## **Results of Proficiency Test** Ethanol (Fuel grade) December 2017

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

ing. G.A. Oosterlaken-Buijs Authors: dr. R.G. Visser & ing. R.J. Starink Correctors:

Report: iis17C15

March 2018

## **CONTENTS**

1	INTRODUCTION	. 3
2	SET UP	. 3
2.1	ACCREDITATION	. 3
2.2	PROTOCOL	. 3
2.3	CONFIDENTIALITY STATEMENT	. 3
2.4	SAMPLES	. 4
2.5	STABILITY OF THE SAMPLES	. 5
2.6	ANALYSES	. 5
3	RESULTS	. 6
3.1	STATISTICS	. 6
3.2	GRAPHICS	. 7
3.3	Z-SCORES	. 7
4	EVALUATION	8
4.1	EVALUATION PER SAMPLE AND PER TEST	. 8
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	11
4.3	COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2017 WITH PREVIOUS PTS	12
Appe	endices:	
1.	Data and statistical results	14
2.	Number of participants in alphabetical country order	54

3.

## 1 Introduction

Since 1995, the Institute for Interlaboratory Studies (iis) organizes a proficiency test (PT) for Ethanol (Fuel grade) in accordance with the latest applicable version of the specifications EN15376 and ASTM D4806.

In this interlaboratory study 61 laboratories from 31 different countries for the PT on Ethanol (Fuel grade) did register for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2017 interlaboratory study on Ethanol (Fuel grade) are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

## 2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. In this proficiency test the participants received two different samples of Ethanol (Fuel grade), a 1 litre bottle (labelled #17240) and a 50 ml bottle (labelled #17241) for Chloride (Inorganic), Sulphate and total Sulphur. only. Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for the statistical evaluation.

### 2.1 ACCREDITATION

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

### 2.2 PROTOCOL

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

## 2.3 CONFIDENTIALITY STATEMENT

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

Fuel/Bio-Ethanol iis17C15 page 3 of 55

### 2.4 SAMPLES

The necessary bulk material for sample #17240 was obtained from an European supplier. The approximately 100 litres bulk material was homogenised in a pre-cleaned drum. After homogenisation 83 amber glass bottles of 1 litre were filled and labelled #17240. The homogeneity of the subsamples #17240 was checked by determination of Density in accordance with ASTM D4052 and Water in accordance with ASTM E203 on 8 stratified randomly selected samples.

	Density at 20°C in kg/L	Water in %M/M
Sample #17240-1	0.79016	0.228
Sample #17240-2	0.79017	0.232
Sample #17240-3	0.79016	0.232
Sample #17240-4	0.79016	0.231
Sample #17240-5	0.79015	0.231
Sample #17240-6	0.79015	0.232
Sample #17240-7	0.79015	0.232
Sample #17240-8	0.79015	0.231

Table 1: homogeneity test results of subsamples #17240

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the reference test methods in agreement with the procedure of ISO 13528, Annex B2 in the next table.

	Density at 20°C in kg/L	Water in %M/M
r (observed)	0.00002	0.004
reference test method	ISO12185:96	EN15489:07
0.3 x R (ref. test method)	0.00015	0.007

Table 2: evaluation of the repeatabilities of subsamples #17240

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, homogeneity of the subsamples of #17240 was assumed.

The necessary bulk material for sample #17241 was obtained from an European supplier. To approximately 3.9 kg of this material, the components (dissolved in water) were added:

Component	Amount
Sodium Chloride (NaCl)	37 mg
Sodium Sulfate (Na <sub>2</sub> SO <sub>4</sub> )	73 mg

Table 3: preparation table for sample #17241

After homogenisation, 97 PE bottles of 50 mL were 60% filled and labelled #17241. The homogeneity of subsamples #17241 was checked by determination of Chloride (inorganic) as CI in accordance with EN15492 on 8 stratified randomly selected samples.

Page 4 of 55 Fuel/Bio-Ethanol iis17C15

	Chloride (inorganic) in mg Cl/kg
Sample #17241-1	5.00
Sample #17241-2	5.36
Sample #17241-3	5.43
Sample #17241-4	5.24
Sample #17241-5	5.34
Sample #17241-6	5.50
Sample #17241-7	5.77
Sample #17241-8	5.57

Table 4: homogeneity test results of subsamples #17241

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the reference test methods in agreement with the procedure of ISO 13528, Annex B2 in the next table.

	Chloride (inorganic) in mg Cl/kg
r (observed)	0.64
reference method	Horwitz
0.3 x R (ref. method)	0.56

Table 5: evaluation of the repeatabilities of subsamples #17241

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples of #17241 was assumed.

To each of the participating laboratories, 1 x 1 litre bottle (labelled #17240) and 1 x 50 ml bottle (60% filled and labelled #17241) was sent on November 8, 2017. A SDS was added to the sample package.

### 2.5 STABILITY OF THE SAMPLES

The stability of Ethanol (Fuel grade), packed in an amber glass bottle and in a PE bottle, was checked. The material was found sufficiently stable for the period of the proficiency test.

## 2.6 ANALYSES

The participants were asked to determine on sample #17240: Acidity, Appearance, Copper as Cu, Density at 20°C, Electrical conductivity at 25°C, Nonvolatile matter, Nitrogen, pHe, Phosphorus as P, Water (coulometric and titrimetric), Ethanol incl. higher alcohols (acc. EN15721), Higher Alcohols (acc. EN15721), Impurities (acc. EN15721), Methanol (acc. EN15721) and Ethanol by mass and by volume (acc. ASTM D5501).

The participants were asked to determine on sample #17241: Chloride (Inorganic), Sulphate and total Sulphur.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but

Fuel/Bio-Ethanol iis17C15 page 5 of 55

report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

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

## 3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.

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

### 3.1 STATISTICS

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

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

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

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by

Page 6 of 55 Fuel/Bio-Ethanol iis17C15

G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

### 3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

### 3.3 Z-SCORES

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

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

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

Fuel/Bio-Ethanol iis17C15 page 7 of 55

The z-scores were calculated according to:

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

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

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

```
|z| < 1 good

1 < |z| < 2 satisfactory

2 < |z| < 3 questionable

3 < |z| unsatisfactory
```

## 4 **EVALUATION**

In this proficiency test some problems were encountered with the dispatch of the samples. Participants in Brasil, Peru and Turkey received the samples late or not at all. Six participants reported the test results after the final reporting date and two participants did not report any test results at all. Not all laboratories were able to report all analyses requested.

In total 59 laboratories reported 537 numerical test results. Observed were 22 outlying test results, which is 4.1% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

### 4.1 EVALUATION PER SAMPLE AND PER TEST

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

Unfortunately, a suitable standard test method, providing the precision data, is not available for all determinations. For the test, that have no available precision data, the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D5501) and an added designation for the year that the method was adopted or revised (e.g. D5501:12). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D5501:12 (2016)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

Page 8 of 55 Fuel/Bio-Ethanol iis17C15

## Sample #17240

Acidity: This determination may be problematic. One statistical outlier was observed.

The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN15491:07 (and ASTM D1613:17 and

ASTM D7795-B:15).

<u>Appearance:</u> This determination was not problematic. All reporting participants agreed about

the appearance as Pass (Clear and Bright).

Copper: Almost all laboratories reported a 'less than' test result. Therefore no statistical

conclusions were drawn.

Density at 20°C: 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 ISO12185:96.

<u>Electrical Conductivity:</u> This determination was very problematic. Three statistical outliers were

observed. The calculated reproducibility after rejection of the statistical outliers

is not at all in agreement with the requirements of EN15938:10.

Nonvolatile matter: All test results were below the application range of the method EN15691:09

(10 – 25 mg/100ml). Therefore no significant conclusions were drawn.

Nitrogen: This determination was problematic at the low level of 0.5 mg/kg. No statistical

outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D4629:12. The low number of

reported test results may (partly) explain the large variation.

<u>pHe:</u> It is known that the pHe determined with a LiCl electrode will be lower than the

pHe determined with a KCI electrode. Two test methods are available for the determination of the pHe of Ethanol: ASTM D6423, that describes the use of a KCI electrode and EN15490, that describes the use of a LiCl electrode. Both test methods are used in this PT and therefore the reported pHe test results

for were split up into pHe (KCI) and pHe (LiCI) and evaluated separately.

<u>pHe (KCI)</u>: This determination was not problematic. No statistical outliers were observed.

The calculated reproducibility is in full agreement with the requirements of

D6423:14.

<u>pHe (LiCI)</u>: 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 EN15490:07. The low number of reported

test results may (partly) explain the large variation.

<u>Phosphorus:</u> Almost all test results were near or below the application range of method

EN15487:07 (0.15 – 1.50 mg/kg). Therefore no statistical conclusions were

drawn.

Fuel/Bio-Ethanol iis17C15 page 9 of 55

Water (coulometric): This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of EN15489:07 (and in agreement with ASTM E1064:16 and ASTM D6304:16e1).

Water (titrimetric): 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 ASTM E203:16 (and in agreement with EN15692:09).

## GC general:

In previous round robins it became clear that the test results reported for the Ethanol content is depending on the test method used by the laboratory. The test method EN15721 uses a different definition for Ethanol than ASTM D5501 does. Therefore, the participants in this proficiency test were requested to report the ethanol content for each of the two definitions.

Ethanol (EN15721): In EN15721 the purity (the ethanol content) is defined as:

Ethanol (incl. higher alcohols) = 100% - impurity% - methanol %, where the higher alcohols consequently are <u>not</u> included in "impurity%".

This determination was problematic. One statistical outlier was observed. Two other laboratories probably did not include the "higher alcohols" in the Ethanol content. The test results of both laboratories were excluded from the statistical calculations. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN15721:13.

Higher Alcohols (EN15721): In EN15721 the higher alcohol content is defined as:

the sum of n-propanol%, n-butanol%, sec-butanol%, isopropanol%, 2-methyl-1-butanol% and 3-methyl-1-butanol%.

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 EN15721:13.

Impurities (EN15721): This determination may be problematic. In EN15721 the impurity content is defined as: content of all components except for Ethanol%, Methanol% and the "higher alcohols"%. No statistical outlier was observed. One laboratory did probably include the "higher alcohols" in the Impurities content and three other laboratories showed statistical outliers in the Ethanol and/or MeOH content. The test result of these four laboratories were excluded from the statistical calculations. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility limits using the Horwitz equation (nine components).

## Methanol:

This determination was problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated requirements calculated using the Horwitz equation, but in agreement with the estimated requirements of ASTM D5501:12. When using standard EN15721:13 a negative value for the reproducibility is found at this concentration level.

Page 10 of 55 Fuel/Bio-Ethanol iis17C15

Ethanol (D5501): This determination was not problematic for Ethanol by mass and Ethanol by volume. No statistical outliers were observed. The test result of one laboratory was excluded as the concentration of Ethanol by mass should be lower as the concentration of Ethanol by volume. For both Ethanol by mass and Ethanol by volume the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D5501:12 (2016).

## **Sample #17241**

Chloride, Inorganic: This determination was not problematic. Two statistical outliers were

observed and one test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the estimated requirements calculated using the Horwitz equation (and in agreement with ASTM D7319:17, but not in agreement with EN15492:12). The average recovery of Inorganic Chloride (theoretical increment of 5.70 mg Chloride/kg) may be good (<92%), the actual Chloride content is unknown.

Sulphate:

This determination was very problematic. No statistical outliers were observed. The reported test results appear to be bimodally distributed. Therefore, no significant conclusions were drawn. Sodium Sulfate was added to sample #17241 (theoretical increment of 12.53 mg SO<sub>4</sub>/kg), the actual Sulfate content is unknown. Regretfully it is unknown if all laboratories reported the Sulfate content as mg SO<sub>4</sub>/kg. In future PTs the Sulfate content will be requested as mg SO₄/kg.

Total Sulphur:

This determination may be problematic at the low level of 1.6 mg/kg, depending on the test method used for evaluation. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of EN15485:07. However, the calculated reproducibility is not in agreement with the reproducibility requirements of EN15486:07.

### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities, derived from reference test methods (in casu ASTM, ISO and EN test methods) are compared in the next tables.

Fuel/Bio-Ethanol iis17C15 page 11 of 55

Parameter	unit	n	average	2.8 *sd	R (lit)
Acidity as Acetic acid, Total	mg/kg	49	24.7	16.8	13.7
Appearance		47	Pass	n.a.	n.a.
Copper as Cu	mg/kg	18	<0.07	n.a.	n.a.
Density at 20°C	kg/L	54	0.7902	0.0002	0.0005
Electrical conductivity at 25°C	μS/cm	27	0.5	0.3	0.1
Nonvolatile matter	mg/100mL	27	0.8	1.7	(0.2)
Nitrogen	mg/kg	9	0.5	0.7	0.5
pHe (KCI)		20	6.71	1.22	1.11
pHe (LiCl)		10	5.72	0.72	0.55
Phosphorus as P	mg/L	21	<0.15	n.a.	n.a.
Water (coulometric)	%M/M	42	0.240	0.022	0.023
Water (titrimetric)	%M/M	26	0.244	0.038	0.078
Ethanol incl. Higher Alcohols (EN15721)	%M/M	29	99.925	0.077	0.045
Higher Alcohols (EN15721)	%M/M	31	0.352	0.103	0.101
Impurities (EN15721)	%M/M	25	0.071	0.050	0.036 *)
Methanol	%M/M	37	0.010	0.003	0.002 *)
Ethanol (D5501)	%M/M	18	99.458	0.556	0.994
Ethanol (D5501)	%V/V	17	99.586	0.601	0.994

Table 6: reproducibilities of tests on sample #17240

Results between brackets should be used with care, because the average was near or below the application range.

<sup>\*)</sup> Reproducibility via the Horwitz equation

Parameter	unit	n	average	2.8 *sd	R (lit)
Chloride, Inorganic as Cl	mg/kg	26	5.3	1.3	1.8 *)
Sulphate as SO <sub>4</sub>	mg/kg	25	7.5	6.8	(1.7)
Total Sulphur	mg/kg	25	1.6	2.3	3.4

Table 7: reproducibilities of tests on sample #17241

Reproducibility between brackets is estimated and should be used with care.

Without further statistical calculations, it can be concluded that for several tests there is a good compliance of the group of participating laboratories with the relevant test methods or the rather strict calculated estimates using the Horwitz equation. The problematic tests have been discussed in paragraph 4.1.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2017 WITH PREVIOUS PTS

	December 2017	December 2016	December 2015	November 2014	November 2013
Number of reporting labs	59	57	68	68	71
Number of results reported	537	476	899	817	880
Statistical outliers	22	31	39	42	41
Percentage outliers	4.1%	6.5%	4.3%	5.1%	4.7%

Table 8: comparison with previous proficiency tests

Page 12 of 55 Fuel/Bio-Ethanol iis17C15

<sup>\*)</sup> Reproducibility via the Horwitz equation

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

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

Determination	December 2017	December 2016	December 2015	November 2014	November 2013
Acidity as Acetic Acid	-	+	+/-	+/-	+
Copper as Cu	n.e.	n.e.	n.e.	n.e.	n.e.
Density at 20°C	++	++	++	++	+
Electrical conductivity			-		
Nonvolatile matter	()	()	()	()	()
Nitrogen	-				-
рНе	-	+/-	n.e.	n.e.	n.e.
Phosphorus as P	n.e.	n.e.	n.e.	(+/-)	()
Water (coulometric)	+/-	+	+/-	+/-	+
Water (titrimetric)	++	++	++	++	++
Ethanol (EN15721)	-	-		n.e.	n.e.
Higher Alcohols (EN15721)	+/-	-	-	n.e.	n.e.
Impurities (EN15721)	-			n.e.	n.e.
Methanol	-	-		++	++
Ethanol (D5501)	+	++	++		++
Chloride, Inorganic as Cl	+	(++)	-	+	-
Sulphate as SO4	*)		()	()	()
Total Sulphur	+	+	++	++	++

Table 9: Comparison determinations against the reference test method

Results between brackets should used with care, because the average was near or below the application range.

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

++: group performed much better than the reference test method

+ : group performed better than the reference test method

+/-: group performance equals the reference test method

- : group performed worse than the reference test method

-- : group performed much worse than the reference test method

n.e.: not evaluated

Fuel/Bio-Ethanol iis17C15 page 13 of 55

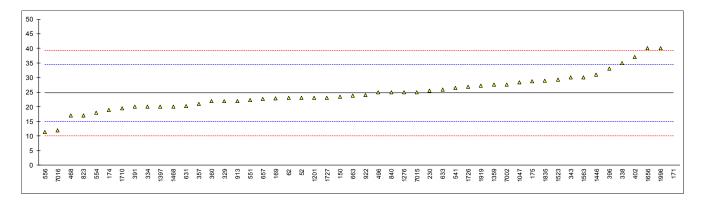
<sup>\*)</sup> Performance results should be used with care, because the reproducibility is estimated.

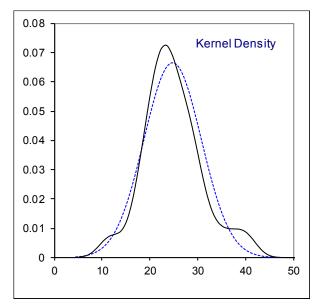
## **APPENDIX 1**

Determination of Acidity, Total as Acetic Acid on sample #17240; results in mg/kg

Detern	nination of Acidity	, Total as	S ACETIC A	Acia on s	sample #17240; results in mg/kg
lab	method	value	mark	z(targ)	remarks
52	D1613	23		-0.35	
62	D1613	23		-0.35	
150	D1613	23.4		-0.27	
169	D7795	22.8		-0.39	
171	EN15491	300	R(0.01)	56.26	
174	D1613	18.9		-1.19	
175	D7795	28.7		0.82	
230	D1613	25.5		0.16	
311	EN15491	<30			
323	EN15491	< 0.003	U		probably reported in a different unit (%M/M ?)
329	EN15491	22		-0.55	
333	EN15491	<30			
334	EN15491	20		-0.96	
337	=111=101				
338	EN15491	35		2.10	
343	EN15491	30		1.08	
357	EN15491	21		-0.76	
360	EN15491	22		-0.55	
391	EN15491	20		-0.96	
396	EN15491	33		1.70	
402	EN15491	37		2.51	
444 469	EN15401	 17		1 50	
468 496	EN15491 EN15491	17 25		-1.58 0.06	
496 511	EN15491	25 		0.06	
511 541	D1613	26.5		0.37	
551	D1613	20.5 22.27		-0.50	
554	D1613	18.0		-1.37	
556	NBR16047	11.4		-2.72	
631	D1613	20.3		-0.90	
633	D1613	26		0.26	
657	D1613	22.64		-0.42	
663	D1613	23.8		-0.19	
823	D1613	17		-1.58	
840	D1613	25.0		0.06	
902					
913	D1613	22.0		-0.55	
922	D1613	24		-0.14	
1047	EN15491	28.3		0.73	
1191					
1201	EN15491	23		-0.35	
1276	EN15491	25		0.06	
1359	EN15491	27.60		0.59	
1397	EN15491	20		-0.96	
1446	EN15491	31		1.29	
1468	EN15491	20		-0.96	
1523	ISO1388-2	29.3		0.94	
1563	EN15491	30		1.08	
1605 1656	EN15401	40		 3 13	
1656 1667	EN15491	40		3.13	
1710	EN15491	19.4		-1.08	
1710	EN15491	26.9		0.45	
1727	EN15491	23		-0.35	
1835	EN15491	29		0.88	
1852					
1919	EN15491	27.3		0.53	
1996	EN15491	40		3.13	
7002		27.6		0.59	
7015		25		0.06	
7016		12		-2.60	
	normality	OK			
	n	49			
	outliers	1			
	mean (n)	24.706			
	st.dev. (n)	6.0004			
	R(calc.)	16.801			
	st.dev.(EN15491:07)	4.8929			application range; 20, 450 mg/l/g
	R(EN15491:07)	13.7			application range: 30 - 150 mg/kg
	For comparison: R(D1613:17)	14			application range: <500 mg/kg
	R(D1013.17) R(D7795-B:15)	13.065			application range: <500 mg/kg
	(27.700 B.10)	. 5.555			application range. Lee myrky

Page 14 of 55 Fuel/Bio-Ethanol iis17C15





Fuel/Bio-Ethanol iis17C15 page 15 of 55

# Determination of Appearance on sample #17240;

lab	method	value	mark	z(targ)	remarks
52	D4176	Pass	murk	<u> </u>	Tomano
62	D4170				
150	E2680	Pass			
169	D4176	Pass			
171	EN15769	Clear and colourless			
174	Visual	Clear & free			
175	D4176	Clear and bright			
230	Visual	Clear & Bright			
311	EN15769	clear & colourless			
323	E2680	clear & bright			
329	Visual	clear			
333	EN15769	clear and colourless			
334	EN 15709				
337	Visual	Colourless			
338	Visual	Clear and Bright			
343	INH-1608	C&B			
357	E2680	Pass			
360	EN15376	Clear and Colourless			
391	E2680	Pass			
	E2680				
396	E2000	Pass 			
402	EN4.5700				
444	EN15769	Pass			
468	EN15769	C&C			
496	Visual	clear colourless			
511	F0000	 D			
541	E2680	Pass			
551	Visual	Clear			and the de Ole and and the affirm a second and another
554	Visual	CFFSM			reported: Clear and free from suspended matter
556	\ r \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \				
631	Visual	Clear and Bright			
633	Visual	Clear & Bright			
657	E2680	Pass			
663	Visual	Clear & Bright			
823	E2680	Pass			
840	E2680	Pass			
902	=				
913	E2680	CFSM			
922	Visual	clear and bright			
1047	EN15769	Clear&Colourless			
1191	D4176	C&B			
1201	Visual	Bright and Clear			
1276	EN15769	C&C			
1359	EN15769	Clear and colourless			
1397					
1446					
1468					
1523	EN145700				
1563	EN15769	clear & colourless			
1605	EN145700	D			
1656	EN15769	Pass			
1667	EN45700	Class and Calavidas			
1710	EN15769	Clear and Colourless			
1726	EN15769	Clear and colourless			
1727	Visual	Clear and colourless			
1835	EN15769	CCL			
1852	Visual	clear and bright			
1919	Viewel	 Lp. p. p.			
1996	Visual	l.p.p.s			
7002					
7015		clear			
7016		clear and without impurity			
	_	47			
	n maan (n)	47			
	mean (n)	Pass (Clear & Bright)			

Page 16 of 55 Fuel/Bio-Ethanol iis17C15

## Determination of Copper as Cu on sample #17240; results in mg/kg

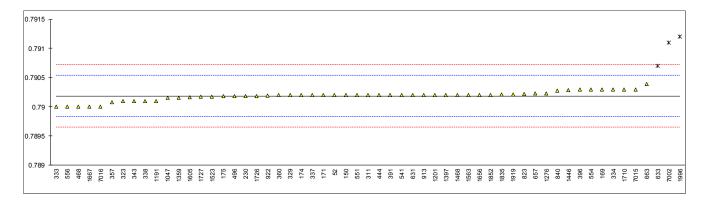
lab	method	value	mark	z(targ)	remarks
52	D1688	<0.05			
62	D4000				
150 169	D1688	<0.5 			
171	D1688	<0.05			
174					
175	D1688	0.001			
230 311	EN15488	<0.0050			
323	EN15488	< 0.070			
329					
333	EN15488	<0.07			
334 337					
338					
343	EN15488	<0.07			
357	EN15027	 < 0.050			
360 391	EN15837	< 0.050 			
396					
402					
444 468	EN15488	<0.1			
496	LIN 13400				
511					
541					
551 554	INH-2047	<0.04			
556					
631	D1688	<0.05			
633					
657 663	INH-12414	0.002			
823	D1688	< 0.05			
840	D1688	<0.05			
902					
913 922	D1688	<0.05			
1047	EN15837	<0.03			
1191					
1201	EN15488	<1			
1276 1359	EN15488	0.2083			false positive test result?
1397					
1446					
1468 1523	EN15837	<0.1 			
1563	EN15488	<0.070			
1605					
1656	D1688-A	<0.01			
1667 1710					
1726					
1727					
1835					
1852 1919					
1996	EN15837	<0.05			
7002					
7015 7016					
7010					
	normality n	unknown 18			
	outliers	n.a.			
	mean (n)	<0.07			
	st.dev. (n)	n.a.			
	R(calc.) st.dev.(EN15488:07)	n.a. n.a.			
	R(EN15488:07)	n.a.			application range: 0.07 – 0.20 mg/kg
	For comparison				
	R(D1688:12)	n.a.			

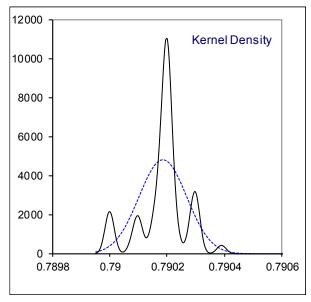
Fuel/Bio-Ethanol iis17C15 page 17 of 55

## Determination of Density at 20°C on sample #17240; results in kg/L

lab	method	value	mark	z(targ)	remarks
52	D4052	0.7902		0.08	
62 150	D4052	0.7902		0.08	
169	D4052	0.7903		0.64	
171	ISO12185	0.7902		0.08	
174 175	D4052 D4052	0.7902 0.79018		0.08 -0.03	
230	D4052	0.79018		-0.03	
311	ISO12185	0.7902		0.08	
323 329	D4052 D4052	0.7901 0.7902		-0.48 0.08	
333	ISO12185	0.7902		-1.04	
334	ISO12185	0.7903		0.64	
337 338	ISO12185 ISO12185	0.7902 0.7901		0.08 -0.48	
343	ISO12185	0.7901		-0.48	
357	D4052	0.79008		-0.59	
360 391	D4052 ISO12185	0.7902 0.7902		0.08 0.08	
396	D4052	0.7902		0.06	
402					
444 468	D4052 ISO12185	0.7902 0.7900		0.08 -1.04	
496	ISO12185	0.7900	С	-0.03	first reported 0.70918
511					
541	D4052	0.79020		0.08	
551 554	D4052 D4052	0.7902 0.7903		0.08 0.64	
556	NBR5992	0.7900		-1.04	
631 633	D4052 D4052	0.79020 0.7907	R(0.01)	0.08 2.88	
657	D4052 D4052	0.7907	K(0.01)	0.25	
663	D4052	0.79039		1.14	
823	D4052	0.79022		0.19	
840 902	D4052	0.79028		0.53	
913	D4052	0.7902		0.08	
922 1047	D4052 ISO12185	0.79019 0.79015		0.02 -0.20	
1191	ISO12185	0.7901		-0.48	
1201	ISO12185	0.7902		0.08	
1276 1359	ISO12185 ISO12185	0.79023 0.79015		0.25 -0.20	
1397	ISO12185	0.7902		0.08	
1446	ISO12185	0.79029		0.58	
1468 1523	ISO12185 D4052	0.7902 0.790172		0.08 -0.08	
1563	INH-035	0.79020		0.08	
1605	D4052	0.790161		-0.14	
1656 1667	D4052 ISO3675	0.7902 0.7900		0.08 -1.04	
1710	ISO12185	0.7903		0.64	
1726	D4052	0.79018		-0.03	
1727 1835	D4052 ISO12185	0.79017 0.79021		-0.09 0.14	
1852	ISO12185	0.7902		0.08	
1919	ISO12185	0.790212	D(0.04)	0.15	
1996 7002	ISO12185 ISO12185	0.7912 0.7911	R(0.01) R(0.01)	5.68 5.12	
7015	ISO12185	0.7903	11(0.01)	0.64	
7016		0.790		-1.04	
	normality	OK			
	n	54			
	outliers mean (n)	3 0.79019			
	st.dev. (n)	0.000083			
	R(calc.)	0.00023			
	st.dev.(ISO12185:96) R(ISO12185:96)	0.000179 0.0005			
	()				

Page 18 of 55 Fuel/Bio-Ethanol iis17C15



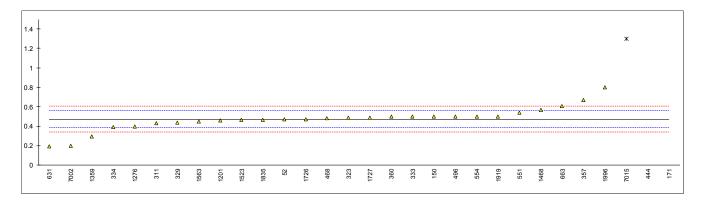


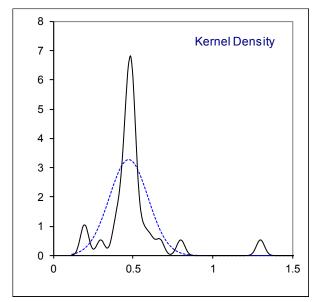
Fuel/Bio-Ethanol iis17C15 page 19 of 55

## Determination of Electrical conductivity at 25°C on sample #17240; results in $\mu S/cm$

lab	method	value	mark	z(targ)	remarks
52		0.47		-0.08	
62	=111=000				
150 169	EN15938	0.50		0.59 	
171	EN15938	70.00	R(0.01)	1558.66	
174	D1125	<10	11(0.01)		
175					
230	EN45000				
311 323	EN15938 EN15938	0.43 0.49		-0.98 0.37	
329	EN15938	0.49		-0.75	
333	EN15938	0.50		0.59	
334	EN15938	0.39		-1.87	
337					
338					
343 357	EN15938	0.67		4.40	
360	EN15938	0.50		0.59	
391					
396					
402	EN45000		0.0(0.04)	74.00	South was asted 0.40
444 468	EN15938 EN15938	3.81 0.48	C,R(0.01)	74.80 0.14	first reported 2.19
496	EN15938	0.40		0.14	
511					
541					
551	NBR10547	0.54		1.49	
554	NBR10547	0.5		0.59	
556 631	D1125	0.193	С	 -6.29	first reported 221.5; reported 193 mS/cm
633	D1120		Ü		mot reported 221.0, reported 100 morom
657					
663	D1125	0.61		3.06	
823					
840 902					
913					
922					
1047					
1191	EN45020	0.460		0.26	
1201 1276	EN15938 EN15938	0.462 0.4		-0.26 -1.65	
1359	EN15938	0.2975		-3.95	
1397					
1446					
1468	EN15938 D2624	0.57 0.465		2.16	
1523 1563	EN15938	0.465		-0.19 -0.53	
1605	21110000				
1656	EN15938	<1			
1667					
1710 1726	EN15938	0.470		-0.08	
1727	EN15938	0.470		0.37	
1835	EN15938	0.468		-0.12	
1852					
1919	EN15938	0.50		0.59	
1996 7002	EN15938	0.8 0.2		7.32 -6.13	
7015		1.3	R(0.01)	18.53	
7016			/		
	normality	not OK			
	n outliers	27 3			
	mean (n)	0.474			
	st.dev. (n)	0.1214			
	R(calc.)	0.340			
	st.dev.(EN15938:10)	0.0446			
	R(EN15938:10)	0.125			

Page 20 of 55 Fuel/Bio-Ethanol iis17C15



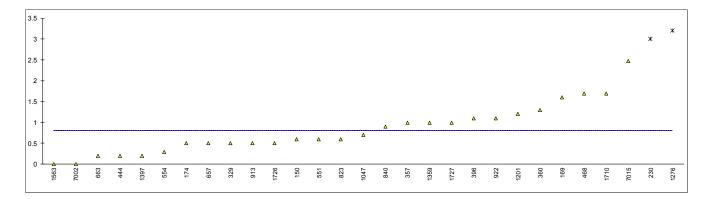


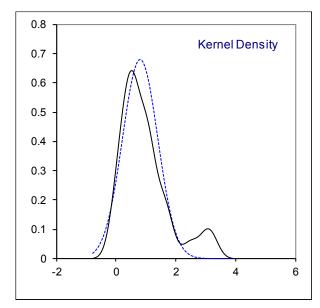
Fuel/Bio-Ethanol iis17C15 page 21 of 55

## Determination of Nonvolatile matter on sample #17240; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
52	D1353	<1			
62					
150 169	D1353 D1353	0.6 1.6			
171	EN15691	<10			
174	D1353	0.5			
175	D4050		0.0(0.05)		Free transported 4.0
230 311	D1353 EN15691	3 <10	C,R(0.05)		first reported 4.0
323	EN15691	< 10			
329	D1353	0.5			
333					
334 337					
338					
343	EN15691	<10			
357	EN15691	1.0 1.3			
360 391	EN15691	1.5			
396	D1353	1.1			
402	EN45004				
444 468	EN15691 EN15691	0.2 1.7			
496	21110001				
511					
541	D1353	<0.1			
551 554	D1353 D1353	0.6 0.3			
556	51000				
631					
633 657	D1353	0.5			
663	D1353	0.20			
823	D1353	0.6			
840	D1353	0.9			
902 913	D1353	0.5			
922	D1353	1.1			
1047	EN15691	0.7			
1191	EN15601	1.2			
1201 1276	EN15691 EN15691	3.2	R(0.05)		
1359	EN15691	1.0	. 1(0.00)		
1397	EN15691	0.2			
1446 1468	EN15691	 <1			
1523	LIVIOUSI				
1563	EN15691	0			
1605	EN15601				
1656 1667	EN15691	<1 			
1710	EN15691	1.7			
1726	EN15691	0.5			
1727 1835	EN15691 EN15691	1 <10			
1852	LIN 1309 I				
1919					
1996					
7002 7015		0.0013 2.48			
7016		Zero			
	normality	suspect 27			
	n outliers	2			
	mean (n)	0.81			
	st.dev. (n)	0.588			
	R(calc.) st.dev.(EN15691:09)	1.65 (0.054)			
	R(EN15691:09)	(0.054)			application range: 10- 25 mg/100mL
	For comparison				
	R(D1353:13)	(0.35)			

Page 22 of 55 Fuel/Bio-Ethanol iis17C15



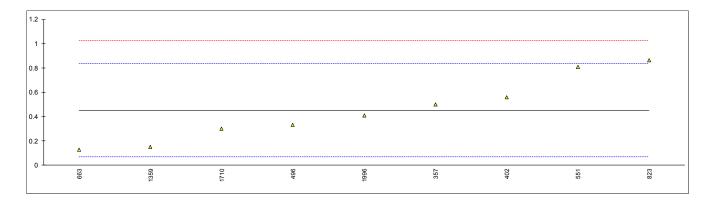


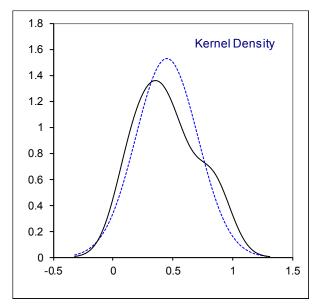
Fuel/Bio-Ethanol iis17C15 page 23 of 55

## Determination of Nitrogen on sample #17240; results in mg/kg

lab	method	value	mark z(targ)	remarks
52				
62				
150				
169				
171	D4629	<0.3		
174 175				
175 230				
311	D4629	<0.3		
323	D4629	< 1		
329				
333	D4629	<0.3		
334				
337				
338				
343 357	D4629	0.5	0.26	
360	D4029	0.5	0.20	
391				
396				
402	D4629	0.56	0.57	
444				
468	D4629	<0.5		
496	D4629	0.33	-0.63 	
511 541				
551	D4629	0.81	1.88	
554	2.020			
556				
631				
633	D. 1000			
657	D4629	< 0.3	4.07	
663 823	D4629 D4629	0.13 0.86	-1.67 2.14	
840	D4029	0.00	2.14	
902				
913				
922	D4629	<0.3		
1047				
1191	D 1000			
1201	D4629	<1		
1276 1359	D4629	0.15	 -1.57	
1397	D-1020			
1446				
1468				
1523				
1563				
1605	D4620			
1656 1667	D4629	<1 		
1710	D4629	0.3	-0.78	
1726	2.020			
1727				
1835				
1852				
1919	D.4000			
1996 7002	D4629	0.41	-0.21	
7002				
7016				
	normality	OK		
	n	9		
	outliers	0		
	mean (n)	0.450		
	st.dev. (n) R(calc.)	0.2606 0.730		
	st.dev.(D4629:12)	0.730		
	R(D4629:12)	0.537		application range: 0.3 – 100 mg/kg
	,			

Page 24 of 55 Fuel/Bio-Ethanol iis17C15



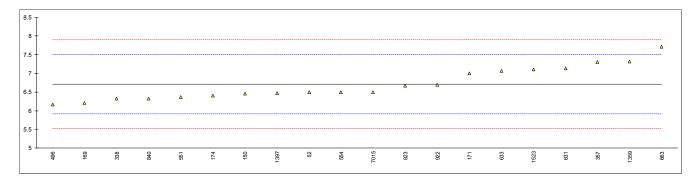


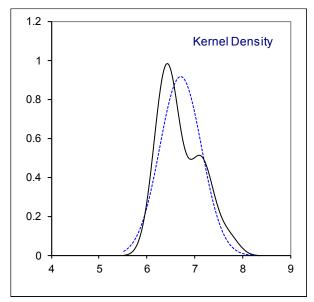
Fuel/Bio-Ethanol iis17C15 page 25 of 55

## Determination of pHe (KCI) on sample #17240;

Jak	mathad	volus	morle	7/40-01	romarka
<b>lab</b> 52	method D6423	value 6.5	mark	<b>z(targ)</b> -0.53	remarks
52 62	D0423	0.5		-0.53	
150	D6423	6.46		-0.63	
169	D6423	6.2	С	-1.29	first reported 10.18
171	D6423	7.0		0.73	no details on which type of electrode was used
174	D6423	6.4		-0.78	•
175					
230					
311 323					
329					
333					
334					
337					
338	EN15490	6.32		-0.99	
343 357	D6423	7.3	С	1.49	first reported as LiCl electrode
360	D0423	7.5	O		mst reported as Lioi electrode
391					
396					
402					
444					
468 406	D6423	6.17		 -1.37	
496 511	D6423	0.17		-1.37	
541					
551	D6423	6.37		-0.86	
554	D6423	6.5		-0.53	
556	D0.100	 		4.00	
631	D6423	7.13 7.07		1.06 0.91	
633 657	D6423	7.07			
663	D6423	7.71		2.52	
823	D6423	6.672		-0.10	
840	D6423	6.32		-0.99	
902					
913 922	D6423	6.7		-0.03	
1047	D0 <del>4</del> 23			-0.03	
1191					
1201					
1276					
1359	EN45400	7.32		1.54	
1397 1446	EN15490	6.47		-0.61 	
1468					
1523	D6423	7.1		0.98	
1563					
1605					
1656 1667					
1710					
1716					
1727					
1835					
1852					
1919 1996					
7002					
7015		6.5		-0.53	no details on which type of electrode was used
7016					•
		OK			
	normality	OK			
	n outliers	20 0			
	mean (n)	6.711			
	st.dev. (n)	0.4343			
	R(calc.)	1.216			
	st.dev.(D6423:14)	0.3958			
	R(D6423:14)	1.108			

Page 26 of 55 Fuel/Bio-Ethanol iis17C15



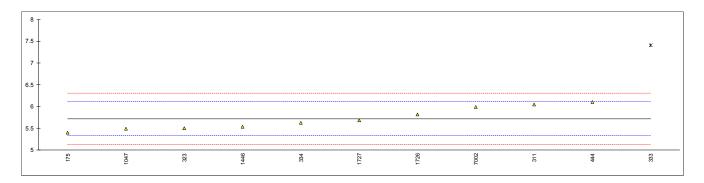


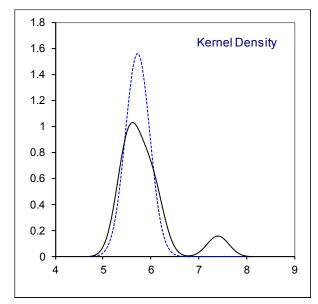
Fuel/Bio-Ethanol iis17C15 page 27 of 55

## Determination of pHe (LiCl) on sample #17240;

lab	method	value	mark	z(targ)	remarks
52					
62					
150					
169 171					
174					
175	D6423	5.4		-1.62	
230					
311	EN15490	6.05		1.70	
323 329	EN15490	5.5 		-1.11 	
333	EN15490	7.4	D(0.01)	8.58	
334	EN15490	5.62	2(0.0.)	-0.50	
337					
338					
343 357					
360					
391					
396					
402	EN45400			4.05	
444 468	EN15490	6.1 		1.95 	
496					
511					
541					
551					
554					
556 631					
633					
657					
663					
823 840					
902					
913					
922	EN45400	 		4.40	
1047 1191	EN15490	5.485		-1.19 	
1201					
1276					
1359 1397					
1446	EN15490	5.53		-0.96	
1468					
1523					
1563 1605					
1656					
1667					
1710	=11.4=400				
1726 1727	EN15490 EN15490	5.82 5.68		0.52 -0.19	
1835	LN 13490	5.00		-0.19	
1852					
1919					
1996		 E 00		1 20	no details on which type of electrode was used
7002 7015		5.99 		1.39	no details on which type of electrode was used
7016					
		014			
	normality n	OK 10			
	outliers	10			
	mean (n)	5.717			
	st.dev. (n)	0.2560			
	R(calc.) st.dev.(EN15490:07)	0.717 0.1960			
	R(EN15490:07)	0.549			
	•				

Page 28 of 55 Fuel/Bio-Ethanol iis17C15





Fuel/Bio-Ethanol iis17C15 page 29 of 55

-- Empty page --

Page 30 of 55 Fuel/Bio-Ethanol iis17C15

## Determination of Phosphorus as P on sample #17240; results in mg/L

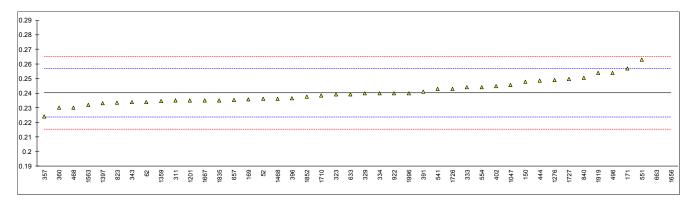
lab	method	value	mark	z(targ)	remarks
52	D3231	<0.20	mark	<u> </u>	Tomarko
62	D3231	<0.2			
150					
169					
171	EN15487	<0.15			
174					
175					
230	EN145407				
311	EN15487	<0.13			
323 329	EN15487 EN15487	< 0.10 <0.15			
333	EN15487	<0.15			
334	LITTOTO				
337					
338					
343					
357					
360	EN15837	0.05			
391					
396 402	EN15487	<0.15			
444	EN15487	0.005			
468	21110107				
496					
511					
541					
551	INH-2047	<0.13			
554					
556					
631 633					
657					
663					
823	UOP389	<0.11			
840					
902					
913					
922	EN145027	<0.09			
1047 1191	EN15837	<0.09 			
1201	EN15487	<0.15	С		first reported 6.7
1276	EN15487	0.01	_		
1359	EN15487	0.00475			
1397					
1446	=>=.				
1468	EN15487	<0.1			
1523 1563	EN15487	0.00			
1605	LIN 13407				
1656	EN15487	<0.01			
1667					
1710					
1726	EN15487	0.0059			
1727	EN15487	<0.01			
1835	EN15487	<0.15			
1852 1919					
1919	EN15487	<0.15			
7002	LIVIOTOI				
7015					
7016					
	normality	unknown			
	n	21			
	outliers	0			
	mean (n) st.dev. (n)	<0.15			
	R(calc.)	n.a. n.a.			
	st.dev.(EN15487:07)	n.a.			
	R(EN15487:07)	n.a			application range: 0.15 – 1.50 mg/L
	,				··· •

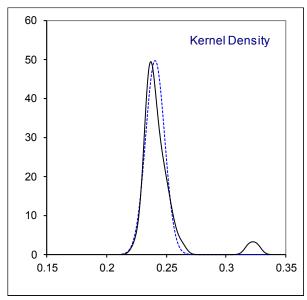
Fuel/Bio-Ethanol iis17C15 page 31 of 55

## Determination of Water (coulometric) on sample #17240; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	E1064	0.236	mark	-0.53	Tomaria
62	E1064	0.234		-0.55	
150	E1064	0.248		0.91	
169	E1064	0.2359		-0.55	
171	EN15489	0.257		1.99	
174					
175					
230					
311	EN15489	0.235		-0.65	
323	EN15489	0.2390		-0.17	
329	E1064	0.2398		-0.08	
333	EN15489	0.244		0.43	
334	EN15489	0.24		-0.05	
337 338					
343	EN15489	0.234		 -0.77	
357	E1064	0.234		-1.97	
360	E1064	0.2300		-1.25	
391	EN15489	0.241		0.07	
396	EN15489	0.2365		-0.47	
402	EN15489	0.245		0.55	
444	EN15489	0.2486		0.98	
468	EN15489	0.23		-1.25	
496	EN15489	0.254		1.63	
511					
541	E1064	0.2428		0.28	
551	E1064	0.263		2.71	
554	D6304	0.2442		0.45	
556 631					
633	D6304	0.2391		-0.16	
657	E1064	0.2355		-0.59	
663	E1064	0.3195	R(0.01)	9.49	
823	E1064	0.2334	(5.5.)	-0.85	
840	E1064	0.2506		1.22	
902					
913					
922	D6304	0.24		-0.05	
1047	EN15489	0.2456		0.62	
1191	=111=100				
1201	EN15489	0.235		-0.65	
1276	EN15489	0.2489		1.02	
1359 1397	EN15489 EN15489	0.23476 0.233		-0.68 -0.89	
1446	LN 13409	0.233		-0.09	
1468	EN15489	0.2361		-0.52	
1523	2.11.0.00				
1563	EN15489	0.232		-1.01	
1605					
1656	EN15489	0.3260	C,R(0.01)	10.27	first reported 2890 mg/kg
1667	EN15489	0.235	С	-0.65	first reported 0.198
1710	EN15489	0.2386		-0.22	
1726	EN15489	0.243		0.31	
1727	EN15489	0.2497		1.11	
1835	EN15489	0.2352		-0.63	
1852 1919	EN15489 EN15489	0.2375 0.25383	С	-0.35 1.61	first reported 0.29516
1996	E1064	0.23363	C	-0.05	first reported 0.28516
7002	L 1004	0.240		-0.03	
7015					first reported as Water (titrimetric)
7016					()
	normality	OK			
	n	42			
	outliers	2			
	mean (n)	0.24044			
	st.dev. (n)	0.008008			
	R(calc.)	0.02242			
	st.dev.(EN15489:07) R(EN15489:07)	0.008329			
	For comparison	0.02332			
	R(E1064:16)	0.04112			
	R(D6304:16e1)	0.18042			
	` '				

Page 32 of 55 Fuel/Bio-Ethanol iis17C15



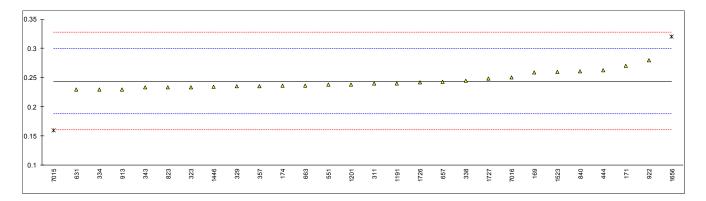


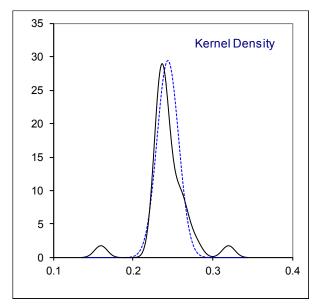
Fuel/Bio-Ethanol iis17C15 page 33 of 55

## Determination of Water (titrimetric) on sample #17240; results in %M/M

lab	method	value	mark	z(targ)	remarks
52					
62 150					
169	E203	0.2592		0.55	
171 174	E203 E203	0.270 0.2359		0.93 -0.29	
175					
230 311	E203	0.240		 -0.14	
323	E203	0.2335		-0.38	
329 333	E203	0.2356		-0.30 	
334	E203	0.23		-0.50	
337 338	E203	 0.2445		0.02	
343	E203	0.233		-0.39	
357 360	E203	0.2357		-0.30 	
391					
396 402					
444	E203	0.2624		0.66	
468 496					
511					
541 551	E203	0.238		-0.21	
554	L203			-0.21	
556 631	E203	0.2295		-0.52	
633					
657 663	E203 E203	0.2425 0.2363		-0.05 -0.28	
823	E203	0.2333		-0.38	
840 902	E203	0.2610		0.61	
913	E203	0.23		-0.50	
922 1047	E203	0.28		1.29	
1191	ISO10336	0.24		-0.14	
1201 1276	E203	0.238		-0.21 	
1359					
1397 1446	ISO760	0.2340		-0.36	
1468					
1523 1563	E203	0.26		0.58	
1605					
1656 1667	E203	0.3200	R(0.01)	2.73	
1710					
1726 1727	EN15692 EN15692	0.2420 0.2489		-0.07 0.18	
1835	21410002				
1852 1919					
1996					
7002 7015		0.16	C,R(0.01)	-3.01	first reported as Water (coulometric)
7016		0.25	0,11(0.01)	0.22	instreported as water (could metric)
	normality	suspect			
	n	26			
	outliers mean (n)	2 0.24397			
	st.dev. (n)	0.013520			
	R(calc.) st.dev.(E203:16)	0.03785 0.027857			
	R(E203:16)	0.078			
	For comparison R(EN15692:09)	0.09788			
	.(=::::300=:30)	2.20.00			

Page 34 of 55 Fuel/Bio-Ethanol iis17C15



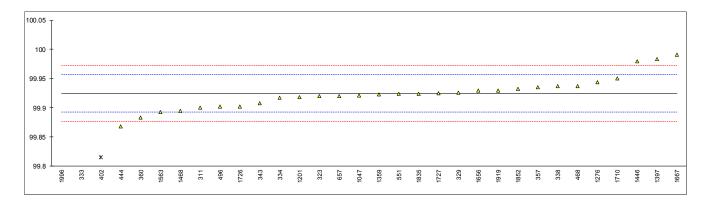


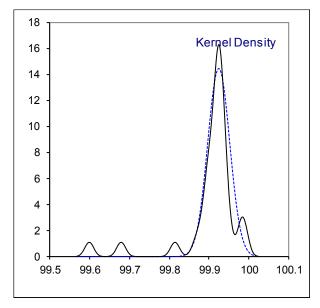
Fuel/Bio-Ethanol iis17C15 page 35 of 55

## Determination of Ethanol incl. Higher Alcohols acc. to EN15721 on sample #17240 in %M/M

lab	method	value	mark	z(targ)	remarks
52					
62 150					
150 169					
171					
174					
175					
230 311	EN15721	99.90		-1.56	
323	EN15721	99.920		-0.30	
329	EN15721	99.926		80.0	
333 334	EN15721 EN15721	99.68 99.917	ex	-15.39 -0.49	result excluded: probably without higher alcohols
337	LIVISIZI				
338	EN15721	99.937		0.77	
343	EN15721	99.908	С	-1.06	first reported 99.674
357 360	EN15721 EN15721	99.935 99.8830		0.64 -2.63	
391	LIVIO721			-2.00	
396					
402	EN15721	99.815	C,R(0.05)	-6.90	first reported 99.72435
444 468	EN15721 EN15721	99.868 99.937	С	-3.57 0.77	first reported 99.63
496	EN15721	99.9021		-1.43	
511					
541 551	INH-1313	99.9238		-0.06	
554	11411-1313			-0.00	
556					
631					
633 657	INH-02	99.9206		-0.26	
663	02				
823					
840 902					
913					
922	<b>-</b> 11101				
1047 1191	EN15721	99.921		-0.24 	
1201	EN15721	99.918		-0.43	
1276	EN15721	99.944	_	1.21	
1359 1397	EN15721 EN15721	99.923 99.983	С	-0.11 3.66	first reported 99.6885
1446	EN15721 EN15721	99.98		3.47	
1468	EN15721	99.895		-1.87	
1523	EN45704				
1563 1605	EN15721	99.893		-2.00 	
1656	EN15721	99.93		0.33	
1667	EN15721	99.991		4.16	
1710 1726	EN15721 EN15721	99.950 99.9022		1.58 -1.42	
1727	EN15721	99.925		0.01	
1835	EN15721	99.924		-0.05	
1852	EN15721	99.9323		0.47	
1919 1996	EN15721 IMPCA001Mod.	99.93 99.600	ex	0.33 -20.42	result excluded: probably without higher alcohols
7002	07.0000		<b></b>		room oxonesses productly mandating for alcoholo
7015					
7016					
	normality	OK			
	n	29			
	outliers mean (n)	1 (+2 ex) 99.92480			
	st.dev. (n)	0.027579			
	R(calc.)	0.07722			
	st.dev.(EN15721:13)	0.015906			
	R(EN15721:13)	0.04454			

Page 36 of 55 Fuel/Bio-Ethanol iis17C15



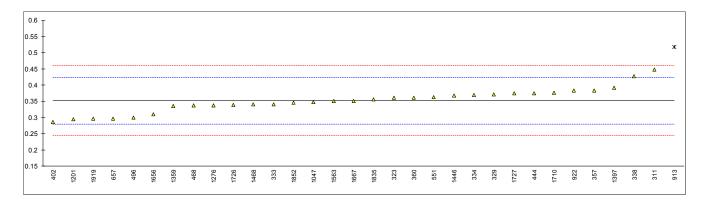


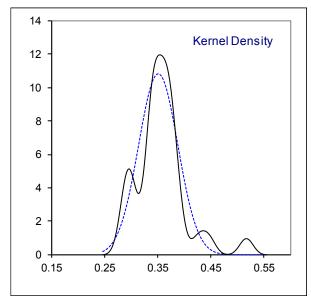
Fuel/Bio-Ethanol iis17C15 page 37 of 55

## Determination of Higher Alcohols acc. to EN15721 on sample #17240; results in %M/M

lab	method	value	mark	z(targ)	remarks
52					
62 150					
150 169					
171					
174					
175 230					
311	EN15721	0.447		2.63	
323	EN15721	0.361		0.25	
329	EN15721	0.3710		0.52	
333 334	EN15721 EN15721	0.341 0.370		-0.31 0.50	
337	LIVIOIZI				
338	EN15721	0.4272		2.08	
343	EN15701	0.384		0.88	
357 360	EN15721 EN15721	0.3610		0.88	
391	21110721				
396	=111==01				
402 444	EN15721 EN15721	0.285868 0.3751		-1.84 0.64	
468	EN15721	0.3731		-0.42	
496	EN15721	0.2996		-1.46	
511					
541 551	INH-1313	0.3622		0.28	
554	11411-1010				
556					
631					
633 657	INH-02	0.2965		-1.54	
663	1111 02				
823					
840 902					
913	INH-001	0.518	R(0.01)	4.60	
922	INH-001	0.3837	( /	0.88	
1047	EN15721	0.347		-0.14	
1191 1201	EN15721	0.294		 -1.61	
1276	EN15721	0.337		-0.42	
1359	EN15721	0.3348		-0.48	
1397	EN15721	0.392		1.11	
1446 1468	EN15721 EN15721	0.3682 0.34		0.45 -0.34	
1523	21110721				
1563	EN15721	0.351		-0.03	
1605 1656	EN15721	0.31		 -1.17	
1667	EN15721	0.351	С	-0.03	first reported 0.173
1710	EN15721	0.376		0.66	·
1726	EN15721	0.3387		-0.37	
1727 1835	EN15721 EN15721	0.3748 0.3562		0.63 0.11	
1852	EN15721	0.3464		-0.16	
1919	EN15721	0.2959		-1.56	
1996					
7002 7015					
7016					
	normality	OK			
	normality n	OK 31			
	outliers	1			
	mean (n)	0.35210			
	st.dev. (n)	0.036919			
	R(calc.) st.dev.(EN15721:13)	0.10337 0.036065			
	R(EN15721:13)	0.10098			

Page 38 of 55 Fuel/Bio-Ethanol iis17C15



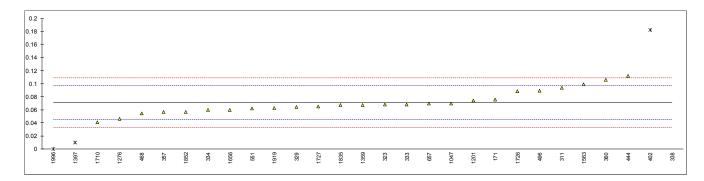


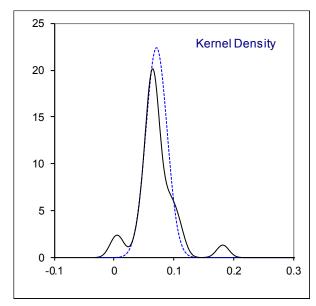
Fuel/Bio-Ethanol iis17C15 page 39 of 55

## Determination of Impurities acc. to EN15721 on sample #17240; results in %M/M

lab	method	value	mark	z(targ)	remarks
52					
62					
150					
169 171	EN15721	0.076		0.38	
174	LIVISIZI	0.070			
175					
230					
311	EN15721	0.094	С	1.80	first reported 0.551
323	EN15721	0.068		-0.25	
329 333	EN15721 EN15721	0.0640 0.068		-0.56 -0.25	
334	EN15721	0.0600	С	-0.23	first reported 0.453
337			· ·		mot reported across
338	EN15721	0.4939	ex	33.28	result excluded: probably included higher alcohols
343	EN45704				
357	EN15721	0.057		-1.11	
360 391	EN15721	0.1062		2.76	
396					
402	EN15721	0.182	C,ex	8.73	first reported 0.27398, result excluded: outlier in MeOH + EtOH
444	EN15721	0.1117		3.19	
468	EN15721	0.0547		-1.29	
496 511	EN15721	0.0891		1.42	
541					
551	INH-1313	0.0619		-0.73	
554					
556					
631 633					
657	INH-02	0.0695		-0.13	
663					
823					
840					
902 913					
922					
1047	EN15721	0.07		-0.09	
1191			_		
1201	EN15721	0.0740	С	0.23	first reported 0.376
1276 1359	EN15721 EN15721	0.046 0.06740		-1.98 -0.29	
1397	EN15721	0.010	ex	-4.81	result excluded: outlier in MeOH
1446					
1468					
1523	EN145704	0.000		2.10	
1563 1605	EN15721	0.099		2.19	
1656	EN15721	0.06		-0.88	
1667					
1710	EN15721	0.041		-2.37	
1726 1727	EN15721 EN15721	0.08837 0.0653		1.36 -0.46	
1835	EN15721	0.0671		-0.32	
1852	EN15721	0.0571		-1.10	
1919	EN15721	0.0626		-0.67	
1996	IMPCA001Mod.	0.0003955	ex	-5.57	result excluded: outlier in MeOH
7002 7015					
7016					
	normality	OK			
	n outliers	25 0 (+4 ex)			
	mean (n)	0 (+4 ex) 0.07112			
	st.dev. (n)	0.017769			
	R(calc.)	0.04975			
	st.dev.(Horwitz (n=9))	0.012705			
	R(Horwitz (n=9))	0.03557			

Page 40 of 55 Fuel/Bio-Ethanol iis17C15



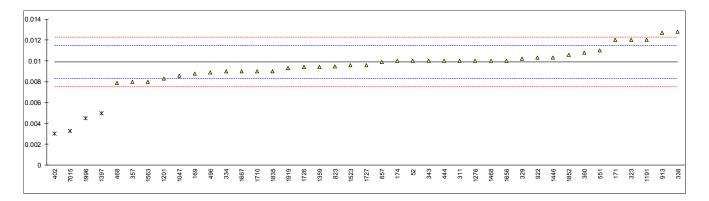


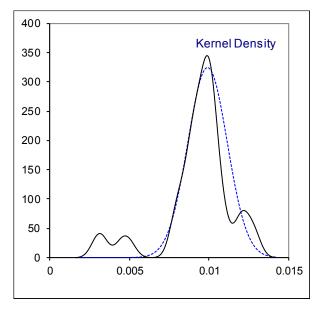
Fuel/Bio-Ethanol iis17C15 page 41 of 55

## Determination of Methanol on sample #17240; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D5501	0.01		0.14	
62 150					
169	INH-02	0.00879		-1.39	
171	EN15721	0.012		2.66	
174	D5501	0.01		0.14	
175 230					
311	EN15721	0.010		0.14	
323	EN15721	0.012		2.66	
329	EN15721	0.0102		0.39	
333 334	EN15721 EN15721	<0.100 0.009		 -1.12	
337					
338	EN15721	0.0128		3.67	
343 357	EN15721 EN15721	0.010 0.008		0.14 -2.38	
360	EN15721	0.0108		1.15	
391					
396 402	EN15721	0.003	C,R(0.05)	-8.69	first reported 0.001753
444	EN15721	0.003	C,11(0.03)	0.14	mat reported 0.001700
468	EN15721	0.0079		-2.51	
496	EN15721	0.0089		-1.25	
511 541					
551	INH-1313	0.0110		1.40	
554					
556					
631 633					
657	INH-02	0.0099		0.01	
663	D5501	<0.01		0.40	
823 840	D5501	0.0095		-0.49 	
902					
913	INH-0001	0.0127		3.55	
922 1047	INH-0001 EN15721	0.0103 0.0086		0.52 -1.63	
1191	D5501	0.000		2.66	
1201	EN15721	0.0083		-2.00	
1276 1359	EN15721 EN15721	0.010 0.00941		0.14 -0.60	
1397	EN15721	0.00541	R(0.05)	-6.17	
1446	EN15721	0.0103	(===,	0.52	
1468	EN15721	0.01		0.14	
1523 1563	D5501 EN15721	0.009582 0.008		-0.39 -2.38	
1605	21110721				
1656	EN15721	0.01		0.14	
1667 1710	EN15721 EN15721	0.009 0.009		-1.12 -1.12	
1726	EN15721	0.00940		-0.62	
1727	EN15721	0.0096		-0.36	
1835	EN15721 EN15721	0.009		-1.12	
1852 1919	EN15721 EN15721	0.0106 0.0093		0.90 -0.74	
1996	IMPCA001Mod.	0.0045	R(0.05)	-6.80	
7002			0.0(0.05)		first assessment of 0.005
7015 7016		0.0033	C,R(0.05)	-8.31 	first reported 0.005
. 5 10					
	normality	OK			
	n outliers	37 4			
	mean (n)	0.00989			
	st.dev. (n)	0.001232			
	R(calc.) st.dev.(Horwitz)	0.00345 0.000792			
	R(Horwitz)	0.000792			
	For comparison				
	R(D5501:12)	0.013733			application range: 0.01 – 0.6 %M/M
	R(EN15721:13)	-0.00299			application range: 0.1 – 3 %M/M

Page 42 of 55 Fuel/Bio-Ethanol iis17C15



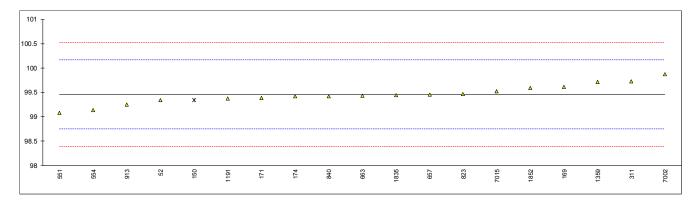


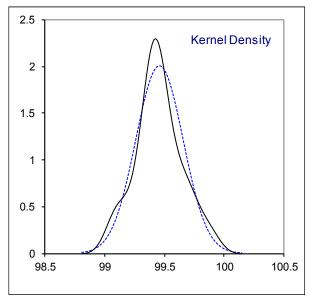
Fuel/Bio-Ethanol iis17C15 page 43 of 55

## Determination of Ethanol acc. to ASTM D5501 on sample #17240; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D5501	99.34	mark	-0.33	Telliares
62	D330 I			-0.55	
150	D5501	99.34	ex	-0.33	result excluded as Ethanol %M/M is < Ethanol %V/V
169	INH-02	99.61	CA.	0.43	result excluded as Ethanol ///////
171	D5501	99.39		-0.19	
174	D5501	99.42		-0.11	
175	D0001				
230					
311	D5501	99.72		0.74	
323	D0001				
329					
333					
334					
337					
338					
343					
357					
360					
391					
396					
402					
444					
468					
496					
511					
541					
551	D5501	99.08		-1.06	
554	D5501	99.13		-0.92	
556					
631					
633					
657	D5501	99.4561		0.00	
663	D5501	99.434		-0.07	
823	D5501	99.4676		0.03	
840	D5501	99.424		-0.09	
902					
913	D5501	99.25		-0.58	
922					
1047					
1191	D5501	99.378		-0.22	
1201					
1276	DEEOA			0.74	
1359	D5501	99.71		0.71	
1397					
1446					
1468 1523					
1563 1605					
1656					
1667					
1710					
1716					
1727					
1835	D5501	99.447		-0.03	
1852	EN15721	99.5859		0.36	
1919	LITIOILI				
1996					
7002		99.874		1.17	
7015		99.52		0.18	
7016					
	normality	OK			
	n	18			
	outliers	0 (+1 ex)			
	mean (n)	99.45760			
	st.dev. (n)	0.198610			
	R(calc.)	0.55611			
	st.dev.(D5501:12)	0.355124			
	R(D5501:12)	0.99435			

Page 44 of 55 Fuel/Bio-Ethanol iis17C15



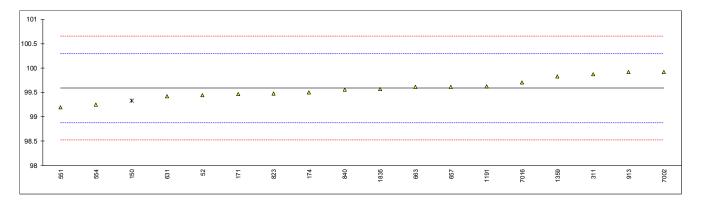


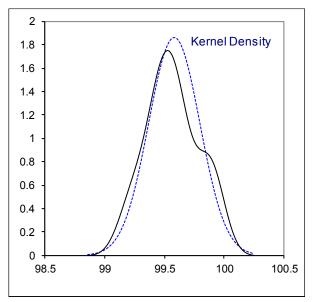
Fuel/Bio-Ethanol iis17C15 page 45 of 55

## Determination of Ethanol acc. to ASTM D5501 on sample #17240; results in %V/V

lab	method	value	mark	z(targ)	remarks
52	D5501	99.44	IIIai K	-0.41	Tomarka
62					
150	D5501	99.33	ex	-0.72	result excluded as Ethanol %M/M is < Ethanol %V/V
169 171	D5501	99.46		-0.35	
174	D5501	99.50		-0.33	
175					
230					
311 323	D5501	99.87 		0.80	
323					
333					
334					
337 338					
343					
357					
360					
391					
396 402					
444					
468					
496					
511 541					
551	D5501	99.19		-1.11	
554	D5501	99.25		-0.95	
556	D5504			0.47	
631 633	D5501	99.42		-0.47 	
657	D5501	99.6112		0.07	
663	D5501	99.610		0.07	
823	D5501	99.4760		-0.31	
840 902	D5501	99.557 		-0.08	
913	D5501	99.92		0.94	
922					
1047	D5504				
1191 1201	D5501	99.627		0.12	
1276					
1359	D5501	99.83		0.69	
1397					
1446 1468					
1523					
1563					
1605					
1656 1667					
1710					
1726					
1727	DEEOA			0.04	
1835 1852	D5501	99.571 		-0.04 	
1919					
1996					
7002 7015		99.923		0.95	
7015		99.7		0.32	
	normality	OK			
	n outliers	17 0 (+1 ex)			
	mean (n)	99.58559			
	st.dev. (n)	0.214508			
	R(calc.)	0.60062			
	st.dev.(D5501:12) R(D5501:12)	0.354850 0.99358			
	11(00001.12)	0.99000			

Page 46 of 55 Fuel/Bio-Ethanol iis17C15



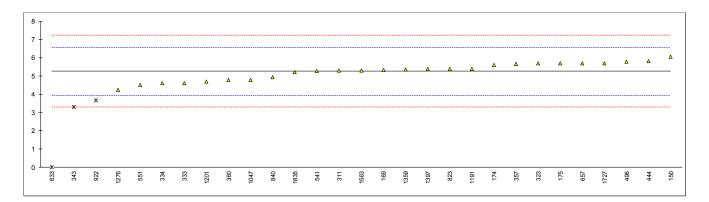


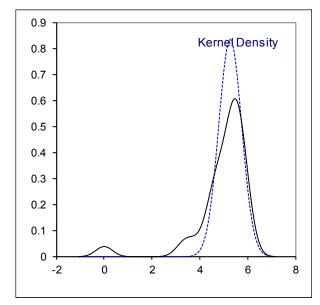
Fuel/Bio-Ethanol iis17C15 page 47 of 55

## Determination of Chlorides, Inorganic as CI on sample #17241; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52					
62 150	D7328	6.06		1.22	
169	D7328 D7319	5.322		0.09	
171	D7210	 5 G		0.52	
174 175	D7319 D7319	5.6 5.7		0.52 0.67	
230	INH-20404	>2			
311 323	EN15492 EN15492	5.3 5.7		0.06 0.67	
329	LIV10402				
333	EN15492	4.6		-1.01 1.01	
334 337	EN15492	4.6 		-1.01 	
338	<b>51115100</b>		50/0 ==:		
343 357	EN15492 EN15492	3.3 5.67	DG(0.05)	-2.99 0.62	
360	EN15492	4.78		-0.73	
391					
396 402					
444	EN15492	5.8		0.82	
468 496	EN15492	 5.79		0.81	
511	LINTUMBL	5.79			
541	D7328	5.26		0.00	
551 554	D7319	4.5 		-1.16 	
556					
631 633	D512	0	ΔV	-8.02	result excluded: 0 is not a value
657	D7328	5.7	ex	0.67	TOSUIT GADIAUGU. O IS HOL A VAIUG
663		 E 4		0.21	
823 840	D7319 D7319	5.4 4.94		0.21 -0.49	
902					
913 922	D7328	3.65	DG(0.05)	 -2.46	
1047	EN15492	4.78	(٥.٥٥)	-0.73	
1191	EN15492	5.4		0.21	
1201 1276	EN15492 EN15492	4.7 4.23		-0.86 -1.57	
1359	EN15492	5.360		0.15	
1397 1446	EN15492	5.38		0.18	
1446					
1523	ENAFACO				
1563 1605	EN15492	5.3		0.06	
1656	EN15492	<1		<-6.26	false negative test result?
1667 1710					
1726					
1727	EN15492	5.7		0.67	
1835 1852	In house	5.21 		-0.08	
1919					
1996 7002					
7015					
7016					
	normality	OK			
	n	26			
	outliers mean (n)	2 (+1 ex) 5.261			spike 5.70 (recovery <92%)
	mean (n) st.dev. (n)	0.4788			J. 10 (1600VGI y \32 /0)
	R(calc.)	1.341			
	st.dev.(Horwitz) R(Horwitz)	0.6556 1.836			
	For comparison				
	R(EN15492:12) R(D7319:17)	0.895 1.334			application range: 1 – 30 mg/kg application range: 0.75 – 50 mg/kg
	14(01018.11)	1.007			application range. 0.70 - 50 mg/kg

Page 48 of 55 Fuel/Bio-Ethanol iis17C15



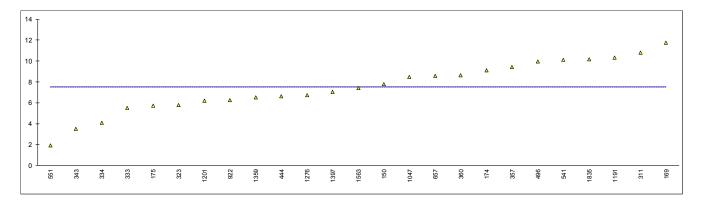


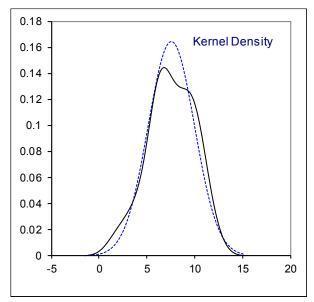
Fuel/Bio-Ethanol iis17C15 page 49 of 55

# Determination of Sulphate as SO<sub>4</sub> on sample #17241; results in mg/kg

lab	method	value	mark z(targ)	remarks
52				
62				
150	D7328	7.76		
169	D7319	11.774		
171 174	D7319	9.1		
175	D7319 D7319	5.7		
230	B1010			
311	EN15492	10.8		
323	EN15492	5.8		
329	=111=100			
333	EN15492	5.5 4.1		
334 337	EN15492			
338				
343	EN15492	3.5		
357	EN15492	9.43		
360	EN15492	8.64		
391 396				
402				
444	EN15492	6.6		
468				
496	EN15492	9.98		
511 541	D7328	 10.10		
551	D7328 D7319	1.9		
554	21010			
556				
631				
633 657	D7328	8.6		
663	D1320	6.0		
823				
840				
902				
913 922	D7328	6.23		
1047	EN15492	8.48		
1191	EN15492	10.3		
1201	EN15492	6.2		
1276	EN15492	6.71		
1359	EN15492	6.541		
1397 1446	EN15492	7.03		
1468				
1523				
1563	EN15492	7.4		
1605	EN45400			
1656 1667	EN15492	<1 		false negative test result?
1710				
1726				
1727				
1835	In house	10.16		
1852 1919				
1919				
7002				
7015				
7016				
	normality	OK		
	normality n	OK 25		
	outliers	0		<u>spike</u>
	mean (n)	7.533		12.53 mg SO₄/kg
	st.dev. (n)	2.4300		
	R(calc.)	6.804		
	st.dev.(EN15492:12) R(EN15492:12)	(0.6116) (1.712)		application range: 1 – 20 mg/kg
	For comparison	(1.1 12)		approation range. 1 20 mg/kg
	R(D7319:17)	(5.753)		application range: 1 – 20 mg/kg
	R(D7328:16)	(2.356)		application range: 0.55 – 20 mg/kg

Page 50 of 55 Fuel/Bio-Ethanol iis17C15



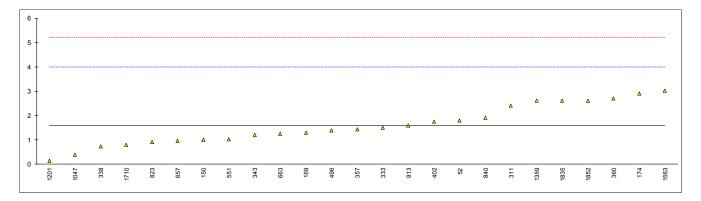


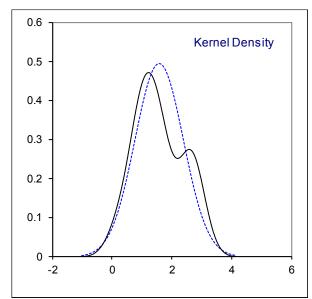
Fuel/Bio-Ethanol iis17C15 page 51 of 55

# Determination of total Sulphur on sample #17241; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D5453	1.8	murk	0.17	Tomaria
62	50700	1.0			
150	D5453	1.0		-0.49	
169	D5453	1.3	С	-0.25	
171					
174	D5453	2.9		1.08	
175					
230					
311	D5453	2.4		0.67	
323	EN15486	< 5			
329 333	ISO20846	1.5		-0.08	
334	13020040	1.5		-0.06	
337					
338	ISO20846	0.72		-0.73	
343	D5453	1.2		-0.33	
357	D5453	1.44		-0.13	
360	EN15486	2.7		0.92	
391					
396	10000010	4.70			
402	ISO20846	1.76		0.14	
444	EN15485				
468 496	ISO20846	<2 1.39		 -0.17	
511	10020070	1.39		-0.17	
541	D5453	<1.0			
551	D5453	1.03		-0.47	
554					
556					
631					
633					
657	D5453	0.949		-0.54	
663	D5453	1.25		-0.29	
823	D5453	0.9 1.90		-0.58 0.25	
840 902	D5453	1.90		0.25	
913	D5453	1.6		0.00	
922	D5453	<1.0			
1047	EN15486	0.4		-0.99	
1191					
1201	EN15485	0.15		-1.20	
1276	EN15405	2.60		0.02	
1359 1397	EN15485	2.60		0.83	
1446					
1468					
1523					
1563	EN15486	3.01		1.17	
1605	<b></b>				
1656	EN15486	<1			
1667 1710	ISO20846	0.8		-0.66	
1726	10020040			-0.00	
1727					
1835	EN15486	2.6		0.83	
1852	ISO20846	2.60		0.83	
1919					
1996					
7002 7015					
7015					
7010					
	normality	OK			
	n	25			
	outliers	0			
	mean (n)	1.596			
	st.dev. (n)	0.8086			
	R(calc.)	2.264 1.2042			
	st.dev.(EN15485:07) R(EN15485:07)	3.372			application range: 7 – 20 mg/kg
	For comparison	0.012			application range. 7 – 20 mg/kg
	R(EN15486:07)	1.925			application range: 5 – 20 mg/kg
	R(D5453:16e1)	0.823			at a concentration < 400 mg/kg, application range: 1-8000 mg/kg

Page 52 of 55 Fuel/Bio-Ethanol iis17C15





Fuel/Bio-Ethanol iis17C15 page 53 of 55

### **APPENDIX 2**

### Number of participating laboratories per country:

- 1 lab in ARGENTINA
- 1 lab in AUSTRIA
- 3 labs in BELGIUM
- 3 labs in BRAZIL
- 2 labs in BULGARIA
- 2 labs in CANADA
- 1 lab in CROATIA
- 1 lab in CZECH REPUBLIC
- 2 labs in FINLAND
- 4 labs in FRANCE
- 2 labs in GERMANY
- 1 lab in HUNGARY
- 1 lab in INDIA
- 3 labs in IRAN, Islamic Republic of
- 3 labs in ITALY
- 1 lab in MAURITIUS
- 4 labs in NETHERLANDS
- 1 lab in PAKISTAN
- 1 lab in PERU
- 2 labs in PHILIPPINES
- 1 lab in POLAND
- 1 lab in ROMANIA
- 1 lab in SINGAPORE
- 1 lab in SOUTH KOREA
- 4 labs in SPAIN
- 2 labs in SWEDEN
- 1 lab in THAILAND
- 2 labs in TURKEY
- 3 labs in UNITED KINGDOM
- 5 labs in UNITED STATES OF AMERICA
- 1 lab in VIETNAM

Page 54 of 55 Fuel/Bio-Ethanol iis17C15

#### **APPENDIX 3**

### Abbreviations:

C = final test result after checking of first reported suspect test result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test

DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test

R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
E = probably an error in calculations

U = test result probably reported in a different unit
W = test result withdrawn on request participant
ex = test result excluded from statistical evaluation

n.a. = not applicable
n.e = not evaluated
n.d. = not detected
fr. = first reported
SDS = Safety Data Sheet

#### Literature:

- 1 iis, Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
- W. Horwitz and R. Albert, J. AOAC Int., Vol. 79, 3, p. 589, (1996)
- 3 ASTM E178:02
- 4 ASTM E1301:95(2003)
- 5 ISO 5725-86
- 6 ISO 5725, parts 1-6, 1994
- 7 ISO 13528
- 8 M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 9 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 10 IP 367/84
- 11 DIN 38402 T41/42
- 12 P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
- 13 J.N. Miller, Analyst, 118, 455, (1993)
- 14 Analytical Methods Committee Technical brief, No 4.January 2001
- 15 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, Analyst, 2002, 127, page 1359-1364.
- Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)
- 17 M.A. Gonçalves et.al., Sensors and Actuators B158, (2011), pages 327-332

Fuel/Bio-Ethanol iis17C15 page 55 of 55