# Results of Proficiency Test
# Biodiesel 100% FAME (B100)
# April 2014


Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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**CONTENTS**

# 1 INTRODUCTION

Since 2001, a proficiency test for Fatty Acid Methyl Esters (FAME) used as Biodiesel B100 is organised every year by the Institute for Interlaboratory Studies (iis).
In this interlaboratory study 73 laboratories from 33 different countries have participated. See appendix 2 for a list of number of participants per country. In this report the results of the 2014 Biodiesel B100 proficiency test are presented and discussed.

# 2 SET UP

In this proficiency test Biodiesel B100, a sample Rapeseed methyl ester was used. Sample analyses for fit-for-use and homogeneity testing were subcontracted. 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 mL | Purpose | Spiked |
|---|---|---|---|
| #14045 | 1000 | For regular analysis | - |
| #14046 | 100 | Analysis of Phosphorus, Potassium, Sodium and Calcium & Magnesium | Phosphorus, Sodium Calcium & Magnesium |
| #14047 | 1000 | Total Contamination test | Quartz materal |
| #14048 | 500 | Cold Soak Test | - |

table 1: four different Biodiesel B100 samples used in iis14G02

The test scope was set up according to both EN14214+A1:2014 and ASTM D6751:12 specifications. Participants were requested to report the analytical results as “rounded and unrounded results” and to use the indicated units on the report form(s). The unrounded results were preferably used for statistical evaluation.

## 2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, 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% confidentially 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).

## 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 Biodiesel B100 was obtained from a European producer. After fit-for-use testing and homogenisation in a precleaned metal drum, the B100 was transferred to 81 brown glass bottles of 1 litre and labelled #14045.
The homogeneity of the subsamples #14045 was checked by the determination of Density in accordance with ASTM D4052 on 8 stratified randomly selected samples:

| | Density at 15ºC in kg/m$^3$ |
|---|---|
| sample 1 #14045-1 | 883.40 |
| sample 2 #14045-2 | 883.41 |
| sample 3 #14045-3 | 883.41 |
| sample 4 #14045-4 | 883.41 |
| sample 5 #14045-5 | 883.43 |
| sample 6 #14045-6 | 883.46 |
| sample 7 #14045-7 | 883.35 |
| sample 8 #14045-8 | 883.38 |

table 2: homogeneity test of subsamples #14045

| | Density at 15ºC in kg/m$^3$ |
|---|---|
| r (sample #14045) | 0.09 |
| reference test | ISO12185:96 |
| 0.3*$R_{(reference\ test)}$ | 0.15 |

table 3: repeatability of subsamples #14045

For Subsample #14046, 3.0 kg Biodiesel #13182 used in iis13G05M that was positive on Calcium & Magnesium, Phosphorus and Sodium was diluted with the Rapeseed methyl ester to 6.7 kg. The concentrations after dilution are approx: Calcium & Magnesium (10.6 mg/kg), Phosphorus (6.0 mg/kg) and Sodium (3.7 mg/kg). The material was subsequently divided over 66 glass bottles of 0.1L and labelled #14046. The homogeneity of the subsamples #14046 was checked by determination of Phosphorus on 8 stratified randomly selected samples:

| | Phosphorus in mg/kg |
|---|---|
| sample 1 #14046-1 | 6.6 |
| sample 2 #14046-2 | 6.6 |
| sample 3 #14046-3 | 6.6 |
| sample 4 #14046-4 | 6.7 |
| sample 5 #14046-5 | 6.5 |
| sample 6 #14046-6 | 6.7 |
| sample 7 #14046-7 | 6.6 |
| sample 8 #14046-8 | 6.6 |

table 4: homogeneity test of subsamples #14046

| | Phosphorus in mg/kg |
|---|---|
| r (sample #14046) | 0.2 |
| reference test | EN14106:03 |
| $0.3*R_{(reference\ test)}$ | 0.4 |

table 5: repeatability of subsamples #14046

For Total Contamination, out of the same batch of Biodiesel B100, another 50 amber glass bottles of 1 litre with inner and outer caps were filled.
Each sample bottle was spiked (approx 15 mg/kg) with a fresh prepared and well shaken particulate quartz material BCR-070 (∅ 1.2 – 20.0 µm) in oil suspension.
Therefore, an amount of the quartz suspension was weighed in the bottle. This bottle was filled up to 850 mL and subsequently labelled #14047.
After homogenization, a random sample was taken to check the Total Contamination.

For the “Cold Soak Test” determination 23 bottles of 1 litre with regular Biodiesel B100 were filled and labelled #14048. The homogeneity of the subsamples was checked by the determination of density in accordance with ISO12185.

The calculated repeatabilities for samples #14045 and #14046 were less than 0.3 times the corresponding reproducibilities of the respective reference method. Therefore, homogeneity of the subsamples was assumed.

Depending on the registration of the participant, one 1 litre bottle labelled #14045, and/or and/or one 0.1 litre bottle labelled #14046, and/or 1 litre bottle labelled #14047, and/or one 0.5 litre bottle labelled #14048, were dispatched to each of the participating laboratories on April 02, 2014.

### 2.5 STABILITY OF THE SAMPLES

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

## 2.6 ANALYSES

The tests methods to be used by the participating laboratories should be in accordance with the requirements of EN14214:12+A1:2014 and/or ASTM D6751:12, e.g.:

| Parameter | EN14214:12+A1:2014 | Parameter | ASTM D6751:12 |
|---|---|---|---|
| Acid Value | EN14104 | Acid Number | ASTM D664 |
| Carbon Residue on 10% dist.* | ISO10370* | | |
| CFPP | EN116 | | |
| | | Cloud Point | ASTM D2500 |
| Copper Strip Corrosion | ISO2160 | Copper Strip Corrosion | ASTM D130 |
| Density @ 15°C | ISO12185 | | |
| Flash Point (Recc) | ISO3679 | Flash Point | ASTM D93 |
| Flash Point (PMcc) | ISO2719 | | |
| Iodine Value | EN14111 | | |
| Kin. Visc. @ 40°C | ISO3104 | Kin. Visc. @ 40°C | ASTM D445 |
| Oxidation Stability | EN14112 | Oxidation Stability | EN15751 |
| Sulphated Ash | ISO3987 | Sulphated Ash | ASTM D874 |
| Sulphur | ISO20846 | Sulphur | ASTM D5453 |
| Water | ISO12937 | Water and Sediment | ASTM D2709 |
| Calcium + Magnesium | EN14538 | Calcium + Magnesium | EN14538 |
| Phosphorus | EN14107 | Phosphorus | ASTM D4951 |
| Polyunsaturated esters | EN15779 | | |
| Potassium + Sodium | EN14108/14109 | Potassium + Sodium | EN14538 |
| Methanol | EN14110 | Methanol | EN14110 |
| mono-, di-, tri-Glycerides | EN14105 | | |
| Free + Total Glycerol | EN14105 | Free + Total Glycerol | ASTM D6584 |
| Total ester content | EN14103 | | |
| Linolenic Acid | EN14103 | | |
| Total Contamination | EN12662 | | |

table 6: requirements and test methods acc. to specifications EN14214:12+A1:2014 and ASTM D6751:12
* = not applicable for B100 according to EN14214:12+A1:2014

## 3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to the laboratories that had not reported results at that moment. Shortly after the deadline, the available results were screened for suspect data. A 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 results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

### 3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, April 2014 version 3.3). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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 results should be used with due care.
In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and Rosner outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test (see appendix 3, no.15). Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05). Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

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

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

### 3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; Nos.13 and 14). Also a normal Gauss curve was projected over the Kernel Density Graph.

### 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 spread 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 in accordance with:

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

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

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

| | |
|---|---|
| \|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: 12 participants reported test results after the final reporting date and 5 participants did not report any test results at all.
For the Total Contamination PT: 11 participants reported the test results after the final reporting date and 3 participants did not report any test results at all.
For the Metals in Biodiesel PT: 7 participants reported the test results after the final reporting date and 6 participants did not report any test results at all.
For the Cold Soak Test PT: 3 participants reported the test results after the final reporting date and 5 participants did not report any test results at all.

Finally, 68 participants reported in total 1093 numerical results. Observed were 54 outlying results, which is 5.2%. 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 acc. to EN14214:12+A1:2014 and ASTM D6751:12 were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the reported data. The abbreviations, used in these tables, are listed in appendix 3.
In the iis PT reports, ASTM methods are referred to with a number (e.g. D2086) and an added designation for the year that the method was adopted or revised (e.g. D2086-08). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2086-08 (2013)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

The majority of the data sets proved to have a normal distribution. For some other tests the number of reported test results was too small to determine whether the data set was normally distributed. In these cases the results of the statistical evaluations should be used with care.

**For Biodiesel B100 sample #14045**

| | |
|---|---|
| Acid Value (EN) | 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 EN14104:03. |
| Acid Number (ASTM) | This determination was not problematic. One statistical outlier was The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D664:11a (method B). |
| Carbon Residue on 10% distillation residue | This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ISO10370:93. |
| Carbon Residue on 100% | The consensus value found was near or below the applicable lower limit of D4530:11 (<0.1%M/M). Therefore no significant conclusions were drawn. |
| CFPP | 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 EN116:97. |
| Cloud Point | This determination was problematic for a number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D2500:11. |
| Copper Corrosion D130 / ISO2160 | No problems have been observed. All participants agreed on a result of 1. |

| | |
|---|---|
| Density @15°C | This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is agreement with the requirements of ISO12185:96. |
| Flash Point conform EN spec. | This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ISO3679:04. |
| Flash Point PMcc ISO2719 / D93 | This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D93-C:13e. |
| Iodine Number | This determination was problematic for a number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers, is in agreement with the requirements of EN14111:03. |
| Kin.Visco. @ 40°C ISO3104 / D445 | The determination of this component may be problematic for a number of laboratories, depending on the test method used by the laboratory. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO3104:96. However, the calculated reproducibility is not in agreement with the requirements of ASTM D445:12. |
| Oxidation Stability | 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 EN14112:03. |
| Sulphur ISO20846 | 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 ISO20846:11. |
| Sulphur ASTM D5453 | 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 D5453:12. |
| 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. |

| | |
|---|---|
| Water | This determination was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12937:00. Remarkably, eight laboratories made probably a unit error in the reported test result. |
| Methanol | 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 full agreement with the requirements of EN14110:03 which are the same for procedure A (automatic headspace system) and B (manual procedure).<br>When the EN14110-A and EN14110-B methods were evaluated separately, the calculated reproducibilities are respectively not and in full agreement with the requirements of EN14110:03. |
| mono-Glycerides | This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN14105:11 and also in good agreement with the requirements of ASTM D6584:13. |
| di-Glycerides | This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of EN14105:11 and also in good agreement with ASTM D6584:13. |
| tri-Glycerides | This determination was problematic for number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN14105:11 and also in good agreement with ASTM D6584:13. |
| Free Glycerol | This determination was not problematic. Two statistical outliers were observed and one result was excluded. However, the calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of EN14105:11. |
| Total Glycerol | This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN14105:11. |
| Total Ester content | This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN14103:11. |

Five laboratories reported to have used the 2003 version and one laboratory the 2009 version of EN14103 instead of the 2011 version. The 2011 version of EN14103 is mentioned in EN14214:2012+A1:2014 as the test method to be used.

Linolenic Acid Methyl Ester: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of EN14103:11.
Three laboratories reported to have used the 2003 version and one laboratory the 2009 version of EN14103 instead of the 2011 version. The 2011 version of EN14103 is mentioned in EN14214:2012+A1:2014 as the test method to be used.

Polyunsaturated: All reported results, were near or below the lower application limit of EN15779:09 (0.3 – 3.0 %M/M). Therefore no significant conclusions were drawn.

**For Biodiesel B100 sample #14046**

Calcium and Magnesium: This determination was not problematic. Two statistical outliers and one false negative test result were observed. However, the calculated reproducibility, after rejection the suspect data is in agreement with the requirements of EN141538:06.
The samples were spiked with Calcium and Magnesium. The average recovery of 103% Calcium and Magnesium (theoretical increment of 10.6 mg/kg) can be considered as good.

Phosphorus: This determination was very problematic. One false negative test result but no statistical outliers were observed. The calculated reproducibility, after rejection of the suspect test results, is not at all in agreement with the requirements of EN14107:03.
The samples were spiked with Phosphorus. The average recovery of 112% Phosphorus (theoretical increment of 6.0 mg/kg) can be considered as satisfactory.

Potassium: All reported results except one were near or below the lower application limit of EN14109:03 (0.5 mg/kg). Therefore no significant conclusions were drawn.

Sodium: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN14108:03.
The samples were spiked with Sodium. The average recovery of 109% Sodium (theoretical increment of 3.7 mg/kg) can be considered as satisfactory.

**For Biodiesel B100 sample #14047**

Total Contamination: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of EN12662:14.
When the EN12662:14 data were evaluated separately, the calculated reproducibility is in full agreement with the requirements of EN12662:14
The samples were spiked with particulate quartz material BCR. Therefore the minimum contamination concentration to be found was known (added amount = 15.31 mg/kg). The laboratories should be able to find at least 6.80 mg/kg [15.31 mg/kg$_{(added\ amount)}$ – 8.51 mg/kg$_{(R\ EN12662:14)}$]. No laboratories reported lower amounts than 6.8 mg/kg.

**For Biodiesel B100 sample #14048**

Cold Soak test: Two test results were excluded because these laboratories probably reported a test result for filter blocking tendency instead of a cold soak test result in seconds. The other five reported test results vary over a large range: 63 – 720 s. Therefore no significant conclusions were drawn. Important aspects such as conditioning of test samples that might have been cooled to temperatures below 20 ºC (68 ºF) (§ 8.4 - 8.6), the choice and the preparation of the glass and following the correct filtration procedure (§11) are important aspects to attain comparable results.

Filter Blocking Tendency: The five reported test results vary from 3.73 – 10.05. Therefore no significant conclusions were drawn.

### 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 Value (EN14104) | mg KOH/g | 42 | 0.158 | 0.048 | 0.060 |
| Acid Number (D664-B) | mg KOH/g | 28 | 0.147 | 0.061 | 0.082 |
| Carbon Residue on 10% dist. | %M/M | 14 | 0.17 | 0.14 | 0.07 |
| Carbon Residue on 100% FAME | %M/M | 7 | 0.02 | 0.08 | (0.14) |
| Cold Filter Plugging Point | °C | 53 | -14.5 | 2.3 | 3.3 |
| Cloud Point | °C | 53 | - 4.3 | 2.1 | 3.0 |
| Copper Strip Corrosion | | 52 | 1 | n.a. | n.a. |
| Density @ 15°C | kg/m$^3$ | 63 | 883.3 | 0.3 | 0.5 |
| Flash Point EN spec. | °C | 26 | 174.9 | 15.2 | 15.0 |
| Flash Point (PMcc) ISO/ASTM | °C | 36 | 167.1 | 15.3 | 14.7 |
| Iodine Value | g $I_2$/100g | 41 | 113.1 | 4.5 | 5.0 |
| Kin. Viscosity @ 40°C | mm$^2$/s | 52 | 4.552 | 0.040 | 0.045 |

| | | | | | |
|---|---|---|---|---|---|
| Oxidation Stability EN14112 | hours | 51 | 8.38 | 1.41 | 2.41 |
| Sulphur (ISO20846) | mg/kg | 32 | 2.04 | 1.34 | 1.35 |
| Sulphur (D5453) | mg/kg | 21 | 2.06 | 0.74 | 1.00 |
| Sulphated Ash | %M/M | 14 | 0.0011 | 0.0023 | (0.0006) |
| Water | %M/M | 60 | 0.033 | 0.007 | 0.013 |
| Methanol | %M/M | 34 | 0.021 | 0.007 | 0.008 |
| mono-Glycerides | %M/M | 35 | 0.59 | 0.12 | 0.18 |
| di-Glycerides | %M/M | 35 | 0.13 | 0.05 | 0.05 |
| tri-Glycerides | %M/M | 33 | 0.10 | 0.06 | 0.08 |
| Free Glycerol | %M/M | 23 | 0.0026 | 0.0060 | 0.0066 |
| Total Glycerol | %M/M | 36 | 0.181 | 0.034 | 0.046 |
| Total Ester Content | %M/M | 45 | 98.51 | 2.69 | 4.16 |
| Linolenic Acid Methyl Ester | %M/M | 43 | 9.71 | 0.68 | 0.66 |
| Polyunsat. Methyl esters | %M/M | 16 | 0.22 | 0.43 | (0.27) |

table 7: comparison of the observed and target reproducibilities of Biodiesel B100 sample #14045

| Parameter | unit | n | average | R (Calc.) | R (lit) |
|---|---|---|---|---|---|
| Calcium & Magnesium | mg/kg | 26 | 10.9 | 3.1 | 2.8 |
| Phosphorus | mg/kg | 26 | 6.7 | 3.1 | 1.3 |
| Potassium | mg/kg | 10 | 0.2 | 0.5 | (1.0) |
| Sodium | mg/kg | 28 | 4.1 | 2.9 | 2.4 |

table 8: comparison of the observed and target reproducibilities of Biodiesel B100 sample #14046

| Parameter | unit | n | average | R (Calc.) | R (lit) |
|---|---|---|---|---|---|
| Total Contamination | mg/kg | 39 | 27.1 | 17.4 | 8.5 |

table 9: comparison of the observed and target reproducibilities of Biodiesel B100 sample #14047

| Parameter | unit | n | average | R (Calc.) | R (lit) |
|---|---|---|---|---|---|
| Cold Soak Filter Test | s | 4 | 95.0 | 63.0 | (10.9) |
| Filter Blocking Tendency | | 5 | 6.70 | 6.50 | (2.78) |

table 10: comparison of the observed and target reproducibilities of Biodiesel B100 sample #14048
* Values between brackets were below the application range of the respective reference test method, therefore results should be used with care.

Without further statistical calculations it can be concluded that for many tests there is not 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 APRIL 2014 WITH PREVIOUS PTS

| | April 2014 | October 2013 | April 2013 | October 2012 |
|---|---|---|---|---|
| Type of FAME | Rapeseed | Fat of Offal | Rapeseed | Used cooking oil |
| Number of reporting labs | 68 | 58 | 75 | 46 |
| Number of results reported | 1093 | 768 | 1010 | 728 |
| Number of statistical outliers | 54 | 44 | 67 | 40 |
| Percentage statistical outliers | 5.2% | 5.7% | 6.6% | 5.5% |

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 standards. The conclusions are given the following table:

| Determination | April 2014 | October 2013 | April 2013 | October 2012 |
|---|---|---|---|---|
| Acid Value (EN14104) | + | -- | + | +/- |
| Acid Number (D664-B) | + | -- | + | + |
| Carbon Residue on 10% dist. | (--) | (--) | -- | -- |
| Carbon Residue on 100% FAME | (++) | n.e. | ++ | n.e. |
| Cold Filter Plugging Point | + | -- | + | - |
| Cloud Point | + | + | + | + |
| Density @15°C | + | ++ | ++ | ++ |
| Flash Point EN spec. | +/- | ++ | - | ++ |
| Flash Point PMcc ISO/ASTM | +/- | +/- | - | +/- |
| Iodine Value | + | -- | +/- | - |
| Kin. Viscosity @ 40°C | +/- | -- | - | - |
| Oxidation Stability | + | -- | + | ++ |
| Sulphated Ash | (--) | (-) | (--) | (--) |
| Sulphur (EN spec.) | +/- | -- | - | - |
| Sulphur (D5453) | + | + | + | - |
| Water | + | + | ++ | + |
| Methanol | +/- | n.e. | -- | -- |
| mono-Glycerides | + | (-) | + | - |
| di-Glycerides | +/- | (-) | + | +/- |
| tri-Glycerides | + | (+) | + | -- |
| Free Glycerol | +/- | + | + | ++ |
| Total Glycerol | + | -- | +/- | +/- |
| Total Ester content | + | -- | ++ | - |
| Linolenic Acid Methyl Ester | +/- | -- | - | ++ |
| Polyunsat. Methyl esters | (-) | -- | (--) | - |
| Total Contamination | -- | -- | -- | (--) |
| Calcium and Magnesium | - | (--) | (--) | (++) |
| Phosphorus | -- | -- | -- | (--) |
| Potassium | (++) | (++) | +/- | (++) |
| Sodium | - | - | +/- | + |
| Cold Soak Filter Test | (--) | n.e. | (--) | n.e. |
| Filter Blocking Tendency | (--) | n.e. | (--) | n.e. |

table 12 : 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:

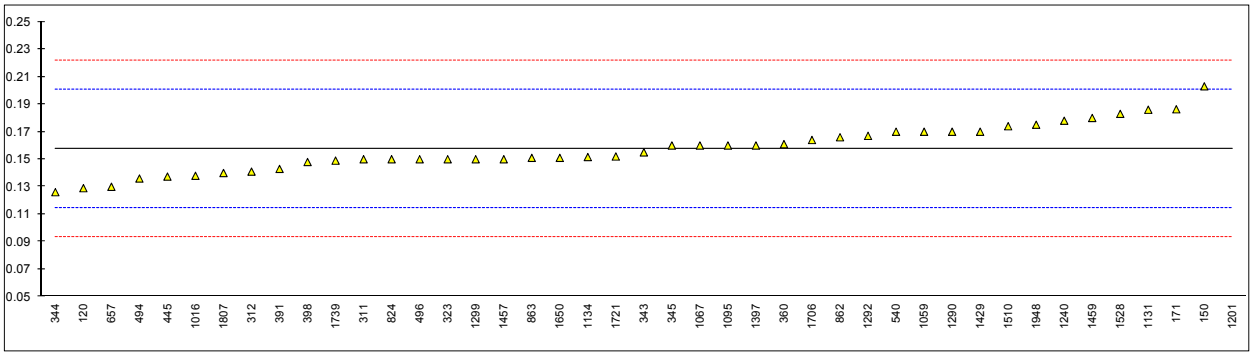
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- - : group performed worse than the standard
- -- : group performed much worse than the standard
- n.e.: not evaluated

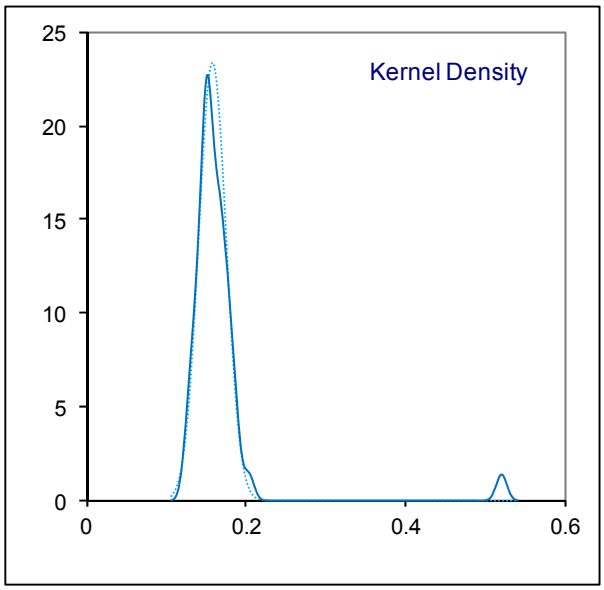
## APPENDIX 1

Determination of Acid Value conform EN spec. on sample #14045; results in mg KOH/g

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | EN14104 | 0.129 | | -1.34 | |
| 150 | EN14104 | 0.203 | | 2.12 | |
| 171 | EN14104 | 0.1863 | | 1.34 | |
| 311 | EN14104 | 0.15 | | -0.36 | |
| 312 | EN14104 | 0.141 | | -0.78 | |
| 323 | EN14104 | 0.15 | | -0.36 | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | EN14104 | 0.155 | | -0.12 | |
| 344 | EN14104 | 0.1261 | | -1.47 | |
| 345 | EN14104 | 0.16 | | 0.11 | |
| 360 | EN14104 | 0.161 | | 0.16 | |
| 391 | EN14104 | 0.143 | | -0.68 | |
| 398 | EN14104 | 0.148 | | -0.45 | |
| 445 | EN14104 | 0.1374 | | -0.95 | |
| 447 | | ----- | | ----- | |
| 494 | EN14104 | 0.136 | | -1.01 | |
| 496 | EN14104 | 0.150 | | -0.36 | |
| 529 | | ----- | | ----- | |
| 540 | EN14104 | 0.17 | | 0.58 | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN14104 | 0.13 | | -1.29 | |
| 824 | EN14104 | 0.15 | | -0.36 | |
| 862 | EN14104 | 0.166 | | 0.39 | |
| 863 | EN14104 | 0.151 | | -0.31 | |
| 1016 | EN14104 | 0.138 | | -0.92 | |
| 1033 | | ----- | | ----- | |
| 1059 | EN14104 | 0.17 | | 0.58 | |
| 1067 | EN14104 | 0.16 | | 0.11 | |
| 1095 | EN14104 | 0.16 | | 0.11 | |
| 1131 | EN14104 | 0.186 | | 1.32 | |
| 1134 | EN14104 | 0.1516 | | -0.28 | |
| 1161 | | ----- | | ----- | |
| 1199 | | ----- | | ----- | |
| 1201 | EN14104 | 0.52 | R(0.01) | 16.91 | |
| 1213 | | ----- | | ----- | |
| 1227 | | ----- | | ----- | |
| 1231 | | ----- | | ----- | |
| 1240 | EN14104 | 0.178 | | 0.95 | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | EN14104 | 0.17 | | 0.58 | |
| 1292 | EN14104 | 0.167 | | 0.44 | |
| 1299 | EN14104 | 0.15 | | -0.36 | |
| 1316 | | ----- | | ----- | |
| 1397 | EN14104 | 0.16 | | 0.11 | |
| 1402 | | ----- | | ----- | |
| 1429 | EN14104 | 0.17 | | 0.58 | |
| 1443 | | ----- | | ----- | |
| 1457 | EN14104 | 0.150 | | -0.36 | |
| 1459 | EN14104 | 0.18 | | 1.04 | |
| 1494 | | ----- | | ----- | |
| 1510 | EN14104 | 0.174 | | 0.76 | |
| 1528 | EN14104 | 0.183 | | 1.18 | |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | EN14104 | 0.151 | | -0.31 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | EN14104 | 0.164 | | 0.30 | |
| 1721 | EN14104 | 0.152 | | -0.26 | |
| 1739 | EN14104 | 0.149 | | -0.40 | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | EN14104 | 0.14 | | -0.82 | |
| 1948 | EN14104 | 0.1751 | | 0.81 | |

| | |
|---|---|
| normality | OK |
| n | 42 |
| outliers | 1 |
| mean (n) | 0.1577 |
| st.dev. (n) | 0.01710 |
| R(calc.) | 0.0479 |
| R(EN14104:03) | 0.0600 |

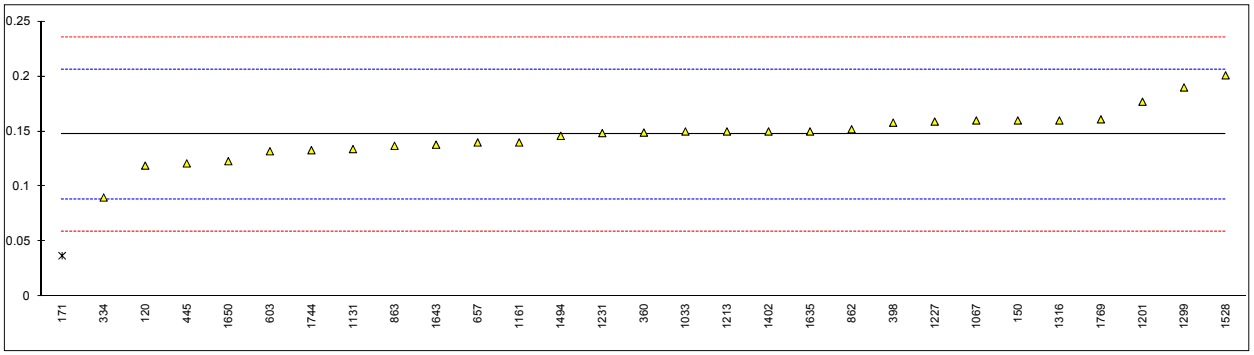


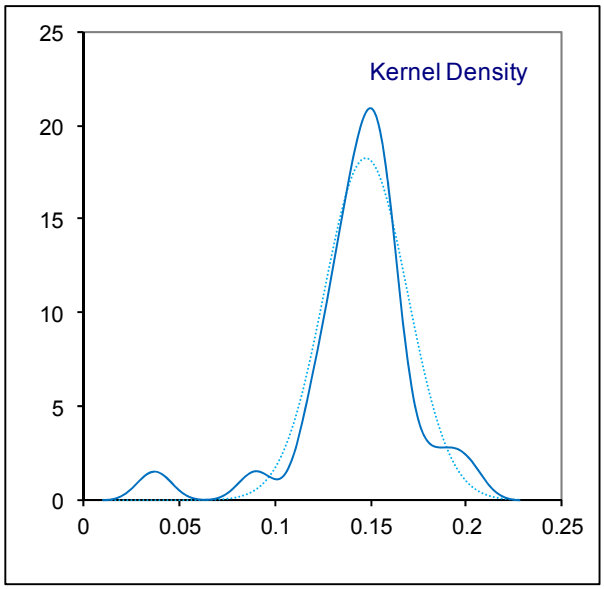



Determination of Acid Number conform ASTM spec. on sample #14045; results in mg KOH/g

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | D664-B | 0.119 | | -0.97 | |
| 150 | D664-B | 0.16 | | 0.43 | |
| 171 | D664-B | 0.0371 | R(0.01) | -3.75 | |
| 311 | | ----- | | ----- | |
| 312 | | ----- | | ----- | |
| 323 | | ----- | | ----- | |
| 334 | D664-B | 0.09 | | -1.95 | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | | ----- | | ----- | |
| 344 | | ----- | | ----- | |
| 345 | | ----- | | ----- | |
| 360 | D664-B | 0.149 | | 0.05 | |
| 391 | | ----- | | ----- | |
| 398 | D664-B | 0.158 | | 0.36 | |
| 445 | D664 | 0.121 | | -0.90 | |
| 447 | | ----- | | ----- | |
| 494 | | ----- | | ----- | |
| 496 | | ----- | | ----- | |
| 529 | | ----- | | ----- | |
| 540 | | ----- | | ----- | |
| 554 | | ----- | | ----- | |
| 603 | D664-B | 0.132 | | -0.53 | |
| 657 | D664-B | 0.14 | | -0.25 | |
| 824 | | ----- | | ----- | |
| 862 | D664-B | 0.152 | | 0.15 | |
| 863 | D664-B | 0.137 | | -0.36 | |
| 1016 | | ----- | | ----- | |
| 1033 | D974 | 0.15 | | 0.09 | |
| 1059 | | ----- | | ----- | |
| 1067 | D664-B | 0.16 | | 0.43 | |
| 1095 | | ----- | | ----- | |
| 1131 | D664-A | 0.134 | | -0.46 | |
| 1134 | | ----- | | ----- | |
| 1161 | D664 | 0.140 | | -0.25 | |
| 1199 | | ----- | | ----- | |
| 1201 | D664-B | 0.177 | | 1.01 | |
| 1213 | D664 | 0.15 | | 0.09 | |
| 1227 | D974 | 0.159 | | 0.39 | |
| 1231 | D664 | 0.1485 | | 0.04 | |
| 1240 | | ----- | | ----- | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | | ----- | | ----- | |
| 1299 | D664-B | 0.190 | | 1.45 | |
| 1316 | D664-B | 0.16 | | 0.43 | |
| 1397 | | ----- | | ----- | |
| 1402 | D664-A | 0.15 | | 0.09 | |
| 1429 | | ----- | | ----- | |
| 1443 | | ----- | | ----- | |
| 1457 | | ----- | | ----- | |
| 1459 | | ----- | | ----- | |
| 1494 | D664-B | 0.14600 | | -0.05 | |
| 1510 | | ----- | | ----- | |
| 1528 | D664-B | 0.201 | | 1.82 | |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | D664-B | 0.15 | | 0.09 | |
| 1643 | D664 | 0.138 | | -0.32 | |
| 1650 | D664-B | 0.123 | | -0.83 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | | ----- | | ----- | |
| 1739 | | ----- | | ----- | |
| 1744 | D664-B | 0.133 | | -0.49 | |
| 1769 | D664-B | 0.161 | | 0.46 | |
| 1807 | | ----- | | ----- | |
| 1948 | | ----- | | ----- | |

| | |
|---|---|
| normality | suspect |
| n | 28 |
| outliers | 1 |
| mean (n) | 0.1474 |
| st.dev. (n) | 0.02182 |
| R(calc.) | 0.0611 |
| R(D664B:11a) | 0.0823 |



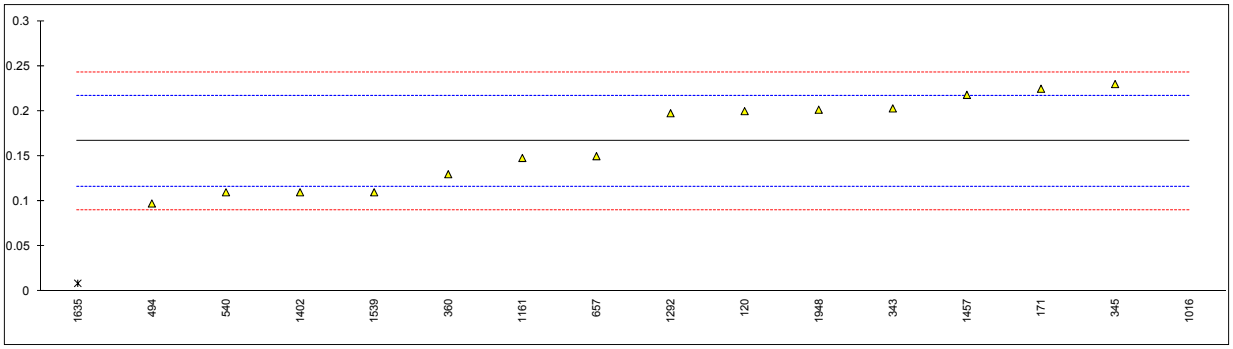



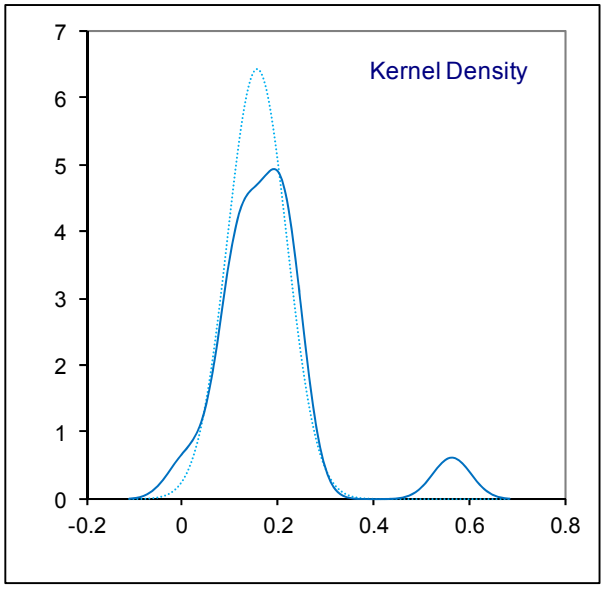
Determination of Carbon Residue on 10% distillation residue on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | ISO10370 | 0.200 | | 1.27 | |
| 150 | | ----- | | ----- | |
| 171 | ISO10370 | 0.225 | | 2.20 | |
| 311 | | ----- | | ----- | |
| 312 | | ----- | | ----- | |
| 323 | ISO10370 | <0.10 | | ----- | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | ISO10370 | 0.203 | | 1.38 | |
| 344 | | ----- | | ----- | |
| 345 | ISO10370 | 0.230 | | 2.40 | |
| 360 | ISO10370 | 0.130 | | -1.38 | |
| 391 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 445 | | ----- | | ----- | |
| 447 | | ----- | | ----- | |
| 494 | ISO10370 | 0.098 | | -2.60 | |
| 496 | | ----- | | ----- | |
| 529 | | ----- | | ----- | |
| 540 | ISO10370 | 0.110 | | -2.13 | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | ISO10370 | 0.150 | | -0.62 | |
| 824 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 863 | | ----- | | ----- | |
| 1016 | ISO10370 | 0.564 | G(0.01) | 15.01 | false positive result? |
| 1033 | | ----- | | ----- | |
| 1059 | | ----- | | ----- | |
| 1067 | | ----- | | ----- | |
| 1095 | | ----- | W | ----- | result withdrawn, first reported: 0.67 |
| 1131 | | ----- | | ----- | |
| 1134 | | ----- | | ----- | |
| 1161 | ISO10370 | 0.148 | | -0.70 | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | | ----- | | ----- | |
| 1227 | | ----- | | ----- | |
| 1231 | | ----- | | ----- | |
| 1240 | | ----- | | ----- | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | ISO10370 | 0.198 | | 1.18 | |
| 1299 | | ----- | | ----- | |
| 1316 | | ----- | | ----- | |
| 1397 | | ----- | | ----- | |
| 1402 | ISO10370 | 0.110 | | -2.13 | |
| 1429 | | ----- | | ----- | |
| 1443 | | ----- | | ----- | |
| 1457 | ISO10370 | 0.218 | | 1.95 | |
| 1459 | | ----- | | ----- | |
| 1494 | | ----- | | ----- | |
| 1510 | | ----- | | ----- | |
| 1528 | | ----- | | ----- | |
| 1539 | ISO10370 | 0.110 | | -2.13 | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | ISO10370 | 0.009 | G(0.05) | -5.94 | |
| 1643 | | ----- | | ----- | |
| 1650 | | ----- | | ----- | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | | ----- | | ----- | |
| 1739 | | ----- | | ----- | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | | ----- | | ----- | |
| 1948 | ISO10370 | 0.202 | | 1.32 | |

| | |
|---|---|
| normality | OK |
| n | 14 |
| outliers | 2 |
| mean (n) | 0.1665 |
| st.dev. (n) | 0.04884 |
| R(calc.) | 0.1368 |
| R(ISO10370:93) | 0.0742 |

application range: 0.10 – 30.0 %M/M



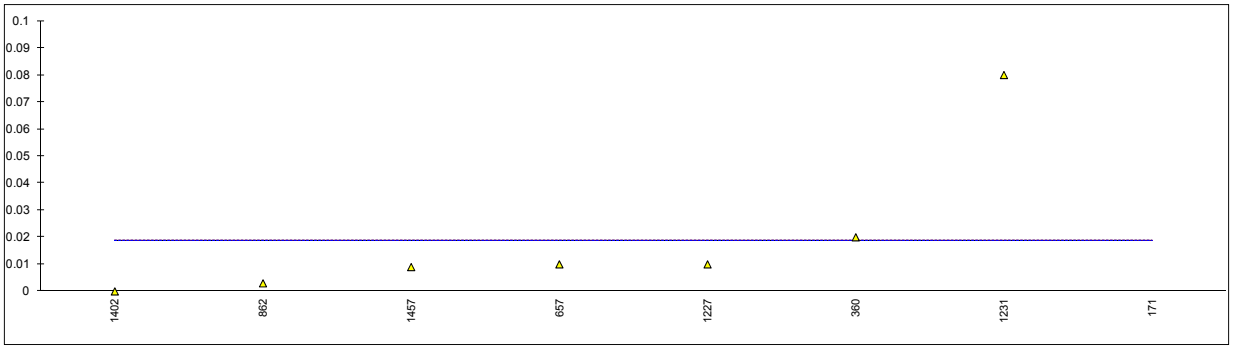



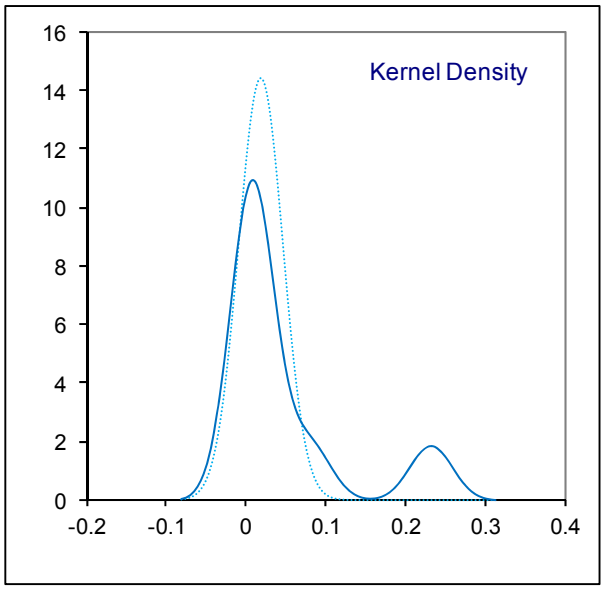
Determination of Carbon Residue on 100% FAME on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | D4530 | 0.2319 | G(0.01) | ----- | |
| 311 | | ----- | | ----- | |
| 312 | | ----- | | ----- | |
| 323 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | | ----- | | ----- | |
| 344 | | ----- | | ----- | |
| 345 | | ----- | | ----- | |
| 360 | D4530 | 0.02 | | ----- | |
| 391 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 445 | | ----- | | ----- | |
| 447 | | ----- | | ----- | |
| 494 | | ----- | | ----- | |
| 496 | | ----- | | ----- | |
| 529 | | ----- | | ----- | |
| 540 | | ----- | | ----- | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | D4530 | 0.01 | | ----- | |
| 824 | | ----- | | ----- | |
| 862 | D4530 | 0.003 | | ----- | |
| 863 | | ----- | | ----- | |
| 1016 | | ----- | | ----- | |
| 1033 | | ----- | | ----- | |
| 1059 | | ----- | | ----- | |
| 1067 | | ----- | | ----- | |
| 1095 | | ----- | | ----- | |
| 1131 | | ----- | | ----- | |
| 1134 | | ----- | | ----- | |
| 1161 | | ----- | | ----- | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | | ----- | | ----- | |
| 1227 | D4530 | 0.01 | | ----- | |
| 1231 | D4530 | 0.08 | | ----- | |
| 1240 | | ----- | | ----- | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | | ----- | | ----- | |
| 1299 | | ----- | | ----- | |
| 1316 | | ----- | | ----- | |
| 1397 | D4530 | <0.01 | | ----- | |
| 1402 | D4530 | 0 | | ----- | |
| 1429 | | ----- | | ----- | |
| 1443 | | ----- | | ----- | |
| 1457 | D4530 | 0.009 | | ----- | |
| 1459 | | ----- | | ----- | |
| 1494 | | ----- | | ----- | |
| 1510 | | ----- | | ----- | |
| 1528 | | ----- | | ----- | |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | | ----- | | ----- | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | | ----- | | ----- | |
| 1739 | | ----- | | ----- | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | | ----- | | ----- | |
| 1948 | | ----- | | ----- | |

| | |
|---|---|
| normality | unknown |
| n | 7 |
| outliers | 1 |
| mean (n) | 0.019 |
| st.dev. (n) | 0.0277 |
| R(calc.) | 0.078 |
| R(D4530:11) | (0.141) |

application range: 0.10 – 30.0 %M/M

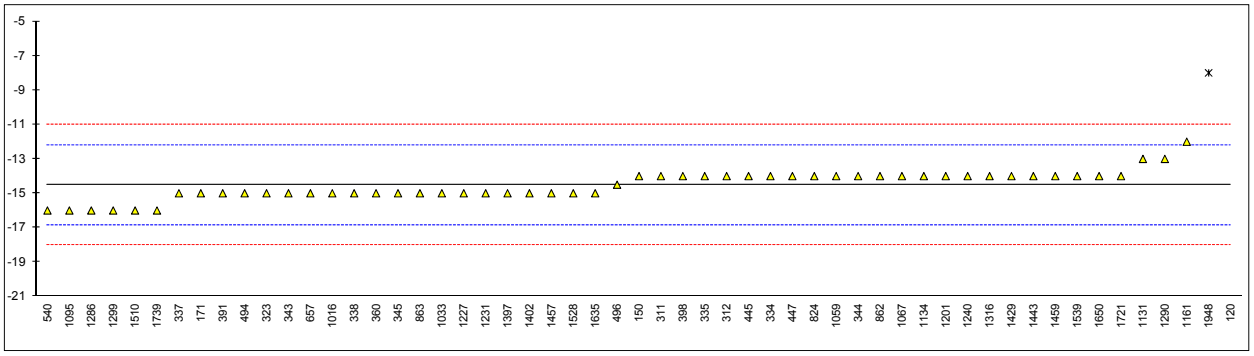


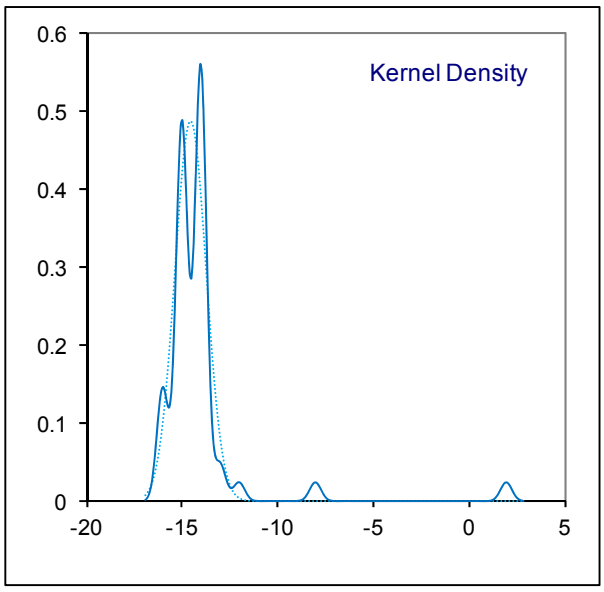



## Determination of Cold Filter Plugging Point on sample #14045; results in °C

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | EN116 | 2 | C,R(0.01) | 14.17 | first reported: -4 |
| 150 | EN116 | -14.0 | | 0.46 | |
| 171 | EN116 | -15 | | -0.40 | |
| 311 | EN116 | -14 | | 0.46 | |
| 312 | EN116 | -14 | | 0.46 | |
| 323 | EN116 | -15 | | -0.40 | |
| 334 | EN116 | -14 | | 0.46 | |
| 335 | EN116 | -14 | | 0.46 | |
| 337 | EN116 | -15 | | -0.40 | |
| 338 | EN116 | -15 | | -0.40 | |
| 343 | EN116 | -15 | | -0.40 | |
| 344 | EN116 | -14 | | 0.46 | |
| 345 | EN116 | -15 | | -0.40 | |
| 360 | EN116 | -15 | | -0.40 | |
| 391 | EN116 | -15 | | -0.40 | |
| 398 | EN116 | -14 | | 0.46 | |
| 445 | IP309 | -14 | | 0.46 | |
| 447 | IP309 | -14 | | 0.46 | |
| 494 | EN116 | -15 | | -0.40 | |
| 496 | EN116 | -14.5 | | 0.03 | |
| 529 | | ----- | | ----- | |
| 540 | EN116 | -16 | | -1.25 | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN116 | -15 | | -0.40 | |
| 824 | EN116 | -14 | | 0.46 | |
| 862 | EN116 | -14 | | 0.46 | |
| 863 | IP309 | -15 | | -0.40 | |
| 1016 | EN116 | -15 | | -0.40 | |
| 1033 | IP309 | -15 | | -0.40 | |
| 1059 | EN116 | -14 | | 0.46 | |
| 1067 | EN116 | -14 | | 0.46 | |
| 1095 | EN116 | -16 | | -1.25 | |
| 1131 | EN116 | -13 | | 1.32 | |
| 1134 | IP309 | -14 | | 0.46 | |
| 1161 | EN116 | -12 | | 2.17 | |
| 1199 | | ----- | | ----- | |
| 1201 | EN116 | -14 | | 0.46 | |
| 1213 | | ----- | | ----- | |
| 1227 | EN116 | -15 | | -0.40 | |
| 1231 | D6371 | -15 | | -0.40 | |
| 1240 | EN116 | -14.0 | | 0.46 | |
| 1268 | | ----- | | ----- | |
| 1286 | EN116 | -16.0 | | -1.25 | |
| 1290 | EN116 | -13 | | 1.32 | |
| 1292 | | ----- | | ----- | |
| 1299 | EN116 | -16 | | -1.25 | |
| 1316 | EN116 | -14.0 | | 0.46 | |
| 1397 | EN116 | -15 | | -0.40 | |
| 1402 | EN116 | -15 | | -0.40 | |
| 1429 | EN116 | -14 | | 0.46 | |
| 1443 | EN116 | -14 | | 0.46 | |
| 1457 | EN116 | -15 | | -0.40 | |
| 1459 | EN116 | -14 | | 0.46 | |
| 1494 | | ----- | | ----- | |
| 1510 | IP309 | -16 | | -1.25 | |
| 1528 | EN116 | -15 | | -0.40 | |
| 1539 | EN116 | -14 | | 0.46 | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | EN116 | -15 | | -0.40 | |
| 1643 | | ----- | | ----- | |
| 1650 | EN116 | -14 | | 0.46 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | EN116 | -14 | | 0.46 | |
| 1739 | EN116 | -16 | | -1.25 | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | | ----- | | ----- | |
| 1948 | EN116 | -8 | C,R(0.01) | 5.60 | first reported: -5 |

| | |
|---|---|
| normality | OK |
| n | 53 |
| outliers | 2 |
| mean (n) | -14.54 |
| st.dev. (n) | 0.820 |
| R(calc.) | 2.29 |
| R(EN116:97) | 3.27 |

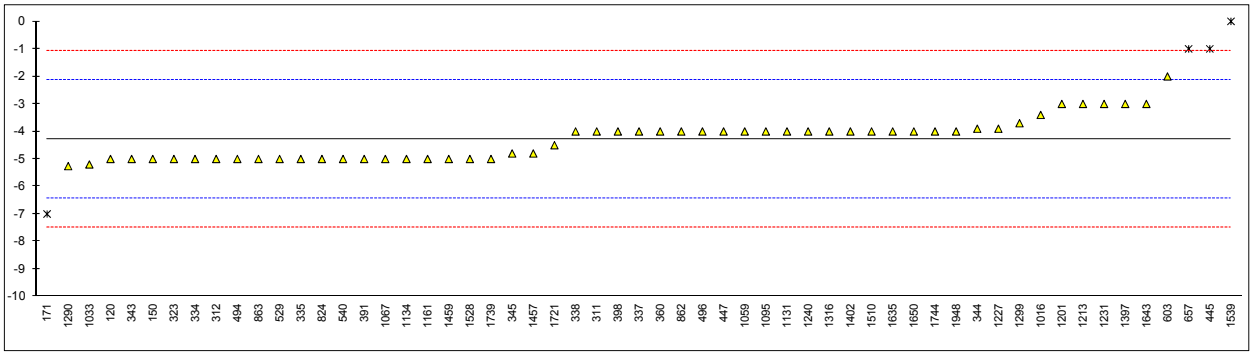


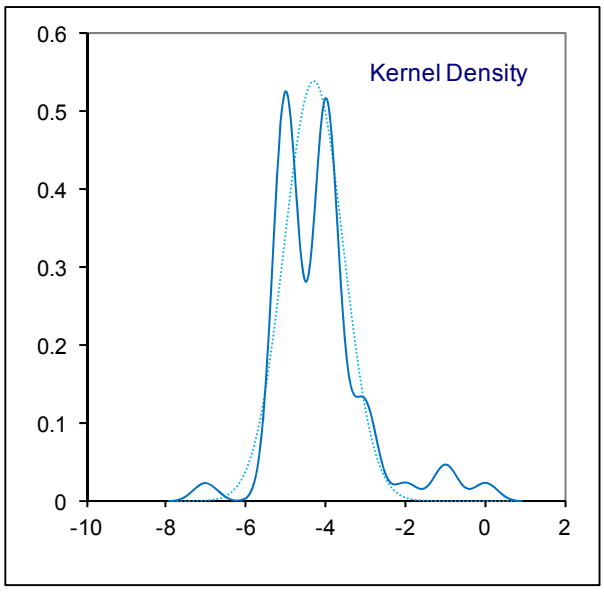



## Determination of Cloud Point on sample #14045; results in °C

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | D2500 | -5 | | -0.66 | |
| 150 | D2500/EN23015 | -5 | | -0.66 | |
| 171 | D2500/EN23015 | -7 | R(0.05) | -2.53 | |
| 311 | D2500/EN23015 | -4 | | 0.27 | |
| 312 | EN23015 | -5 | | -0.66 | |
| 323 | EN23015 | -5 | | -0.66 | |
| 334 | EN23015 | -5 | | -0.66 | |
| 335 | D2500/EN23015 | -5 | | -0.66 | |
| 337 | D2500/EN23015 | -4 | | 0.27 | |
| 338 | EN23015 | -4.0 | | 0.27 | |
| 343 | D2500/EN23015 | -5 | | -0.66 | |
| 344 | D2500 | -3.9 | | 0.37 | |
| 345 | D5771 | -4.8 | | -0.47 | |
| 360 | EN23015 | -4 | | 0.27 | |
| 391 | D2500/EN23015 | -5 | | -0.66 | |
| 398 | EN23015 | -4 | | 0.27 | |
| 445 | EN23015 | -1 | R(0.01) | 3.07 | |
| 447 | D2500 | -4 | | 0.27 | |
| 494 | EN23015 | -5 | | -0.66 | |
| 496 | EN23015 | -4.0 | | 0.27 | |
| 529 | D2500 | -5 | | -0.66 | |
| 540 | D2500 | -5 | | -0.66 | |
| 554 | | ----- | | ----- | |
| 603 | D2500 | -2 | | 2.14 | |
| 657 | D2500 | -1 | R(0.01) | 3.07 | |
| 824 | D2500 | -5 | | -0.66 | |
| 862 | D2500 | -4 | | 0.27 | |
| 863 | D2500 | -5 | | -0.66 | |
| 1016 | D2500/EN23015 | -3.4 | | 0.83 | |
| 1033 | IP219 | -5.2 | | -0.85 | |
| 1059 | EN23015 | -4 | | 0.27 | |
| 1067 | D2500/EN23015 | -5 | | -0.66 | |
| 1095 | D2500 | -4 | | 0.27 | |
| 1131 | EN23015 | -4 | | 0.27 | |
| 1134 | IP219 | -5 | | -0.66 | |
| 1161 | EN23015 | -5 | | -0.66 | |
| 1199 | | ----- | | ----- | |
| 1201 | D2500/EN23015 | -3 | | 1.21 | |
| 1213 | D2500 | -3 | C | 1.21 | first reported: 1 |
| 1227 | D2500 | -3.9 | | 0.37 | |
| 1231 | D2500 | -3 | | 1.21 | |
| 1240 | EN23015 | -4.0 | | 0.27 | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | D2500 | -5.26 | | -0.90 | |
| 1292 | | ----- | | ----- | |
| 1299 | D2500/EN23015 | -3.7 | | 0.55 | |
| 1316 | EN23015 | -4.0 | | 0.27 | |
| 1397 | D2500/EN23015 | -3 | | 1.21 | |
| 1402 | D2500/EN23015 | -4 | | 0.27 | |
| 1429 | | ----- | | ----- | |
| 1443 | | ----- | | ----- | |
| 1457 | D2500/EN23015 | -4.8 | | -0.47 | |
| 1459 | EN23015 | -5 | | -0.66 | |
| 1494 | | ----- | | ----- | |
| 1510 | D2500 | -4 | | 0.27 | |
| 1528 | D2500/EN23015 | -5 | | -0.66 | |
| 1539 | ISO3015 | 0 | R(0.01) | 4.01 | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | EN23015 | -4 | | 0.27 | |
| 1643 | D2500 | -3 | | 1.21 | |
| 1650 | D5771 | -4.0 | | 0.27 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | D2500/EN23015 | -4.5 | | -0.19 | |
| 1739 | EN23015 | -5 | | -0.66 | |
| 1744 | D2500 | -4 | | 0.27 | |
| 1769 | | ----- | | ----- | |
| 1807 | | ----- | | ----- | |
| 1948 | D2500/EN23015 | -4 | | 0.27 | |

| | |
|---|---|
| normality | OK |
| n | 53 |
| outliers | 4 |
| mean (n) | -4.29 |
| st.dev. (n) | 0.741 |
| R(calc.) | 2.07 |
| R(D2500:11) | 3.00 |



# Determination of Copper Strip Corrosion 3 hrs/50°C on sample #14045

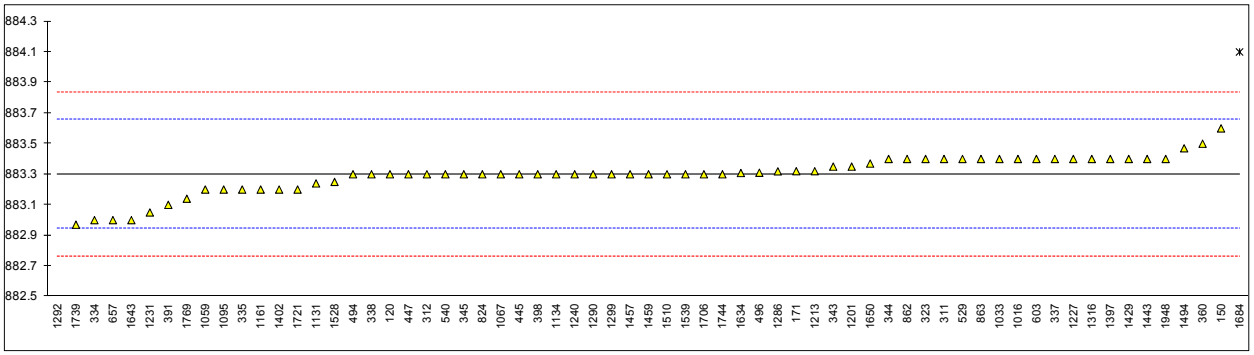
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | D130 | 1A | | ----- | |
| 150 | D130/ISO2160 | 1A | | ----- | |
| 171 | D130/ISO2160 | 1A | | ----- | |
| 311 | D130/ISO2160 | 1A | | ----- | |
| 312 | D130 | 1A | | ----- | |
| 323 | ISO2160 | 1A | | ----- | |
| 334 | | ----- | | ----- | |
| 335 | D130/ISO2160 | 1 | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | D130/ISO2160 | 1A | | ----- | |
| 344 | D130 | 1A | | ----- | |
| 345 | ISO2160 | 1A | | ----- | |
| 360 | D130 | 1A | | ----- | |
| 391 | D130/ISO2160 | 1A | | ----- | |
| 398 | ISO2160 | 1A | | ----- | |
| 445 | ISO2160 | 1A | | ----- | |
| 447 | D130 | 1A | | ----- | |
| 494 | D130/ISO2160 | 1A | | ----- | |
| 496 | ISO2160 | 1A | | ----- | |
| 529 | D130 | 1A | | ----- | |
| 540 | D130 | 1A | | ----- | |
| 554 | | ----- | | ----- | |
| 603 | D130 | 1A | | ----- | |
| 657 | D130 | 1A | | ----- | |
| 824 | D130 | 1A | | ----- | |
| 862 | D130 | 1A | | ----- | |
| 863 | D130 | 1A | | ----- | |
| 1016 | D130/ISO2160 | 1A | | ----- | |
| 1033 | IP154 | 1B | | ----- | |
| 1059 | ISO2160 | 1A | | ----- | |
| 1067 | D130/ISO2160 | 1A | | ----- | |
| 1095 | ISO2160 | 1A | | ----- | |
| 1131 | ISO2160 | 1A | | ----- | |
| 1134 | D130/ISO2160 | 1A | | ----- | |
| 1161 | ISO2160 | 1 | | ----- | |
| 1199 | | ----- | | ----- | |
| 1201 | D130/ISO2160 | 1A | | ----- | |
| 1213 | D130 | 1A | | ----- | |
| 1227 | D130 | 1A | | ----- | |
| 1231 | D130 | 1A | | ----- | |
| 1240 | | ----- | | ----- | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | D130/ISO2160 | 1A | | ----- | |
| 1299 | D130/ISO2160 | 1A | | ----- | |
| 1316 | D130 | 1A | | ----- | |
| 1397 | D130/ISO2160 | 1 | | ----- | |
| 1402 | D130/ISO2160 | 1A | | ----- | |
| 1429 | D130 | 1A | | ----- | |
| 1443 | | ----- | | ----- | |
| 1457 | D130/ISO2160 | 1A | | ----- | |
| 1459 | | ----- | | ----- | |
| 1494 | | ----- | | ----- | |
| 1510 | D130 | 1A | | ----- | |
| 1528 | D130/ISO2160 | 1A | | ----- | |
| 1539 | ISO2160 | 1A | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | D130/ISO2160 | 1A | | ----- | |
| 1635 | ISO2160 | 1A | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | ISO2160 | 1A | | ----- | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | D130/ISO2160 | 1 | | ----- | |
| 1739 | ISO2160 | 1A | | ----- | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | | ----- | | ----- | |
| 1948 | D130/ISO2160 | 1A | | ----- | |

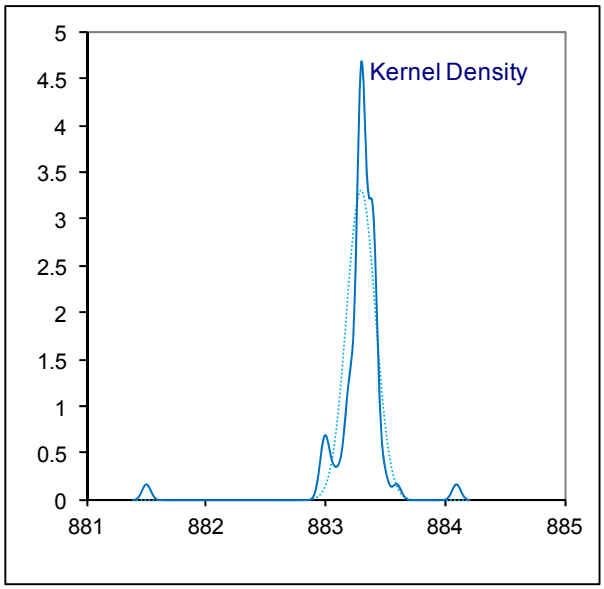
| | |
|---|---|
| normality | n.a |
| n | 52 |
| outliers | n.a |
| mean (n) | 1(1A) |
| st.dev. (n) | n.a |
| R(calc.) | n.a |
| R(D130:12) | n.a |

Determination of Density @ 15°C conform EN spec. on sample #14045; results in kg/m$^3$

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | ISO12185 | 883.3 | | 0.00 | |
| 150 | ISO12185 | 883.6 | | 1.68 | |
| 171 | ISO12185 | 883.32 | | 0.12 | |
| 311 | ISO12185 | 883.4 | | 0.56 | |
| 312 | ISO12185 | 883.3 | | 0.00 | |
| 323 | ISO12185 | 883.4 | | 0.56 | |
| 334 | ISO12185 | 883.0 | C | -1.68 | first reported:882.5 |
| 335 | ISO12185 | 883.2 | | -0.56 | |
| 337 | ISO12185 | 883.4 | | 0.56 | |
| 338 | ISO12185 | 883.3 | | 0.00 | |
| 343 | ISO12185 | 883.35 | | 0.28 | |
| 344 | ISO12185 | 883.4 | | 0.56 | |
| 345 | ISO12185 | 883.3 | | 0.00 | |
| 360 | ISO12185 | 883.5 | | 1.12 | |
| 391 | ISO12185 | 883.1 | | -1.12 | |
| 398 | ISO12185 | 883.3 | | 0.00 | |
| 445 | ISO12185 | 883.3 | | 0.00 | |
| 447 | D4052 | 883.3 | | 0.00 | |
| 494 | ISO12185 | 883.3 | | 0.00 | |
| 496 | ISO12185 | 883.31 | | 0.06 | |
| 529 | D4052 | 883.4 | | 0.56 | |
| 540 | ISO12185 | 883.3 | | 0.00 | |
| 554 | | ----- | | ----- | |
| 603 | ISO12185 | 883.4 | | 0.56 | |
| 657 | ISO12185 | 883.0 | | -1.68 | |
| 824 | ISO12185 | 883.3 | | 0.00 | |
| 862 | ISO12185 | 883.4 | | 0.56 | |
| 863 | ISO12185 | 883.40 | | 0.56 | |
| 1016 | ISO12185 | 883.4 | | 0.56 | |
| 1033 | IP365 | 883.4 | | 0.56 | |
| 1059 | ISO12185 | 883.2 | | -0.56 | |
| 1067 | ISO12185 | 883.3 | | 0.00 | |
| 1095 | ISO12185 | 883.2 | | -0.56 | |
| 1131 | ISO12185 | 883.24 | | -0.33 | |
| 1134 | ISO12185 | 883.3 | | 0.00 | |
| 1161 | ISO12185 | 883.2 | | -0.56 | |
| 1199 | | ----- | | ----- | |
| 1201 | ISO12185 | 883.35 | | 0.28 | |
| 1213 | D4052 | 883.32 | | 0.12 | |
| 1227 | D4052 | 883.4 | | 0.56 | |
| 1231 | D4052 | 883.05 | C | -1.40 | probably unit error, reported 0.88305 |
| 1240 | ISO12185 | 883.3 | | 0.00 | |
| 1268 | | ----- | | ----- | |
| 1286 | ISO12185 | 883.319 | | 0.11 | |
| 1290 | ISO12185 | 883.3 | | 0.00 | |
| 1292 | ISO12185 | 881.5 | R(0.01) | -10.08 | |
| 1299 | ISO12185 | 883.3 | | 0.00 | |
| 1316 | ISO12185 | 883.4 | | 0.56 | |
| 1397 | ISO12185 | 883.4 | | 0.56 | |
| 1402 | ISO12185 | 883.2 | | -0.56 | |
| 1429 | ISO12185 | 883.4 | | 0.56 | |
| 1443 | ISO12185 | 883.40 | | 0.56 | |
| 1457 | ISO12185 | 883.3 | | 0.00 | |
| 1459 | ISO12185 | 883.30 | | 0.00 | |
| 1494 | D4052 | 883.470 | | 0.96 | |
| 1510 | IP365 | 883.3 | | 0.00 | |
| 1528 | ISO12185 | 883.25 | | -0.28 | |
| 1539 | ISO12185 | 883.3 | | 0.00 | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | ISO12185 | 883.308 | | 0.05 | |
| 1635 | | ----- | | ----- | |
| 1643 | ISO12185 | 883.0 | C | -1.68 | first reported: 0.8830 |
| 1650 | ISO12185 | 883.37 | | 0.40 | |
| 1654 | | ----- | | ----- | |
| 1684 | ISO3675 | 884.1 | R(0.01) | 4.48 | |
| 1706 | ISO12185 | 883.3 | | 0.00 | |
| 1721 | ISO12185 | 883.2 | | -0.56 | |
| 1739 | ISO3675 | 882.97 | | -1.84 | |
| 1744 | D2500 | 883.30 | | 0.00 | |
| 1769 | D4052 | 883.14 | C | -0.89 | first reported:0.88314 |
| 1807 | | ----- | | ----- | |
| 1948 | ISO12185 | 883.4 | | 0.56 | |

| | |
|---|---|
| normality | Suspect |
| n | 63 |
| outliers | 2 |
| mean (n) | 883.299 |
| st.dev. (n) | 0.1207 |
| R(calc.) | 0.338 |
| R(ISO12185:96) | 0.500 |

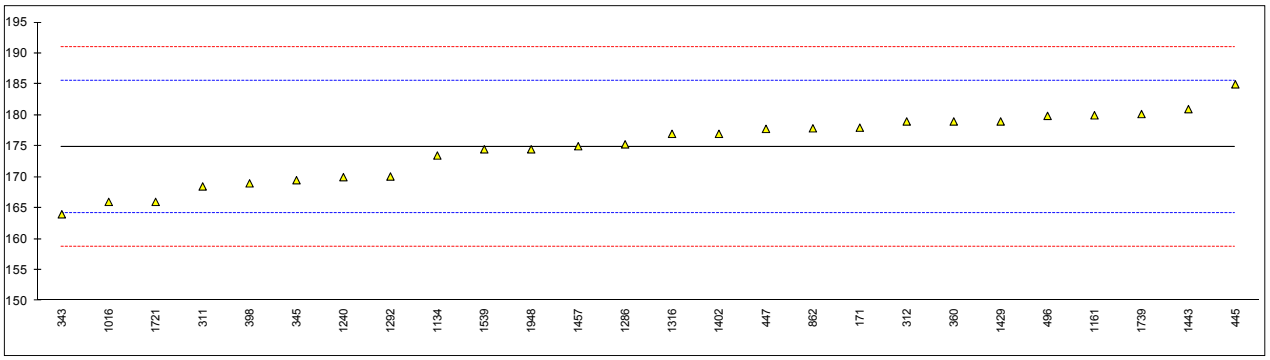


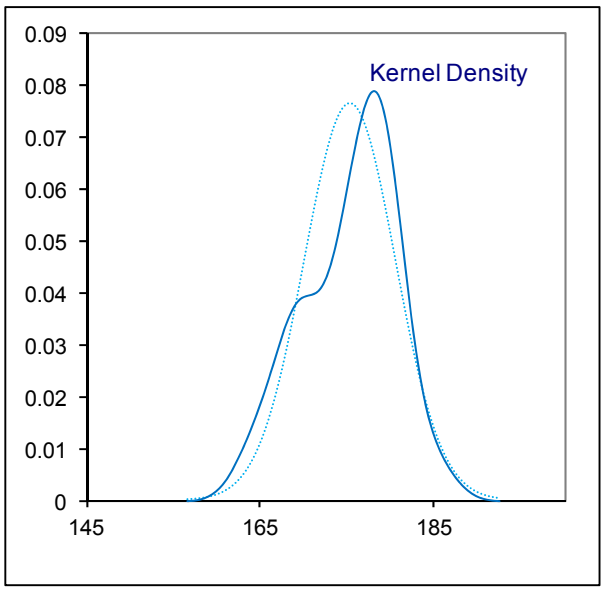



Determination of Flash Point conform EN spec. on sample #14045; results in °C

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | ISO3679 | 178 | | 0.58 | |
| 311 | ISO3679 | 168.5 | | -1.19 | |
| 312 | ISO3679 | 179.0 | | 0.77 | |
| 323 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | ISO3679 | 164.0 | | -2.03 | |
| 344 | | ----- | | ----- | |
| 345 | ISO3679 | 169.5 | | -1.00 | |
| 360 | ISO3679 | 179.0 | | 0.77 | |
| 391 | | ----- | | ----- | |
| 398 | ISO3679 | 169.0 | | -1.10 | |
| 445 | IP523 | 185.0 | | 1.89 | |
| 447 | IP523 | 177.8 | | 0.55 | |
| 494 | | ----- | | ----- | |
| 496 | ISO3679 | 179.9 | | 0.94 | |
| 529 | | ----- | | ----- | |
| 540 | | ----- | | ----- | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | | ----- | | ----- | |
| 824 | | ----- | | ----- | |
| 862 | ISO3679 | 177.9 | | 0.57 | |
| 863 | | ----- | | ----- | |
| 1016 | ISO3679 | 166.0 | | -1.66 | result was reported under flash point (PMcc) |
| 1033 | | ----- | | ----- | |
| 1059 | | ----- | | ----- | |
| 1067 | | ----- | | ----- | |
| 1095 | | ----- | | ----- | |
| 1131 | | ----- | | ----- | |
| 1134 | ISO3679 | 173.5 | | -0.26 | |
| 1161 | ISO3679 | 180 | | 0.96 | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | | ----- | | ----- | |
| 1227 | | ----- | | ----- | |
| 1231 | | ----- | | ----- | |
| 1240 | ISO3679 | 170.0 | | -0.91 | |
| 1268 | | ----- | | ----- | |
| 1286 | ISO3679 | 175.3 | | 0.08 | |
| 1290 | | ----- | | ----- | |
| 1292 | ISO3679 | 170.1 | | -0.89 | |
| 1299 | | ----- | | ----- | |
| 1316 | ISO3679 | 177 | | 0.40 | |
| 1397 | | ----- | | ----- | |
| 1402 | ISO3679 | 177 | | 0.40 | |
| 1429 | ISO3679 | 179 | | 0.77 | |
| 1443 | ISO3679 | 181.0 | | 1.14 | |
| 1457 | ISO3679 | 175.0 | | 0.02 | |
| 1459 | | ----- | | ----- | |
| 1494 | | ----- | | ----- | |
| 1510 | | ----- | | ----- | |
| 1528 | | ----- | | ----- | |
| 1539 | ISO3679 | 174.5 | | -0.07 | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | | ----- | | ----- | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | ISO3679 | 166 | | -1.66 | |
| 1739 | ISO3679 | 180.2 | | 0.99 | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | | ----- | | ----- | |
| 1948 | ISO3679 | 174.5 | | -0.07 | |

| | |
|---|---|
| normality | OK |
| n | 26 |
| outliers | 0 |
| mean (n) | 174.87 |
| st.dev. (n) | 5.427 |
| R(calc.) | 15.19 |
| R(ISO3679:04) | 15.00 |

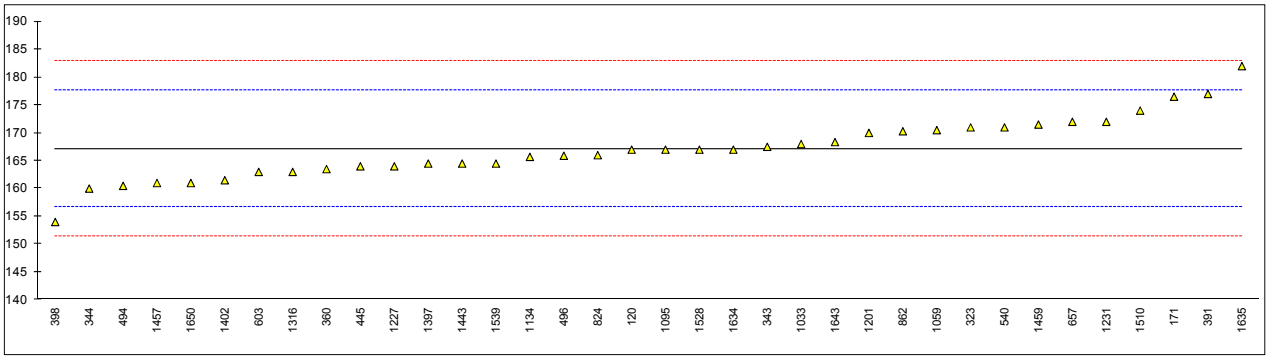


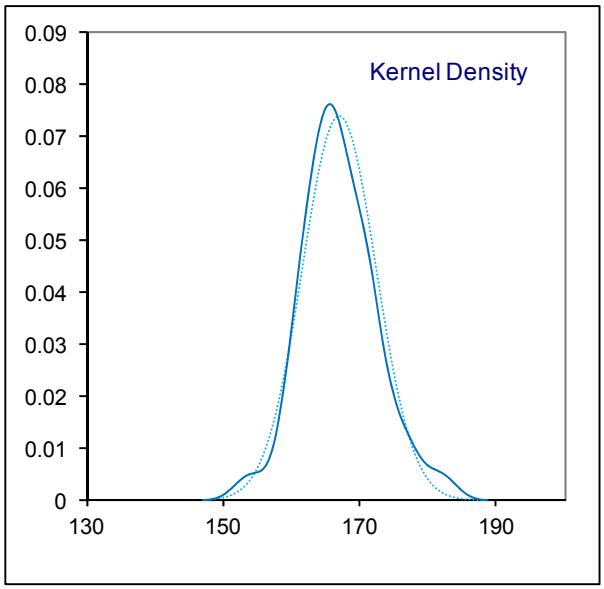



Determination of Flash Point (PMcc) conform ISO/ASTM spec. on sample #14045; results in °C

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | D93 | 167 | | -0.02 | |
| 150 | D93/ISO2719 | >130 | | ----- | |
| 171 | D93/ISO2719 | 176.5 | | 1.79 | |
| 311 | | ----- | | ----- | |
| 312 | | ----- | | ----- | |
| 323 | ISO2719 | 171.0 | | 0.74 | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | D93/ISO2719 | 167.5 | | 0.07 | |
| 344 | D93 | 160.0 | | -1.36 | |
| 345 | | ----- | | ----- | |
| 360 | D93 | 163.5 | | -0.69 | |
| 391 | D93/ISO2719 | 177 | | 1.88 | |
| 398 | ISO2719 | 154.0 | | -2.50 | |
| 445 | ISO2719 | 164.0 | | -0.59 | |
| 447 | | ----- | | ----- | |
| 494 | D93/ISO2719 | 160.5 | | -1.26 | |
| 496 | ISO2719 | 165.9 | | -0.23 | |
| 529 | | ----- | | ----- | |
| 540 | D93 | 171.0 | | 0.74 | |
| 554 | | ----- | | ----- | |
| 603 | D93 | 163.0 | | -0.78 | |
| 657 | D93 | 172 | | 0.93 | |
| 824 | ISO2719 | 166.0 | | -0.21 | |
| 862 | D93 | 170.3 | | 0.61 | |
| 863 | | ----- | | ----- | |
| 1016 | | ----- | | ----- | |
| 1033 | IP34 | 168.0 | | 0.17 | |
| 1059 | D93/ISO2719 | 170.5 | | 0.64 | |
| 1067 | | ----- | | ----- | |
| 1095 | ISO2719 | 167.0 | | -0.02 | |
| 1131 | | ----- | | ----- | |
| 1134 | ISO2719 | 165.7 | | -0.27 | |
| 1161 | | ----- | | ----- | |
| 1199 | | ----- | | ----- | |
| 1201 | D93/ISO2719 | 170 | | 0.55 | |
| 1213 | | ----- | | ----- | |
| 1227 | D93 | 164 | | -0.59 | |
| 1231 | D93 | 172.0 | | 0.93 | |
| 1240 | | ----- | | ----- | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | | ----- | | ----- | |
| 1299 | D93/ISO2719 | >120.0 | | ----- | |
| 1316 | D93/ISO2719 | 163.0 | | -0.78 | |
| 1397 | D93/ISO2719 | 164.5 | | -0.50 | |
| 1402 | D93/ISO2719 | 161.5 | | -1.07 | |
| 1429 | | ----- | | ----- | |
| 1443 | ISO2719 | 164.5 | | -0.50 | |
| 1457 | D93/ISO2719 | 161.0 | | -1.17 | |
| 1459 | ISO2719 | 171.5 | | 0.83 | |
| 1494 | | ----- | | ----- | |
| 1510 | D93 | 174 | | 1.31 | |
| 1528 | D93/ISO2719 | 167.0 | | -0.02 | |
| 1539 | ISO2719 | 164.5 | | -0.50 | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | D93/ISO2719 | 167.0 | | -0.02 | |
| 1635 | ISO2719 | 182 | | 2.83 | |
| 1643 | D93 | 168.35 | | 0.23 | |
| 1650 | ISO2719 | 161.0 | | -1.17 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | | ----- | | ----- | |
| 1739 | | ----- | | ----- | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | | ----- | | ----- | |
| 1948 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 36 |
| outliers | 0 |
| mean (n) | 167.12 |
| st.dev. (n) | 5.474 |
| R(calc.) | 15.33 |
| R(D93-C:13e) | 14.70 |

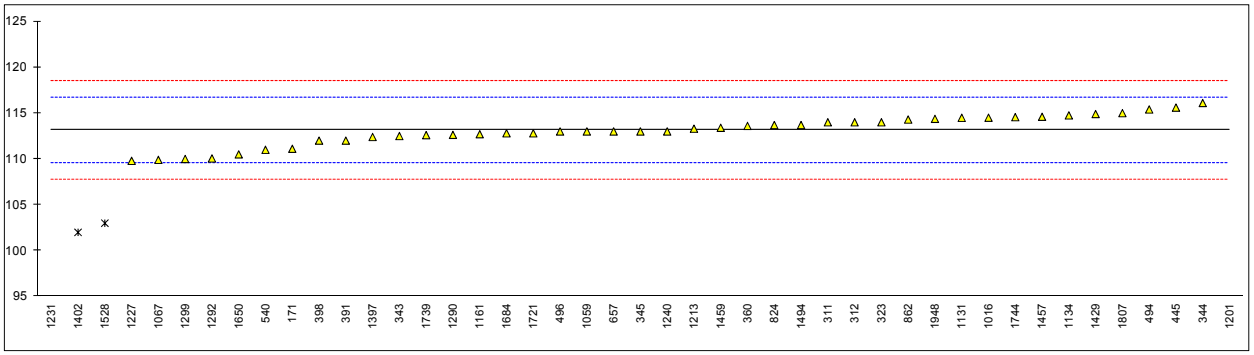


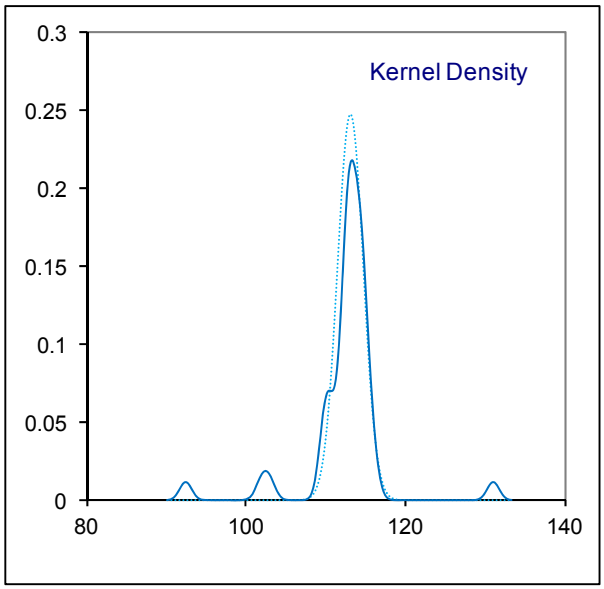



Determination of Iodine Value conform EN spec. on sample #14045; results in g $I_2$/100g

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | EN14111 | 111.11 | | -1.13 | |
| 311 | EN14111 | 114 | | 0.49 | |
| 312 | EN14111 | 114.0 | | 0.49 | |
| 323 | EN14111 | 114 | | 0.49 | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | EN14111 | 112.5 | | -0.35 | |
| 344 | EN14111 | 116.1 | | 1.67 | |
| 345 | EN14111 | 113 | | -0.07 | |
| 360 | EN14111 | 113.6 | | 0.27 | |
| 391 | EN14111 | 112 | | -0.63 | |
| 398 | EN14111 | 112.0 | | -0.63 | |
| 445 | EN14111 | 115.6 | | 1.39 | |
| 447 | | ----- | | ----- | |
| 494 | EN14111 | 115.4 | | 1.27 | |
| 496 | EN14111 | 113 | | -0.07 | |
| 529 | | ----- | | ----- | |
| 540 | EN14111 | 111 | | -1.19 | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN14111 | 113 | | -0.07 | |
| 824 | EN14111 | 113.7 | | 0.32 | |
| 862 | EN14111 | 114.3 | | 0.66 | |
| 863 | | ----- | | ----- | |
| 1016 | EN14111 | 114.5 | | 0.77 | |
| 1033 | | ----- | | ----- | |
| 1059 | EN14111 | 113 | | -0.07 | |
| 1067 | EN14111 | 109.9 | | -1.81 | |
| 1095 | | ----- | | ----- | |
| 1131 | EN14111 | 114.49 | | 0.77 | |
| 1134 | EN14111 | 114.75 | | 0.91 | |
| 1161 | EN14111 | 112.7 | | -0.24 | |
| 1199 | | ----- | | ----- | |
| 1201 | EN14111 | 131 | R(0.01) | 10.01 | |
| 1213 | EN14111 | 113.3 | | 0.10 | |
| 1227 | EN14111 | 109.8 | | -1.86 | |
| 1231 | EN14111 | 92.5 | R(0.01) | -11.55 | |
| 1240 | EN16300 | 113.0 | | -0.07 | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | EN14111 | 112.63 | | -0.28 | |
| 1292 | EN14111 | 110.06 | | -1.72 | |
| 1299 | EN14111 | 110 | | -1.75 | |
| 1316 | | ----- | | ----- | |
| 1397 | EN16300 | 112.4 | | -0.41 | |
| 1402 | EN14111 | 102 | R(0.01) | -6.23 | |
| 1429 | EN14111 | 114.9 | | 0.99 | |
| 1443 | | ----- | | ----- | |
| 1457 | EN14111 | 114.6 | | 0.83 | |
| 1459 | EN16300 | 113.4 | | 0.15 | |
| 1494 | EN14111 | 113.700 | | 0.32 | |
| 1510 | | ----- | | ----- | |
| 1528 | EN14111 | 103 | C,R(0.01) | -5.67 | first reported: 90.9 |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | EN14111 | 110.5 | | -1.47 | |
| 1654 | | ----- | | ----- | |
| 1684 | EN14111 | 112.8 | | -0.18 | |
| 1706 | | ----- | | ----- | |
| 1721 | EN14111 | 112.8 | | -0.18 | |
| 1739 | EN14111 | 112.6 | | -0.29 | |
| 1744 | EN14111 | 114.56 | | 0.80 | |
| 1769 | | ----- | | ----- | |
| 1807 | EN14111 | 115 | | 1.05 | |
| 1948 | EN14111 | 114.374 | | 0.70 | |

| | |
|---|---|
| normality | OK |
| n | 41 |
| outliers | 4 |
| mean (n) | 113.12 |
| st.dev. (n) | 1.614 |
| R(calc.) | 4.52 |
| R(EN14111:03) | 5.00 |



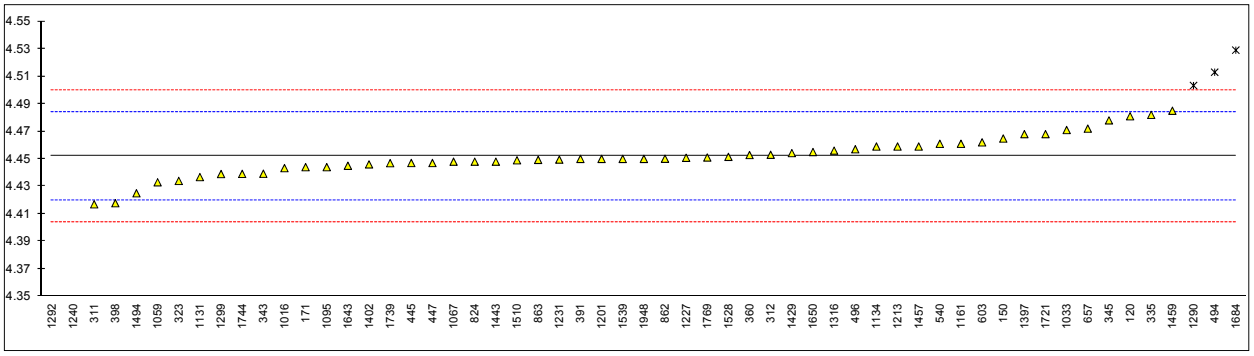



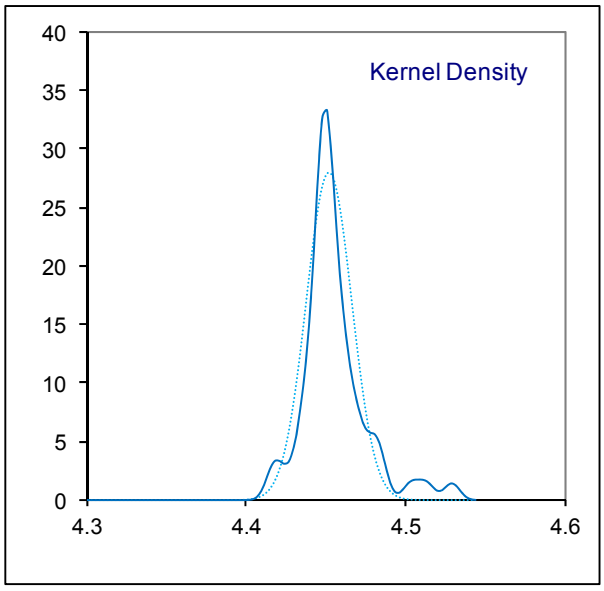
Determination of Kinematic Viscosity @ 40°C on sample #14045; results in $mm^2/s$

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | D445 | 4.481 | | 1.82 | |
| 150 | D445/ISO3104 | 4.4648 | | 0.81 | |
| 171 | D445/ISO3104 | 4.444 | | -0.49 | |
| 311 | D445/ISO3104 | 4.417 | | -2.18 | |
| 312 | ISO3104 | 4.453 | | 0.07 | |
| 323 | ISO3104 | 4.434 | | -1.12 | |
| 334 | | ----- | | ----- | |
| 335 | D445/ISO3104 | 4.482 | | 1.89 | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | D445/ISO3104 | 4.4392 | | -0.79 | |
| 344 | | ----- | | ----- | |
| 345 | ISO3104 | 4.478 | | 1.64 | |
| 360 | ISO3104 | 4.4528 | | 0.06 | |
| 391 | D445/ISO3104 | 4.450 | | -0.12 | |
| 398 | ISO3104 | 4.4178 | | -2.13 | |
| 445 | ISO3104 | 4.447 | | -0.31 | |
| 447 | D445 | 4.447 | | -0.31 | |
| 494 | D445/ISO3104 | 4.513 | C,R(0.05) | 3.83 | first reported:4.523 |
| 496 | ISO3104 | 4.4570 | | 0.32 | |
| 529 | | ----- | | ----- | |
| 540 | ISO3104 | 4.461 | | 0.57 | |
| 554 | | ----- | | ----- | |
| 603 | D445 | 4.462 | | 0.63 | |
| 657 | D445 | 4.472 | C | 1.26 | first reported:4.555 |
| 824 | ISO3104 | 4.448 | | -0.24 | |
| 862 | D445 | 4.4501 | | -0.11 | |
| 863 | D445 | 4.4493 | | -0.16 | |
| 1016 | D445/ISO3104 | 4.4433 | | -0.54 | |
| 1033 | IP71 | 4.471 | | 1.20 | |
| 1059 | D445/ISO3104 | 4.433 | | -1.18 | |
| 1067 | D445/ISO3104 | 4.448 | | -0.24 | |
| 1095 | ISO3104 | 4.444 | | -0.49 | |
| 1131 | D445 | 4.4368 | | -0.94 | |
| 1134 | ISO3104 | 4.459 | | 0.45 | |
| 1161 | ISO3104 | 4.461 | | 0.57 | |
| 1199 | | ----- | | ----- | |
| 1201 | D445/ISO3104 | 4.450 | | -0.12 | |
| 1213 | D445 | 4.459 | | 0.45 | |
| 1227 | D445 | 4.4508 | | -0.07 | |
| 1231 | D445 | 4.4495 | | -0.15 | |
| 1240 | ISO3104 | 4.265 | C,R(0.01) | -11.71 | first reported:4.383 |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | D7042 | 4.5033 | R(0.05) | 3.22 | |
| 1292 | D445/ISO3104 | 3.79285 | R(0.01) | -41.28 | |
| 1299 | D445/ISO3104 | 4.439 | | -0.81 | |
| 1316 | ISO3104 | 4.456 | | 0.26 | |
| 1397 | D445/ISO3104 | 4.468 | | 1.01 | |
| 1402 | D445/ISO3104 | 4.446 | | -0.37 | |
| 1429 | D445 | 4.4542 | | 0.14 | |
| 1443 | ISO3104 | 4.4480 | | -0.24 | |
| 1457 | D445/ISO3104 | 4.459 | | 0.45 | |
| 1459 | D7042 | 4.485 | | 2.07 | |
| 1494 | D445 | 4.42500 | | -1.68 | |
| 1510 | D445 | 4.449 | | -0.18 | |
| 1528 | D445/ISO3104 | 4.45149 | | -0.02 | |
| 1539 | ISO3104 | 4.450 | | -0.12 | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | D445 | 4.445 | | -0.43 | |
| 1650 | ISO3104 | 4.4550 | | 0.20 | |
| 1654 | | ----- | | ----- | |
| 1684 | ISO3104 | 4.529 | R(0.01) | 4.83 | |
| 1706 | | ----- | | ----- | |
| 1721 | D445/ISO3104 | 4.468 | | 1.01 | |
| 1739 | ISO3104 | 4.4469 | | -0.31 | |
| 1744 | D445 | 4.4391 | | -0.80 | |
| 1769 | D445 | 4.4510 | | -0.06 | |
| 1807 | | ----- | | ----- | |
| 1948 | D445/ISO3104 | 4.450 | | -0.12 | |

| | |
|---|---|
| normality | OK |
| n | 52 |
| outliers | 5 |
| mean (n) | 4.4519 |
| st.dev. (n) | 0.01428 |
| R(calc.) | 0.0400 |
| R(ISO3104:94) | 0.0447 |

Compare R(445:12) = 0.0338

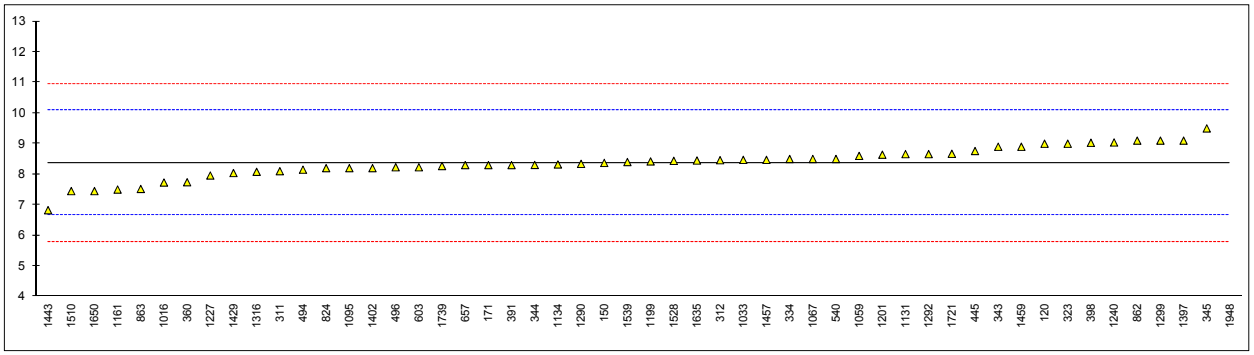


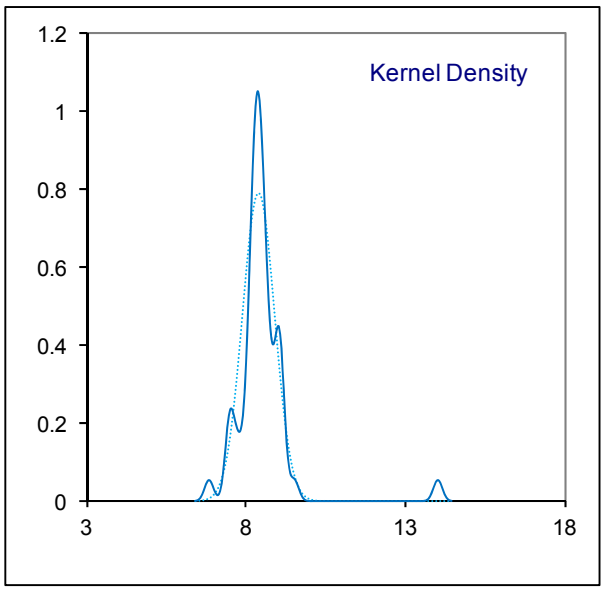



## Determination of Oxidation Stability on sample #14045; results in hours

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | EN14112 | 9 | | 0.72 | |
| 150 | EN14112 | 8.37 | | -0.01 | |
| 171 | EN14112 | 8.3 | | -0.09 | |
| 311 | EN14112 | 8.1 | | -0.33 | |
| 312 | EN14112 | 8.46 | | 0.09 | |
| 323 | EN14112 | 9.0 | | 0.72 | |
| 334 | EN14112 | 8.5 | | 0.14 | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | EN14112 | 8.9 | | 0.60 | |
| 344 | EN14112 | 8.305 | | -0.09 | |
| 345 | EN14112 | 9.5 | | 1.30 | |
| 360 | EN14112 | 7.74 | | -0.74 | |
| 391 | EN14112 | 8.3 | | -0.09 | |
| 398 | EN14112 | 9.03 | | 0.76 | |
| 445 | EN15751 | 8.76 | | 0.44 | |
| 447 | | ----- | | ----- | |
| 494 | EN14112 | 8.15 | | -0.27 | |
| 496 | EN14112 | 8.23 | | -0.17 | |
| 529 | | ----- | | ----- | |
| 540 | EN14112 | 8.5 | | 0.14 | |
| 554 | | ----- | | ----- | |
| 603 | EN14112 | 8.23 | | -0.17 | |
| 657 | EN14112 | 8.3 | | -0.09 | |
| 824 | EN14112 | 8.2 | | -0.21 | |
| 862 | EN14112 | 9.10 | | 0.84 | |
| 863 | EN14112 | 7.52 | | -1.00 | |
| 1016 | EN14112 | 7.73 | | -0.76 | |
| 1033 | EN14112 | 8.47 | | 0.10 | |
| 1059 | EN14112 | 8.6 | | 0.26 | |
| 1067 | EN14112 | 8.5 | | 0.14 | |
| 1095 | EN14112 | 8.2 | | -0.21 | |
| 1131 | EN14112 | 8.66 | | 0.33 | |
| 1134 | EN14112 | 8.32 | | -0.07 | |
| 1161 | EN14112 | 7.5 | | -1.02 | |
| 1199 | EN14112 | 8.42 | | 0.05 | |
| 1201 | EN14112 | 8.64 | | 0.30 | |
| 1213 | | ----- | | ----- | |
| 1227 | EN14112 | 7.96 | | -0.49 | |
| 1231 | | ----- | | ----- | |
| 1240 | EN15751 | 9.04 | | 0.77 | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | EN14112 | 8.34 | | -0.05 | |
| 1292 | EN14112 | 8.66 | | 0.33 | |
| 1299 | EN15751 | 9.1 | | 0.84 | |
| 1316 | EN14112 | 8.08 | | -0.35 | |
| 1397 | EN14112 | 9.1 | | 0.84 | |
| 1402 | EN14112 | 8.2 | | -0.21 | |
| 1429 | EN14112 | 8.04 | | -0.40 | |
| 1443 | EN14112 | 6.825 | | -1.81 | |
| 1457 | EN14112 | 8.47 | | 0.10 | |
| 1459 | EN15751 | 8.9 | | 0.60 | |
| 1494 | | ----- | | ----- | |
| 1510 | EN14112 | 7.45 | | -1.08 | |
| 1528 | EN14112 | 8.44 | | 0.07 | |
| 1539 | EN14112 | 8.4 | | 0.02 | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | EN14112 | 8.45 | | 0.08 | |
| 1643 | | ----- | | ----- | |
| 1650 | EN14112 | 7.45 | | -1.08 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | EN14112 | 8.67 | | 0.34 | |
| 1739 | EN14112 | 8.27 | | -0.13 | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | | ----- | | ----- | |
| 1948 | EN14112 | 14.01 | C,R(0.01) | 6.54 | first reported:25.09 |

| | |
|---|---|
| normality | suspect |
| n | 51 |
| outliers | 1 |
| mean (n) | 8.380 |
| st.dev. (n) | 0.5049 |
| R(calc.) | 1.414 |
| R(EN14112:03) | 2.409 |



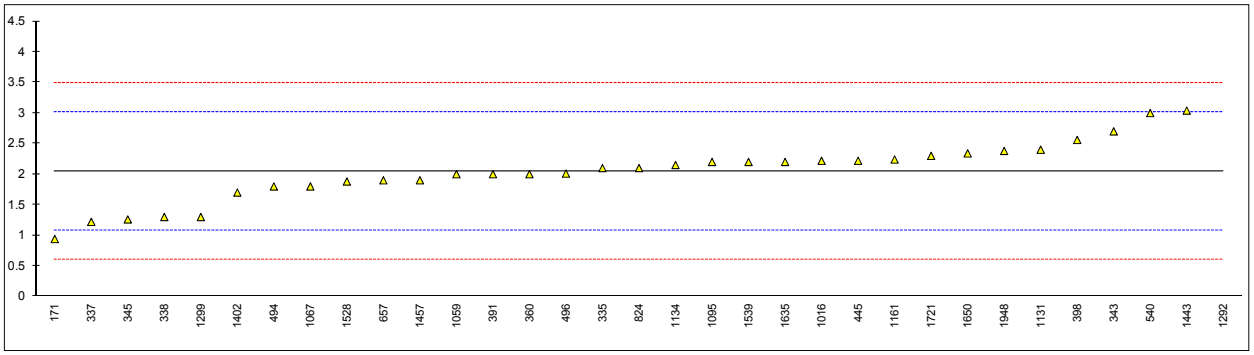



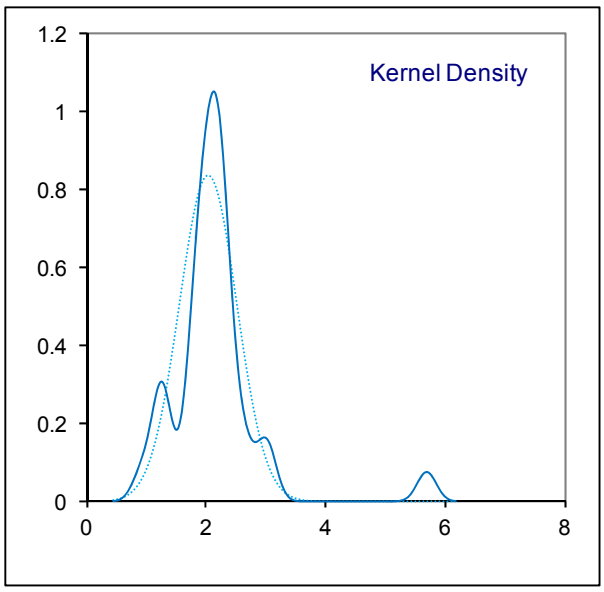
Determination of Sulphur (EN spec.) on sample #14045; results in mg/kg

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | ISO20846 | 0.94 | | -2.29 | |
| 311 | ISO20846 | <3 | | ----- | |
| 312 | | ----- | | ----- | |
| 323 | ISO20846 | <3.0 | | ----- | |
| 334 | | ----- | | ----- | |
| 335 | ISO20846 | 2.1 | | 0.12 | |
| 337 | ISO20846 | 1.22 | | -1.71 | |
| 338 | ISO20846 | 1.3 | | -1.54 | |
| 343 | ISO20846 | 2.7 | | 1.37 | |
| 344 | | ----- | | ----- | |
| 345 | ISO20846 | 1.26 | | -1.62 | |
| 360 | ISO20846 | 2.0 | | -0.09 | |
| 391 | ISO20846 | 2.0 | | -0.09 | |
| 398 | ISO20846 | 2.56 | | 1.07 | |
| 445 | IP490 | 2.22 | | 0.37 | |
| 447 | | ----- | | ----- | |
| 494 | ISO20846 | 1.8 | | -0.50 | |
| 496 | ISO20846 | 2.01 | | -0.07 | |
| 529 | | ----- | | ----- | |
| 540 | ISO20846 | 3.0 | | 1.99 | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | ISO20846 | 1.9 | | -0.30 | |
| 824 | ISO20846 | 2.1 | | 0.12 | |
| 862 | | ----- | | ----- | |
| 863 | | ----- | | ----- | |
| 1016 | ISO20846 | 2.219 | | 0.37 | |
| 1033 | | ----- | | ----- | |
| 1059 | ISO20884 | 2.0 | | -0.09 | |
| 1067 | ISO20846 | 1.8 | | -0.50 | |
| 1095 | ISO20846 | 2.2 | | 0.33 | |
| 1131 | ISO20846 | 2.4 | | 0.74 | |
| 1134 | ISO20846 | 2.15 | | 0.22 | |
| 1161 | ISO20846 | 2.24 | | 0.41 | |
| 1199 | ISO20884 | <5.0 | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | | ----- | | ----- | |
| 1227 | | ----- | | ----- | |
| 1231 | | ----- | | ----- | |
| 1240 | | ----- | | ----- | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | W | ----- | result withdrawn, first reported: 17.72 |
| 1292 | in house | 5.695 | R(0.01) | 7.58 | |
| 1299 | ISO20846 | 1.3 | | -1.54 | |
| 1316 | | ----- | | ----- | |
| 1397 | | ----- | | ----- | |
| 1402 | ISO20846 | 1.7 | | -0.71 | |
| 1429 | | ----- | | ----- | |
| 1443 | ISO20846 | 3.04 | | 2.07 | |
| 1457 | ISO20846 | 1.9 | | -0.30 | |
| 1459 | in house | <2 | | ----- | |
| 1494 | | ----- | | ----- | |
| 1510 | | ----- | | ----- | |
| 1528 | ISO20846 | 1.88 | | -0.34 | |
| 1539 | ISO20846 | 2.2 | | 0.33 | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | ISO20846 | 2.2 | | 0.33 | |
| 1643 | | ----- | | ----- | |
| 1650 | ISO20846 | 2.34 | | 0.62 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | ISO20846 | 2.3 | | 0.53 | |
| 1739 | | ----- | | ----- | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | | ----- | | ----- | |
| 1948 | ISO20846 | 2.38 | | 0.70 | |

| | |
|---|---|
| normality | OK |
| n | 32 |
| outliers | 1 |
| mean (n) | 2.042 |
| st.dev. (n) | 0.4792 |
| R(calc.) | 1.342 |
| R(ISO20846:11) | 1.349 |

application range 3 – 500 mg/kg



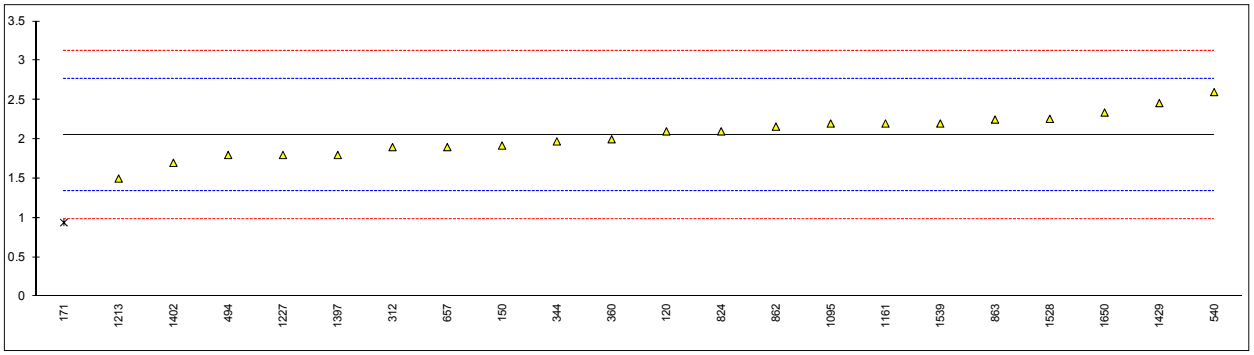



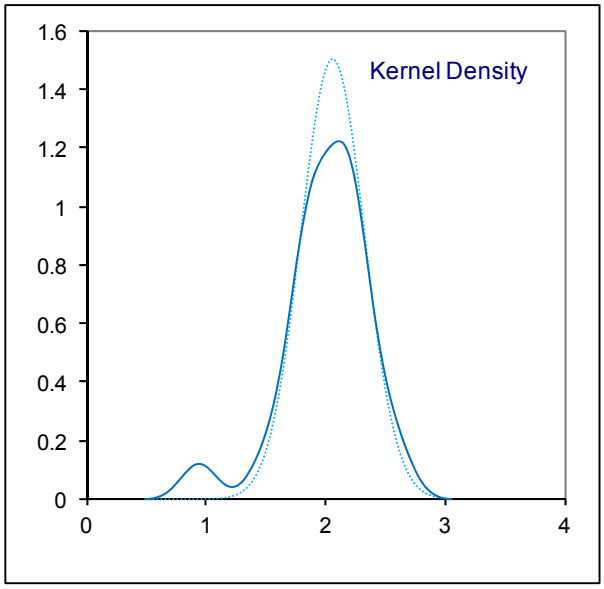
## Determination of Sulphur (ASTM D5453) on sample #14045; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | D5453 | 2.1 | C | 0.13 | first reported: 3.1 |
| 150 | D5453 | 1.92 | | -0.38 | |
| 171 | D5453 | 0.94 | R(0.05) | -3.14 | |
| 311 | | ----- | | ----- | |
| 312 | D5453 | 1.9 | | -0.44 | |
| 323 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | | ----- | | ----- | |
| 344 | D5453 | 1.973 | | -0.23 | |
| 345 | | ----- | | ----- | |
| 360 | D5453 | 2.0 | | -0.16 | |
| 391 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 445 | | ----- | | ----- | |
| 447 | | ----- | | ----- | |
| 494 | D5453 | 1.8 | | -0.72 | |
| 496 | | ----- | | ----- | |
| 529 | | ----- | | ----- | |
| 540 | D5453 | 2.6 | | 1.53 | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | D5453 | 1.9 | | -0.44 | |
| 824 | D5453 | 2.1 | | 0.13 | |
| 862 | D5453 | 2.16 | | 0.29 | |
| 863 | D5453 | 2.25 | | 0.55 | |
| 1016 | | ----- | | ----- | |
| 1033 | | ----- | | ----- | |
| 1059 | | ----- | | ----- | |
| 1067 | | ----- | | ----- | |
| 1095 | D5453 | 2.2 | | 0.41 | |
| 1131 | | ----- | | ----- | |
| 1134 | | ----- | | ----- | |
| 1161 | D5453 | 2.20 | | 0.41 | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | D5453 | 1.50 | | -1.56 | |
| 1227 | D5453 | 1.8 | | -0.72 | |
| 1231 | | ----- | | ----- | |
| 1240 | | ----- | | ----- | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | | ----- | | ----- | |
| 1299 | | ----- | | ----- | |
| 1316 | | ----- | | ----- | |
| 1397 | D5453 | 1.8 | | -0.72 | |
| 1402 | D5453 | 1.7 | | -1.00 | |
| 1429 | D5453 | 2.46 | | 1.14 | |
| 1443 | | ----- | | ----- | |
| 1457 | | ----- | | ----- | |
| 1459 | | ----- | | ----- | |
| 1494 | | ----- | | ----- | |
| 1510 | | ----- | | ----- | |
| 1528 | D5453 | 2.26 | | 0.58 | |
| 1539 | D5453 | 2.2 | | 0.41 | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | D5453 | 2.34 | | 0.80 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | | ----- | | ----- | |
| 1739 | | ----- | | ----- | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | | ----- | | ----- | |
| 1948 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 21 |
| outliers | 1 |
| mean (n) | 2.055 |
| st.dev. (n) | 0.2651 |
| R(calc.) | 0.742 |
| R(D5453:12) | 0.995 |

Application range: 1- 8000 mg/kg



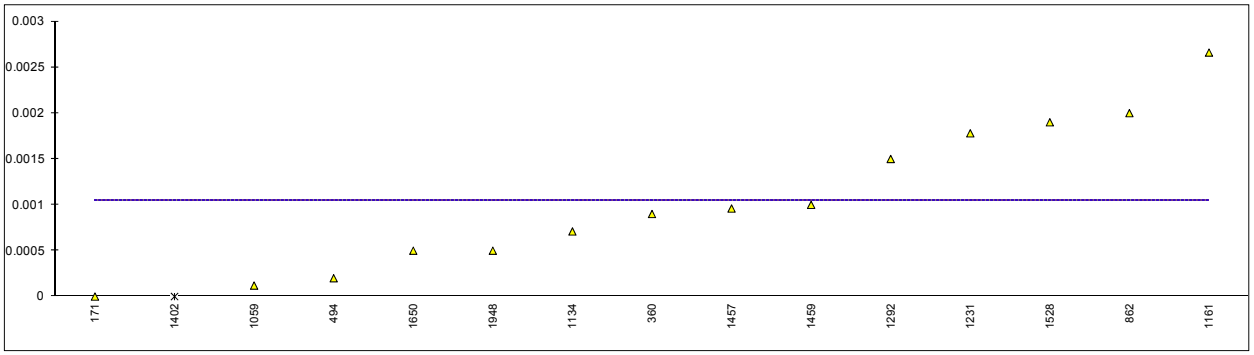



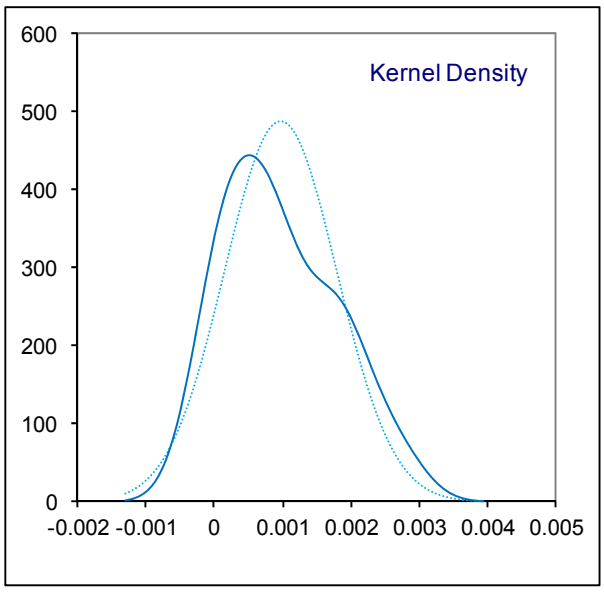
## Determination of Sulphated Ash on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | D874 | <0.005 | | ----- | |
| 150 | D874/ISO3987 | <0.001 | | ----- | |
| 171 | D874/ISO3987 | 0.000 | | ----- | |
| 311 | | ----- | | ----- | |
| 312 | | ----- | | ----- | |
| 323 | ISO3987 | <0.005 | | ----- | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | D874/ISO3987 | <0.005 | | ----- | |
| 344 | ISO3987 | <0.005 | | ----- | |
| 345 | ISO3987 | <0.005 | | ----- | |
| 360 | D874 | 0.0009 | | ----- | |
| 391 | | ----- | | ----- | |
| 398 | ISO3987 | <0.005 | | ----- | |
| 445 | ISO3987 | <0.01 | | ----- | |
| 447 | | ----- | | ----- | |
| 494 | D874/ISO3987 | 0.0002 | | ----- | |
| 496 | ISO3987 | <0.001 | | ----- | |
| 529 | | ----- | | ----- | |
| 540 | ISO3987 | <0.005 | | ----- | |
| 554 | | ----- | | ----- | |
| 603 | D874 | <0.005 | | ----- | |
| 657 | D874 | <0.005 | | ----- | |
| 824 | | ----- | | ----- | |
| 862 | D874 | 0.002 | | ----- | |
| 863 | D874 | <0.001 | | ----- | |
| 1016 | D874 | <0.001 | | ----- | |
| 1033 | | ----- | | ----- | |
| 1059 | ISO3987 | 0.00012 | | ----- | |
| 1067 | | ----- | | ----- | |
| 1095 | | ----- | | ----- | |
| 1131 | | ----- | | ----- | |
| 1134 | ISO3987 | 0.00071 | | ----- | |
| 1161 | ISO3987 | 0.00266 | | ----- | |
| 1199 | | ----- | | ----- | |
| 1201 | D874/ISO3987 | <0.001 | | ----- | |
| 1213 | D874 | <0.001 | | ----- | |
| 1227 | | ----- | | ----- | |
| 1231 | D874 | 0.00178 | | ----- | |
| 1240 | | ----- | | ----- | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | D874/ISO3987 | 0.0015 | | ----- | |
| 1299 | | ----- | | ----- | |
| 1316 | | ----- | | ----- | |
| 1397 | | ----- | | ----- | |
| 1402 | D874/ISO3987 | 0 | ex | ----- | result excluded, zero is not a real value |
| 1429 | | ----- | | ----- | |
| 1443 | | ----- | W | ----- | result withdrawn, first reported:0.0146 |
| 1457 | D874/ISO3987 | 0.00096 | | ----- | |
| 1459 | ISO3987 | 0.0010 | | ----- | |
| 1494 | | ----- | | ----- | |
| 1510 | | ----- | | ----- | |
| 1528 | D874/ISO3987 | 0.0019 | | ----- | |
| 1539 | ISO3987 | <0.005 | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | ISO3987 | 0.0005 | | ----- | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | D874/ISO3987 | <0.005 | | ----- | |
| 1739 | ISO3987 | <0.01 | | ----- | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | ISO3987 | <0.005 | | ----- | |
| 1948 | D874/ISO3987 | 0.0005 | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 14 |
| outliers | 1 + 1excl. |
| mean (n) | 0.00105 |
| st.dev. (n) | 0.000804 |
| R(calc.) | 0.00225 |
| R(D874:13a) | (0.00056) |

Compare R(ISO3987:10) = 0.00013
Applicable lower limit of 0.005 %M/M

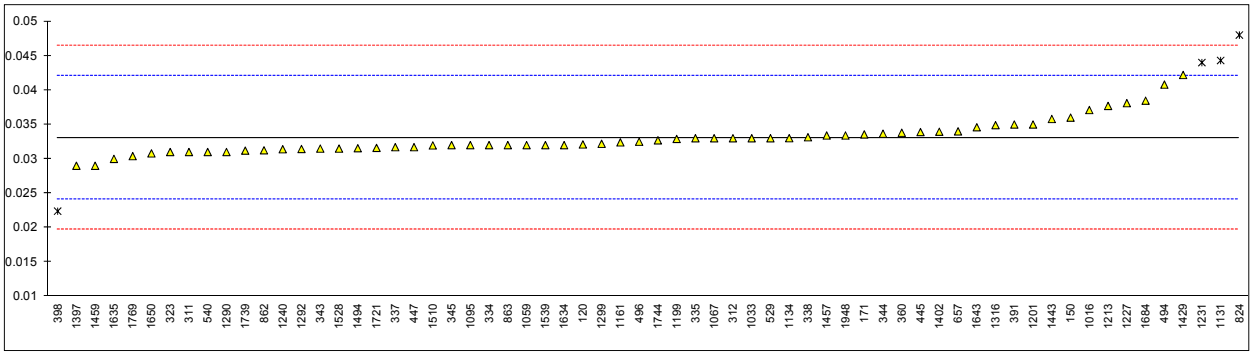


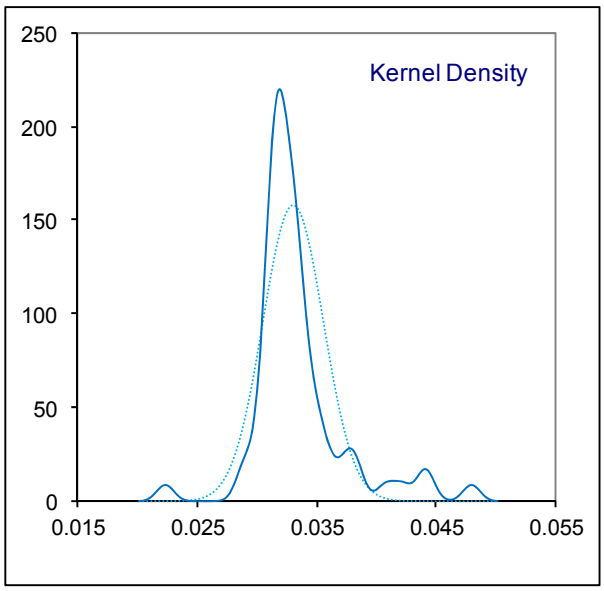



## Determination of Water on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | ISO12937 | 0.0321 | | -0.22 | |
| 150 | ISO12937 | 0.036 | | 0.65 | |
| 171 | ISO12937 | 0.03356 | C | 0.11 | probably unit error, reported 335.6 |
| 311 | ISO12937 | 0.031 | | -0.47 | |
| 312 | ISO12937 | 0.0330 | | -0.02 | |
| 323 | ISO12937 | 0.0310 | C | -0.47 | first reported: 310 |
| 334 | ISO12937 | 0.032 | | -0.24 | |
| 335 | ISO12937 | 0.033 | | -0.02 | |
| 337 | ISO12937 | 0.0317 | | -0.31 | |
| 338 | ISO12937 | 0.033144 | C | 0.01 | probably unit error, reported 331.44 |
| 343 | ISO12937 | 0.0315 | | -0.36 | |
| 344 | ISO12937 | 0.03366 | | 0.13 | |
| 345 | ISO12937 | 0.032 | | -0.24 | |
| 360 | ISO12937 | 0.0338 | | 0.16 | |
| 391 | ISO12937 | 0.035 | C | 0.43 | probably unit error, reported 350 |
| 398 | ISO12937 | 0.0224 | R(0.01) | -2.39 | |
| 445 | ISO12937 | 0.0339 | | 0.18 | |
| 447 | IP438 | 0.031705 | C | -0.31 | probably unit error, reported 317.05 |
| 494 | ISO12937 | 0.0408 | C | 1.73 | first reported: 408 |
| 496 | ISO12937 | 0.0325 | C | -0.13 | first reported: 352.5 |
| 529 | E1064 | 0.033 | | -0.02 | |
| 540 | ISO12937 | 0.031 | | -0.47 | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | ISO12937 | 0.034 | | 0.20 | |
| 824 | ISO12937 | 0.048 | R(0.01) | 3.34 | |
| 862 | ISO12937 | 0.03125 | C | -0.41 | probably unit error, reported 312.5 |
| 863 | ISO12937 | 0.032 | | -0.24 | |
| 1016 | ISO12937 | 0.0371 | C | 0.90 | first reported: 371.2 |
| 1033 | IP438 | 0.033 | | -0.02 | |
| 1059 | ISO12937 | 0.032 | C | -0.24 | probably unit error, reported 320 |
| 1067 | ISO12937 | 0.033 | | -0.02 | |
| 1095 | ISO12937 | 0.032 | | -0.24 | |
| 1131 | ISO12937 | 0.0443 | R(0.01) | 2.51 | |
| 1134 | ISO12937 | 0.0330115 | C | -0.02 | probably unit error, reported 330.115 |
| 1161 | ISO12937 | 0.0324 | | -0.15 | |
| 1199 | ISO12937 | 0.0329 | | -0.04 | |
| 1201 | ISO12937 | 0.035 | | 0.43 | |
| 1213 | D6304 | 0.0377 | | 1.03 | |
| 1227 | D6304 | 0.0381 | | 1.12 | |
| 1231 | ISO12937 | 0.044 | R(0.01) | 2.44 | |
| 1240 | ISO12937 | 0.0314 | | -0.38 | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | ISO12937 | 0.031 | | -0.47 | |
| 1292 | ISO12937 | 0.03144 | | -0.37 | |
| 1299 | ISO12937 | 0.0322 | | -0.20 | |
| 1316 | E1064 | 0.0349 | C | 0.41 | first reported: 349 |
| 1397 | ISO12937 | 0.029 | | -0.92 | |
| 1402 | ISO12937 | 0.033947 | C | 0.19 | first reported: 339.47 |
| 1429 | ISO12937 | 0.0422 | | 2.04 | |
| 1443 | ISO12937 | 0.0358 | | 0.61 | |
| 1457 | ISO12937 | 0.0334 | C | 0.07 | first reported: 334 |
| 1459 | ISO12937 | 0.029 | | -0.92 | |
| 1494 | E203 | 0.031550 | | -0.34 | |
| 1510 | IP438 | 0.03196 | C | -0.25 | probably unit error, reported 319.6 |
| 1528 | ISO12937 | 0.0315 | | -0.36 | |
| 1539 | ISO12937 | 0.032 | | -0.24 | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | ISO12937 | 0.032 | | -0.24 | |
| 1635 | ISO12937 | 0.03 | C | -0.69 | first reported: 326 |
| 1643 | ISO12937 | 0.0346 | | 0.34 | |
| 1650 | ISO12937 | 0.0308 | | -0.51 | |
| 1654 | | ----- | | ----- | |
| 1684 | ISO12937 | 0.03846 | | 1.20 | |
| 1706 | | ----- | | ----- | |
| 1721 | ISO12937 | 0.0316 | | -0.33 | |
| 1739 | ISO12937 | 0.0312 | | -0.42 | |
| 1744 | E203 | 0.0327 | | -0.09 | |
| 1769 | ISO12937 | 0.0304 | | -0.60 | |
| 1807 | | ----- | | ----- | |
| 1948 | ISO12937 | 0.0334 | C | 0.07 | first reported: 333.8 |

| | |
|---|---|
| normality | not OK |
| n | 60 |
| outliers | 4 |
| mean (n) | 0.03309 |
| st.dev. (n) | 0.002529 |
| R(calc.) | 0.00708 |
| R(ISO12937:00) | 0.01251 |

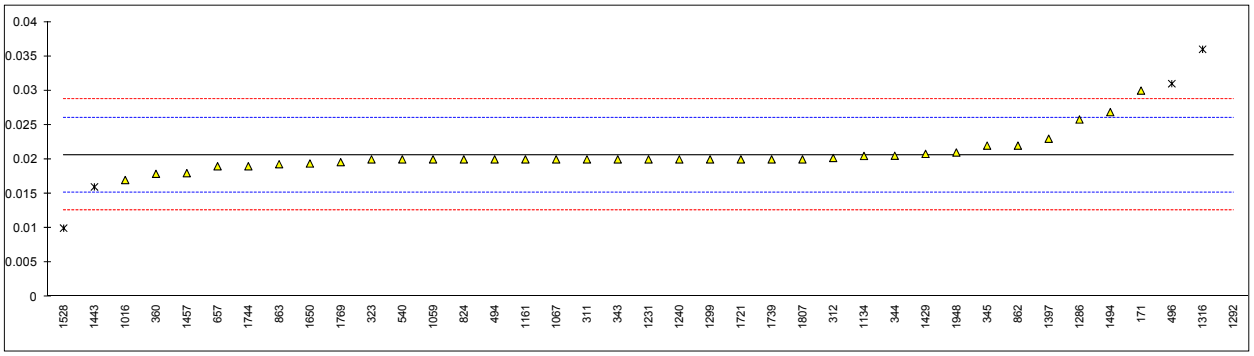


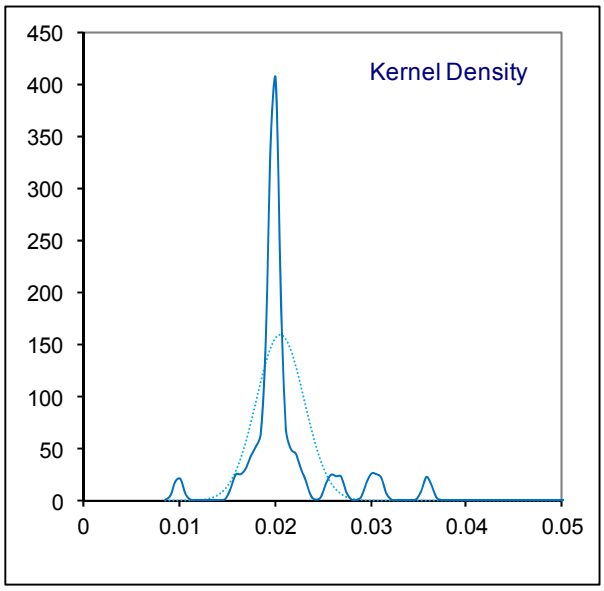



## Determination of Methanol on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | EN14110 | <0.01 | | <-2.94 | false negative result? |
| 150 | | ----- | | ----- | |
| 171 | EN14110-A | 0.03 | | 3.46 | |
| 311 | EN14110-B | 0.02 | | -0.24 | |
| 312 | EN14110-B | 0.0202 | | -0.16 | |
| 323 | EN14110-B | 0.02 | | -0.24 | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | EN14110-A | 0.02 | | -0.24 | |
| 344 | EN14110-A | 0.02053 | | -0.04 | |
| 345 | EN14110-B | 0.022 | C | 0.50 | first reported: 0.04 |
| 360 | EN14110-B | 0.0179 | | -1.02 | |
| 391 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 445 | | ----- | | ----- | |
| 447 | | ----- | | ----- | |
| 494 | EN14110-A | 0.02 | | -0.24 | |
| 496 | EN14110-B | 0.031 | R(0.01) | 3.83 | |
| 529 | | ----- | | ----- | |
| 540 | EN14110-A | 0.02 | | -0.24 | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN14110-A | 0.019 | | -0.61 | |
| 824 | EN14110-B | 0.02 | | -0.24 | |
| 862 | EN14110-A | 0.022 | | 0.50 | |
| 863 | EN14110-A | 0.0193 | | -0.50 | |
| 1016 | EN14110-B | 0.017 | | -1.35 | |
| 1033 | | ----- | | ----- | |
| 1059 | EN14110-B | 0.02 | | -0.24 | |
| 1067 | EN14110-B | 0.02 | | -0.24 | |
| 1095 | | ----- | | ----- | |
| 1131 | | ----- | | ----- | |
| 1134 | EN14110-A | 0.0205 | | -0.05 | |
| 1161 | EN14110-A | 0.02 | | -0.24 | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | | ----- | | ----- | |
| 1227 | | ----- | | ----- | |
| 1231 | EN14110 | 0.02 | | -0.24 | |
| 1240 | EN14110-A | 0.020 | | -0.24 | |
| 1268 | | ----- | | ----- | |
| 1286 | EN14110-B | 0.0258 | | 1.91 | |
| 1290 | | ----- | | ----- | |
| 1292 | EN14110-B | 0.141 | R(0.01) | 44.56 | |
| 1299 | EN14110-B | 0.02 | | -0.24 | |
| 1316 | EN14110-B | 0.036 | R(0.01) | 5.69 | |
| 1397 | EN14110 | 0.023 | | 0.87 | |
| 1402 | | ----- | | ----- | |
| 1429 | EN14110- | 0.0208 | | 0.06 | |
| 1443 | EN14110-B | 0.016 | R(0.01) | -1.72 | |
| 1457 | EN14110-B | 0.018 | | -0.98 | |
| 1459 | | ----- | | ----- | |
| 1494 | EN14110-B | 0.02687 | C | 2.31 | first reported: 0.0277 |
| 1510 | | ----- | | ----- | |
| 1528 | EN14110-AMod. | 0.01 | R(0.01) | -3.94 | |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | EN14110Mod. | 0.0194 | | -0.46 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | EN14110-B | 0.020 | C | -0.24 | first reported: 0.014 |
| 1739 | EN14110 | 0.020 | | -0.24 | |
| 1744 | EN14110-B | 0.019 | | -0.61 | |
| 1769 | EN14110-B | 0.0196 | | -0.39 | |
| 1807 | EN14110 | 0.02 | | -0.24 | |
| 1948 | EN14110 | 0.021 | | 0.13 | |

| | | Method A | Method B |
|---|---|---|---|
| normality | not OK | not OK | not OK |
| n | 34 | 11 | 19 |
| outliers | 5 | 0 | 3 |
| mean (n) | 0.0206 | 0.0210 | 0.0215 |
| st.dev. (n) | 0.00250 | 0.00307 | 0.00501 |
| R(calc.) | 0.0070 | 0.0086 | 0.0076 |
| R(EN14110:03) A+B | 0.0076 | 0.0076 | 0.0074 |



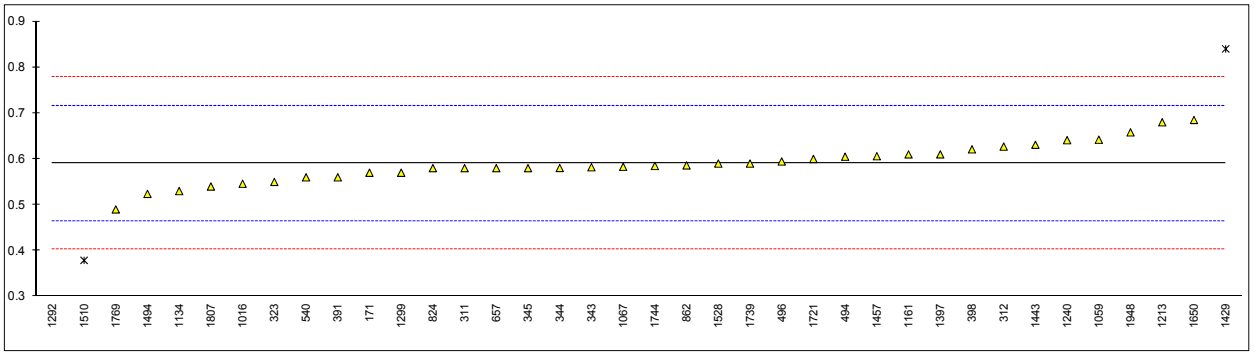



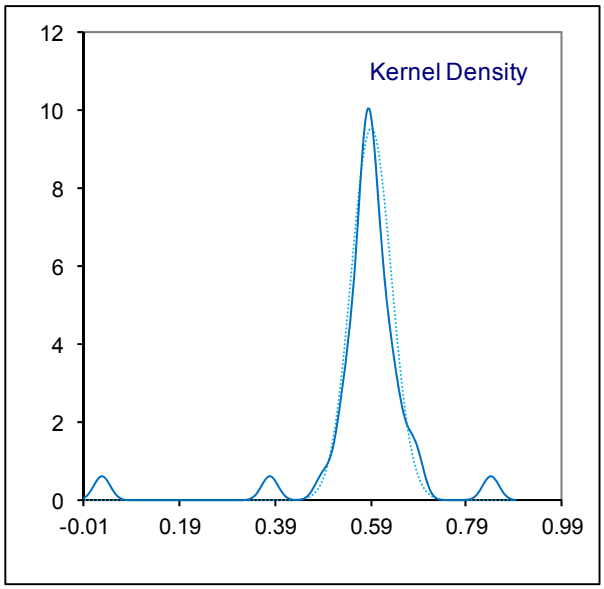
## Determination of mono-Glycerides on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | EN14105 | 0.57 | | -0.33 | |
| 311 | EN14105 | 0.58 | | -0.17 | |
| 312 | EN14105 | 0.627 | | 0.58 | |
| 323 | EN14105 | 0.55 | | -0.65 | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | EN14105 | 0.582 | | -0.14 | |
| 344 | EN14105 | 0.5804 | | -0.16 | |
| 345 | EN14105 | 0.58 | | -0.17 | |
| 360 | | ----- | | ----- | |
| 391 | EN14105 | 0.56 | | -0.49 | |
| 398 | EN14105 | 0.621 | | 0.49 | |
| 445 | | ----- | | ----- | |
| 447 | | ----- | | ----- | |
| 494 | EN14105 | 0.605 | | 0.23 | |
| 496 | EN14105 | 0.5946 | | 0.07 | |
| 529 | | ----- | | ----- | |
| 540 | EN14105 | 0.56 | | -0.49 | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN14105 | 0.58 | | -0.17 | |
| 824 | EN14105 | 0.58 | | -0.17 | |
| 862 | EN14105 | 0.586 | | -0.07 | |
| 863 | | ----- | | ----- | |
| 1016 | EN14105 | 0.546 | | -0.71 | |
| 1033 | | ----- | | ----- | |
| 1059 | EN14105 | 0.642 | | 0.82 | |
| 1067 | EN14105 | 0.583 | | -0.12 | |
| 1095 | | ----- | | ----- | |
| 1131 | | ----- | | ----- | |
| 1134 | EN14105 | 0.530 | | -0.96 | |
| 1161 | EN14105 | 0.61 | | 0.31 | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | D6584 | 0.680 | | 1.43 | |
| 1227 | | ----- | | ----- | |
| 1231 | | ----- | | ----- | |
| 1240 | EN14105 | 0.641 | | 0.81 | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | EN14105 | 0.029 | R(0.01) | -8.95 | |
| 1299 | EN14105 | 0.57 | | -0.33 | |
| 1316 | | ----- | | ----- | |
| 1397 | EN14105 | 0.61 | | 0.31 | |
| 1402 | | ----- | | ----- | |
| 1429 | EN14105 | 0.84 | R(0.01) | 3.98 | |
| 1443 | EN14105 | 0.631 | | 0.65 | |
| 1457 | EN14105 | 0.606 | | 0.25 | |
| 1459 | | ----- | | ----- | |
| 1494 | D6584 | 0.5239 | | -1.06 | |
| 1510 | EN14105 | 0.379 | R(0.01) | -3.37 | |
| 1528 | EN14105 | 0.59 | C | -0.01 | first reported: 0.424 |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | EN14105 | 0.685 | | 1.51 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | EN14105 | 0.60 | | 0.15 | |
| 1739 | EN14105 | 0.59 | | -0.01 | |
| 1744 | D6584 | 0.5848 | | -0.09 | |
| 1769 | D6584 | 0.490 | | -1.60 | |
| 1807 | EN14105 | 0.54 | | -0.80 | |
| 1948 | EN14105 | 0.658 | | 1.08 | |

| | |
|---|---|
| normality | OK |
| n | 35 |
| outliers | 3 |
| mean (n) | 0.5905 |
| st.dev. (n) | 0.04191 |
| R(calc.) | 0.1173 |
| R(EN14105:11) | 0.1756 |

Compare R(D6584:13) = 0.3318
Application limit EN14105:11 = 0.10 %M/M



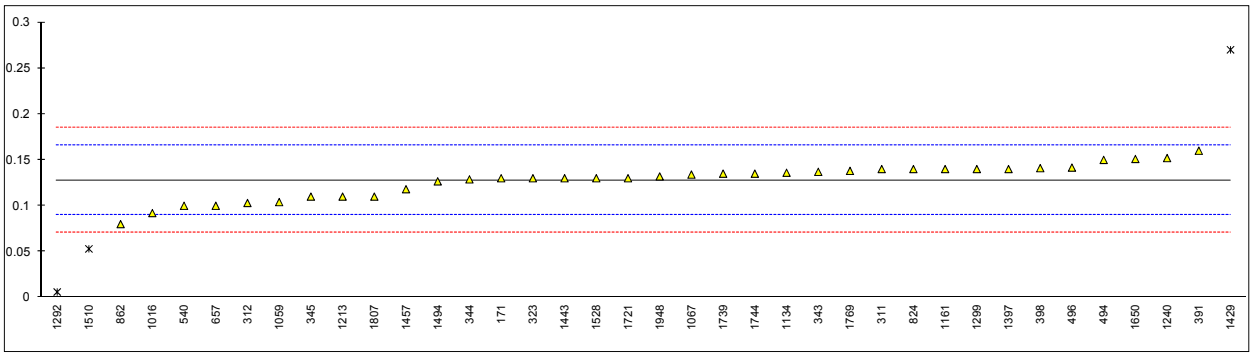



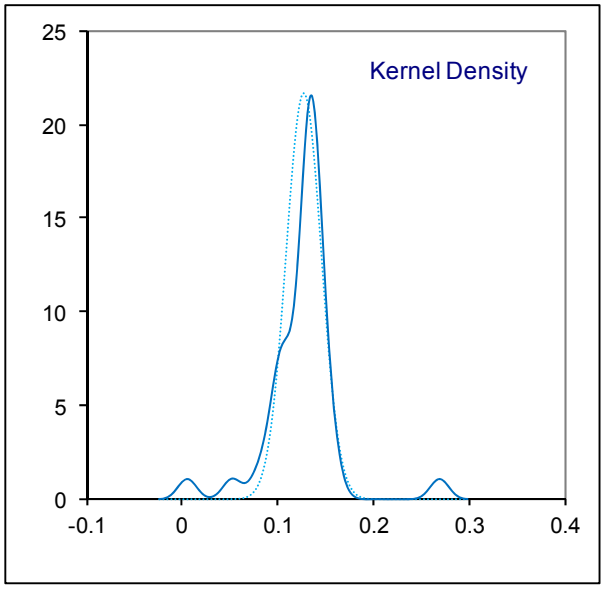
## Determination of di-Glycerides on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | EN14105 | 0.13 | | 0.11 | |
| 311 | EN14105 | 0.14 | | 0.64 | |
| 312 | EN14105 | 0.103 | | -1.31 | |
| 323 | EN14105 | 0.13 | | 0.11 | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | EN14105 | 0.137 | | 0.48 | |
| 344 | EN14105 | 0.1288 | | 0.05 | |
| 345 | EN14105 | 0.11 | | -0.94 | |
| 360 | | ----- | | ----- | |
| 391 | EN14105 | 0.16 | | 1.70 | |
| 398 | EN14105 | 0.141 | | 0.69 | |
| 445 | | ----- | | ----- | |
| 447 | | ----- | | ----- | |
| 494 | EN14105 | 0.15 | | 1.17 | |
| 496 | EN14105 | 0.1416 | | 0.73 | |
| 529 | | ----- | | ----- | |
| 540 | EN14105 | 0.10 | | -1.47 | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN14105 | 0.10 | | -1.47 | |
| 824 | EN14105 | 0.14 | | 0.64 | |
| 862 | EN14105 | 0.080 | | -2.53 | |
| 863 | | ----- | | ----- | |
| 1016 | EN14105 | 0.092 | | -1.89 | |
| 1033 | | ----- | | ----- | |
| 1059 | EN14105 | 0.104 | | -1.26 | |
| 1067 | EN14105 | 0.134 | | 0.32 | |
| 1095 | | ----- | | ----- | |
| 1131 | | ----- | | ----- | |
| 1134 | EN14105 | 0.136 | | 0.43 | |
| 1161 | EN14105 | 0.14 | | 0.64 | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | D6584 | 0.110 | | -0.94 | |
| 1227 | | ----- | | ----- | |
| 1231 | | ----- | | ----- | |
| 1240 | EN14105 | 0.152 | | 1.28 | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | EN14105 | 0.006 | R(0.01) | -6.44 | |
| 1299 | EN14105 | 0.14 | | 0.64 | |
| 1316 | | ----- | | ----- | |
| 1397 | EN14105 | 0.14 | | 0.64 | |
| 1402 | | ----- | | ----- | |
| 1429 | EN14105 | 0.27 | R(0.01) | 7.51 | |
| 1443 | EN14105 | 0.130 | | 0.11 | |
| 1457 | EN14105 | 0.118 | | -0.52 | |
| 1459 | | ----- | | ----- | |
| 1494 | D6584 | 0.1266 | C | -0.07 | first reported: 0.2502 |
| 1510 | EN14105 | 0.053 | R(0.05) | -3.95 | |
| 1528 | EN14105 | 0.13 | C | 0.11 | first reported: 0.075 |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | EN14105 | 0.151 | | 1.22 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | EN14105 | 0.13 | | 0.11 | |
| 1739 | EN14105 | 0.135 | | 0.38 | |
| 1744 | D6584 | 0.1350 | | 0.38 | |
| 1769 | D6584 | 0.138 | | 0.54 | |
| 1807 | EN14105 | 0.11 | C | -0.94 | first reported: 0.07 |
| 1948 | EN14105 | 0.132 | | 0.22 | |

| | |
|---|---|
| normality | OK |
| n | 35 |
| outliers | 3 |
| mean (n) | 0.1279 |
| st.dev. (n) | 0.01843 |
| R(calc.) | 0.0516 |
| R(EN14105:11) | 0.0530 |

Compare R(D6584:13) = 0.1158
Application limit EN14105:11 = 0.10 %M/M



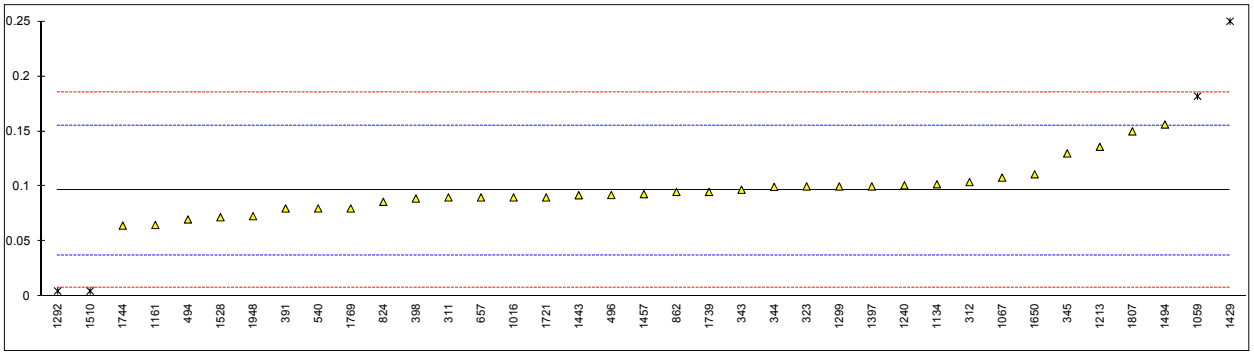



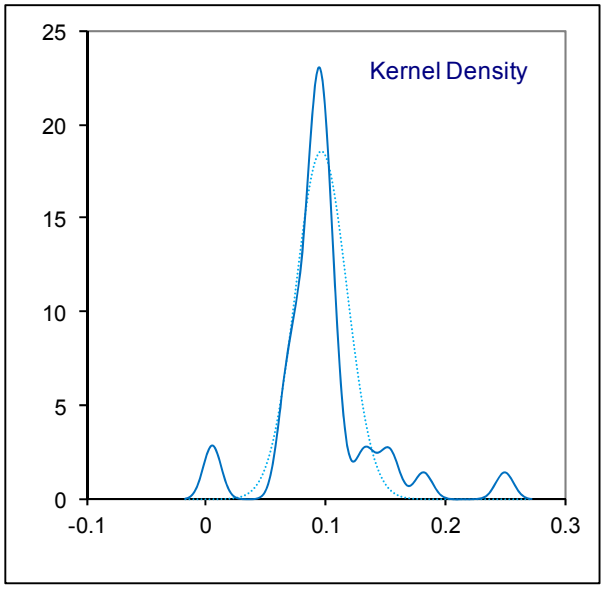
Determination of tri-Glyceriden on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | EN14105 | <0.10 | | ----- | |
| 311 | EN14105 | 0.09 | | -0.22 | |
| 312 | EN14105 | 0.104 | | 0.26 | |
| 323 | EN14105 | 0.10 | | 0.12 | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | EN14105 | 0.097 | | 0.02 | |
| 344 | EN14105 | 0.0996 | | 0.11 | |
| 345 | EN14105 | 0.13 | | 1.14 | |
| 360 | | ----- | | ----- | |
| 391 | EN14105 | 0.08 | | -0.56 | |
| 398 | EN14105 | 0.089 | | -0.25 | |
| 445 | | ----- | | ----- | |
| 447 | | ----- | | ----- | |
| 494 | EN14105 | 0.07 | | -0.89 | |
| 496 | EN14105 | 0.0922 | | -0.14 | |
| 529 | | ----- | | ----- | |
| 540 | EN14105 | 0.08 | | -0.56 | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN14105 | 0.09 | | -0.22 | |
| 824 | EN14105 | 0.086 | | -0.35 | |
| 862 | EN14105 | 0.095 | | -0.05 | |
| 863 | | ----- | | ----- | |
| 1016 | EN14105 | 0.09 | C | -0.22 | first reported: 0.013 |
| 1033 | | ----- | | ----- | |
| 1059 | EN14105 | 0.182 | R(0.05) | 2.90 | |
| 1067 | EN14105 | 0.108 | | 0.39 | |
| 1095 | | ----- | | ----- | |
| 1131 | | ----- | | ----- | |
| 1134 | EN14105 | 0.102 | | 0.19 | |
| 1161 | EN14105 | 0.065 | | -1.06 | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | D6584 | 0.136 | | 1.34 | |
| 1227 | | ----- | | ----- | |
| 1231 | | ----- | | ----- | |
| 1240 | EN14105 | 0.101 | | 0.16 | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | EN14105 | 0.005 | R(0.05) | -3.10 | |
| 1299 | EN14105 | 0.10 | | 0.12 | |
| 1316 | | ----- | | ----- | |
| 1397 | EN14105 | 0.10 | | 0.12 | |
| 1402 | | ----- | | ----- | |
| 1429 | EN14105 | 0.25 | R(0.01) | 5.20 | |
| 1443 | EN14105 | 0.092 | | -0.15 | |
| 1457 | EN14105 | 0.093 | | -0.12 | |
| 1459 | | ----- | | ----- | |
| 1494 | D6584 | 0.1562 | | 2.03 | |
| 1510 | EN14105 | 0.005 | R(0.05) | -3.10 | |
| 1528 | EN14105 | 0.072 | | -0.83 | |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | EN14105 | 0.111 | | 0.49 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | EN14105 | 0.09 | | -0.22 | |
| 1739 | EN14105 | 0.095 | | -0.05 | |
| 1744 | D6584 | 0.0644 | | -1.08 | |
| 1769 | D6584 | 0.080 | | -0.56 | |
| 1807 | EN14105 | 0.15 | | 1.82 | |
| 1948 | EN14105 | 0.073 | C | -0.79 | first reported: 0.016 |

| | |
|---|---|
| normality | not OK |
| n | 33 |
| outliers | 4 |
| mean (n) | 0.0964 |
| st.dev. (n) | 0.02142 |
| R(calc.) | 0.0600 |
| R(EN14105:11) | 0.0827 |

Compare R(6584:13) = 0.2165
Application limit EN14105:11 = 0.10 %M/M



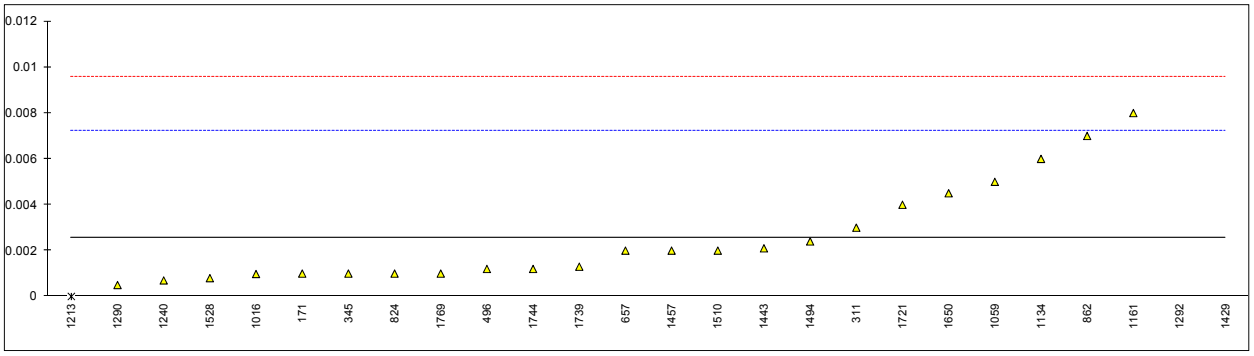



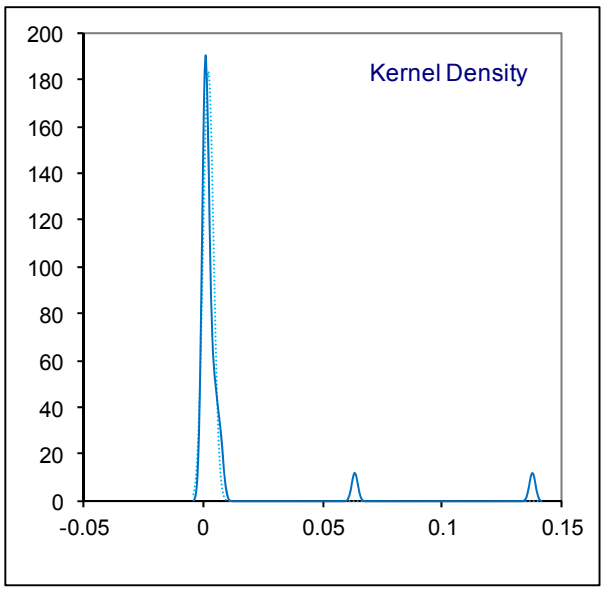
## Determination of Free Glycerol on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | EN14105 | 0.001 | | -0.66 | |
| 311 | EN14105 | 0.003 | | 0.19 | |
| 312 | EN14105 | <0.001 | | ----- | |
| 323 | EN14105 | <0.01 | | ----- | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | EN14105 | <0.005 | | ----- | |
| 344 | EN14105 | <0.05 | | ----- | |
| 345 | EN14105 | 0.001 | | -0.66 | |
| 360 | | ----- | | ----- | |
| 391 | EN14105 | <0.01 | | ----- | |
| 398 | EN14105 | <0.01 | | ----- | |
| 445 | | ----- | | ----- | |
| 447 | | ----- | | ----- | |
| 494 | EN14105 | <0.001 | | ----- | |
| 496 | EN14105 | 0.0012 | | -0.58 | |
| 529 | | ----- | | ----- | |
| 540 | EN14105 | <0.001 | | ----- | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN14105 | 0.002 | | -0.24 | |
| 824 | EN14105 | 0.001 | | -0.66 | |
| 862 | EN14105 | 0.007 | | 1.90 | |
| 863 | | ----- | | ----- | |
| 1016 | EN14105 | 0.00098 | | -0.67 | |
| 1033 | | ----- | | ----- | |
| 1059 | EN14105 | 0.005 | | 1.04 | |
| 1067 | EN14105 | <0.010 | | ----- | |
| 1095 | | ----- | | ----- | |
| 1131 | | ----- | | ----- | |
| 1134 | EN14105 | 0.006 | | 1.47 | |
| 1161 | EN14105 | 0.008 | | 2.32 | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | D6584 | 0 | ex | -1.09 | result excluded, zero is not a real value |
| 1227 | | ----- | | ----- | |
| 1231 | | ----- | | ----- | |
| 1240 | EN14105 | 0.0007 | | -0.79 | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | in house | 0.0005 | | -0.87 | |
| 1292 | EN14105 | 0.0637 | R(0.01) | 26.07 | |
| 1299 | EN14105 | <0.01 | C | ----- | first reported: 0.01 |
| 1316 | | ----- | | ----- | |
| 1397 | EN14105 | <0.005 | | ----- | |
| 1402 | | ----- | | ----- | |
| 1429 | EN14105 | 0.138 | R(0.01) | 57.75 | |
| 1443 | EN14105 | 0.0021 | | -0.19 | |
| 1457 | EN14105 | 0.002 | | -0.24 | |
| 1459 | | ----- | | ----- | |
| 1494 | D6584 | 0.0024 | | -0.06 | |
| 1510 | EN14105 | 0.002 | | -0.24 | |
| 1528 | EN14105 | 0.0008 | | -0.75 | |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | EN14105 | 0.0045 | | 0.83 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | EN14105 | 0.004 | | 0.62 | |
| 1739 | EN14105 | 0.0013 | | -0.53 | |
| 1744 | D6584 | 0.0012 | | -0.58 | |
| 1769 | D6584 | 0.001 | | -0.66 | |
| 1807 | EN14105 | <0.001 | | ----- | |
| 1948 | EN14105 | <0.001 | | ----- | |

| | |
|---|---|
| normality | suspect |
| n | 23 |
| outliers | 2 +1 excl |
| mean (n) | 0.00255 |
| st.dev. (n) | 0.002160 |
| R(calc.) | 0.00605 |
| R(EN14105:11) | 0.00657 |

Compare R(6584:13) = 0.0050
Application limit EN14105:11 = 0.001 %M/M



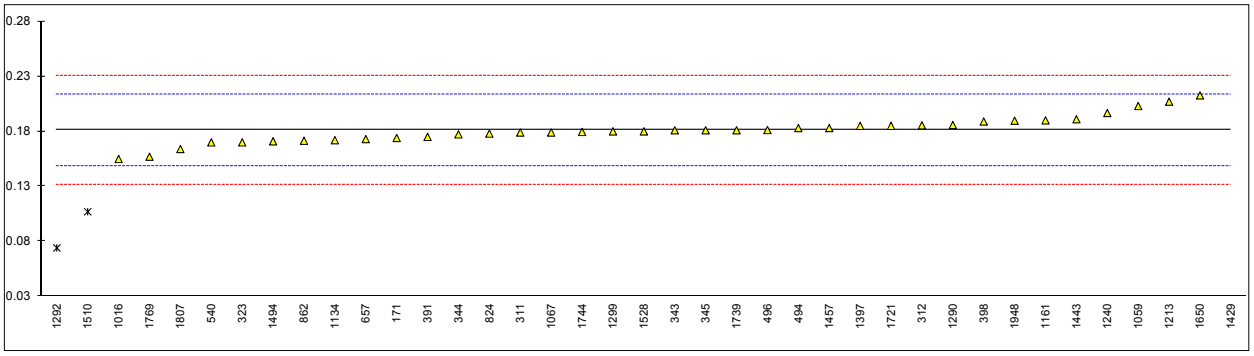



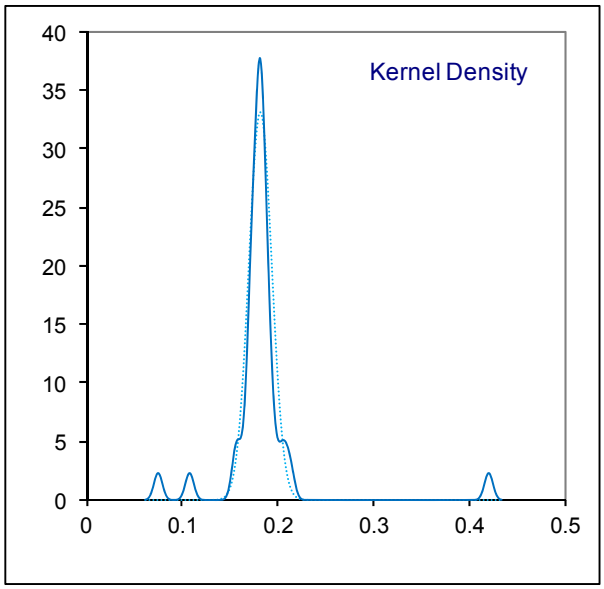
## Determination of Total Glycerol on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | EN14105 | 0.174 | | -0.43 | |
| 311 | EN14105 | 0.179 | | -0.12 | |
| 312 | EN14105 | 0.1855 | | 0.27 | |
| 323 | EN14105 | 0.17 | | -0.67 | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | EN14105 | 0.181 | | 0.00 | |
| 344 | EN14105 | 0.1773 | | -0.23 | |
| 345 | EN14105 | 0.181 | | 0.00 | |
| 360 | | ----- | | ----- | |
| 391 | EN14105 | 0.175 | | -0.37 | |
| 398 | EN14105 | 0.189 | | 0.49 | |
| 445 | | ----- | | ----- | |
| 447 | | ----- | | ----- | |
| 494 | EN14105 | 0.183 | | 0.12 | |
| 496 | EN14105 | 0.1812 | | 0.01 | |
| 529 | | ----- | | ----- | |
| 540 | EN14105 | 0.17 | | -0.67 | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN14105 | 0.173 | | -0.49 | |
| 824 | EN14105 | 0.178 | | -0.18 | |
| 862 | EN14105 | 0.1715 | | -0.58 | |
| 863 | | ----- | | ----- | |
| 1016 | EN14105 | 0.1549 | | -1.59 | |
| 1033 | | ----- | | ----- | |
| 1059 | EN14105 | 0.203 | | 1.34 | |
| 1067 | EN14105 | 0.179 | | -0.12 | |
| 1095 | | ----- | | ----- | |
| 1131 | | ----- | | ----- | |
| 1134 | EN14105 | 0.172 | | -0.55 | |
| 1161 | EN14105 | 0.19 | | 0.55 | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | D6584 | 0.207 | | 1.59 | |
| 1227 | | ----- | | ----- | |
| 1231 | | ----- | | ----- | |
| 1240 | EN14105 | 0.1967 | | 0.96 | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | in house | 0.1858 | | 0.29 | |
| 1292 | EN14105 | 0.0741 | R(0.01) | -6.52 | |
| 1299 | EN14105 | 0.18 | | -0.06 | |
| 1316 | | ----- | | ----- | |
| 1397 | EN14105 | 0.185 | | 0.24 | |
| 1402 | | ----- | | ----- | |
| 1429 | EN14105 | 0.42 | R(0.01) | 14.57 | |
| 1443 | EN14105 | 0.1910 | | 0.61 | |
| 1457 | EN14105 | 0.183 | | 0.12 | |
| 1459 | | ----- | | ----- | |
| 1494 | D6584 | 0.1710 | C | -0.61 | first reported: 0.1912 |
| 1510 | EN14105 | 0.107 | R(0.01) | -4.51 | |
| 1528 | EN14105 | 0.18 | C | -0.06 | first reported: 0.127 |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | EN14105 | 0.2127 | | 1.93 | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | EN14105 | 0.185 | | 0.24 | |
| 1739 | EN14105 | 0.181 | | 0.00 | |
| 1744 | D6584 | 0.1795 | | -0.09 | |
| 1769 | D6584 | 0.157 | | -1.46 | |
| 1807 | EN14105 | 0.164 | | -1.04 | |
| 1948 | EN14105 | 0.1897 | | 0.53 | |

| | |
|---|---|
| normality | suspect |
| n | 36 |
| outliers | 3 |
| mean (n) | 0.1810 |
| st.dev. (n) | 0.01202 |
| R(calc.) | 0.0337 |
| R(EN14105:11) | 0.0459 |

Compare R(6584:13) = 0.0833
Application limit EN14105:11 = 0.10 %M/M



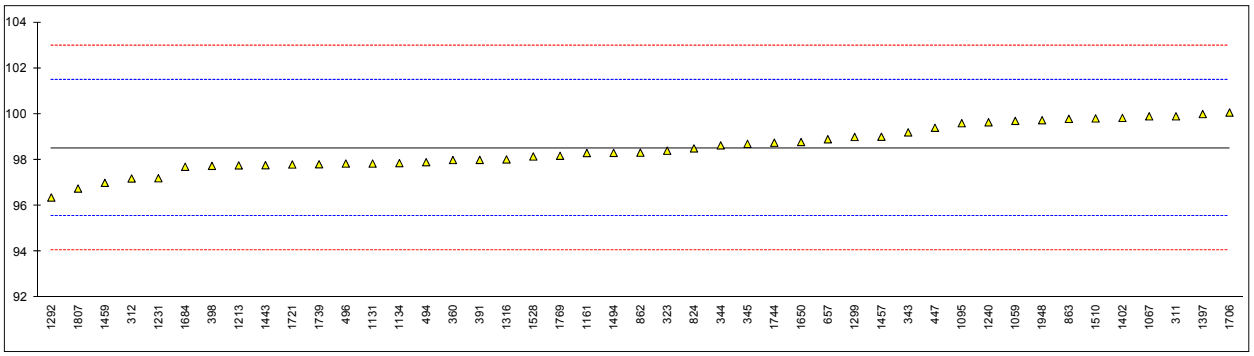



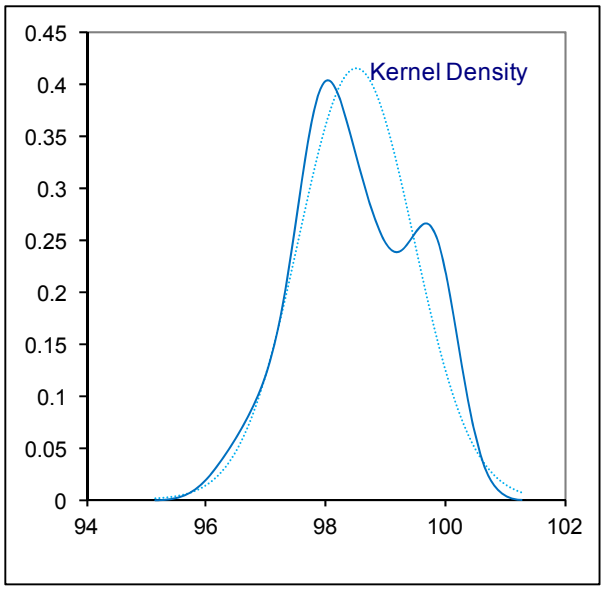
## Determination of Total Ester content on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | | ----- | | ----- | |
| 311 | EN14103:11 | 99.9 | | 0.93 | |
| 312 | EN14103:11 | 97.19 | | -0.89 | |
| 323 | EN14103:11 | 98.4 | | -0.08 | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | EN14103:11 | 99.2 | | 0.46 | |
| 344 | EN14103:11 | 98.634 | | 0.08 | |
| 345 | EN14103:03 | 98.7 | | 0.13 | |
| 360 | EN14103:11 | 97.994 | | -0.35 | |
| 391 | EN14103:11 | 98.0 | | -0.34 | |
| 398 | EN14103:11 | 97.74 | | -0.52 | |
| 445 | | ----- | | ----- | |
| 447 | EN14103 | 99.4 | | 0.60 | |
| 494 | EN14103:11 | 97.9 | | -0.41 | |
| 496 | EN14103:11 | 97.84 | | -0.45 | |
| 529 | | ----- | | ----- | |
| 540 | | ----- | | ----- | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN14103:11 | 98.9 | | 0.26 | |
| 824 | EN14103:11 | 98.5 | | -0.01 | |
| 862 | EN14103:11 | 98.317 | | -0.13 | |
| 863 | EN14103:11 | 99.79 | | 0.86 | |
| 1016 | | ----- | | ----- | |
| 1033 | | ----- | | ----- | |
| 1059 | EN14103:11 | 99.7 | | 0.80 | |
| 1067 | EN14103:11 | 99.9 | | 0.93 | |
| 1095 | EN14103:03 | 99.6 | | 0.73 | |
| 1131 | EN14103:11 | 97.84 | | -0.45 | |
| 1134 | EN14103:11 | 97.86 | | -0.44 | |
| 1161 | EN14103:11 | 98.3 | | -0.14 | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | EN14103:03 | 97.76 | | -0.51 | |
| 1227 | | ----- | | ----- | |
| 1231 | EN14103:11 | 97.2 | | -0.88 | |
| 1240 | EN14103:11 | 99.64 | | 0.76 | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | EN14103 | 96.36 | | -1.45 | |
| 1299 | EN14103:11 | 99.0 | | 0.33 | |
| 1316 | EN14103:11 | 98.02 | | -0.33 | |
| 1397 | EN14103 | 100.0 | | 1.00 | |
| 1402 | EN14103:11 | 99.830 | | 0.89 | |
| 1429 | EN14103:11 | >99 | | ----- | |
| 1443 | EN14103:11 | 97.77 | | -0.50 | |
| 1457 | EN14103:11 | 99.01 | | 0.34 | |
| 1459 | EN14103:11 | 97.0 | | -1.02 | |
| 1494 | EN14103:11 | 98.3065 | | -0.14 | |
| 1510 | EN14103:11 | 99.81 | | 0.87 | |
| 1528 | EN14103:11 | 98.15 | | -0.24 | |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | EN14103:11 | 98.78 | | 0.18 | |
| 1654 | | ----- | | ----- | |
| 1684 | EN14103 | 97.7 | | -0.55 | |
| 1706 | EN14103:09 | 100.066 | | 1.05 | |
| 1721 | EN14103:11 | 97.8 | | -0.48 | |
| 1739 | EN14103 | 97.81 | | -0.47 | |
| 1744 | EN14103:03 | 98.75 | | 0.16 | |
| 1769 | EN14103:03 | 98.18 | | -0.22 | |
| 1807 | EN14103:11 | 96.75 | | -1.19 | |
| 1948 | EN14103:11 | 99.73 | | 0.82 | |

| | |
|---|---|
| normality | OK |
| n | 45 |
| outliers | 0 |
| mean (n) | 98.512 |
| st.dev. (n) | 0.9616 |
| R(calc.) | 2.692 |
| R(EN14103:11) | 4.160 |

Compare R(EN14103:03) = 3.1



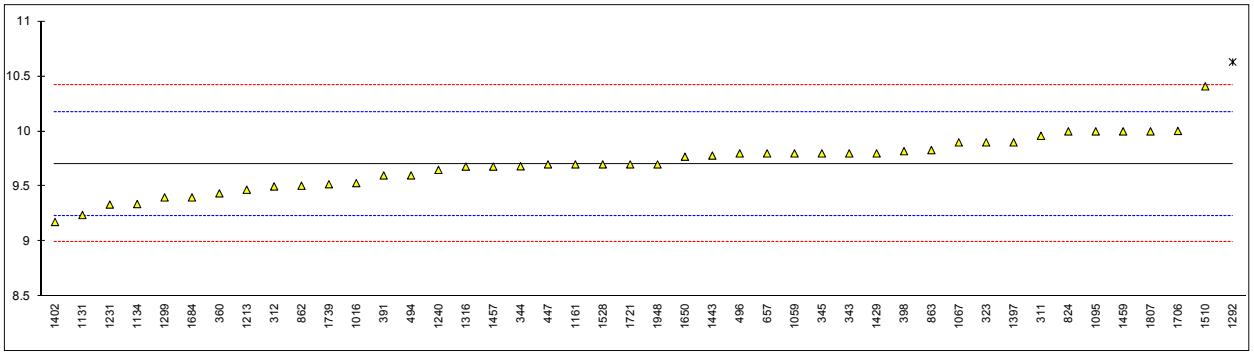



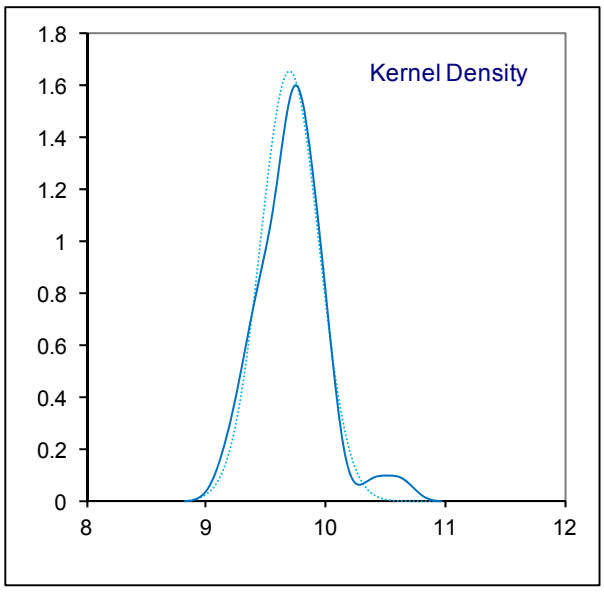
Determination of Linolenic Acid Methyl Ester content on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | | ----- | | ----- | |
| 311 | EN14103:11 | 9.96 | | 1.08 | |
| 312 | EN14103:11 | 9.5 | | -0.87 | |
| 323 | EN14103:11 | 9.9 | | 0.82 | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | EN14103:11 | 9.8 | | 0.40 | |
| 344 | EN14103:11 | 9.683 | | -0.09 | |
| 345 | EN14103:03 | 9.8 | | 0.40 | |
| 360 | EN14103:11 | 9.437 | | -1.13 | |
| 391 | EN14103:11 | 9.6 | | -0.44 | |
| 398 | EN14103:11 | 9.82 | | 0.48 | |
| 445 | | ----- | | ----- | |
| 447 | EN14103 | 9.7 | | -0.02 | |
| 494 | EN14103:11 | 9.6 | | -0.44 | |
| 496 | EN14103:11 | 9.80 | | 0.40 | |
| 529 | | ----- | | ----- | |
| 540 | | ----- | | ----- | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN14103:11 | 9.8 | | 0.40 | |
| 824 | EN14103:11 | 10.0 | | 1.24 | |
| 862 | EN14103:11 | 9.506 | | -0.84 | |
| 863 | EN14103:11 | 9.83 | | 0.53 | |
| 1016 | EN14103:11 | 9.53 | | -0.74 | |
| 1033 | | ----- | | ----- | |
| 1059 | EN14103:11 | 9.8 | | 0.40 | |
| 1067 | EN14103:11 | 9.9 | | 0.82 | |
| 1095 | EN14103:03 | 10.0 | | 1.24 | |
| 1131 | EN14103:11 | 9.24 | | -1.96 | |
| 1134 | EN14103:11 | 9.34 | | -1.54 | |
| 1161 | EN14103:11 | 9.7 | | -0.02 | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | EN14103:03 | 9.47 | | -0.99 | |
| 1227 | | ----- | | ----- | |
| 1231 | EN14103:11 | 9.335 | | -1.56 | |
| 1240 | EN14103:11 | 9.65 | | -0.23 | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | EN14103 | 10.63 | G(0.05) | 3.90 | |
| 1299 | EN14103:11 | 9.4 | | -1.29 | |
| 1316 | EN14103:11 | 9.68 | | -0.11 | |
| 1397 | EN14103 | 9.9 | | 0.82 | |
| 1402 | EN14103:11 | 9.177 | | -2.23 | |
| 1429 | EN14103:11 | 9.8 | | 0.40 | |
| 1443 | EN14103:11 | 9.78 | | 0.32 | |
| 1457 | EN14103:11 | 9.68 | | -0.11 | |
| 1459 | EN14103:11 | 10.0 | | 1.24 | |
| 1494 | | ----- | | ----- | |
| 1510 | EN14103:11 | 10.409 | | 2.97 | |
| 1528 | EN14103:11 | 9.7 | | -0.02 | |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | EN14103:11 | 9.77 | | 0.27 | |
| 1654 | | ----- | | ----- | |
| 1684 | EN14103 | 9.40 | | -1.29 | |
| 1706 | EN14103:09 | 10.004 | | 1.26 | |
| 1721 | EN14103:11 | 9.7 | | -0.02 | |
| 1739 | EN14103 | 9.52 | | -0.78 | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | EN14103:11 | 10.0 | | 1.24 | |
| 1948 | EN14103:11 | 9.70 | | -0.02 | |

| | |
|---|---|
| normality | OK |
| n | 43 |
| outliers | 1 |
| mean (n) | 9.705 |
| st.dev. (n) | 0.2419 |
| R(calc.) | 0.677 |
| R(EN14103:11) | 0.664 |

Compare R(EN14103:03) = 3.038



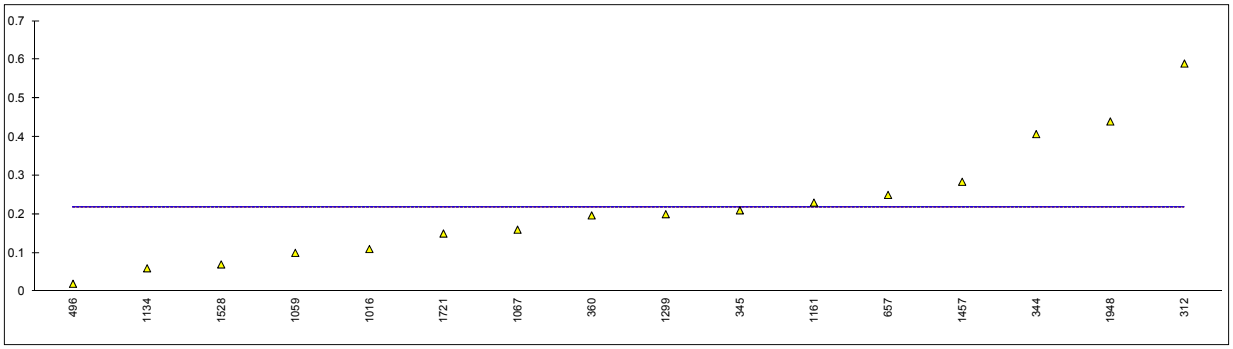



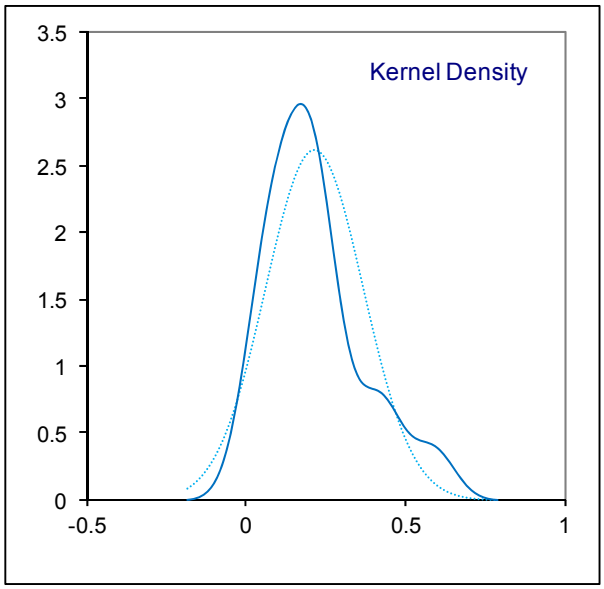
Determination of Polyunsaturated Methyl Esters content on sample #14045; results in %M/M

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | | ----- | | ----- | |
| 311 | EN15779 | <0.7 | | ----- | |
| 312 | EN15779 | 0.59 | | ----- | false positive result? |
| 323 | EN15779 | <0.6 | | ----- | |
| 334 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 343 | EN15779 | <0.30 | | ----- | |
| 344 | EN15779 | 0.4073 | | ----- | |
| 345 | EN15779 | 0.21 | | ----- | |
| 360 | EN15779 | 0.197 | | ----- | |
| 391 | | ----- | | ----- | |
| 398 | EN15779 | <0.1 | | ----- | |
| 445 | | ----- | | ----- | |
| 447 | | ----- | | ----- | |
| 494 | | ----- | | ----- | |
| 496 | EN15779 | 0.02 | | ----- | |
| 529 | | ----- | | ----- | |
| 540 | | ----- | | ----- | |
| 554 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN15779 | 0.25 | | ----- | |
| 824 | EN15779 | <0.6 | | ----- | |
| 862 | | ----- | | ----- | |
| 863 | | ----- | | ----- | |
| 1016 | EN15779 | 0.11 | | ----- | |
| 1033 | | ----- | | ----- | |
| 1059 | EN15779 | 0.1 | | ----- | |
| 1067 | EN15779 | 0.16 | | ----- | |
| 1095 | | ----- | | ----- | |
| 1131 | | ----- | | ----- | |
| 1134 | EN15779 | 0.06 | | ----- | |
| 1161 | EN15779 | 0.23 | | ----- | |
| 1199 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1213 | | ----- | | ----- | |
| 1227 | | ----- | | ----- | |
| 1231 | | ----- | | ----- | |
| 1240 | | ----- | | ----- | |
| 1268 | | ----- | | ----- | |
| 1286 | | ----- | | ----- | |
| 1290 | | ----- | | ----- | |
| 1292 | | ----- | | ----- | |
| 1299 | EN15779 | 0.2 | | ----- | |
| 1316 | | ----- | | ----- | |
| 1397 | | ----- | | ----- | |
| 1402 | | ----- | | ----- | |
| 1429 | | ----- | | ----- | |
| 1443 | | ----- | | ----- | |
| 1457 | EN15779 | 0.284 | | ----- | |
| 1459 | | ----- | | ----- | |
| 1494 | | ----- | | ----- | |
| 1510 | | ----- | | ----- | |
| 1528 | in house | 0.07 | | ----- | |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |
| 1588 | | ----- | | ----- | |
| 1634 | | ----- | | ----- | |
| 1635 | | ----- | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | | ----- | | ----- | |
| 1654 | | ----- | | ----- | |
| 1684 | | ----- | | ----- | |
| 1706 | | ----- | | ----- | |
| 1721 | EN15779 | 0.15 | | ----- | |
| 1739 | EN15779 | <0.6 | | ----- | |
| 1744 | | ----- | | ----- | |
| 1769 | | ----- | | ----- | |
| 1807 | | ----- | | ----- | |
| 1948 | EN15779 | 0.44 | | ----- | |

| | |
|---|---|
| normality | not OK |
| n | 16 |
| outliers | 0 |
| mean (n) | 0.217 |
| st.dev. (n) | 0.1526 |
| R(calc.) | 0.427 |
| R(EN15779:09) | (0.270) |

Application range 0.3 -3.0 %M/M

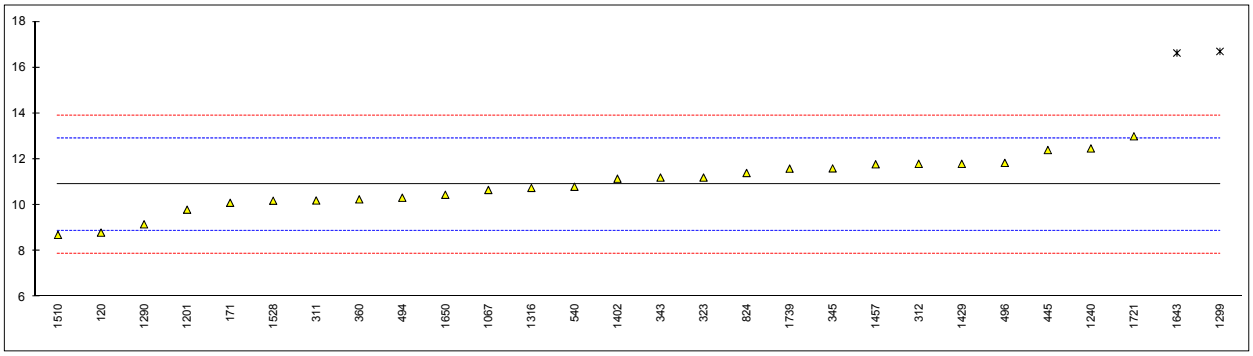


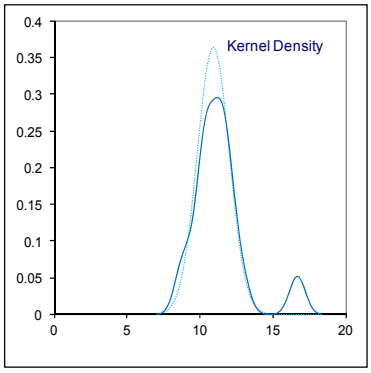



## Determination of sum of Calcium and Magnesium on sample #14046; results in mg/kg

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | EN14538 | 8.8 | C | -2.09 | first reported: 2.5 |
| 171 | EN14538 | 10.1 | | -0.80 | |
| 311 | EN14538 | 10.2 | | -0.70 | |
| 312 | EN14538 | 11.8 | | 0.90 | |
| 323 | EN14538 | 11.2 | | 0.30 | |
| 343 | EN14538 | 11.2 | | 0.30 | |
| 345 | EN14538 | 11.6 | | 0.70 | |
| 360 | EN14538 | 10.25 | | -0.65 | |
| 391 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 445 | EN14538 | 12.40 | | 1.49 | |
| 447 | | ----- | | ----- | |
| 494 | EN14538 | 10.32 | | -0.58 | |
| 496 | EN14538 | 11.84 | | 0.94 | |
| 540 | EN14538 | 10.8 | | -0.10 | |
| 603 | | ----- | | ----- | |
| 657 | | ----- | | ----- | |
| 824 | EN14538 | 11.4 | | 0.50 | |
| 1067 | EN14538 | 10.66 | | -0.24 | |
| 1134 | | ----- | | ----- | |
| 1161 | EN14538 | <0.5 | | <-10.36 | false negative result? |
| 1201 | D5185mod | 9.8 | | -1.10 | |
| 1240 | EN14538 | 12.47 | | 1.56 | |
| 1268 | | ----- | | ----- | |
| 1290 | EN14538 | 9.162 | | -1.73 | |
| 1299 | EN14538 | 16.7 | R(0.01) | 5.78 | |
| 1316 | D7111 | 10.75 | | -0.15 | |
| 1402 | EN14538 | 11.15 | | 0.25 | |
| 1429 | EN14538 | 11.8 | | 0.90 | |
| 1457 | EN14538 | 11.78 | | 0.88 | |
| 1510 | EN14538 | 8.7 | | -2.19 | |
| 1528 | D4628 | 10.19 | | -0.71 | |
| 1643 | D5185 | 16.631 | R(0.01) | 5.71 | |
| 1650 | EN14538 | 10.45 | | -0.45 | |
| 1721 | EN14538 | 13.0 | | 2.09 | |
| 1739 | EN14538 | 11.59 | | 0.69 | |

| | | | |
|---|---|---|---|
| normality | OK | Spike | |
| n | 26 | | |
| outliers | 2 | | |
| mean (n) | 10.90 | 10.59 | recovery: <103% |
| st.dev. (n) | 1.096 | | |
| R(calc.) | 3.07 | | |
| R(EN14538:06) | 2.81 | | application range: 1- 10 mg/kg |

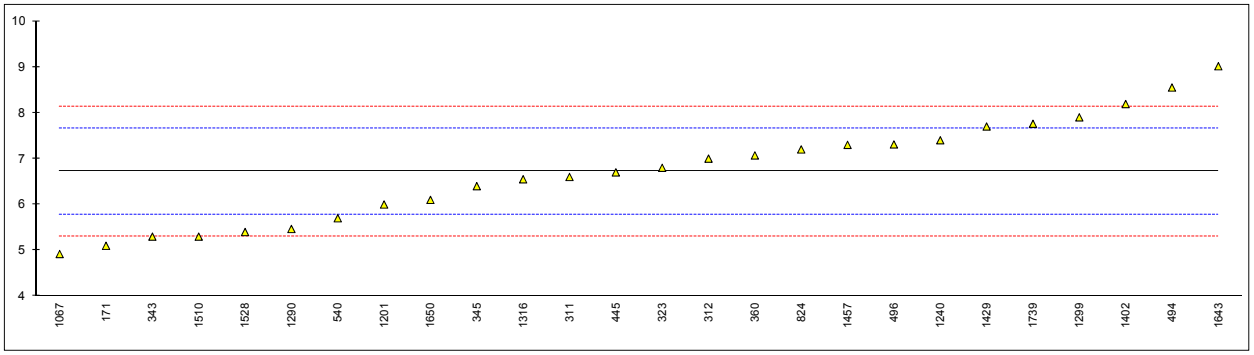


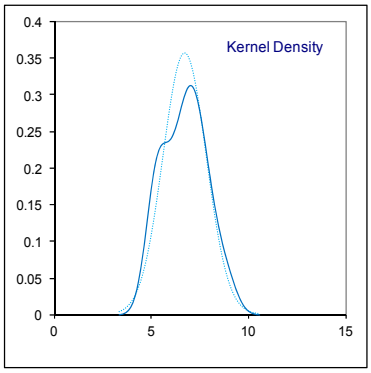



## Determination of Phosphorus on sample #14046; results in mg/kg

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 171 | EN14107 | 5.1 | | -3.45 | |
| 311 | EN14107 | 6.6 | | -0.26 | |
| 312 | EN14107 | 7.0 | | 0.60 | |
| 323 | EN14107 | 6.8 | | 0.17 | |
| 343 | EN14107 | 5.3 | | -3.02 | |
| 345 | EN14107 | 6.4 | | -0.68 | |
| 360 | EN14107 | 7.07 | | 0.74 | |
| 391 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 445 | EN14107 | 6.70 | | -0.04 | |
| 447 | | ----- | | ----- | |
| 494 | EN14107 | 8.55 | | 3.89 | |
| 496 | EN14107 | 7.31 | | 1.26 | |
| 540 | EN14107 | 5.7 | | -2.17 | |
| 603 | | ----- | | ----- | |
| 657 | | ----- | | ----- | |
| 824 | EN14107 | 7.2 | | 1.02 | |
| 1067 | EN14107 | 4.92 | | -3.83 | |
| 1134 | | ----- | | ----- | |
| 1161 | EN14107 | <1 | | <-12.18 | false negative result? |
| 1201 | D5185mod | 6.0 | | -1.53 | |
| 1240 | EN16294 | 7.40 | | 1.45 | |
| 1268 | | ----- | | ----- | |
| 1290 | EN14107 | 5.466 | | -2.67 | |
| 1299 | EN14107 | 7.9 | | 2.51 | |
| 1316 | D7111 | 6.55 | | -0.36 | |
| 1402 | EN14107 | 8.190 | | 3.13 | |
| 1429 | EN14107 | 7.7 | | 2.09 | |
| 1457 | EN14107 | 7.30 | | 1.23 | |
| 1510 | EN14107 | 5.3 | | -3.02 | |
| 1528 | D3231 | 5.4 | C | -2.81 | first reported: 1.81 |
| 1643 | D5185 | 9.014 | | 4.88 | |
| 1650 | EN14107 | 6.10 | | -1.32 | |
| 1721 | EN14107 | <1 | | <-12.18 | false negative result? |
| 1739 | EN14107 | 7.76 | | 2.21 | |

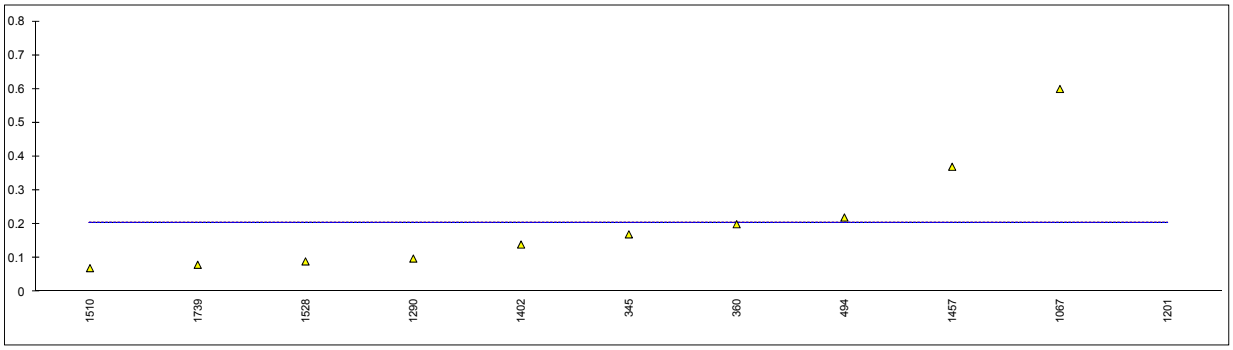
| | | | |
|---|---|---|---|
| normality | OK | Spike | |
| n | 26 | | |
| outliers | 0 | | |
| mean (n) | 6.72 | 6.01 | recovery: <111.8% |
| st.dev. (n) | 1.115 | | |
| R(calc.) | 3.12 | | |
| R(EN14107:03) | 1.32 | | application range: EN14107:03 = 4 -20 mg/kg |

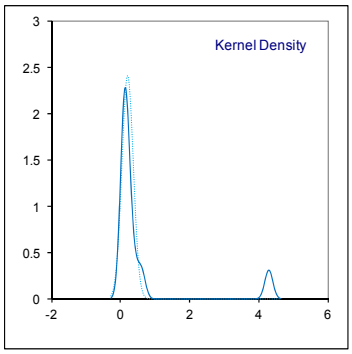


## Determination of Potassium on sample #14046; results in mg/kg

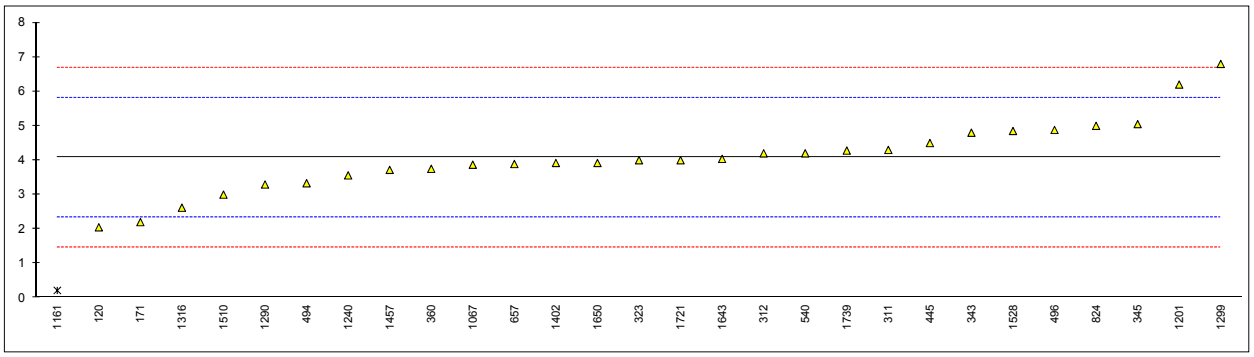
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | EN14109 | <0.10 | C | ----- | first reported: 0.83 |
| 171 | EN14109 | <0.1 | | ----- | |
| 311 | EN14109 | <1 | | ----- | |
| 312 | EN14109 | <0.5 | | ----- | |
| 323 | EN14538 | <1.0 | | ----- | |
| 343 | EN14109 | <0.5 | | ----- | |
| 345 | EN14538 | 0.17 | | ----- | |
| 360 | EN14538 | 0.2 | | ----- | |
| 391 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 445 | EN14538 | <1 | | ----- | |
| 447 | | ----- | | ----- | |
| 494 | EN14538 | 0.22 | | ----- | |
| 496 | EN14538 | <1.0 | | ----- | |
| 540 | EN14538 | <0.5 | | ----- | |
| 603 | | ----- | | ----- | |
| 657 | EN14109 | <0.5 | | ----- | |
| 824 | EN14109 | <1 | | ----- | |
| 1067 | EN14538 | 0.6 | | ----- | |
| 1134 | | ----- | | ----- | |
| 1161 | EN14109 | <1 | | ----- | |
| 1201 | D5185mod | 4.3 | G(0.01) | ----- | false positive result? |
| 1240 | EN14538 | <1.0 | | ----- | |
| 1268 | | ----- | | ----- | |
| 1290 | EN14538 | 0.0984 | | ----- | |
| 1299 | EN14109 | <1.0 | C | ----- | first reported: 1.0 |
| 1316 | D7111 | <0.50 | | ----- | |
| 1402 | EN14538 | 0.14 | | ----- | |
| 1429 | EN14107 | <1.0 | | ----- | |
| 1457 | EN14538 | 0.37 | | ----- | |
| 1510 | EN14538 | 0.07 | | ----- | |
| 1528 | EN14109 | 0.09 | | ----- | |
| 1643 | | ----- | | ----- | |
| 1650 | EN14109 | <0.5 | | ----- | |
| 1721 | EN14109 | <1 | | ----- | |
| 1739 | EN14538 | 0.08 | | ----- | |
| | normality | not OK | | | |
| | n | 10 | | | |
| | outliers | 1 | | | |
| | mean (n) | 0.20 | | | |
| | st.dev. (n) | 0.166 | | | |
| | R(calc.) | 0.46 | | | |
| | R(EN14109:03) | (1.03) | | | application range: >0.5 mg/kg |

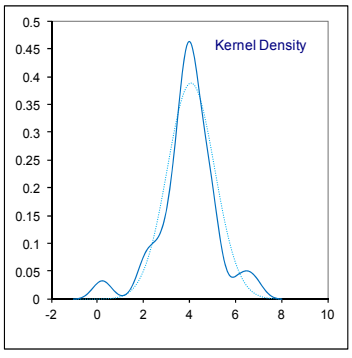


## Determination of Sodium on sample #14046; results in mg/kg

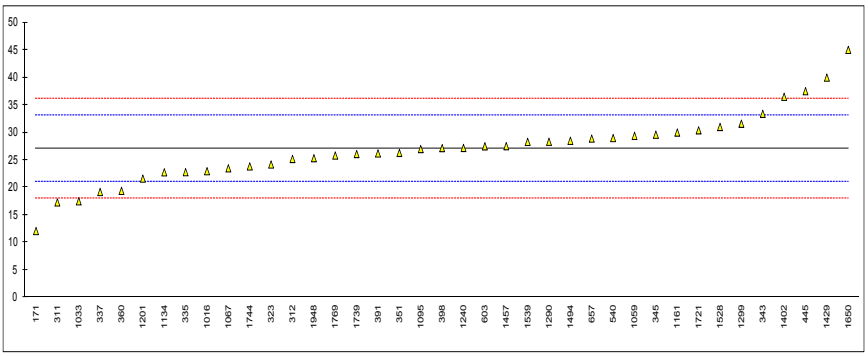
| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 120 | EN14108 | 2.05 | | -2.34 | |
| 171 | EN14108 | 2.2 | | -2.17 | |
| 311 | EN14108 | 4.3 | | 0.25 | |
| 312 | EN14108 | 4.2 | | 0.14 | |
| 323 | EN14538 | 4.0 | | -0.09 | |
| 343 | EN14108 | 4.8 | | 0.83 | |
| 345 | EN14538 | 5.05 | | 1.12 | |
| 360 | EN14538 | 3.75 | | -0.38 | |
| 391 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 445 | EN14538 | 4.50 | | 0.49 | |
| 447 | | ----- | | ----- | |
| 494 | EN14538 | 3.33 | | -0.86 | |
| 496 | EN14538 | 4.88 | | 0.92 | |
| 540 | EN14538 | 4.2 | | 0.14 | |
| 603 | | ----- | | ----- | |
| 657 | EN14108 | 3.89 | | -0.22 | |
| 824 | EN14108 | 5.0 | | 1.06 | |
| 1067 | EN14358 | 3.87 | | -0.24 | |
| 1134 | | ----- | | ----- | |
| 1161 | EN14108 | 0.2139 | R(0.05) | -4.46 | |
| 1201 | D5185mod | 6.2 | | 2.45 | |
| 1240 | EN14538 | 3.56 | | -0.60 | |
| 1268 | | ----- | | ----- | |
| 1290 | EN14538 | 3.294 | | -0.91 | |
| 1299 | EN14108 | 6.8 | | 3.14 | |
| 1316 | D7111 | 2.62 | | -1.68 | |
| 1402 | EN14538 | 3.92 | | -0.18 | |
| 1429 | | ----- | | ----- | |
| 1457 | EN14538 | 3.72 | | -0.41 | |
| 1510 | EN14358 | 3.0 | | -1.24 | |
| 1528 | EN14108 | 4.85 | | 0.89 | |
| 1643 | D5185 | 4.038 | | -0.05 | |
| 1650 | EN14108 | 3.92 | | -0.18 | |
| 1721 | EN14108 | 4.0 | | -0.09 | |
| 1739 | EN14538 | 4.28 | | 0.23 | |
| | normality | suspect | | Spike | |
| | n | 28 | | | |
| | outliers | 1 | | | |
| | mean (n) | 4.08 | | 3.74 | recovery: < 109 % |
| | st.dev. (n) | 1.028 | | | |
| | R(calc.) | 2.88 | | | |
| | R(EN14108:03) | 2.43 | | | Application range: ≥ 1 mg/kg |

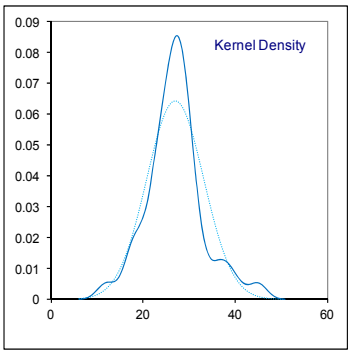


## Determination of Total Contamination on sample #14047; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 171 | EN12662:08 | 12.1 | | -4.93 | |
| 311 | EN12662:14 | 17.3 | | -3.22 | |
| 312 | EN12662:14 | 25.2 | | -0.62 | |
| 323 | EN12662:14 | 24.2 | | -0.95 | |
| 334 | EN12662:08 | >30 | | ----- | |
| 335 | EN12662:14 | 22.8 | | -1.41 | |
| 337 | EN12662:08 | 19.2 | | -2.59 | |
| 343 | EN12662:98 | 33.4 | | 2.08 | |
| 345 | EN12662:98 | 29.6 | | 0.83 | |
| 351 | EN12662:14 | 26.3 | | -0.26 | |
| 360 | EN12662:08 | 19.4 | | -2.53 | |
| 391 | EN12662:14 | 26.2 | | -0.29 | |
| 398 | EN12662:98 | 27.17 | | 0.03 | |
| 445 | EN12662:98 | 37.51 | | 3.43 | |
| 540 | EN12662:98 | 29.0 | | 0.63 | |
| 603 | EN12662 | 27.5 | | 0.14 | |
| 657 | EN12662:08 | 28.9 | | 0.60 | |
| 862 | | ----- | | ----- | |
| 1016 | EN12662:14 | 22.97 | | -1.35 | |
| 1033 | IP440/EN12662:08 | 17.5 | | -3.15 | |
| 1059 | EN12662 | 29.4 | | 0.76 | |
| 1067 | EN12662:14 | 23.5 | | -1.18 | |
| 1095 | EN12662:98 | 27.0 | | -0.03 | |
| 1134 | EN12662:08 | 22.77 | | -1.42 | |
| 1161 | EN12662 | 30.0 | | 0.96 | |
| 1199 | | ----- | | ----- | |
| 1201 | EN12662:98 | 21.63 | | -1.79 | |
| 1240 | EN12662:14 | 27.2 | | 0.04 | |
| 1290 | EN12662:08 | 28.32 | | 0.41 | |
| 1299 | EN12662:08 | 31.6 | | 1.49 | |
| 1402 | EN12662:99 | 36.5 | | 3.10 | |
| 1429 | EN12662:98 | 39.98 | | 4.24 | |
| 1457 | EN12662:14 | 27.53 | | 0.15 | |
| 1494 | EN12662:08 | 28.50 | | 0.47 | |
| 1528 | EN12662:08 | 31.02 | | 1.30 | |
| 1539 | EN12662:09 | 28.3 | | 0.40 | |
| 1582 | | ----- | | ----- | |
| 1650 | EN12662:08 | 45.02 | | 5.90 | |
| 1721 | EN12662:09 | 30.4 | | 1.09 | recovery: < 177 % |
| 1739 | EN12662:98 | 26.1 | | -0.32 | |
| 1744 | EN12662:08 | 23.85 | | -1.06 | |
| 1769 | EN12662:08 | 25.82 | | -0.41 | |
| 1948 | EN12662 | 25.34 | C | -0.57 | first reported: 10.80 |
| | | | Only EN12662:14 data | | |
| | normality | suspect | not OK | Spike | |
| | n | 39 | 10 | | |
| | outliers | 0 | 0 | | |
| | mean (n) | 27.08 | 24.32 | 15.31 | |
| | st.dev. (n) | 6.207 | 2.999 | | |
| | R(calc.) | 17.38 | 8.40 | | |
| | R(EN12662:14) | 8.51 | 8.51 | | Application range: 12 – 30 mg/kg |

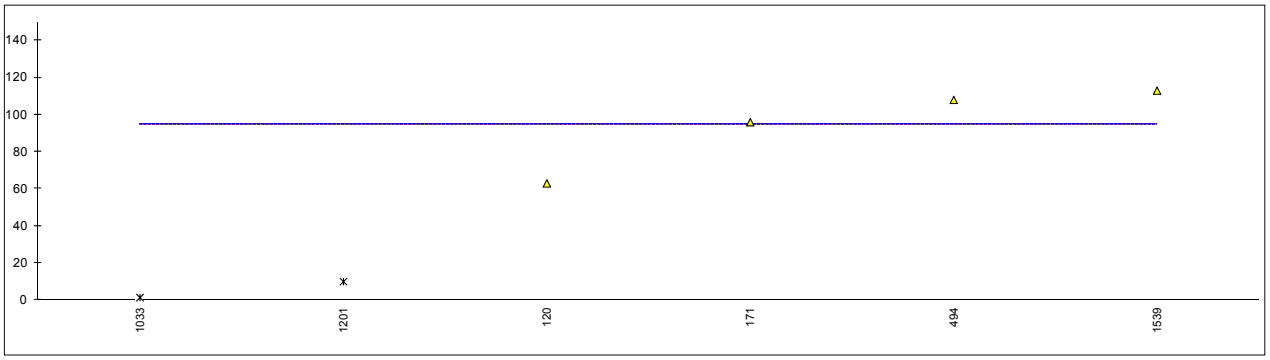


## Determination of Cold Soak Filter Test on sample #14048; results in s

| lab | method | value | mark | z(targ) | filter blocked after (ml) | remarks |
|---|---|---|---|---|---|---|
| 120 | D7501 | 63 | | ----- | ----- | |
| 171 | D7501 | 96 | | ----- | ----- | |
| 311 | | ----- | | ----- | ----- | |
| 343 | | ----- | | ----- | ----- | |
| 398 | | ----- | | ----- | 50 | |
| 494 | D7501 | 108 | | ----- | ----- | |
| 657 | D7501 | >720 | | ----- | 294 | |
| 1033 | IP PM-EA | 1.47 | ex | ----- | 280 | |
| 1067 | | ----- | | ----- | ----- | |
| 1134 | | ----- | | ----- | ----- | |
| 1201 | D7501 | 10.05 | ex | ----- | 30 | |
| 1287 | | ----- | | ----- | ----- | |
| 1402 | | ----- | | ----- | 30 | |
| 1429 | | ----- | | ----- | ----- | |
| 1539 | D7501 | 113 | | ----- | 300 | |
| 1582 | | ----- | | ----- | ----- | |

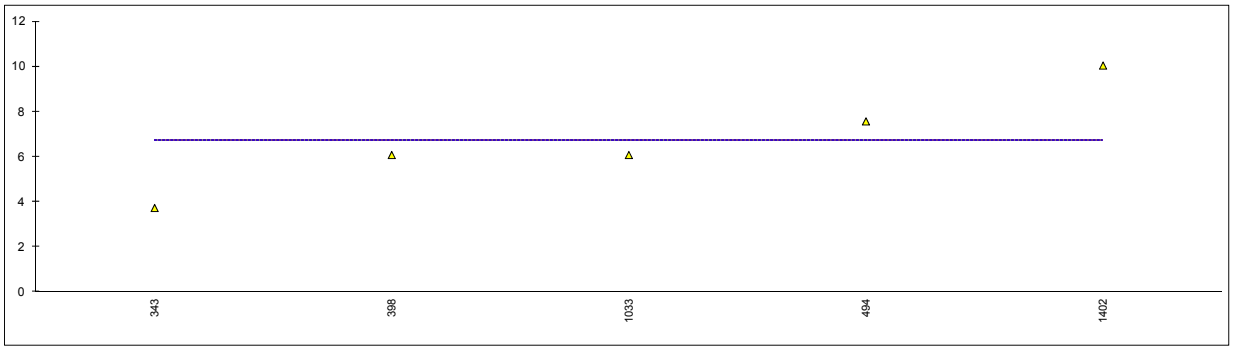
| | |
|---|---|
| normality | unknown |
| n | 4 |
| outliers | 0 + 2 excl |
| mean (n) | 95.0 |
| st.dev. (n) | 22.49 |
| R(calc.) | 63.0 |
| R(D7501:12a) | (10.9) |



## Determination of Filter Blocking Tendency on sample #14048

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 120 | | ----- | | ----- | |
| 171 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 343 | IP387-B | 3.73 | | ----- | |
| 398 | IP387-B | 6.08 | | ----- | |
| 494 | IP387-B | 7.57 | | ----- | |
| 657 | | ----- | | ----- | |
| 1033 | IP387 | 6.08 | | ----- | |
| 1067 | | ----- | | ----- | |
| 1134 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1287 | | ----- | | ----- | |
| 1402 | IP387-B | 10.05 | | ----- | |
| 1429 | | ----- | | ----- | |
| 1539 | | ----- | | ----- | |
| 1582 | | ----- | | ----- | |

| | |
|---|---|
| normality | unknown |
| n | 5 |
| outliers | 0 |
| mean (n) | 6.702 |
| st.dev. (n) | 2.3221 |
| R(calc.) | 6.502 |
| R(IP387B:13) | (2.782) |



## APPENDIX 2

### Number of participants per country

1 lab in ARGENTINA
4 labs in BELGIUM
1 lab in BOSNIA and HERZEGOVINA
2 labs in BRAZIL
3 labs in BULGARIA
3 labs in COLOMBIA
1 lab in CROATIA
1 lab in CZECH REPUBLIC
7 labs in FRANCE
4 labs in GERMANY
2 labs in HONG KONG
2 labs in HUNGARY
2 lab in ITALY
1 lab in LITHUANIA
1 lab in MALAYSIA
4 labs in NETHERLANDS
1 lab in NORWAY
1 lab in PHILIPPINES
4 labs in PORTUGAL
1 lab in SERBIA
1 lab in SLOVENIA
7 labs in SPAIN
4 labs in TURKEY
4 labs in UNITED KINGDOM
3 labs in UNITED STATES OF AMERICA

## APPENDIX 3

**Abbreviations**:

| | |
|---|---|
| C | = final result after checking of first reported suspect 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 |
| ex | = excluded from calculations |
| S | = scope of the reported method is not applicable |
| U | = reported in different unit |
| n.a. | = not applicable |
| n.e. | = not evaluated |
| SDS | = Safety Data Sheet |

**Literature**: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3 ASTM E1301-03

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7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)

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11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)

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13 Analytical Methods Committee Technical Brief, No4 January 2001

14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)

15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983).