.0074	G(0.01)	3.17	
1634		----		----	
1643		----		----	
1744	D6584	0.0020		-0.04	
1769	D6584	0.0028		0.43	
1948	EN14105	0.0035		0.85	
6033		----		----	

normality OK  
n 15  
outliers 2  
mean (n) 0.00207  
st.dev. (n) 0.001077  
R(calc.) 0.00302  
R(D6584:13) 0.00470

Compare R(EN14105) = 0.00648



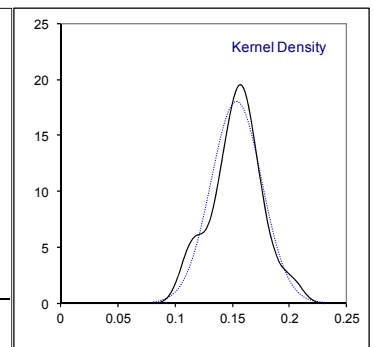
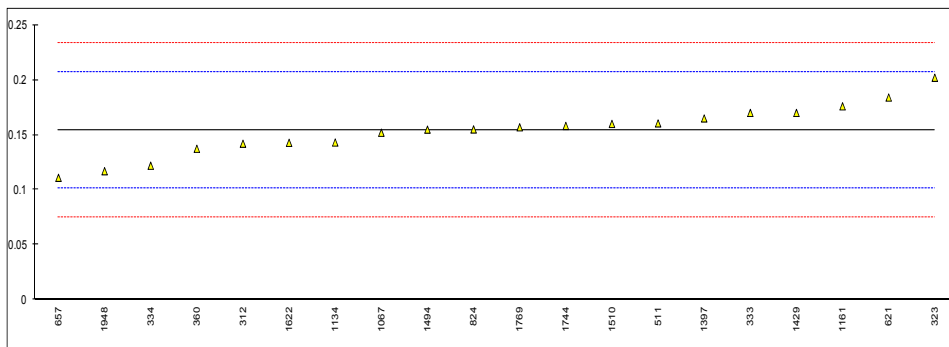


Determination of Total Glycerine on sample #16065; results in %M/M

lab	method	value	mark	z(targ)	remarks
62		----		----	
120		----		----	
150		----		----	
171		----		----	
225		----		----	
228		----		----	
253		----		----	
312	EN14105	0.142		-0.45	
323	EN14105	0.202		1.82	
333	EN14105	0.170		0.61	
334	EN14105	0.122		-1.21	
335		----		----	
336		----		----	
338		----		----	
360	EN14105	0.1374		-0.63	
391		----		----	
445		----		----	
511	D6584	0.1605		0.25	
529		----		----	
540		----		----	
556		----		----	
558		----		----	
621	D6584	0.184		1.14	
657	EN14105	0.111		-1.63	
823		----		----	
824	D6584	0.155		0.04	
862		----		----	
970		----		----	
971		----		----	
1033		----		----	
1067	EN14105	0.152		-0.08	
1134	EN14105	0.1431		-0.41	
1161	EN14105	0.176		0.83	
1199		----		----	
1286		----		----	
1297		----		----	
1397	EN14105	0.165		0.42	
1429	EN14105	0.17	C	0.61	First reported 0.19
1459		----		----	
1494	D6584	0.1548		0.03	
1510	EN14105	0.16		0.23	
1582		----		----	
1622	D6584	0.1428		-0.42	
1634		----		----	
1643		----		----	
1744	D6584	0.1582		0.16	
1769	D6584	0.1569		0.11	
1948	EN14105	0.117		-1.40	
6033		----		----	

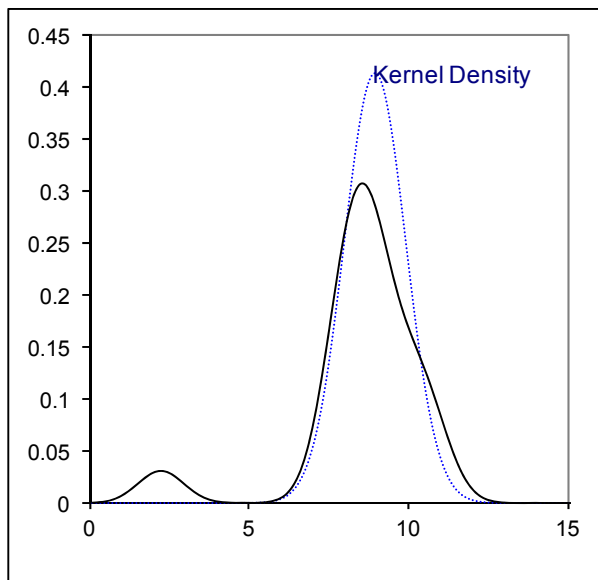
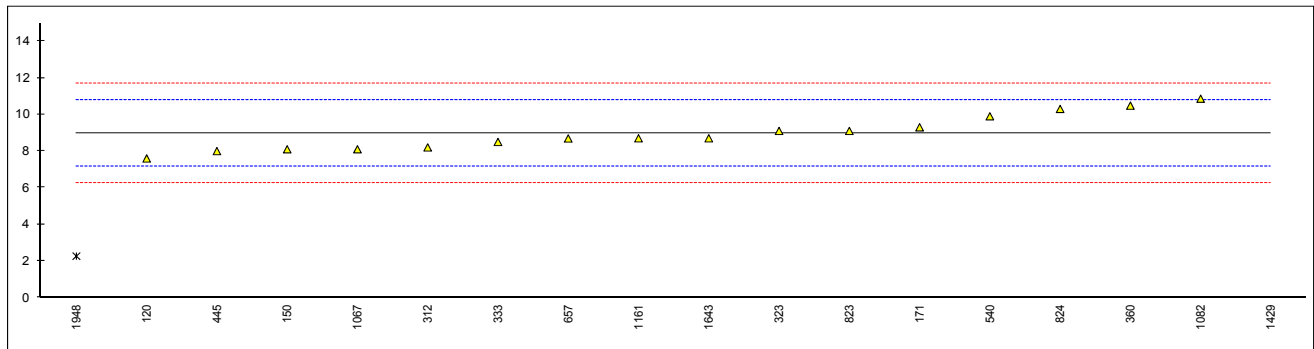
normality OK  
n 20  
outliers 0  
mean (n) 0.1540  
st.dev. (n) 0.02217  
R(calc.) 0.0621  
R(D6584:13) 0.0740

Compare R (EN14105) = 0.0408



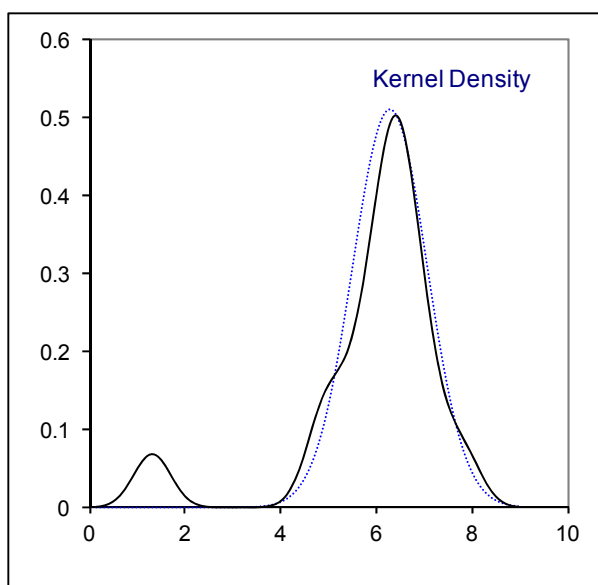
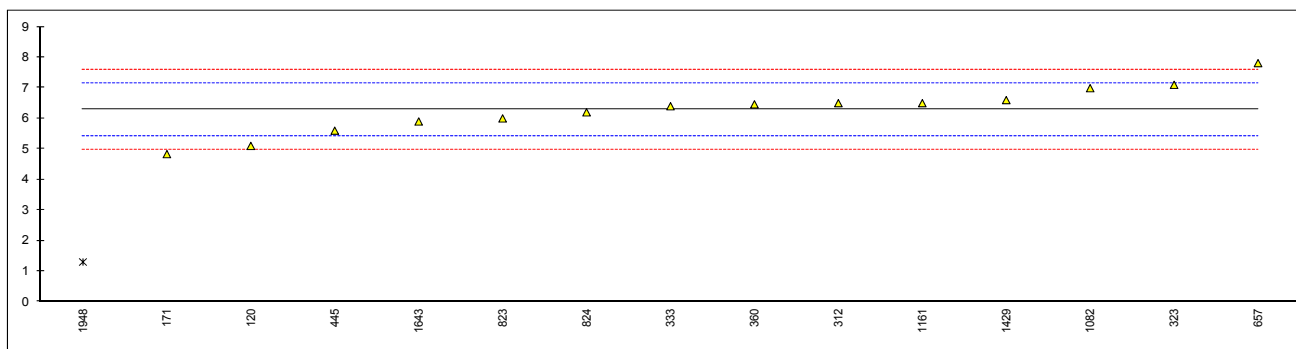
Determination of sum of Calcium and Magnesium (as Ca + Mn) on sample #16066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	EN14538	7.6	C	-1.53	First reported 3.7
150	EN14538	8.1		-0.97	
171	EN14538	9.3		0.36	
312	EN14538	8.2		-0.86	
323	EN14538	9.1		0.14	
333	EN14538	8.5		-0.53	
360	EN14538	10.48		1.67	
391		----		----	
445	EN14538	8.0		-1.08	
540	EN14538	9.9		1.02	
657	EN14538	8.69		-0.32	
823	EN14538	9.1		0.14	
824	EN14538	10.3		1.47	
1067	EN14538	8.1		-0.97	
1082	D5185	10.86		2.09	
1134		----		----	
1161	EN14538	8.69992		-0.31	
1429	EN14538	36.2	C,G(0.01)	30.21	First reported 21.3
1643	D5185	8.7		-0.31	
1948	EN14538	2.27	G(0.01)	-7.44	
normality		OK			
n		16			
outliers		2			
mean (n)		8.977			
st.dev. (n)		0.9660			
R(calc.)		2.705			
R(EN14538:06)		2.524			



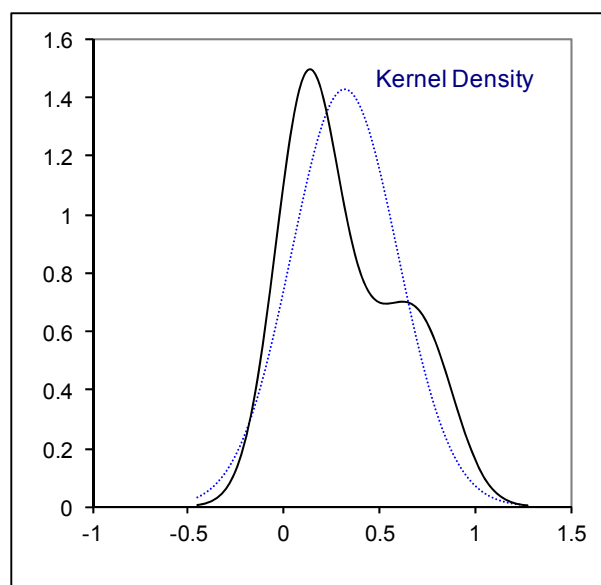
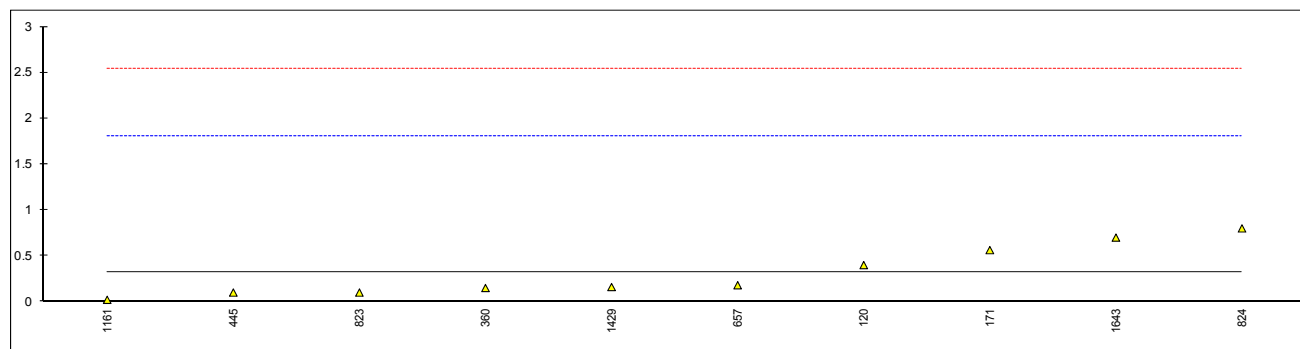
Determination of Phosphorus as P on sample #16066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	D4951	5.1	C	-2.69	First reported 1.5
150					
171	D4951	4.831		-3.31	
312	EN14107	6.5		0.49	
323	EN14107	7.1		1.85	
333	EN14538	6.4		0.26	
360	EN14107	6.46		0.40	
391					
445	EN14107	5.6	C	-1.56	First reported 2.8
540					
657	EN14107	7.81		3.47	
823	D4951	6		-0.65	
824	D4951	6.2		-0.19	
1067					
1082	D5185	6.99		1.60	
1134					
1161	EN14107	6.50	C	0.49	First reported 1.86362
1429	EN14107	6.6		0.72	
1643	D5185	5.9		-0.88	
1948	EN14107	1.30	G(0.01)	-11.33	
normality		OK			
n		14			
outliers		1	Spike		
mean (n)		6.285	5.94		Recovery: <106%
st.dev. (n)		0.7818			
R(calc.)		2.189			
R(EN14107:03)		1.232			Application range 4 – 20 mg/kg
Compare					
R(D4951:14)		0.635			Application range: 0.05 – 0.12 %M/M



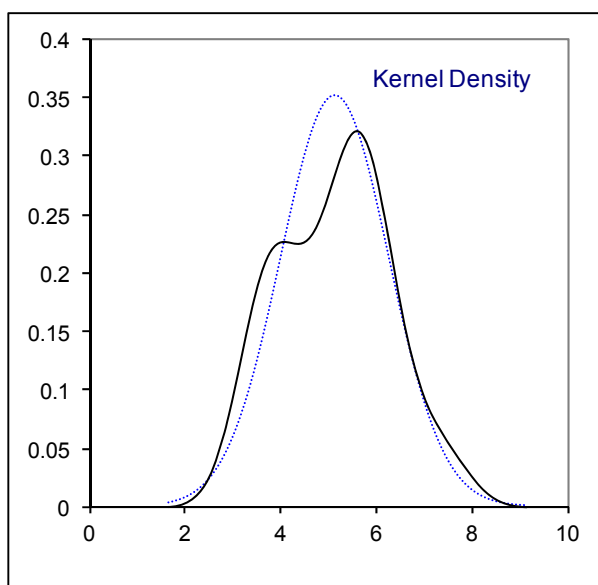
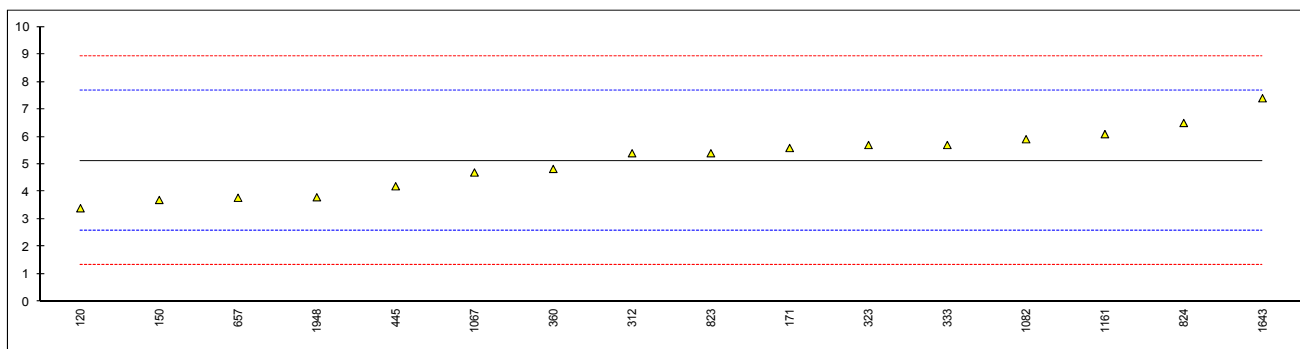
Determination of Potassium as K on sample #16066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	EN14109	0.4		0.11	
150	EN14109	<1.0		----	
171	EN14538	0.563		0.33	
312	EN14109	<0.1		----	
323	EN14538	<1.0		----	
333	EN14538	<1.0		----	
360	EN14538	0.15		-0.23	
391		----		----	
445	EN14109	0.1		-0.29	
540		----		----	
657	EN14109	0.18		-0.19	
823	EN14109	0.1		-0.29	
824	EN14538	0.8		0.65	
1067	EN14538	<1.0		----	
1082		----		----	
1134		----		----	
1161	EN14109	0.02095		-0.40	
1429	EN14538	0.16		-0.21	
1643	D5185	0.7	C	0.52	First reported 1.8
1948	EN14109	<0.1		----	
normality		OK			
n		10			
outliers		0			
mean (n)		0.317			
st.dev. (n)		0.2790			
R(calc.)		0.781			
R(EN14214:14)		2.077			Application range: ≥ 0.5 mg/kg
Compare					
R(EN14109:03)		0.682			



Determination of Sodium as Na on sample #16066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	EN14108	3.4		-1.37	
150	EN14108	3.7		-1.13	
171	EN14538	5.591		0.36	
312	EN14108	5.4		0.21	
323	EN14538	5.7		0.45	
333	EN14538	5.7		0.45	
360	EN14538	4.83		-0.24	
391		----		----	
445	EN14108	4.2		-0.74	
540		----		----	
657	EN14108	3.78		-1.07	
823	EN14108	5.4		0.21	
824	EN14538	6.5		1.08	
1067	EN14538	4.7		-0.34	
1082	D5185	5.91		0.61	
1134		----		----	
1161	EN14108	6.095		0.76	
1429		----		----	
1643	D5185	7.4		1.79	
1948	EN14108	3.80		-1.05	
	normality	OK			
	n	16			
	outliers	0	Spike		
	mean (n)	5.132	5.99		Recovery: <86%
	st.dev. (n)	1.1357			
	R(calc.)	3.180			
	R(EN14214:14)	3.545			Application range: ≥1 mg/kg
Compare					
	R(EN14108:03)	2.705			

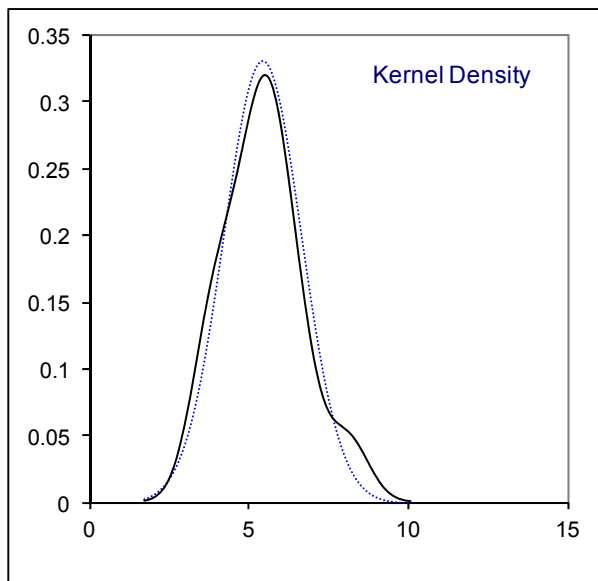
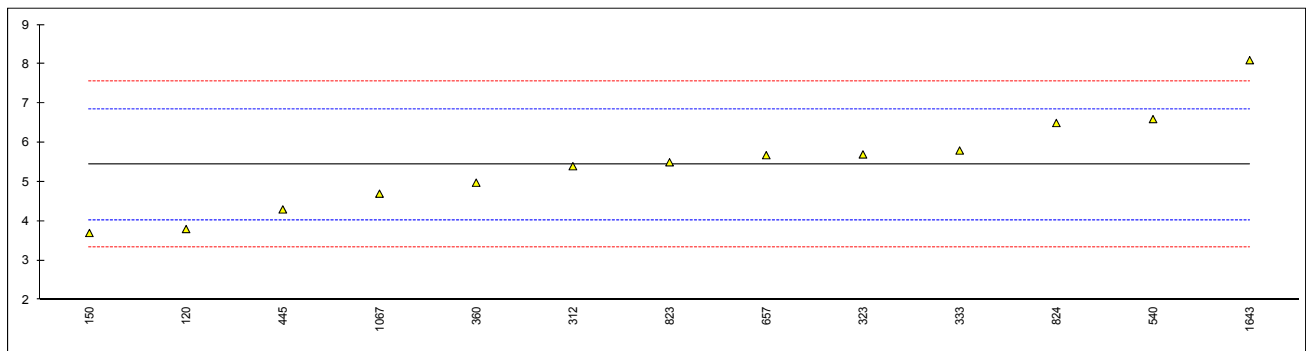


Determination of sum of Potassium and Sodium (as K + Na) on sample #16066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	EN14538	3.8		-2.32	
150	EN14538	3.7		-2.46	
171		----		----	
312	Calculated	5.4		-0.06	
323	EN14538	5.7		0.36	
333	EN14538	5.8		0.50	
360	EN14538	4.98		-0.65	
391		----		----	
445	EN14538	4.3		-1.62	
540	EN14538	6.6		1.64	
657	EN14538	5.68		0.33	
823	EN14538	5.5		0.08	
824	EN14538	6.5		1.49	
1067	EN14538	4.7		-1.05	
1082		----		----	
1134		----		----	
1161		----		----	
1429		----		----	
1643	D5185	8.1	C	3.76	First reported 9.2
1948		----		----	

normality OK  
n 13  
outliers 0  
mean (n) 5.443  
st.dev. (n) 1.2104  
R(calc.) 3.389  
R(EN14538:06) 1.981

Application range: 1- 10 mg/kg

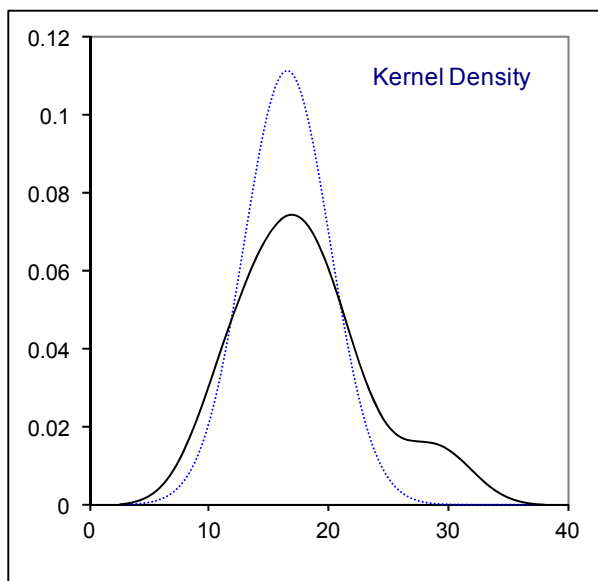
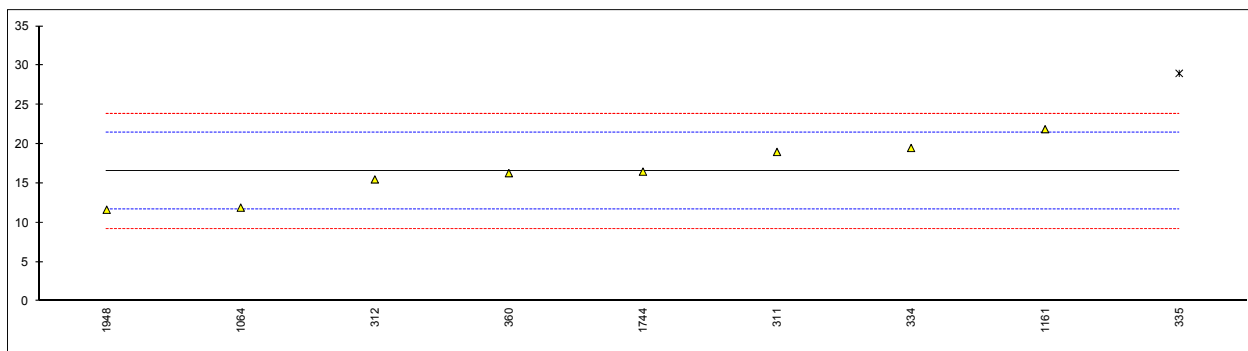


## Determination of Total Contamination (ASTM D7321) on sample #16067; results in mg/L

lab	method	value	mark	z(targ)	remarks
120	D7321	19.9		----	
150		----		----	
171	D7321	3.0		----	
311		----		----	
312		----		----	
323		----		----	
334		----		----	
335		----		----	
360		----		----	
391		----		----	
445		----		----	
540		----		----	
657		----		----	
862		----		----	
1033		----		----	
1064		----		----	
1067		----		----	
1095		----		----	
1134		----		----	
1161		----		----	
1397		----		----	
1429		----		----	
1582		----		----	
1744		----		----	
1948		----		----	
	normality	n.a.			
	n	2			
	outliers	n.a.			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D7321:14)	n.a.			

Determination of Total Contamination (EN12662:2014) on sample #16067; results in mg/kg

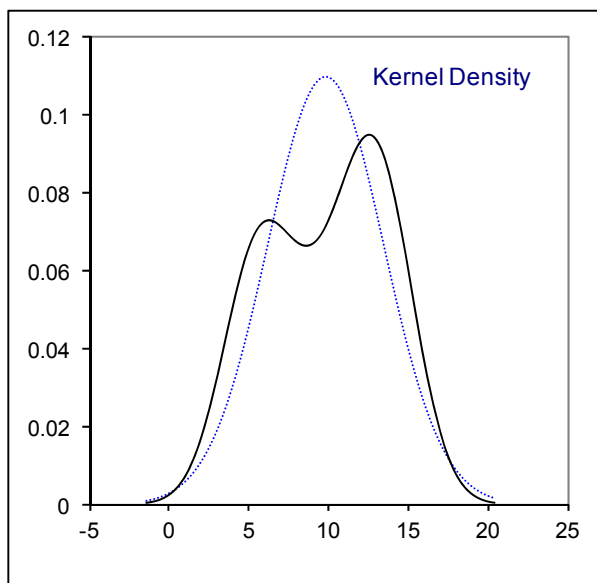
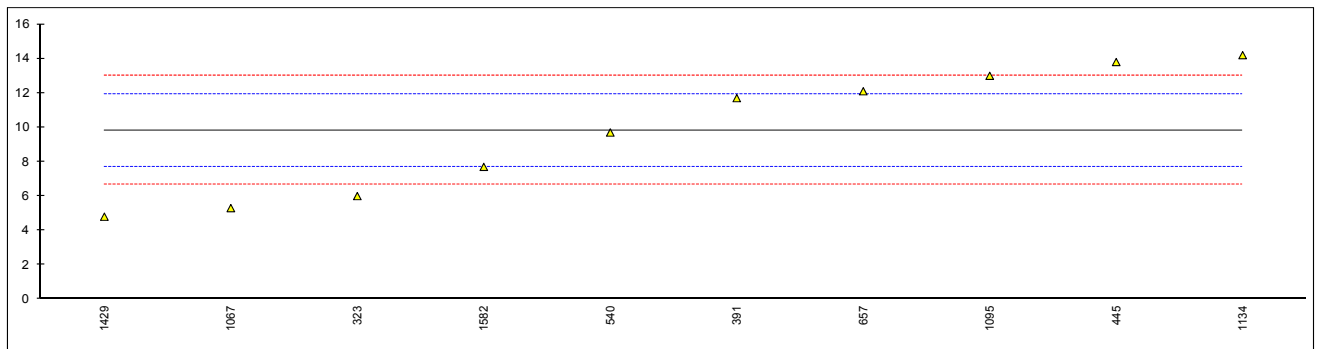
lab	method	value	mark	z(targ)	Vol. filtered (ml)	No. of filtrations	remarks
120		----		----	----	----	
150		----		----	----	----	
171		----		----	----	----	
311	EN12662:2014	19.0		1.01	300	----	
312	EN12662:2014	15.5		-0.42	300	1	
323		----		----	----	----	
334	EN12662:2014	19.5		1.22	290	1	
335	EN12662:2014	29	G(0.05)	5.11	----	----	
360	EN12662:2014	16.3		-0.09	300	----	
391		----		----	----	----	
445		----		----	----	----	
540		----		----	----	----	
657		----		----	----	----	
862		----		----	----	----	
1033		----		----	----	----	
1064	EN12662:2014	11.906		-1.90	272.95	2	
1067		----		----	----	----	
1095		----		----	----	----	
1134		----		----	----	----	
1161	EN12662:2014	21.9		2.20	----	----	
1397		----		----	----	----	
1429		----		----	----	----	
1582		----		----	----	----	
1744	EN12662:2014	16.49		-0.02	300	----	
1948	EN12662:2014	11.64		-2.00	----	----	
normality		OK					
n		8					
outliers		1					
mean (n)		16.529					
st.dev. (n)		3.5890					
R(calc.)		10.049					
R(EN12662:14)		6.828					





Determination of Total Contamination (EN12662:1998(2008)) on sample #16067; results in mg/kg

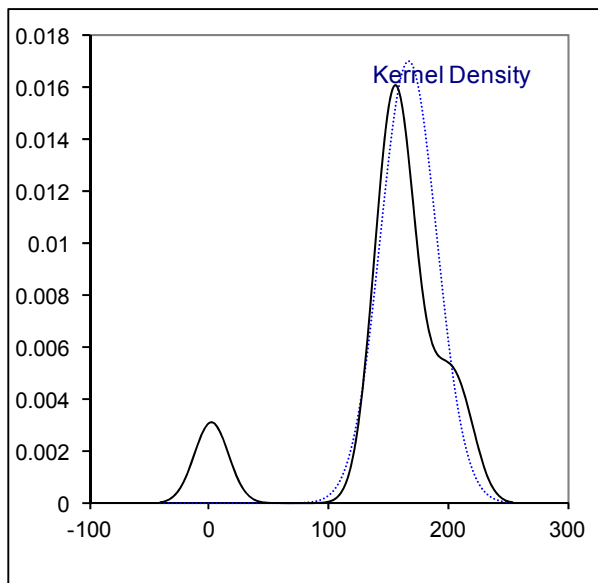
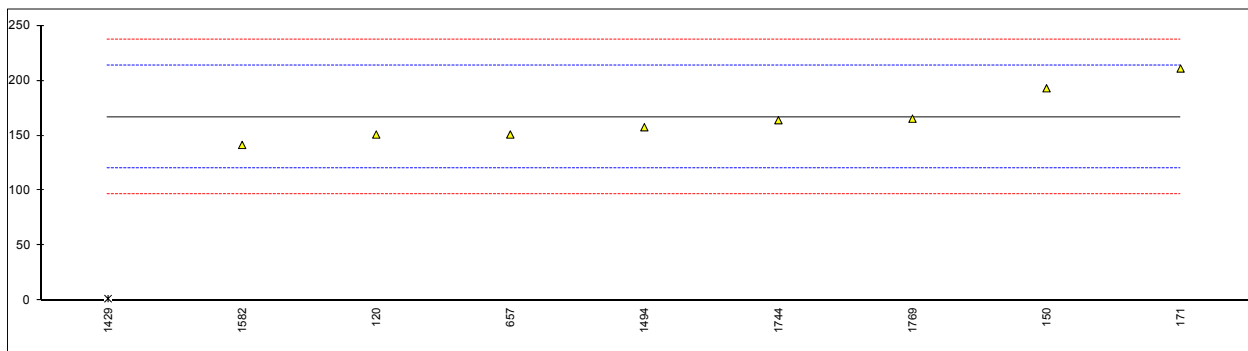
lab	method	value	mark	z(targ)	Vol. filtered (ml)	No. of filtrations	remarks
120		----		----	----	----	
150		----		----	----	----	
171		----		----	----	----	
311		----		----	----	----	
312		----		----	----	----	
323	EN12662:1998	6		-3.64	277.1	2	
334		----		----	----	----	
335		----		----	----	----	
360		----		----	----	----	
391	EN12662:2008	11.7		1.78	----	----	
445	EN12662:1998	13.80		3.77	312	1	
540	EN12662-1998	9.7		-0.12	----	----	
657	EN12662:2008	12.1		2.16	800	1	
862		----		----	----	----	
1033		----		----	----	----	
1064		----		----	----	----	
1067	EN12662:1998	5.3		-4.30	280	----	
1095	EN12662:1998	13		3.01	300	----	
1134	EN12662:1998	14.2		4.15	318	2	
1161		----		----	----	----	
1397		----		----	----	----	
1429	EN12662:1998	4.8		-4.78	400	1	
1582	EN12662:2008	7.70		-2.02	----	----	
1744		----		----	----	----	
1948		----		----	----	----	
normality		OK					
n		10					
outliers		0					
mean (n)		9.830					
st.dev. (n)		3.6301					
R(calc.)		10.164					
R(EN12662:98)		2.949					



Determination of Filter Blocking Potential by Cold Soak Filterability Test (CSFT) on sample #16068; results in s

lab	method	value	mark	z(targ)	Vol. at time >720sec	remarks
120	D7501	151		-0.67	----	
150	D7501	193		1.12	0	
171	D7501	211		1.89	----	
312		----		----	----	
323		----		----	----	
360		----		----	----	
445		----		----	----	
657	D7501	151		-0.67	----	
1033		----		----	----	
1067		----		----	----	
1095		----		----	----	
1134		----		----	----	
1161		----		----	----	
1429	IP PM-EA	1.74	G(0.01)	-7.04	----	
1494	D7501	157.62		-0.39	----	
1582	D7501	141.5		-1.08	----	
1739		----		----	----	
1744	D7501	164		-0.12	----	
1769	D7501	165.38		-0.06	----	
1948		----		----	----	

normality suspect  
n 8  
outliers 1  
mean (n) 166.81  
st.dev. (n) 23.524  
R(calc.) 65.87  
R(D7501:12a) 65.62

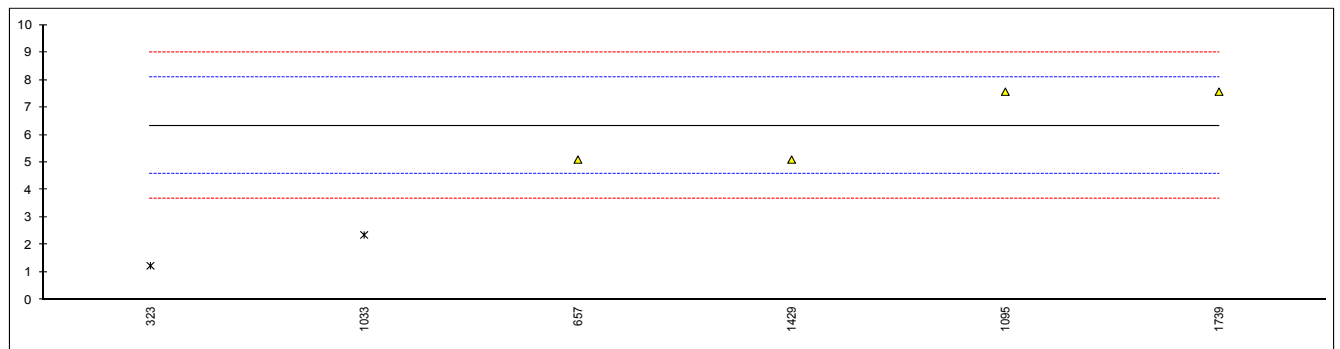


Determination of Filter Blocking Tendency (FBT) on sample #16068

lab	method	value	mark	z(targ)	press. end test (kPa)	vol. pumped (ml)	Remarks
120		----		----	----	----	
150		----		----	----	----	
171		----		----	----	----	
312		----		----	----	----	
323	IP387-B	1.24	ex	-5.77	75	300	Calculated on pressure
360		----		----	----	----	
445		----		----	----	----	
657	D2068-B	5.10		-1.40	105	60	
1033	IP387-B	2.36	G(0.05)	-4.50	105	140	
1067		----		----	----	----	
1095	IP387-B	7.57		1.40	105	40	
1134		----		----	----	----	
1161		----		----	----	----	
1429	IP387-B	5.10		-1.40	105	60	
1494		----		----	----	----	
1582		----		----	----	----	
1739	IP387-B	7.57		1.40	----	40	
1744		----		----	----	----	
1769		----		----	----	----	
1948		----		----	----	----	

normality unknown  
n 4  
outliers 1 (+1 excl)  
mean (n) 6.335  
st.dev. (n) 1.4261  
R(calc.) 3.993  
R(D2068:14) 2.472

Compare R(IP387-B) = 2.472 (methods identical)



**APPENDIX 2****Number of participants per country**

1 lab in ARGENTINA  
1 lab in BELGIUM  
2 labs in BRAZIL  
1 lab in BULGARIA  
1 lab in CANADA  
1 lab in CHINA, People's Republic  
4 labs in COLOMBIA  
1 lab in COTE D'IVOIRE  
1 lab in GREECE  
1 lab in CROATIA  
1 lab in DJIBOUTI  
1 lab in FINLAND  
6 labs in FRANCE  
1 lab in GERMANY  
1 lab in HONG KONG  
2 labs in INDONESIA  
1 lab in ITALY  
1 lab in MEXICO  
4 labs in NETHERLANDS  
1 lab in OMAN  
1 lab in PERU  
3 labs in PORTUGAL  
1 lab in SINGAPORE  
2 labs in SOUTH KOREA  
1 lab in SPAIN  
1 lab in SWEDEN  
1 lab in TOGO  
2 labs in TURKEY  
1 lab in UNITED ARAB EMIRATES  
5 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 outlier test
R(0.05)	= straggler in Rosner outlier test
W	= test result withdrawn on request of participant
ex	= test result excluded from calculations
n.a.	= not applicable
n.d.	= not detected
SDS	= Safety Data Sheet

### Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
- 2 ASTM E178-02
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- 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, No4 January 2001
- 14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson.
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983).