Results of Proficiency Test Ethanol (Bio / Fuel grade) December 2020

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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1 Introduction

Since 1995 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Ethanol (Bio / Fuel grade) in accordance with the latest version of the EN15376 and ASTM D4806 every year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis of Ethanol (Bio / Fuel grade).

In this interlaboratory study 59 laboratories in 32 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the Ethanol (Bio / Fuel grade) proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send three different samples of Ethanol (Bio / Fuel grade), one bottle of 1L labelled #20245 for regular analyzes, one bottle of 50mL labelled #20246 for Inorganic Chloride, Sulfate and total Sulfur determination and one bottle of 250mL labelled #20249 for determination of Nonvolatile matter only.

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

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

For the preparation of the sample for the regular analyzes a batch of approximately 100 liters of Ethanol (Bio / Fuel grade) was obtained from a European supplier and spiked with Bitrex. After homogenization 96 amber glass bottles of 1L were filled and labelled #20245. The homogeneity of the subsamples was checked by determination of Density in accordance with ASTM D4052 at 20°C and Water in accordance with E203 on 8 stratified randomly selected subsamples.

	Density at 20°C in kg/L	Water in %M/M	
Sample #20245-1	0.78975	0.117	
Sample #20245-2	0.78978	0.119	
Sample #20245-3	0.78976	0.118	
Sample #20245-4	0.78976	0.119	
Sample #20245-5	0.78976	0.118	
Sample #20245-6	0.78976	0.118	
Sample #20245-7	0.78976	0.118	
Sample #20245-8	0.78976	0.119	

Table 1: homogeneity test results of subsamples #20245

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

	Density at 20°C in kgL	Water in %M/M
r (observed)	0.00002	0.002
reference test method	ISO12185:96	E203:16
0.3 x R (reference test method)	0.00015	0.023

Table 2: evaluation of the repeatabilities of subsamples #20245

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

For the preparation of the sample for the determination of Inorganic Chloride, Sulfate and total Sulfur in Ethanol (Bio / Fuel grade) a batch of about 5L Ethanol (Bio / Fuel grade) was spiked with Sodium Chloride (NaCl) and Sodium Sulfate (Na2SO4) dissolved in water. After homogenization 90 PE bottles of 50mL were filled and labelled #20246.

The homogeneity of the subsamples was checked by determination of Chloride as Cl in accordance with EN15492 on 7 stratified randomly selected subsamples.

	Chloride as Cl in mg/kg
sample #20246-1	2.1
sample #20246-2	2.0
sample #20246-3	1.9
sample #20246-4	1.9
sample #20246-5	1.9
sample #20246-6	2.0
sample #20246-7	2.0

Table 3: homogeneity test results of subsamples #20246

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

	Chloride as Cl in mg/kg
r (observed)	0.21
reference test method	D7319:17
0.3 x R (reference test method)	0.20

Table 4: evaluation of the repeatability of subsamples ##20246

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

For the preparation of the sample for the determination of Nonvolatile matter in Ethanol (Bio / Fuel grade) a batch of about 25L Ethanol (Bio / Fuel grade) was spiked with Sodium Chloride (NaCl) dissolved in water. After homogenization 96 amber glass bottles of 250mL were filled and labelled #20249.

The homogeneity of the subsamples was checked by determination of Nonvolatile matter in accordance with EN15691 on 7 stratified randomly selected subsamples.

	Nonvolatile matter in mg/100mL
sample #20249-1	34
sample #20249-2	34
sample #20249-3	33
sample #20249-4	33
sample #20249-5	33
sample #20249-6	34
sample #20249-7	33

Table 5: homogeneity test results of subsamples #20249

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

	Nonvolatile matter in mg/100mL
r (observed)	1.5
reference test method	EN15691:09
0.3 x R (reference test method)	1.9

Table 6: evaluation of the repeatability of subsamples ##20249

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

To each of the participating laboratories one sample labelled #20245, one sample labelled #20246 and one sample labelled #20249 was sent on November 11, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Ethanol (Bio / Fuel grade) packed in amber glass bottles and PE bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine on sample #20245: Total Acidity as Acetic Acid, Appearance, Copper as Cu, Density at 20°C, Electrical Conductivity at 25°C, Nonvolatile matter, Nitrogen, pHe (LiCl and KCl), Phosphorus as P, Water (Coulometric and Volumetric), Ethanol incl. higher alcohols (acc. EN15721), Higher alcohols (acc. EN15721), Impurities (acc. EN15721), Methanol, Ethanol by mass and by volume (acc. ASTM D5501), Bitrex and Gum (solvent washed).

On sample #20246 it was requested to determine Inorganic Chloride as CI, Sulfate as SO₄ and Sulfur.

On sample #20249 it was requested to determine Nonvolatile matter.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

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

3 RESULTS

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

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

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

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

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

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

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

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) 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. 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

```
z_{\text{(target)}} = \text{(test result - average of PT)} / \text{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. Therefore, 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**

Some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another three weeks. When considering the test results of the three samples together one participant reported test results after the final reporting date and ten participants did not report any test results. Not all participants were able to report all tests requested.

In total 49 participants reported 507 numerical test results. Observed were 18 outlying test results, which is 3.6%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER SAMPLE AND PER TEST

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

Unfortunately, a suitable reference test method, providing the precision data, is not available for all determinations. For these tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D5501) and an added designation for the year that the test method was adopted or revised (e.g. D5501:20).

Sample #20245

<u>Total Acidity</u>: This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in agreement with the

requirements of EN15491:07 and ASTM D1613:17

Appearance: This determination was not problematic. All reporting participants agreed

about the appearance as Pass (Clear and Bright).

Copper as Cu: Almost all of the participants reported a test result near or below the

application range of the method EN15488:07. Therefore, no z-scores were

calculated.

<u>Density at 20°C</u>: This determination was not problematic. Two statistical outliers were

observed. The calculated reproducibility after rejection of the statistical

outliers is in agreement with the requirements of ISO12185:96.

Electrical Conductivity at 25°C: This determination was very problematic. One statistical

outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of

EN15938:10.

Nonvolatile matter: This determination was not problematic. All participants reported a test

result near or below the application range of the method EN15691:09.

Therefore, no z-scores were calculated.

Nitrogen: This determination was problematic. No statistical outliers were observed.

The calculated reproducibility is not in agreement with the requirements of

ASTM D4629:17.

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

the pHe determined with a KCl electrode. Two test methods are available for the determination of the pHe of Ethanol: EN15490, that describes the use of a LiCl electrode and ASTM D6423, that describes the use of a KCl

electrode.

pHe (LiCl): This determination may be problematic. Only 5 participants reported a test

result. Due to the large variation in the test results it was decided not to

calculate z-scores.

<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 D6423:20a.

<u>Phosphorus as P</u>: This determination was not problematic. Almost all of the participants

reported a test result near or below the application range of the method

EN15487:07. Therefore, no z-scores were calculated.

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

<u>Water, Volumetric</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM E203:16 and EN15692:09.

GC general:

The test results reported for the Ethanol content is depending on the test method used by the laboratory. Test method EN15721 uses a different definition for Ethanol than ASTM D5501. 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%" but in Ethanol content.

This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full 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. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of EN15721:13.

Impurities (EN15721): In EN15721 the impurity content is defined as: content of all components except for Ethanol%, Methanol% and the higher alcohols%. This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility using the Horwitz equation based on nine components.

Methanol:

This determination may be problematic depending on the test method used. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility using the Horwitz equation, but is in agreement with the requirements of ASTM D5501:20. A negative value for the reproducibility is found at this concentration level for test method EN15721:13 which is theoretically not correct.

Ethanol (D5501): This determination was not problematic for Ethanol by mass and Ethanol

by volume. No statistical outliers were observed but in total eight test results were excluded. 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 D5501:20.

Bitrex: This determination may be problematic. Only 5 participants reported a test

result. Due to the large variation in the test results it was decided not to

calculate z-scores.

<u>Gum (solvent washed):</u> This determination was not problematic. Almost all of the participants

agreed on a test result <1 mg/100mL. Therefore, no z-scores were

calculated.

Sample #20246

Inorganic Chloride: This determination was problematic. No statistical outliers were observed.

The calculated reproducibility is not in agreement with the requirements of

ASTM D7319:17 or EN15492:12.

Sulfate as SO₄: This determination may be problematic depending on the test method used.

No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7319:17, but is not in

agreement with the requirements of EN15492:12 or ASTM D7328:17.

Sulfur: 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 EN15485:07, EN15486:07

and ASTM D5453:19a.

Sample #20249

Nonvolatile matter: This determination was not problematic. Three statistical outliers were

observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN15691:09 and ASTM

D1353:13.

4.2 Performance evaluation for the group of Laboratories

A comparison has been made between the reproducibility as declared by the reference test method or as declared by the estimated target reproducibility calculated with the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM, EN and ISO test methods) or estimated using the Horwitz equation are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acidity as Acetic Acid	mg/kg	37	16.5	10.4	13.7
Appearance		41	Pass	n.a.	n.a.
Copper as Cu	mg/kg	21	<0.07	n.e.	n.e.
Density at 20°C	kg/L	44	0.7898	0.0002	0.0005
Electrical Conductivity at 25°C	μS/cm	29	1.18	0.42	0.21
Nonvolatile matter	mg/100mL	28	<10	n.e.	n.e.
Nitrogen	mg/kg	16	1.69	1.39	1.06
pHe (LiCl)		5	6.99	2.02	(0.67)
pHe (KCI)		15	7.25	1.09	1.08
Phosphorus as P	mg/L	18	<0.15	n.e.	n.e.
Water, Coulometric	%M/M	38	0.127	0.021	0.021
Water, Volumetric	%M/M	25	0.121	0.020	0.078
Ethanol + higher alcohols (EN15721)	%M/M	30	99.924	0.048	0.045
Higher alcohols (EN15721)	%M/M	29	0.125	0.033	0.034
Impurities (EN15721)	%M/M	24	0.070	0.040	0.035
Methanol	%M/M	26	0.005	0.0018	0.0013
Ethanol (D5501)	%M/M	12	99.733	0.228	0.993
Ethanol (D5501)	%V/V	13	99.787	0.258	0.992
Bitrex	mg/kg	5	<50	n.e.	n.e.
Gum (solvent washed)	mg/100mL	13	<1	n.e.	n.e.

Table 7: reproducibilities of tests on sample #20245

Results between brackets should be used with due care

Parameter	unit	n	average	2.8 * sd	R(lit)
Inorganic Chloride as Cl	mg/kg	24	1.9	0.9	0.6
Sulfate as SO ₄	mg/kg	21	1.1	1.3	1.5
Sulfur	mg/kg	23	1.2	0.6	3.3
Nonvolatile matter	mg/100mL	28	33.5	5.7	6.2

Table 8: reproducibilities of tests on sample #20246 and sample #20249

Without further statistical calculations, it can be concluded that for many tests there is a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2020 WITH PREVIOUS PTS

	December 2020	November 2019	December 2018	December 2017	December 2016
Number of reporting laboratories	49	51	53	59	57
Number of test results	507	457	473	537	476
Number of statistical outliers	18	16	14	22	31
Percentage of statistical outliers	3.6%	3.5%	3.0%	4.1%	6.5%

Table 9: 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 to the requirements of the reference test methods. The conclusions are given in the following table.

Parameter	December 2020	November 2019	December 2018	December 2017	December 2016
Acidity, Total as Acetic Acid	++	+	+	-	+
Density at 20°C	++	++	++	++	++
Electrical Conductivity at 25°C		-			
Nonvolatile matter	n.e./ +	n.e.	()	()	()
Nitrogen	-	-	-	-	
рНе	+/-	+/-	-	-	+/-
Water, Coulometric	+/-	-	+/-	+/-	+
Water, Volumetric	+	+	+	++	++
Ethanol + higher alcohols (EN15721)	+/-	++		-	-
Higher alcohols (EN15721)	+/-	+	+/-	+/-	-
Impurities (EN15721)	-	++	-	-	
Methanol	-			-	-
Ethanol (D5501)	++	++	+	+	++
Inorganic Chloride as Cl	-	-	-	+	(++)
Sulfate as SO ₄	+	-	-		
Sulfur	++	++	+	+	+

Table 9: comparison determinations against the reference test methods

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

The following performance categories were used:

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

+ : group performed better than the reference test method

+/- : group performance equals the reference test method

- : group performed worse than the reference test method

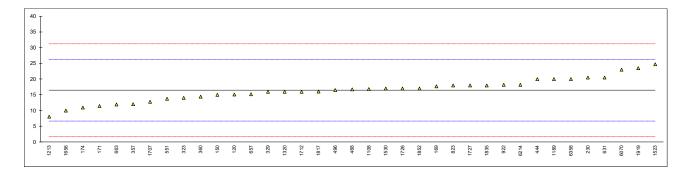
-- : group performed much worse than the reference test method

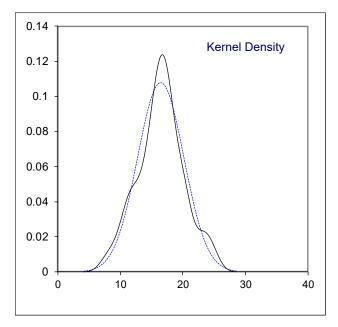
n.e. : not evaluated

APPENDIX 1

Determination of Total Acidity as Acetic Acid on sample #20245; results in mg/kg

		· · · · · · · · · · · · · · · · · · ·	•	•	sample #20245; results in mg/kg
lab 52	method EN15401	<30	mark	z(targ)	remarks
52 120	EN15491 D1613	<30 15.1		-0.28	
150	D1613	15.1		-0.20	
169	D7795	17.7		0.25	
171	D1613	11.4		-1.04	
174	D1613	11		-1.12	
175					
230	D1613	20.5		0.82	
311	EN15491	<30			
323	EN15491	14		-0.51	
329	EN15491	16		-0.10	
333 334	EN15491 EN15491	<30 <30			
337	LIVIOTOI				
343	EN15491	<30			
357	EN15491	12		-0.92	
360	EN15491	14.4		-0.43	
396					
444	EN15491	20		0.72	
468	EN15491	16.7		0.04	
495 496	EN15401	 16.5		0.00	
496 511	EN15491	10.5		0.00	
541					
551	D1613	13.7		-0.57	
554					
558					
621					
631	D1613	20.5		0.82	
633					
634 657	D1613	15.2203		-0.26	
663	D1613	11.9	С	-0.20	first reported 0.00119 mg/kg
823	D1613	18	Ü	0.31	mot reported 0.00 Fro mg/kg
913					
922	D1613	18.2		0.35	
1108	EN15491	16.776		0.06	
1189	EN15491	20		0.72	
1213	D1613	8.1		-1.71	
1320 1397	ISO17315	16 		-0.10 	
1438					
1523	D1388	24.743		1.69	
1530	EN15491	17		0.10	
1656	EN15491	10		-1.33	
1707	D1613	12.8		-0.75	
1712	EN15491	16		-0.10	
1726	EN15491	17		0.10	
1727	EN15491	18		0.31	
1817 1835	ISO1388 EN15491	16.08 18		-0.08 0.31	
1852	EN15491 EN15491	17		0.31	
1919	EN15491	23.5		1.43	
6070	D1613	23.3		1.33	
6072					
6214	EN15491	18.2		0.35	
6297					
6341	EN45404			0.70	
6358	EN15491	20		0.72	
	normality	OK			
	n	37			
	outliers	0			
	mean (n)	16.487			
	st.dev. (n)	3.6977			
	R(calc.)	10.353			
	st.dev.(EN15491:07)	4.8929			1. 1. 00 450 11
	R(EN15491:07)	13.7			application range: 30 – 150 mg/kg
	compare				application range: <500 mg/kg
	R(D1613:17)	14			application range. <p(1) kg<="" mg="" td=""></p(1)>





Determination of Appearance on sample #20245;

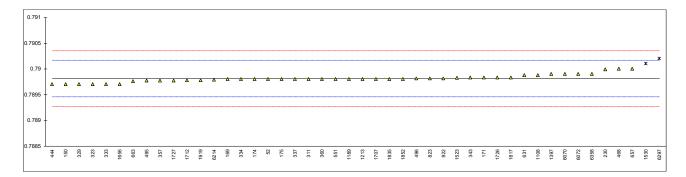
lah	mothod	value	mort	=/to==)	romorko
lab	method	Value	mark	z(targ)	remarks
52 120	EN15769	Clear & Coloured			
120	D4176	C&B			
150 169	E2680 Visual	Pass CBFSM			
171	Visual	CFSM			
174	Visual	Clear & Free			
175	D4176	Pass			
230	Visual	C&B			
311	EN15769	clear & colourless			
323	D4176	c&b			
329	Visual	clear			
333	Visual	Clear and bright			
334	EN15769	clear and bright FFSM			
337	Visual	colourless			
343	Visual	clear & bright			
357	E2680	Pass			
360	EN15769	Clear and Colourless			
396					
444	EN15769	Pass			
468	EN15769	C&C			
495	EN15769	Clear and colourless			
496	Visual	clear&bright			
511					
541					
551	E2680	Pass			
554					
558					
621					
631	Visual	clear & bright			
633					
634					
657	E2680	Pass			
663	Visual	Bright & Clear			
823	E2680	Pass			
913					
922	Visual	Clear and bright			
1108	N.C I				
1189	Visual	C/B			
1213					
1320	EN16360				
1397	EN15769	colourless, bright			
1438 1523					
1523	Vioual				
1656	Visual EN15769	bright and clear pass			
1707	Visual	C&B			
1712	EN15769	C&B			
1712	EN15769	Clear&Colorless			
1727	Visual	Clear&Colorless			
1817	Visual	PASS			
1835	EN15769	C&C			
1852	Visual	clear & bright			
1919	= =:=::				
6070	Visual	Clear and Free			
6072					
6214	EN15769	Clear and colourless			
6297					
6341					
6358	EN15769	clear and colourless			
	n	41			
	mean	Pass (Clear and Bright)			

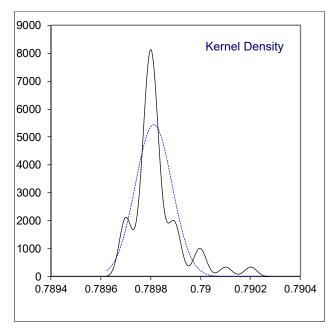
Determination of Copper as Cu on sample #20245; results in mg/kg

lab	method	value	mark z(targ)	remarks
52	EN15837	<0.050	mark Z(targ)	
120	LIN 10001			
150	D1688	<0.1		
169	D1688	<0.05		
171	2.000			
174				
175	D1688	< 0.05		
230				
311	EN15837	< 0.050		
323	EN15488	< 0.070		
329	EN15488	0.002		
333	EN15488	<0.07		
334				
337				
343	EN15837	<0,050		
357				
360	EN15837	< 0.050		
396				
444	EN15837	0.002		
468	EN15488	<0,07		
495				
496				
511				
541	INII I 00 47			
551	INH-2047	<0.04		
554 558				
621				
631	D1688	<0.05		
633	D 1000	-0.00		
634				
657				
663	In house	0.0001		
823	UOP389	< 0.01		
913				
922	D1688	<0.05		
1108				
1189				
1213				
1320				
1397				
1438				
1523				
1530	D4600 A			
1656 1707	D1688-A	<0.1		
1707 1712	EN15488	 <0.07		
1712 1726	LIN 1 3400	<0,07		
1720				
1817				
1835	EN15837	<0.050		
1852				
1919				
6070	D1688	0.025		
6072				
6214	EN15488	0.0013		
6297				
6341				
6358	EN15488	<0,070		
	n	21		
	mean (n)	<0.07		
	application range of te	est method E	N15488:07: 0.07 - 0.20)mg/kg

Determination of Density at 20°C on sample #20245; results in kg/L

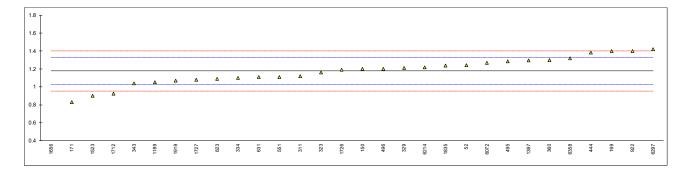
lab	method	value	mark	z(targ)	remarks
52	ISO12185	0.7898	HUIK	-0.07	TOTHATING
120	13012103	0.7090		-0.07	
150	D4052	0.7897		-0.63	
169	D4052	0.7898		-0.07	
171	D4052	0.78983		0.10	
174	D4052	0.7898		-0.07	
175	D4052	0.7898		-0.07	
230	D4052	0.78999		0.99	
311	D4052	0.7898		-0.07	
323	D4052	0.7897		-0.63	
329	D4052	0.7897		-0.63	
333	ISO12185	0.7897		-0.63	
334	ISO12185	0.7898		-0.07	
337	ISO12185	0.7898		-0.07	
343	ISO12185	0.78983	С	0.10	first reported 0.7904
357	D4052	0.78977		-0.24	
360	ISO12185	0.7898		-0.07	
396	D. 40-50				
444	D4052	0.7897		-0.63	
468	ISO12185	0.7900		1.05	
495	ISO12185	0.78977		-0.24	
496	ISO12185	0.78981		-0.01	
511					
541 551	D4052	0.7898		-0.07	
554	D4032	0.7696		-0.07	
558					
621					
631	D4052	0.78988		0.38	
633	D-1032				
634					
657	D4052	0.7900		1.05	
663	D4052	0.78976		-0.29	
823	ISO12185	0.78981		-0.01	
913					
922	D4052	0.78981		-0.01	
1108	D4052	0.78988		0.38	
1189	D4052	0.7898		-0.07	
1213	D4052	0.7898		-0.07	
1320					
1397	ISO12185	0.7899	С	0.49	first reported 789.9 kg/L
1438					
1523	D4052	0.7898245		0.07	
1530	ISO12185	0.79010	R(0.05)	1.61	
1656	D4052	0.7897		-0.63	
1707	D4052	0.7898		-0.07	
1712	ISO12185	0.78978		-0.18	
1726	D4052	0.78983		0.10	
1727	D4052	0.78977		-0.24	
1817 1835	Table OIML	0.78983		0.10	
1835 1852	ISO12185 ISO12185	0.78980 0.7898		-0.07 -0.07	
1919	ISO12185	0.78978		-0.07 -0.18	
6070	D4052	0.7899		0.18	
6072	D4052 D4052	0.7899	С	0.49	first reported 789.9 kg/L
6214	ISO12185	0.78979	5	-0.13	mot reported 7 00.0 kg/L
6297	D4052	0.7902	C,R(0.01)	2.17	reported 790.2 kg/L
6341	_ 100_	0.7902	S, ((0.01)		. 5p3.15d 1 00.E Ng/E
6358	ISO12185	0.7899		0.49	
		-		2	
	normality	suspect			
	n	44			
	outliers	2			
	mean (n)	0.78981			
	st.dev. (n)	0.000073			
	R(calc.)	0.00021			
	st.dev.(ISO12185:96)	0.000179			
	R(ISO12185:96)	0.0005			

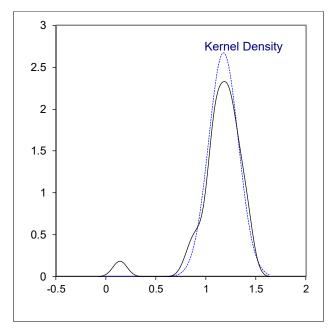




Determination of Electrical Conductivity at 25°C on sample #20245; results in $\mu\text{S/cm}$

lab	method	value	mark	z(targ)	remarks
52	EN15938	1.24		0.83	
120					
150	EN15938	1.2		0.30	
169	NBR10547	1.40	C	2.98	reported 140 μS/cm, possibly a unit error?
171	EN15938	0.83	С	-4.67	reported 83 μS/cm, possibly a unit error?
174	D1125	<10			
175 230					
311	EN15938	1.12		-0.78	
323	EN15938	1.16		-0.76	
329	EN15938	1.21		0.43	
333					
334	EN15938	1.10		-1.04	
337					
343	EN15938	1.04	С	-1.85	first reported 0.72
357	EN4E020	1.2	0	1.64	first reported 0.7
360 396	EN15938	1.3	С	1.64	first reported 0.7
444	EN15938	1.384		2.76	
468	21110000				
495	EN15938	1.286		1.45	
496	EN15938	1.2		0.30	
511					
541					
551	NBR10547	1.11		-0.91	
554					
558 621					
631	D1125	1.11	С	-0.91	first reported 111
633	D1120		O	-0.51	macreported 111
634					
657					
663					
823	D1125	1.090		-1.18	
913	DE204	4.40	0	2.00	first new autod 0.40
922 1108	D5391	1.40 	С	2.98	first reported 0.46
1189	EN15938	1.053		-1.67	
1213	LIV 13930			-1.07	
1320					
1397	EN2788	1.296		1.58	
1438					
1523	D2624	0.90		-3.73	
1530	EN45000		0.0(0.04)		
1656 1707	EN15938	0.14	C,R(0.01)	-13.92	first reported <1
1707 1712	EN15938	0.926		-3.38	
1712	EN15936 EN15938	1.19		-3.36 0.16	
1727	EN15938	1.08		-1.31	
1817	2.11.0000				
1835	EN15938	1.238		0.81	
1852					
1919	EN15938	1.07		-1.45	
6070	NDD 40547	4.000			
6072	NBR10547	1.268		1.21	
6214 6297	EN15938 NBR10547	1.218 1.42		0.54 3.25	reported 142 μS/m
6341	NDIX 10347	1.42		3.23	Teported 142 μο/ΠΙ
6358	EN15938	1.319		1.89	
	normality	OK			
	n	29			
	outliers	1			
	mean (n)	1.178			
	st.dev. (n)	0.1493			
	R(calc.) st.dev.(EN15938:10)	0.418			
	R(EN15938:10)	0.0746 0.209			
	11(LIN 10000.10)	0.203			



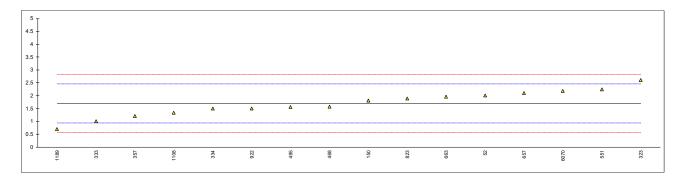


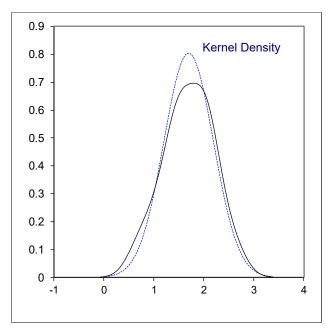
Determination of Nonvolatile matter on sample #20245; results in mg/100mL

lab	method	value	mark z(targ)	remarks
52	EN15691	<10		
120				
150	D1353	1.4		
169	D1353	0.00		
171 174	D1353	3 0.6		
174	D1353	0.0		
230	D1353	2.7		
311	EN15691	<10		
323	EN15691	1.9		
329	EN15691	1.4		
333				
334				
337 343	EN15691	<10		
357	EN15691	1		
360	EN15691	1.1		
396				
444	EN15691	0		
468	EN15691	<1,0		
495	EN15601			
496 511	EN15691	<3.5 		
541				
551	D1353	0.5		
554				
558				
621				
631				
633 634				
657	D1353	1.2		
663				
823	D1353	0.4		
913				
922	D1353	1.0		
1108 1189	EN15691	1.8		
1213	EN 13091	1.0		
1320				
1397	EN15691	<0,1		
1438				
1523				
1530	EN15691	<1		
1656 1707	D1353	1.2		
1712	D 1000	1.2		
1726	EN15691	0.8		
1727	EN15691	<10		
1817	In house	0.6		
1835	EN15691	<10		
1852				
1919 6070	D1353	4.9		
6072	D 1000	4.9		
6214				
6297				
6341				
6358				
	m	00		
	n mean (n)	28 <10		
	application range		EN15691:09: 10 – 25 m	g/100mL

Determination of Nitrogen on sample #20245; results in mg/kg

lab	method	value	mark z(targ)	remarks
52	D4629	2.0	0.81	
120				
150	D4629	1.8	0.28	
169				
171				
174				
175				
230				
311	D. 4000			
323	D4629	2.6	2.39	
329	D.4000	4.00		
333	D4629	1.00	-1.83	
334 337	D4629	1.5	-0.51 	
343				
357	D4629	1.2	-1.30	
360	D4023	1.2	-1.50	
396				
444				
468	D4629	1.57	-0.33	
495	D4629	1.55	-0.38	
496				
511				
541				
551	D4629	2.24	1.44	
554				
558				
621				
631				
633				
634	D. 4000			
657	D4629	2.1	1.07	
663	D4629	1.96	0.70	
823	D4629	1.88	0.49	
913 922	D4630	 1	 -0.51	
1108	D4629 D5762	1.5 1.334	-0.95	
1189	D4629	0.7	-2.62	
1213	D4029	0.7	-2.02	
1320				
1397				
1438				
1523				
1530	D4629	<1		
1656				
1707				
1712				
1726				
1727				
1817				
1835				
1852				
1919	D4620	2 174	1.27	
6070	D4629	2.174	1.27	
6072 6214				
6214 6297				
6341				
6358				
2000				
	normality	OK		
	n	16		
	outliers	0		
	mean (n)	1.694		
	st.dev. (n)	0.4953		
	R(calc.)	1.387		
	st.dev.(D4629:17)	0.3792		
	R(D4629:17)	1.062		



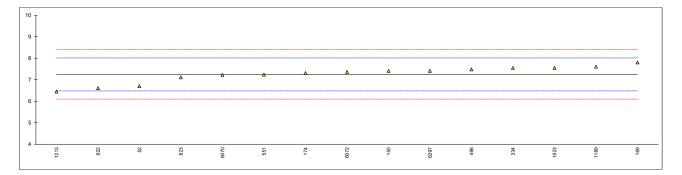


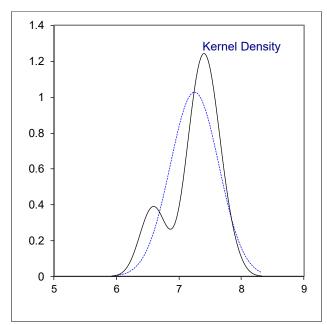
Determination of pHe with LiCl electrode on sample #20245;

lab	method	value	mark z(tar	remarks
52				
120				
150				
169	D0400			
171	D6423	8.01		
174 175				
230				
311				
323				
329				
333				
334				
337 343			 	
357				
360				
396				_
444				
468				
495				
496 511				
541				
551	NBR10891	6.21		
554				
558				
621				
631 633				
634				
657				
663				_
823				
913				
922				
1108 1189				
1213				
1320				
1397				
1438				
1523				
1530 1656				
1707				
1712				
1726	EN15490	7.43		
1727	EN15490	6.59		
1817	EN45400			
1835 1852	EN15490	6.7		
1919				
6070				
6072				-
6214				
6297				
6341 6358				
0000				
	normality	unknown		
	n	5		
	outliers	0		
	mean (n)	6.988		
	st.dev. (n) R(calc.)	0.722 2.0223		
	st.dev.(EN15490:07)	(0.240)		
	R(EN15490:07)	(0.6708)		
	,	` ,		

Determination of pHe with KCI electrode on sample #20245;

lab	method	value	mark	z(targ)	remarks
52	EN15490	6.7		-1.42	
120				-1.72	
150	D6423	7.4		0.39	
169	D6423	7.8		1.43	
171					
174	D6423	7.3		0.13	
175					
230					
311					
323					
329					
333					
334	EN15490	7.55	С	0.78	first reported 4.707
337					
343					
357					
360					
396					
444					
468					
495	EN45400	7.47		0.57	
496	EN15490	7.47		0.57	
511					
541 551	D6423	7.23		-0.05	
554	D0423	1.23		-0.05	
558					
621					
631					
633					
634					
657					
663					
823	D6423	7.104		-0.38	
913					
922	D6423	6.6		-1.68	
1108					
1189	EN15490	7.6		0.91	
1213	D6423	6.45		-2.07	
1320					
1397					
1438					
1523	D6423	7.552		0.79	
1530					
1656					
1707					
1712					
1726					
1727					
1817					
1835					
1852					
1919 6070	D6423	7.22		-0.08	
6070	D6423	7.22 7.36		0.29	
6214	D0423	7.30		0.29	
6297	D6423	7.4		0.39	
6341	53720	7			
6358					
2000					
	normality	OK			
	n	15			
	outliers	0			
	mean (n)	7.249			
	st.dev. (n)	0.3875			
	R(calc.)	1.085			
	st.dev.(D6423:20a)	0.3855			
	R(D6423:20a)	1.079			



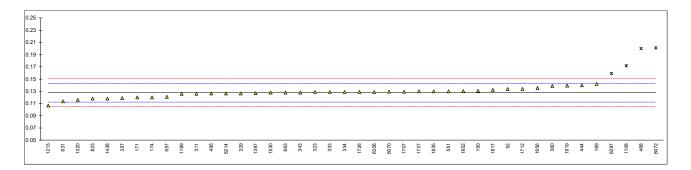


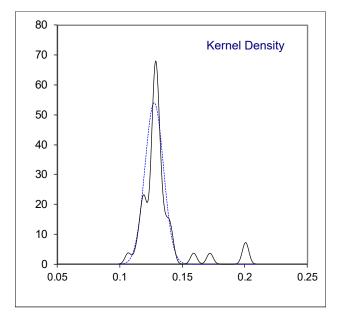
Determination of Phosphorus as P on sample #20245; results in mg/L

lab	method	value	mark z(targ) remarks
52	EN15837	<0.10		
120	D2024			-
150 169	D3231	<0.20 		-
171	D3231	<0.10		• -
174	D3231			-
175				_
230				-
311	EN15837	< 0.13		-
323	EN15487	<0.10		-
329	EN15487	0.004		-
333				-
334				-
337	= 1.1.= 1.0=			-
343	EN15487	<0,13		-
357	EN145007			-
360	EN15837	< 0.10		-
396 444	EN15937	0.09		-
468	EN15837	0.09		-
495				- -
496				_
511				-
541				-
551	INH-2047	< 0.13		-
554				-
558				-
621				-
631				-
633				-
634				-
657 663				-
823	UOP389	<0.11		-
913	001 000			_
922				-
1108	EN15487	0.00		-
1189				-
1213				-
1320				-
1397				-
1438				-
1523				-
1530 1656	EN15487	<0.01		_
1707	LIN 10401	~0.01 		-
1712	EN15487	0.01		_
1726	EN15487	0.023		-
1727	EN15487	<0,15		-
1817	-			-
1835	EN15837	0.03		-
1852				-
1919				-
6070	EN15487	0.4		- possibly a false positive test result?
6072	EN145405			-
6214	EN15487	0.139		-
6297				-
6341	EN15/197	 <0.15		-
6358	EN15487	<0,15		-
	n	18		
	mean (n)	<0.15		

Determination of Water, Coulometric on sample #20245; results in %M/M

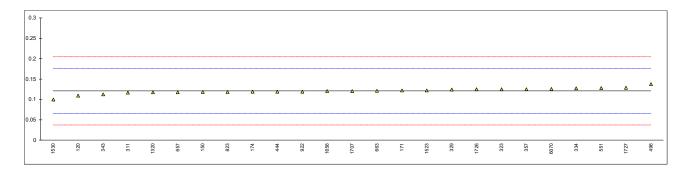
lab	method	value	mark	z(targ)	remarks
52	EN15489	0.134	HMIN	0.86	Tomano
120					
150	E1064	0.131		0.47	
169	E1064	0.142		1.92	
171	EN15489	0.11996		-0.99	
174	E1064	0.120		-0.98	
175					
230	EN45400	0.400		0.40	
311 323	EN15489	0.126		-0.19 0.18	
329	EN15489 D6304	0.1288 0.1263		-0.15	
333	EN15489	0.1203		0.20	
334	EN15489	0.129		0.20	
337					
343	EN15489	0.128		0.07	
357	E1064	0.1188		-1.14	
360	EN15489	0.1389		1.51	
396	=111=100				
444	EN15489	0.1398	0.0(0.04)	1.63	first new cuts d 0.04
468	EN15489	0.20	C,R(0.01)	9.58	first reported 0.24
495 496	EN15489	0.1262		-0.17 	
511					
541					
551	E1064	0.130		0.34	
554					
558					
621					
631	D6304	0.1136		-1.83	
633					
634	E4004	0.4040			
657 663	E1064 E1064	0.1210		-0.85 0.07	
823	E1064	0.1280 0.1182		-1.22	
913	L1004			-1.22	
922					
1108	EN15489	0.172	C,R(0.01)	5.88	reported 0.172 mg/kg
1189	EN15489	0.1257	, ,	-0.23	
1213	D6304	0.10661		-2.75	
1320	ISO12937	0.1162		-1.49	
1397	EN15489	0.127		-0.06	
1438	D6304	0.1182		-1.22	
1523	EN15/190	0.12762		0.02	
1530 1656	EN15489 EN15489	0.12762		1.04	
1707	EN15489	0.1333		0.27	
1712	EN15489	0.1340		0.86	
1726	EN15489	0.129		0.20	
1727	EN15489	0.1296		0.28	
1817	In house	0.1323		0.64	
1835	EN15489	0.1297		0.30	
1852	EN15489	0.1304		0.39	
1919	EN15489	0.13906 0.1293		1.53	
6070 6072	E1064 E1064	0.1293	C,R(0.01)	0.24 9.72	first reported 0.163
6214	EN15489	0.12622	C,IX(0.01)	-0.16	ilist reported 0.103
6297	E1064	0.159	R(0.01)	4.17	
6341			(0.0 .)		
6358	EN15489	0.129		0.20	
	normality	OK			
	n	38			
	outliers	4			
	mean (n)	0.12745			
	st.dev. (n) R(calc.)	0.007389 0.02069			
	st.dev.(EN15489:07)	0.02069			
	R(EN15489:07)	0.007370			
	compare	3.32.120			
	R(E1064:16)	0.02027			
	R(D6304:16e1)	0.20811			

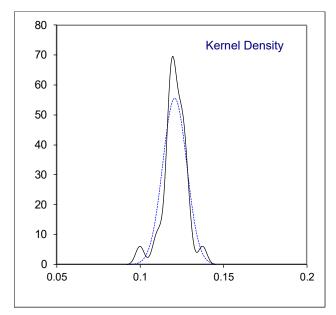




Determination of Water, Volumetric on sample #20245; results in %M/M

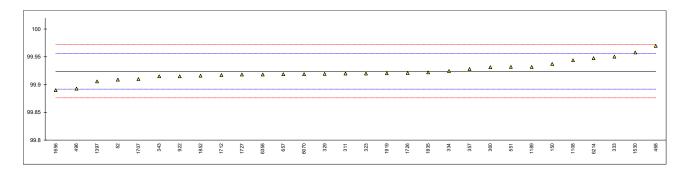
lab	method	value	mark	z(targ)	remarks
52					
120	E203	0.10935		-0.41	
150	E203	0.118		-0.10	
169	F000				
171	E203	0.1217		0.03	
174	E203	0.119		-0.06	
175					
230 311	EN15692	0.117		-0.13	
323	E203	0.117		0.16	
329	E203	0.1245		0.13	
333					
334	E203	0.1270		0.22	
337					
343	E203	0.112		-0.31	
357	E203	0.1252		0.16	
360					
396 444	E203	0.1190		-0.06	
468	L203	0.1190		-0.00	
495					
496	E203	0.1375		0.60	
511					
541					
551	E203	0.128		0.26	
554					
558					
621					
631 633					
634					
657	E203	0.1175		-0.12	
663	E203	0.1214		0.02	
823	D1364	0.1182		-0.09	
913					
922	E203	0.119		-0.06	
1108					
1189					
1213 1320	E203	0.1174		-0.12	
1397	L203			-0.12	
1438					
1523	E203	0.122		0.04	
1530	E203	0.09982		-0.75	
1656	E203	0.1202		-0.02	
1707	E203	0.1202		-0.02	
1712	EN45600	0.1050		0.15	
1726 1727	EN15692 EN15692	0.1250 0.1289		0.15 0.29	
1817	LINIJUJZ	0.1209		0.29	
1835					
1852					
1919					
6070	E203	0.1258		0.18	
6072					
6214					
6297					
6341 6358					
0336					
	normality	not OK			
	n	25			
	outliers	0			
	mean (n)	0.12075			
	st.dev. (n)	0.007194			
	R(calc.)	0.02014			
	st.dev.(E203:16)	0.027857			
	R(E203:16) compare	0.078			
	R(EN15692:09)	0.095			
	()	0.000			

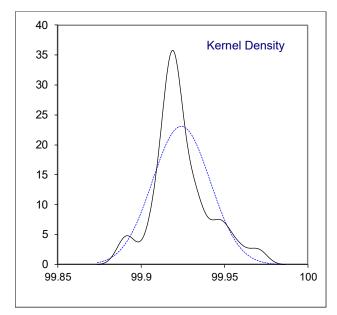




Determination of Ethanol incl. higher alcohols acc. to EN15721 on sample #20245 in %M/M

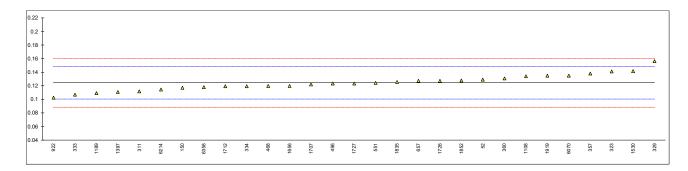
lab	method	value	mark	z(targ)	remarks
52	EN15721	99.909	παικ	-0.94	Tolliano
120					
150	EN15721	99.937		0.81	
169					
171 174					
174					
230					
311	EN15721	99.92		-0.25	
323	EN15721	99.920		-0.25	
329	EN15721	99.9195		-0.28	
333 334	EN15721 EN15721	99.950 99.924		1.62 0.00	
337	LIVIOIZI				
343	EN15721	99.915		-0.57	
357	EN15721	99.928		0.25	
360	EN15721	99.9311		0.44	
396 444					
468	EN15721	99.97		2.87	
495	LIVIOIZI				
496	EN15721	99.8927		-1.96	
511					
541	INII 1040				
551 554	INH-1313	99.932		0.50	
558					
621					
631					
633					
634 657	INH-02	99.9185		-0.35	
663	IINI I-UZ			-0.55	
823					
913					
922	INH-02	99.915		-0.57	
1108	EN15721	99.944		1.25	
1189 1213	EN15721	99.932		0.50	
1320					
1397	EN15721	99.906		-1.13	
1438					
1523	EN45704			0.40	
1530 1656	EN15721 EN15721	99.958 99.89		2.12 -2.13	
1707	EN15721	99.91		-0.88	
1712	EN15721	99.9170		-0.44	
1726	EN15721	99.921		-0.19	
1727	EN15721	99.918		-0.38	
1817	EN45704			0.40	
1835 1852	EN15721 EN15721	99.922 99.9163		-0.13 -0.48	
1919	EN15721	99.9205		-0.22	
6070	EN15721	99.919		-0.32	
6072					
6214	EN15721	99.9477		1.48	
6297					
6341 6358	EN15721	99.918		-0.38	
0000	L. 110/21	50.010		0.00	
	normality	suspect			
	n	30			
	outliers	0 02404			
	mean (n) st.dev. (n)	99.92404 0.017249			
	R(calc.)	0.017249			
	st.dev.(EN15721:13)	0.015989			
	R(EN15721:13)	0.04477			

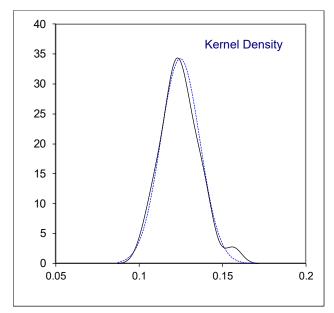




Determination of Higher alcohols acc. to EN15721 on sample #20245; results in % M/M

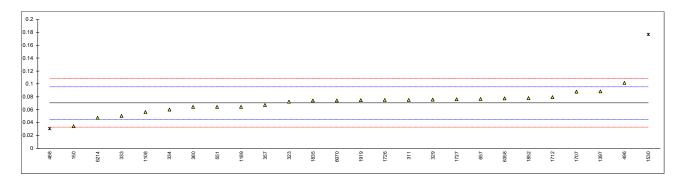
lab	method	value	mark	z(targ)	remarks
52	EN15721	0.129	IIIWIII	0.37	
120					
150	EN15721	0.117		-0.62	
169 171					
174					
175					
230	EN45704			4.04	
311 323	EN15721 EN15721	0.112 0.141		-1.04 1.37	
329	EN15721	0.1562		2.63	
333	EN15721	0.107		-1.46	
334	EN15721	0.1196		-0.41	
337 343					
357	EN15721	0.138		1.12	
360	EN15721	0.1308		0.52	
396					
444 468	EN15721	0.12		-0.38	
495	LIVIOIZI				
496	EN15721	0.1234		-0.09	
511					
541 551	INH-1313	0.12409		-0.04	
554	11411-1010				
558					
621					
631 633					
634					
657	INH-02	0.1272		0.22	
663					
823 913					
922	INH-02	0.1024		-1.84	
1108	EN15721	0.134		0.79	
1189 1213	EN15721	0.109		-1.29 	
1320					
1397	EN15721	0.111		-1.12	
1438					
1523 1530	EN15721	0.1415	С	1.41	first reported 0.2286
1656	EN15721	0.1413	C	-0.38	first reported 0.33
1707	In house	0.122		-0.21	1
1712	EN15721	0.1192		-0.44	
1726 1727	EN15721 EN15721	0.1272 0.1235		0.22 -0.09	
1817	LIVIOIZI	0.1233		-0.03	
1835	EN15721	0.1258		0.11	
1852	EN15721	0.1280		0.29	
1919 6070	EN15721 EN15721	0.1348 0.135		0.85 0.87	
6072	LIVIOIZI				
6214	EN15721	0.1145		-0.83	
6297					
6341 6358	EN15721	 0.118		-0.54	
0000	LINIOIZI	0.110		-0.04	
	normality	OK			
	n 	29			
	outliers mean (n)	0 0.12452			
	st.dev. (n)	0.011675			
	R(calc.)	0.03269			
	st.dev.(EN15721:13)	0.012039			
	R(EN15721:13)	0.03371			

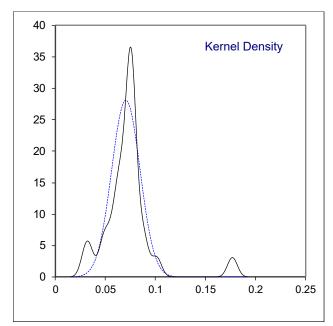




Determination of Impurities acc. to EN15721 on sample #20245; results in % M/M

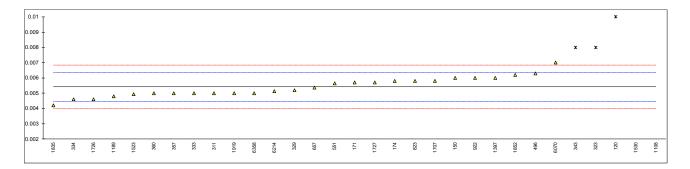
lab	method	value	mark	z(targ)	remarks
52	EN15721	<0.1	mark	2(tary)	Tomarko
120	LINIUIZI	~ 0.1			
150	EN15721	0.034	С	-2.89	first reported 0.177
169	21110721		Ü		mot reported 6.177
171					
174					
175					
230					
311	EN15721	0.075		0.37	
323	EN15721	0.072		0.13	
329	EN15721	0.0753		0.39	
333	EN15721	0.050		-1.62	
334	EN15721	0.060	С	-0.82	first reported 0.1958
337					
343					
357	EN15721	0.067		-0.27	
360	EN15721	0.0639		-0.51	
396					
444	=111==01		D(0.05)		
468	EN15721	0.03	R(0.05)	-3.21	
495	EN145704	0.1010		2.44	
496 511	EN15721	0.1010		2.44	
511 541					
541 551	INIL 1212	0.06400		0.50	
551 554	INH-1313	0.06400		-0.50	
558					
621					
631					
633					
634					
657	INH-02	0.0761		0.46	
663					
823					
913					
922					
1108	EN15721	0.056		-1.14	
1189	EN15721	0.064		-0.50	
1213					
1320					
1397	EN15721	0.088		1.40	
1438					
1523	EN145704		0.0(0.04)		C + + + + 0.0700
1530	EN15721	0.1767	C,R(0.01)	8.45	first reported 0.2702
1656	In house	0.0877		1 20	
1707 1712	In house EN15721	0.0877		1.38 0.70	
1712	EN15721 EN15721	0.0792		0.70	
1720	EN15721	0.0749		0.36	
1817	LINIUIZI	0.070		0.45	
1835	EN15721	0.0739		0.28	
1852	EN15721	0.0775		0.57	
1919	EN15721	0.0745		0.33	
6070	EN15721	0.074		0.29	
6072					
6214	EN15721	0.047137		-1.84	
6297					
6341					
6358	EN15721	0.077		0.53	
	normality	suspect			
	n	24			
	outliers	2			
	mean (n)	0.07034			
	st.dev. (n)	0.014179			
	R(calc.)	0.03970			
	st.dev.(Horwitz (n=9))	0.012586			
	R(Horwitz (n=9))	0.03524			

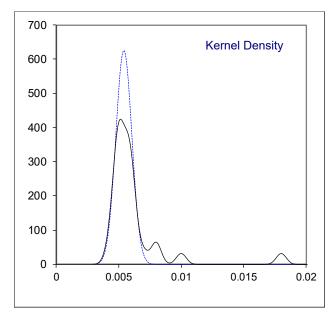




Determination of Methanol on sample #20245; results in %M/M

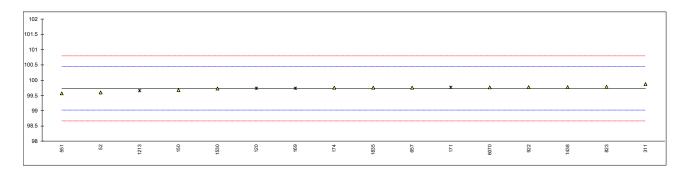
lab	method	value	mark	z(targ)	remarks
52	EN15721	<0.1	MUNIT		Tomand
120	D5501	0.01	R(0.01)	9.65	
150	EN15721	0.006	(0.01)	1.23	
169	- - ·				
171	D5501	0.0057		0.60	
174	D5501	0.0058	С	0.81	first reported 0.01
175					
230	=114==04				
311	EN15721	0.005	D(0.05)	-0.87	
323 329	EN15721 EN15721	0.008 0.0052	R(0.05)	5.44 -0.45	
333	EN15721	0.0052		-0.43	
334	EN15721	0.0046		-1.71	
337					
343	EN15721	0.008	R(0.05)	5.44	
357	EN15721	0.005		-0.87	
360	EN15721	0.0050		-0.87	
396					
444	EN16701	 -0.01			
468 495	EN15721	<0,01 			
495 496	EN15721	0.0063		1.86	
511	TIO/ 2				
541					
551	INH-1313	0.00565		0.50	
554					
558					
621	DEEOA		0		first remarked 0.00
631 633	D5501	<0.01	С		first reported 0.02
634					
657	INH-02	0.005364	С	-0.11	first reported 53.64 %M/M
663			-		
823	D5501	0.0058	С	0.81	first reported 0.0221
913					
922	INH-02	0.0060		1.23	
1108	EN15721	0.029	C,R(0.01)	49.65	first reported 0
1189 1213	EN15721	0.0048	С	-1.29	fr 0.00 corrected into: Not detected with 1.00 = 0.04% v/v
1213 1320	D5501		C		fr. 0.09, corrected into: Not detected with LOD = 0,01% v/v
1320	EN15721	0.006		1.23	
1438					
1523	D5501	0.004931		-1.02	
1530	EN15721	0.0180	C,R(0.01)	26.49	first reported 0.0207
1656	EN15721	<0.01			
1707	In house	0.0058		0.81	
1712 1726	EN15721	<0,010		 1 71	
1726 1727	EN15721 EN15721	0.0046 0.0057		-1.71 0.60	
1817	LINIUIZI	0.0057		0.00	
1835	EN15721	0.0042		-2.56	
1852	EN15721	0.0062		1.65	
1919	EN15721	0.005		-0.87	
6070	EN15721	0.007		3.34	
6072	EN46704	0.00540			
6214	EN15721	0.00513		-0.60	
6297 6341					
6358	EN15721	0.005		-0.87	
2000		3.000		3.01	
	normality	OK			
	n	26			
	outliers	5			
	mean (n)	0.00541			
	st.dev. (n)	0.000638			
	R(calc.) st.dev.(Horwitz)	0.00179 0.000475			
	R(Horwitz)	0.000473			
	compare	5.55.50			
	R(D5501:20)	0.01319			application range: 0.01 – 0.6 %M/M
	R(EN15721:13)	-0.00449			application range: 0.1 – 3 %M/M

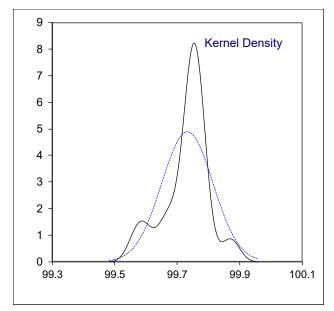




Determination of Ethanol acc. to ASTM D5501 on sample #20245; results in %M/M

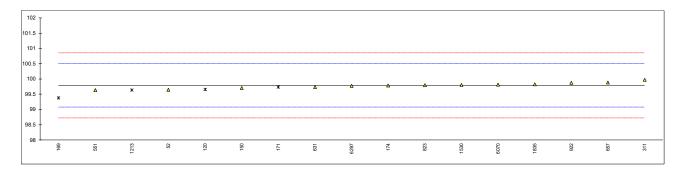
lab	method	value	mark	z(targ)	remarks
52	D5501	99.60	IIIuIK	-0.37	Tomaino
120	D5501	99.73	ex	-0.37 -0.01	result excluded as Ethanol %M/M is > Ethanol %V/V which is not possible
150	D5501	99.68	CX	-0.15	result excluded as Ethanor ////////////////////////////////////
169	D5501	99.7349	ex	0.01	result excluded as Ethanol %M/M is > Ethanol %V/V which is not possible
171	D5501	99.76	ex	0.08	result excluded as Ethanol %M/M is > Ethanol %V/V which is not possible
174	D5501	99.75	CX	0.05	result excluded as Ethanor /00//01/15 - Ethanor /00//0 Which is not possible
175	D0001				
230					
311	D5501	99.87		0.39	
323	D0001				
329					
333					
334					
337					
343					
357					
360					
396					
444					
468					
495					
496					
511					
541					
551	D5501	99.57		-0.46	
554					
558					
621					
631					
633					
634					
657	D5501	99.7589		0.07	
663					
823	D5501	99.7810		0.14	
913	D==0.4				
922	D5501	99.77		0.11	
1108					
1189	DEEOA			0.00	manufactural of a Character of MANA is a Character of MANA is a Character of the control of the
1213	D5501	99.66	ex	-0.20	result excluded as Ethanol %M/M is > Ethanol %V/V which is not possible
1320 1397					
	In house	00.77		0.11	
1438 1523	III IIouse	99.77 		0.11	
1530	D5501	99.7298		-0.01	
1656	D0001				
1707					
1712					
1726					
1727					
1817					
1835	D5501	99.75		0.05	
1852					
1919					
6070	D5501	99.76		0.08	
6072					
6214					
6297					
6341					
6358					
	normality	OK			
	n	12			
	outliers	0 + 4ex			
	mean (n)	99.7325			
	st.dev. (n)	0.08139			
	R(calc.)	0.2279			
	st.dev.(D5501:20)	0.35454			
	R(D5501:20)	0.9927			

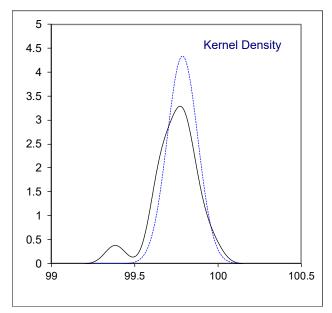




Determination of Ethanol acc. to ASTM D5501 on sample #20245; results in %V/V

lab	method	value	mark	z(targ)	remarks
52	D5501	99.65	HIGH	-0.39	Tomano
120	D5501	99.66	ex	-0.39	result excluded as Ethanol %M/M is > Ethanol %V/V which is not possible
150	D5501	99.71	CA.	-0.22	Toodit oxolded as Ethanol /000/101 is a Ethanol /00/10 willoth is hot possible
169	D5501	99.3832	ex	-0.22 -1.14	result excluded as Ethanol %M/M is > Ethanol %V/V which is not possible
171	D5501	99.73	ex	-0.16	result excluded as Ethanol %M/M is > Ethanol %V/V which is not possible
174	D5501	99.78	ΟΛ	-0.10	100ail 0x010000 do Ediano 7010/10 10 2 Ediano 700/ V Willott 10 Hot possible
175	_ 000 1			-0.02	
230					
311	D5501	99.97		0.52	
323	20001				
329					
333					
334					
337					
343					
357					
360					
396					
444					
468					
495					
496					
511					
541					
551	D5501	99.64		-0.42	
554					
558					
621					
631	D5501	99.733		-0.15	
633					
634					
657	D5501	99.8854		0.28	
663					
823	D5501	99.7948		0.02	
913	B==0.4				
922	D5501	99.87		0.23	
1108					
1189	DEEOA			0.40	
1213	D5501	99.64	ex	-0.42	result excluded as Ethanol %M/M is > Ethanol %V/V which is not possible
1320 1397					
1438 1523					
1530	D5501	99.8		0.04	
1656	D0001			0.04	
1707					
1712					
1726					
1727					
1817					
1835	D5501	99.82		0.09	
1852					
1919					
6070	D5501	99.81		0.06	
6072					
6214					
6297	D5501	99.77		-0.05	
6341					
6358					
	normality	OK			
	n	13			
	outliers	0 + 4ex			
	mean (n)	99.7872			
	st.dev. (n)	0.09196			
	R(calc.)	0.2575			
	st.dev.(D5501:20)	0.35442			
	R(D5501:20)	0.9924			





Determination of Bitrex on sample #20245; results in mg/kg

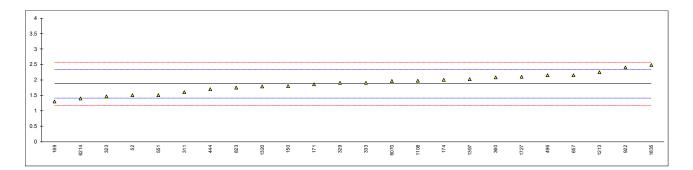
l a la	waath a d	value	-/	wa wa walea
lab 52	method	value	mark z(tar	
52 120				
150				
169				
171				
174				
175				
230 311				
323				
329	In house	35		
333				
334				
337 343	INILI 1077	12 //1		
357	INH-1877	13.41 		
360				
396				
444				
468				
495 496				
511				
541				
551	INH-3284	46.5		
554				
558				
621 631				
633				
634				
657				
663				
823				
913 922				
1108				
1189				
1213				
1320				
1397 1438		38.3		
1523		30.3		
1530				
1656				
1707	In house	12.66		
1712				
1726 1727				
1817				
1835				
1852				
1919				
6070				
6072 6214				
6297				
6341				
6358				
		_		
	n maan (n)	5		
	mean (n)	<50		

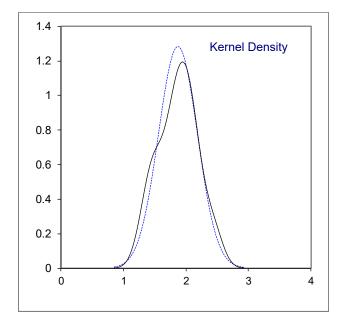
Determination of Gum (solvent washed) on sample #20245; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
52	D381	<0.5	man	Z(tary)	TOTHATING
120	D381	<0.5			
150	D381	<0.5			
169	D381	0.0			
171	D381	<0.5			
174					
175					
230					
311					
323					
329					
333					
334	D381	<0.5			
337					
343					
357	D381	<1			
360	D381	0.8			
396					
444					
468					
495					
496					
511					
541	D391				
551 554	D381	<0.5 			
558					
621					
631					
633					
634					
657					
663					
823	D381	<0.5			
913					
922	D381	1.3			
1108					
1189	D381	0.3			
1213					
1320					
1397					
1438					
1523	D 004				
1530	D381	<1			
1656					
1707					
1712 1726					
1720					
1817					
1835					
1852					
1919					
6070	D381	0.2			
6072					
6214					
6297					
6341					
6358					
	n	13			
	mean (n)	<1			

Determination of Inorganic Chloride as CI on sample #20246; results in mg/kg

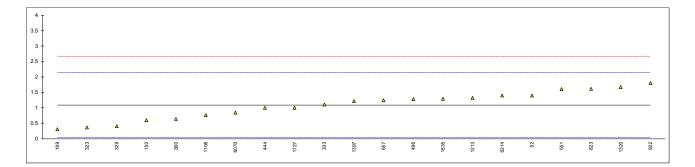
lab	method	value	mark	z(targ)	remarks
52	EN15492	1.5	mann	-1.62	
120					
150	D7319	1.8		-0.33	
169	D7319	1.3		-2.49	
171	D7319	1.854		-0.09	
174	D7319	2		0.54	
175	INILLOO				
230 311	INH-23 EN15492	<2 1.6		 -1.19	
323	EN15492 EN15492	1.6		-1.19 -1.75	
329	EN15492	1.9		0.10	
333	EN15492	1.9		0.10	
334	EN15492	<1.0		<-3.78	possibly a false negative test result?
337					
343					
357	EN45400	2.00			
360 396	EN15492	2.08		0.88	
444	EN15492	1.7		-0.76	
468	21110102				
495					
496	EN15492	2.15		1.19	
511					
541					
551	D7319	1.5		-1.62	
554 558					
621					
631					
633					
634					
657	D7328	2.15	С	1.19	first reported 5.85
663	D7040	4.740			
823 913	D7319	1.749 		-0.55 	
922	D7328	2.4		2.27	
1108	EN15492	1.97		0.41	
1189					
1213	D7328	2.2497		1.62	
1320	EN10304-1	1.79		-0.37	
1397	EN15492	2.02		0.62	
1438 1523					
1530					
1656					
1707					
1712			W		first reported 5.84 with test method EN15484
1726	EN45400				
1727	EN15492	2.1		0.97	
1817 1835	EN15492	2.48		2.61	
1852					
1919					
6070	D7319	1.9596		0.36	
6072					
6214	EN15492	1.3954		-2.08	
6297 6341					
6358	EN15484	<5,0			
0000	LITTOTOT	٠٠,٥			
	normality	OK			
	n	24			
	outliers	0			
	mean (n)	1.876			
	st.dev. (n)	0.3108 0.870			
	R(calc.) st.dev.(D7319:17)	0.870			
	R(D7319:17)	0.2314			application range: 0.75 – 50 mg/kg
	compare				
	R(EN15492:12)	0.641			application range: 1 – 30 mg/kg

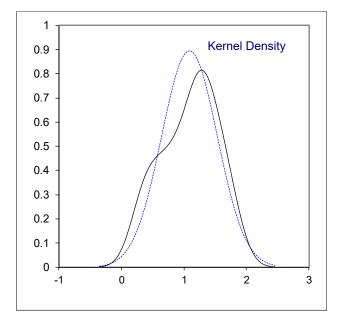




Determination of Sulfate as SO₄ on sample #20246; results in mg/kg

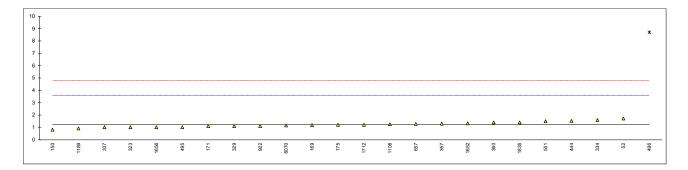
lab	method	value	mark z(targ)	remarks
52	EN15492	1.4	0.59	
120				
150	D7319	0.6	-0.92	
169	D7319	0.3	-1.49	
171	D7319	<1.0		
174 175	D7319	<1 		
230				
311	EN15492	<1.0		
323	EN15492	0.36	-1.38	
329	EN15492	0.4	-1.30	
333	EN15492	1.1	0.03	
334	EN15492	<1.0		
337	EN45400			
343 357	EN15492	<1 		
360	EN15492	0.64	-0.85	
396	2.110.02			
444	EN15492	1.0	-0.16	
468				
495	EN145400	4.00		
496 511	EN15492	1.28	0.37	
511 541				
551	D7319	1.6	0.97	
554	2.010		0.07	
558				
621				
631				
633				
634 657	D7328	 1.244	0.30	
663	D1020			
823	D7319	1.607	0.99	
913				
922	D7328	1.8	1.35	
1108	EN15492	0.76	-0.62	
1189 1213	D7328	 1.3210	0.44	
1320	EN10304-1	1.67	1.11	
1397	EN15492	1.21	0.23	
1438				
1523				
1530				
1656				
1707 1712				
1712				
1727	EN15492	1.0	-0.16	
1817				
1835	EN15492	1.29	0.39	
1852				
1919 6070	D7319	0.8431	-0.46	
6070 6072	פוטום	0.8431	-0.46	
6214	EN15492	1.3973	0.59	
6297				
6341				
6358				
		OV		
	normality n	OK 21		
	n outliers	0		
	mean (n)	1.087		
	st.dev. (n)	0.4465		
	R(calc.)	1.250		
	st.dev.(D7319:17)	0.5265		" " 4 OC "
	R(D7319:17)	1.474		application range: 1 – 20 mg/kg
	compare R(EN15492:12)	0.3780		application range: 1 - 20 mg/kg
	R(D7328:17)	1.0861		application range: 0.55 – 20 mg/kg
	(= · -= 0· · ·)			11

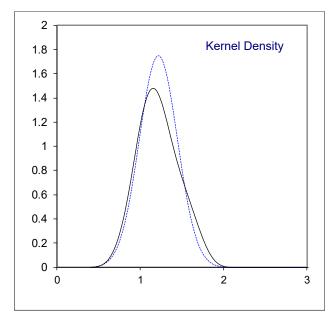




Determination of Sulfur on sample #20246; results in mg/kg

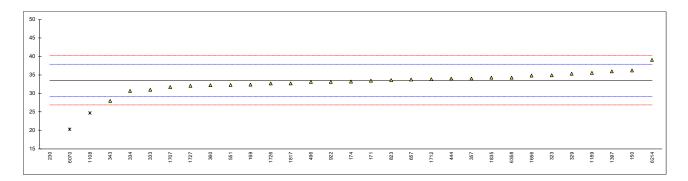
lab	method	value	mark	z(targ)	remarks
52	EN15486	1.7	mun	0.40	Tomario
120	LIVIOTOU	1.7		0.40	
150	D5453	8.0		-0.35	
169	D5453	1.18		-0.03	
171	D5453	1.091		-0.10	
174					
175	D5453	1.2		-0.01	
230					
311	EN15486	<5.0			
323	EN15486	1.0		-0.18	
329	D5453	1.1		-0.10	
333					
334	D5453	1.6		0.32	
337	EN15486	1.0		-0.18	
343	D5453	<1			
357	D5453	1.3		0.07	
360	EN15486	1.4		0.15	
396	EN15406	1.50		0.25	
444 468	EN15486 EN15486	1.52		0.25	
495	ISO20846	<2 1.01		-0.17	
496	EN15485	8.7	R(0.01)	6.26	
511	LN 13403		11(0.01)		
541					
551	D5453	1.5		0.24	
554	20100				
558					
621					
631					
633					
634					
657	D5453	1.28		0.05	
663					
823	D5453	<1.0			
913	DE4E2	4.4		0.40	
922	D5453	1.1		-0.10	
1108 1189	EN15485	1.24		0.02	
1213	D5453	0.9 <2		-0.26	
1320	D0400				
1397					
1438					
1523					
1530					
1656	EN15486	1.0		-0.18	
1707					
1712	EN15486	1.2		-0.01	
1726					
1727					
1817	ENIAE 400	4.4		0.45	
1835 1852	EN15486 ISO20846	1.4 1.31		0.15 0.08	
1919	13020040	1.31		0.00	
6070	D5453	1.146		-0.06	
6072	D0-100				
6214					
6297					
6341					
6358					
	normality	OK			
	n	23			
	outliers	1			
	mean (n)	1.216			
	st.dev. (n)	0.2284 0.640			
	R(calc.) st.dev.(EN15485:07)	1.1947			
	R(EN15485:07)	3.345			application range: 7 – 20 mg/kg
	compare	0.0 10			approximation to marky
	R(EN15468:07)	1.865			application range: 5 – 20 mg/kg
	R(D5453:19a) [′]	0.671			at a concentration < 400 mg/kg; application range: 1-8000 mg/kg

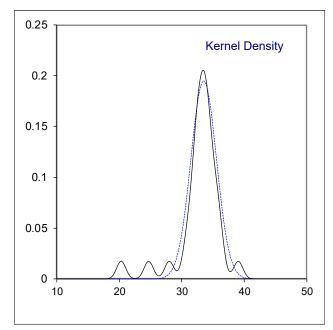




Determination of Nonvolatile matter on sample #20249; results in mg/100mL

lah	mathod	value	mark	7(tara)	romarks
lab 52	method EN15601	value >25	mark	z(targ)	remarks
52 120	EN15691	>25 			
150	D1353	36.2		1.21	
169	D1353	32.4		-0.50	
171	D1353	33.45		-0.03	
174	D1353	33.2		-0.14	
175	21000				
230	D1353	0.9	C,R(0.01)	-14.74	first reported 40.9
311	EN15691	>25	-, (-, -,		
323	EN15691	34.9		0.63	
329	EN15691	35.3		0.81	
333	EN15691	31		-1.14	
334	EN15691	30.7		-1.27	
337					
343	EN15691	28		-2.49	
357	D1353	34		0.22	
360	EN15691	32.2		-0.59	
396	=111=001				m
444	EN15691	34	С	0.22	first reported 17
468					
495	ENACCO4	22.4		0.40	
496 511	EN15691	33.1		-0.19	
541					
551	D1353	32.3		-0.55	
554	D 1000			-0.55	
558					
621					
631					
633					
634					
657	D1353	33.8		0.13	
663					
823	D1353	33.6		0.04	
913					
922	D1353	33.1		-0.19	
1108	EN15691	24.7	R(0.05)	-3.98	
1189	D1353	35.6		0.94	
1213					
1320 1397	EN15691	36		1.12	
1438	LIVIOUSI			1.12	
1523					
1530					
1656	EN15691	34.8		0.58	
1707	EN15691	31.7		-0.82	
1712	EN15691	33.85		0.15	
1726	EN15691	32.7		-0.37	
1727	EN15691	32.1		-0.64	
1817	In house	32.75		-0.35	
1835	EN15691	34.3		0.36	
1852					
1919	D.4050		D(0.64)		
6070	D1353	20.3	R(0.01)	-5.97	
6072	ENACCO4	20.05			
6214	EN15691	39.05		2.50	
6297 6341					
6358	EN15691	34.3		0.36	
0000	LIVIOUSI	04.0		0.50	
	normality	not OK			
	n	28			
	outliers	3			
	mean (n)	33.514			
	st.dev. (n)	2.0470			
	R(calc.)	5.732			
	st.dev.(EN15691:09)	2.2122			
	R(EN15691:09)	6.194			
	compare	14 464			
	R(D1353:13)	14.461			





APPENDIX 2

Number of participants per country

- 1 lab in ARGENTINA
- 3 labs in BELGIUM
- 3 labs in BRAZIL
- 1 lab in BULGARIA
- 1 lab in CANADA
- 3 labs in COLOMBIA
- 1 lab in CROATIA
- 1 lab in FINLAND
- 3 labs in FRANCE
- 4 labs in GERMANY
- 1 lab in GREECE
- 1 lab in HUNGARY
- 1 lab in INDIA
- 1 lab in INDONESIA
- 1 lab in ISRAEL
- 1 lab in ITALY
- 1 lab in MAURITIUS
- 2 labs in NETHERLANDS
- 1 lab in PAKISTAN
- 1 lab in PERU
- 3 labs in PHILIPPINES
- 2 labs in POLAND
- 1 lab in SINGAPORE
- 1 lab in SLOVAKIA
- 1 lab in SOUTH KOREA
- 4 labs in SPAIN
- 2 labs in SWEDEN
- 2 labs in THAILAND
- 1 lab in TURKEY
- 2 labs in UNITED KINGDOM
- 7 labs in UNITED STATES OF AMERICA
- 1 lab in VIETNAM

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 D(0.01) = outlier in Grubbs' outlier test D(0.05) = straggler in Grubbs' outlier test D(0.05) = outlier in Double Grubbs' outlier test D(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 = calculation difference between reported test result and result calculated by iis

W = test result withdrawn on request of participant ex = test result excluded from statistical evaluation

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

Literature

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