Results of Proficiency Test Ethanol (Bio / Fuel grade) November 2019

Organised by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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Report: iis 19C16

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1 Introduction

Since 1995 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Ethanol (Bio / Fuel grade) every year. During the annual proficiency testing (PT) program 2019/2020 it was decided to continue the PT for the analyses of Ethanol (Bio / Fuel grade) in accordance with the latest applicable version of the specifications EN15376 and ASTM D4806. In this interlaboratory study 57 laboratories from 29 different countries registered for participation for the PT on Ethanol (Bio / Fuel grade). See appendix 2 for the number of participants per country. In this report, the results of the 2019 proficiency test on Ethanol (Bio / 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 organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. In this proficiency test the participants received two different samples of Ethanol (Bio / Fuel grade), one bottle of 1L labelled #19250 for regular analyzes and one bottle of 50mL labelled #19251 for Inorganic Chloride, Sulfate and total Sulfur determination only. 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

A batch of approximately 80 liters of Ethanol (Bio / Fuel grade) obtained from a European supplier was used for the preparation of the subsamples for the regular analyzes. After homogenization 77 amber glass bottles of 1L were filled and labelled #19250. The homogeneity of the subsamples #19250 was checked by determination of Density in accordance with ISO12185 and Water in accordance with E203 on 8 stratified randomly selected samples.

	Density at 15°C in kg/L	Water in %M/M
Sample #19250-1	0.79445	0.259
Sample #19250-2	0.79445	0.262
Sample #19250-3	0.79445	0.261
Sample #19250-4	0.79445	0.260
Sample #19250-5	0.79445	0.258
Sample #19250-6	0.79445	0.258
Sample #19250-7	0.79445	0.257
Sample #19250-8	0.79445	0.254

Table 1: homogeneity test results of subsamples #19250

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 ISO13528, Annex B2 in the next table.

	Density at 15°C in kg/L	Water in %M/M
r (observed)	0.00000	0.007
reference test method	ISO12185:96	E203:16
0.3 * R (ref. test method)	0.00015	0.023

Table 2: evaluation of the repeatabilities of subsamples #19250

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, homogeneity of the subsamples was assumed. Please note that the Density for homogeneity purposes was determined at 15°C. The Density determination as requested from the participants was at 20°C.

The remaining part of the batch of Ethanol (Bio / Fuel grade) was spiked with Sodium Chloride (NaCl) and Sodium Sulfate (Na₂SO₄), dissolved in water, especially for Inorganic Chloride, Sulfate and total Sulfur determination. After homogenization 80 PE bottles of 50mL were filled and labelled #19251. The homogeneity of the subsamples #19251 was checked by determination of Sulfate as SO₄ in accordance with EN15492 on 7 stratified randomly selected samples.

	Sulfate as SO4 in mg/kg
Sample #19251-1	4.61
Sample #19251-2	4.10
Sample #19251-3	4.09
Sample #19251-4	3.63
Sample #19251-5	4.02
Sample #19251-6	4.12
Sample #19251-7	3.44

Table 3: homogeneity test results of subsamples #19251

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.

	Sulfate as SO4 in mg/kg
r (observed)	1.06
reference test method	D7319:17
0.3 * R (ref. test method)	1.05

Table 4: evaluation of the repeatability of subsamples #19251

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

To each of the participating laboratories, one bottle of 1L labelled #19250 and one bottle of 50mL labelled #19251 was sent on November 6, 2019. 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 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 #19250: Acidity Total as Acetic Acid, Appearance, Copper as Cu, Density at 20° C, Electrical Conductivity at 25° C, Nonvolatile matter, Nitrogen, pHe (KCl & LiCl), Phosphorus as P, Water (Coulometric and Volumetric), Ethanol incl. higher alcohols (acc. EN15721), Higher alcohols (acc. EN15721), Impurities (acc. EN15721), Methanol and Ethanol by mass and by volume (acc. ASTM D5501). The participants were asked to determine on sample #19251: Inorganic Chloride as Cl, Sulfate as SO_4 and Sulfur.

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

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

3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the 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 June 2018 (iis-protocol, version 3.5).

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

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

According to ISO5725 the original test results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

Finally, the reproducibilities were calculated from the standard deviations by multiplying these 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. The Kernel Density Graph 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 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, 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 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**

In this proficiency test, no major problems were encountered with the dispatch of the samples. Three participants reported the test results after the final reporting date and six participants did not report any test results at all. Not all laboratories were able to report all analyses requested.

In total 51 laboratories reported 457 numerical test results. Observed were 16 outlying test results, which is 3.5% 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, 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 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 (e.g. D5501:12) will be used.

Sample #19250

Acidity, Total: 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 EN15491:07 and ASTM D1613:17, but

not in agreement with the requirements of ASTM D7795-B:15.

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

about the appearance as Pass (Clear and Bright).

<u>Copper as Cu</u>: The majority 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. However, 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 problematic. Three statistical outliers

were observed. The calculated reproducibility after rejection of the

statistical outliers is not in agreement with the requirements of EN15938:10.

Nonvolatile matter: All participants reported a test result near or below the application range

of the method EN15691:09. Therefore no z-scores were calculated.

<u>Nitrogen</u>: 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 D4629:17. The low number of reported test results may (partly) explain the large variation.

pHe: 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 LiCI 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 problematic. No statistical outliers were observed.

However, the calculated reproducibility is not in agreement with the

requirements of D6423:19.

<u>pHe (LiCI)</u>: 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 EN15490:07.

<u>Phosphorus as P</u>: All 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 may be problematic depending on the test method used. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of EN15489:07 but is in agreement with the requirements of 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. One statistical outlier was observed. The calculated reproducibility is in agreement with the requirements of EN15721:13.

<u>Higher alcohols (EN15721)</u>: 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 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 not problematic. No statistical outlier was observed. The calculated reproducibility is 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. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility using the Horwitz equation, but in agreement with the requirements of ASTM D5501:12 (2016). A negative value for the reproducibility is found at this concentration level for test method EN15721:13.

Ethanol (D5501): This determination was not problematic for Ethanol by mass and Ethanol by volume. No statistical outliers were observed but in total three 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 D5501:12 (2016).

Sample #19251

Inorganic Chloride: 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 nor with EN15492:12 or ASTM D7319:17. Because of the strict reproducibility of EN15492:12 and ASTM D7319:17 it was decided to evaluate the test results with the estimated reproducibility using the Horwitz equation.

Sulfate as SO₄: This determination was problematic. No statistical outliers were observed.

The calculated reproducibility is not in agreement with the requirements of

ASTM D7319:17 nor with EN15492:12 or ASTM D7328:17.

Sulfur: This determination is not problematic. No statistical outliers were observed.

> The calculated reproducibility is in agreement with the requirements of EN15485:07, and also in agreement with the requirements of EN15486:07

and ASTM D5453:19a.

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 or as declared by the estimated target reproducibility using the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average results, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM, EN standards) or based on previous proficiency tests or the estimated target reproducibility are presented in the next tables.

Determination	unit	n	average	2.8 * sd	R(lit)
Acidity, Total as Acetic Acid	mg/kg	39	13.9	11.8	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	47	0.7902	0.0002	0.0005
Electrical Conductivity at 25°C	μS/cm	25	0.36	0.18	0.11
Nonvolatile matter	mg/100mL	13	<10	n.e.	n.e.
Nitrogen	mg/kg	8	0.53	1.11	0.58
pHe (KCI)		21	6.98	1.26	1.09
pHe (LiCI)		7	6.40	0.30	0.62
Phosphorus as P	mg/L	20	<0.15	n.e.	n.e.
Water, Coulometric	%M/M	42	0.261	0.039	0.024

Determination	unit	n	average	2.8 * sd	R(lit)
Water, Volumetric	%M/M	26	0.263	0.062	0.078
Ethanol incl. higher alcohols (EN15721)	%M/M	24	99.966	0.011	0.032
Higher alcohols (EN15721)	%M/M	24	0.135	0.025	0.037
Impurities (EN15721)	%M/M	21	0.031	0.009	0.017
Methanol	%M/M	27	0.004	0.002	0.001
Ethanol (D5501)	%M/M	17	99.625	0.298	0.993
Ethanol (D5501)	%V/V	17	99.744	0.339	0.993

Table 5: reproducibilities of tests on sample #19250

Parameter	unit	n	average	2.8 * sd	R (lit)
Inorganic Chloride as Cl	mg/kg	20	3.7	1.6	1.4
Sulfate as SO ₄	mg/kg	21	2.8	4.2	2.7
Sulfur	mg/kg	25	1.9	0.9	3.4

Table 6: reproducibilities of tests on sample #19251

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2019 WITH PREVIOUS PTS

	November 2019	December 2018	December 2017	December 2016	December 2015
Number of reporting laboratories	51	53	59	57	68
Number of test results	457	473	537	476	899
Number of statistical outliers	16	14	22	31	39
Percentage outliers	3.5%	3.0%	4.1%	6.5%	4.3%

Table 7: comparison with previous proficiency tests

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

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

Determination	November 2019	December 2018	December 2017	December 2016	December 2015
Acidity, Total as Acetic Acid	+	+	-	+	+/-
Density at 20°C	++	++	++	++	++
Electrical Conductivity at 25°C	-				-
Nonvolatile matter	n.e.	()	()	()	()
Nitrogen	-	-	-		
рНе	+/-	-	-	+/-	n.e.

Determination	November 2019	December 2018	December 2017	December 2016	December 2015
Water, Coulometric	-	+/-	+/-	+	+/-
Water, Volumetric	+	+	++	++	++
Ethanol incl. higher alcohols (EN15721)	++		-	-	
Higher alcohols (EN15721)	+	+/-	+/-	-	-
Impurities (EN15721)	++	-	-		
Methanol			-	-	
Ethanol (D5501)	++	+	+	++	++
Inorganic Chloride as Cl	-	-	+	(++)	-
Sulfate as SO ₄	-	-			()
Sulfur	++	+	+	+	++

Table 8: 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.

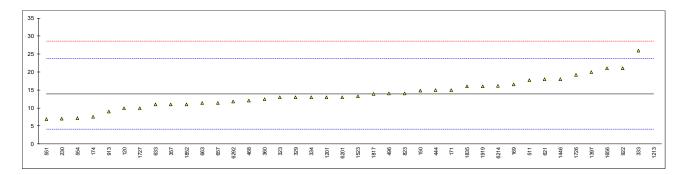
The following performance categories were used:

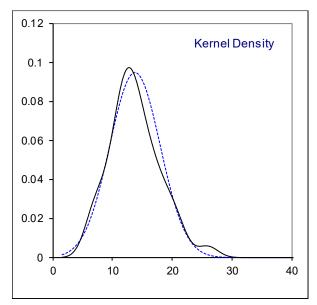
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- : group performed worse than the reference test method
- -- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Acidity, Total as Acetic Acid on sample #19250; results in mg/kg

Detern		_			sample #19250; results in mg/kg
lab	method	value	mark	z(targ)	remarks
52	EN15491	<30			
120	D1613	9.97		-0.79	
150	D7795	14.8		0.19	
169	D7795	16.61		0.56	
171 174	D1613	15 7.6		0.23	
174 175	D1613	7.6 		-1.28 	
194					
230	D1613	6.988		-1.40	
311	D1613	<30			
323	EN15491	13		-0.18	
329	EN15491	13		-0.18	
333	EN15491	26		2.48	
334	EN15491	13		-0.18	
337	EN15491	<30			
343	EN15491	<30		0.50	
357	EN15491	11		-0.58	
360 396	EN15491	12.4		-0.30 	
444	EN15491	15	С	0.23	first reported 0.0034 %M/M
468	EN15491	12	J	-0.38	
496	EN15491	14		0.03	
511	D1613	17.8		0.81	
541					
551	D1613	6.875		-1.43	
554	D1613	7.2		-1.36	
558	D4040	40			
621	D1613	18		0.85	
631 633	D1613	 11		-0.58	
657	D1613	11.4139		-0.50	
663	D1613	11.4		-0.50	
823	D1613	14		0.03	
913	D1613	9		-0.99	
922	D1613	21.1		1.48	
1201	EN15491	13		-0.18	
1213	D1613	52.5	R(0.01)	7.90	
1359	EN45404			4.00	
1397 1446	EN15491 EN15491	20 18		1.26 0.85	
1523	ISO1388/2	13.3		-0.11	
1656	EN15491	21	С	1.46	first reported 0.004 %M/M
1667	LITTO-TOT		Ü		mot reported 0.004 7000/00
1726	EN15491	19.2		1.09	
1727	EN15491	10		-0.79	
1817	ISO1388/2	13.94		0.02	
1835	EN15491	16		0.44	
1852	EN15491	11		-0.58	
1919	EN15491	16		0.44	
6201 6214	EN15491 EN15491	13 16.17		-0.18 0.47	
6214	LIN 1949 I	10.17		0.47	
6262					
6292	NBR9866	11.73		-0.44	
6297					
6303					
6305					
	11.	011			
	normality	OK			
	n outliers	39 1			
	mean (n)	13.859			
	st.dev. (n)	4.2187			
	R(calc.)	11.812			
	st.dev.(EN15491:07)	4.8929			
	R(EN15491:07)	13.70			application range: 30 - 150 mg/kg
	compare				W 41
	R(D1613:17)	14			application range: <500 mg/kg
	R(D7795-B:15)	10.281			application range: <200 mg/kg





Determination of Appearance on sample #19250;

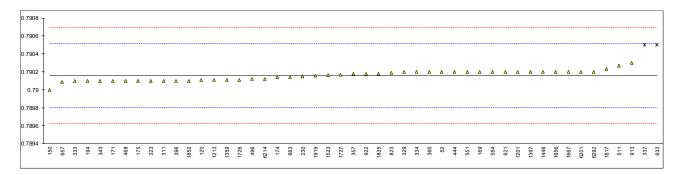
lab	method	value	mark	z(targ)	remarks
52	D4176	Pass		(tai.g/	· · · ·
120					
150	E2680	Pass			
169					
171	Visual	Pass			
174	Visual	Clear & Free			
175	D4176	Pass			
194	Visual	Clear			
230	Visual	Clear & Bright			
311	EN15769	clear & colourless			
323	D4176	clear & bright liquid			
329	Visual				
333	Visual	clear 			
334	Vieuel				
	Visual	clear and bright			
337	Visual	Incolore			
343	Visual	PASS			
357	E2680	Pass			
360	EN15769	Clear and Colourless			
396	Visual	Clear and Bright			
444	EN15769	Pass			
468	EN15769	C&C			
496	EN15769	clear & colourless			
511	EN15769	Clear and colourless			
541					
551	E2680	PASS			
554	Visual	Pass			
558					
621	Visual	Clear & colourless			
631					
633	Visual	Clear & Bright			
657	E2680	Pass			
663	Visual	Clear and Bright			
823	E2680	Pass			
913	E2680	Pass			
922	Visual	Clear and bright			
1201	Visual	Bright and Clear			
1213					
1359	Visual	Clear and bright			
1397	EN15769	colourless, bright			
1446					
1523					
1656	EN15769	pass			
1667		· 			
1726	EN15769	Clear and colourless			
1727	Visual	Clear&Colorless			
1817	Visual	pass			
1835	EN15769	C&C			
1852	Visual	clear & bright			
1919					
6201	EN15769	Clear and Colourless			
6214	EN15769	clear and colourless			
6224					
6262					
6292	Visual	Clear			
6297					
6303					
6305					
0000					
	n	41			
	mean	Pass (Clear & Bright)			
	moan	1 433 (Olcai & Diigili)			

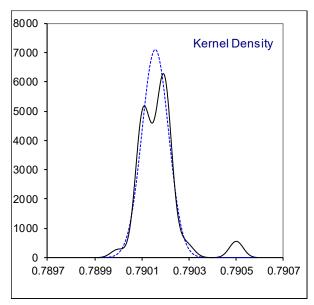
Determination of Copper as Cu on sample #19250; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	EN15837	<0.050			
120					
150					
169					
171					
174					
175	D1688	<0.05			
194					
230	EN15488	0			
311	EN15837	<0.050			
323	EN15488	<0.07			
329	EN15488	<0,07			
333	EN15488	<0.07			
334					
337					
343	EN15837	<0.05			
357					
360	EN15837	< 0.050			
396					
444	EN15837	< 0.05			
468	EN15488	<0,07			
496	EN15837	0.000			
511	D1688	0.006			
541					
551	INH-2047	<0.04			
554					
558					
621					
631					
633					
657	INII 1 40 44 4				
663	INH-12414	0.002			
823	UOP389	<0.01			
913	D4000				
922	D1688	< 0.05			
1201	EN15488	< 0.07			
1213	EN45400	<0.1			
1359	EN15488	<1			
1397					
1446 1523					
1656	D1688-A	 -0.1			
1667	D1000-A	<0.1 			
1726					
1727					
1817					
1835	EN15837	0.03			
1852	LIN 10001	0.03			
1919					
6201	EN15488	0.003			
6214	EN15488	0.00245			
6224	_1110100	0.00240			
6262					
6292					
6297					
6303					
6305					
5500					
	n	21			
	mean (n)	< 0.07			
	application range		N15488·07·	0 07 – 0 20	ma/ka

Determination of Density at 20°C on sample #19250; results in kg/L

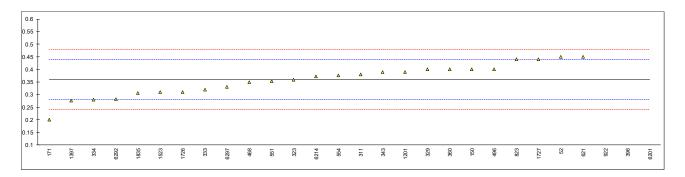
lab	method	value	mark	z(targ)	remarks
52	ISO12185	0.7902	mun	0.24	Tomatino
120	D4052	0.79011	С	-0.27	first reported 790.11 kg/L
150	D4052	0.7900	Ü	-0.88	motroportou roo.rr kg/2
169	D4052	0.7902		0.24	
171	D4052	0.7901		-0.32	
174	D4052	0.79014		-0.10	
175	D4052	0.7901		-0.32	
194	D4052	0.7901	С	-0.32	first reported 0.7944
230	D4052	0.79015		-0.04	
311	D4052	0.7901		-0.32	
323	D4052	0.7901		-0.32	
329	D4052	0.7902		0.24	
333	D4052	0.7901		-0.32	
334	ISO12185	0.7902		0.24	
337	ISO12185	0.7905	R(0.01)	1.92	
343	ISO12185	0.7901		-0.32	
357	D4052	0.79018		0.12	
360	D4052	0.7902		0.24	
396	ISO12185	0.7901		-0.32	
444	D4052	0.7902		0.24	
468	ISO12185	0.7901		-0.32	
496	ISO12185	0.79012	С	-0.21	first reported 790.12 kg/L
511	D4052	0.79027	С	0.63	first reported 0.79172
541					
551	D4052	0.7902		0.24	
554	D4052	0.7902		0.24	
558					
621	D4052	0.7902		0.24	
631					
633	D4052	0.79050	R(0.01)	1.92	
657	D4052	0.79009		-0.38	
663	D4052	0.79014		-0.10	
823	D4052	0.79019		0.18	
913	D4052	0.7903		0.80	
922	D4052	0.79018		0.12	
1201	D4052	0.7902		0.24	
1213	D4052	0.79011		-0.27	
1359	ISO12185	0.79011		-0.27	
1397	ISO12185	0.7902		0.24	
1446	ISO12185	0.79020		0.24	
1523	D4052	0.7901623		0.03	
1656	D4052	0.7902		0.24	
1667	ISO12185	0.7902		0.24	
1726	D4052	0.79011		-0.27	
1727	D4052	0.79017		0.07	
1817	Table OIML	0.79023		0.40	
1835	ISO12185	0.79018	_	0.12	first reported 700.4 kg/l
1852	ISO12185	0.7901	С	-0.32	first reported 790.1 kg/L
1919	ISO12185	0.79016		0.01	
6201	ISO12185	0.79020		0.24	
6214	ISO12185	0.79012		-0.21	
6224					
6262 6292	D4052	0.7902		0.24	
6292 6297	D403Z	0.7902		0.24	
6303					
6305					
0303					
	normality	ОК			
	n	47			
	outliers	2			
	mean (n)	0.79016			
	st.dev. (n)	0.000056			
	R(calc.)	0.00016			
	st.dev.(ISO12185:96)	0.000179			
	R(ISO12185:96)	0.0005			
	11,120.2.30.00)	2.000			

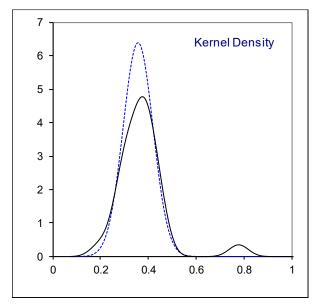




Determination of Electrical Conductivity at 25°C on sample #19250; results in $\mu S/cm$

lab	method	value	mark	z(targ)	remarks
52	EN15938	0.45		2.30	
120	EN145000		0	4.04	Fresh and a series of 40
150 169	EN15938	0.4	С	1.04	first reported 40
171	EN15938	0.20	С	-3.99	first reported 20
174	D1125-A	<10			
175 194					
230					
311	EN15938	0.38		0.54	
323	EN15938	0.36		0.03	
329 333	EN15938 EN15938	0.40 0.32		1.04 -0.97	
334	EN15938	0.32		-1.98	
337					
343 357	EN15938	0.39		0.79 	
360	EN15938	0.40		1.04	
396	EN15938	1.65	R(0.01)	32.51	
444	EN145000				
468 496	EN15938 EN15938	0.35 0.4		-0.22 1.04	
511	LIV10000				
541					
551	NBR10547	0.353		-0.14 0.44	
554 558	NBR10547	0.376		0.44	
621	EN15938	0.45		2.30	
631					
633 657					
663					
823	D1125	0.44		2.05	
913 922	D5391	0.78	R(0.01)	10.61	
1201	EN15938	0.39	11(0.01)	0.79	
1213					
1359 1397		0.276		-2.08	
1446		0.270		-2.00	
1523	D2624	0.31		-1.23	
1656	EN15938	<1			
1667 1726	EN15938	0.31		-1.23	
1727	EN15938	0.44		2.05	
1817	EN145000	0.007		4.20	
1835 1852	EN15938	0.307		-1.30	
1919					
6201	EN15938	2.10	R(0.01)	43.84	
6214 6224	EN15938	0.373		0.36	
6262					
6292	NBR10547	0.282		-1.93	
6297 6303	NBR10547	0.33		-0.72 	
6305					
	normality	OK			
	n outliers	25 3			
	mean (n)	0.359			
	st.dev. (n)	0.0624			
	R(calc.) st.dev.(EN15938:10)	0.175 0.0397			
	R(EN15938:10)	0.111			





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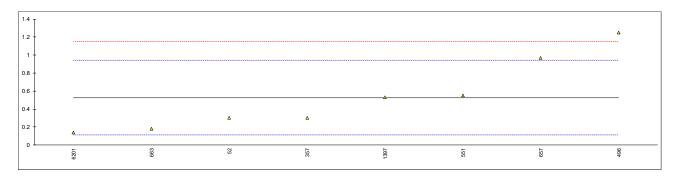
Determination of Nonvolatile matter on sample #19250; results in mg/100mL

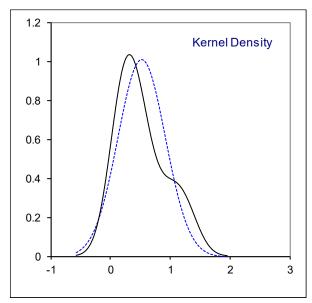
lab	method	value	mark	z(targ)	remarks
52	EN15691	<10			
120					
150	D1353	1.2			
169	D1353	0.00			
171	D1353	<1			
174	D1353	0.2			
175					
194	D. 40=0		0(0.04)		
230	D1353	5.0	G(0.01)		
311	EN15691	<10			
323	EN15691	<1 0.5			
329 333	EN15691 EN15691	0.5 <10			
334	EN15691	<10			
337	LIVIOUSI				
343					
357	EN15691	< 1			
360	EN15691	0.8			
396					
444	EN15691	0.9			
468					
496	EN15691	0			
511					
541					
551	D1353	0.4			
554	D1353	0.3			
558	D4050	0.4			
621	D1353	0.1			
631 633					
657	D1353	0.4			
663	D 1333				
823					
913	D1353	<1			
922	D1353	0.7			
1201	EN15691	<10			
1213					
1359					
1397	EN15691	4	G(0.01)		
1446					
1523					
1656	EN15691	<1			
1667	EN45004				
1726	EN15691	<10			
1727	EN15691	<10			
1817 1835	In house EN15691	0.4 <10			
1852	LIV 1309 I				
1919					
6201	EN15691	0.5			
6214	EN15691	0.3			
6224	*****				
6262					
6292					
6297					
6303					
6305					
		40			
	n	13			
	mean (n)	<10	1145004.00.4		/400

application range of test method EN15691:09: 10 - 25 mg/100mL

Determination of Nitrogen on sample #19250; results in mg/kg

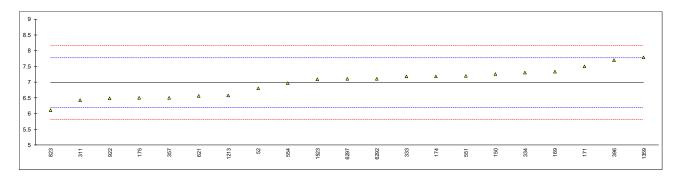
lab	method	value	mark	z(targ)	remarks
52	D4629	0.3	IIIai N	-1.09	IGIIIGI NO
120	D-TUZU			-1.09	
150					
169					
171	D4629	<0.3			
174 175					
173					
230					
311					
323					
329	D4000				
333 334	D4629 D4629	<0.3 <0.3			
337	D4029				
343					
357	D4629	0.3		-1.09	
360					
396	D4629	<0.3			
444 468	D4629 D4629	<0.3 <0,5			
496	D4629	1.25		3.48	
511					
541					
551	D4629	0.55		0.11	
554					
558 621					
631					
633					
657	D4629	0.9679		2.12	
663	D4629	0.18		-1.67	
823 913					
922	D4629	<0.3			
1201	D4629	<1			
1213					
1359					
1397 1446		0.53		0.02	
1523					
1656					
1667					
1726					
1727					
1817 1835					
1852					
1919					
6201	D4629	0.137		-1.88	
6214					
6224					
6262 6292					
6297					
6303					
6305					
	n a rm a litu				
	normality n	unknown 8			
	outliers	0			
	mean (n)	0.527			
	st.dev. (n)	0.3952			
	R(calc.)	1.107			
	st.dev.(D4629:17) R(D4629:17)	0.2078 0.582			application range: 0.3 – 100 mg/kg
	N(D4029.17)	0.002			application range: 0.3 – 100 mg/kg

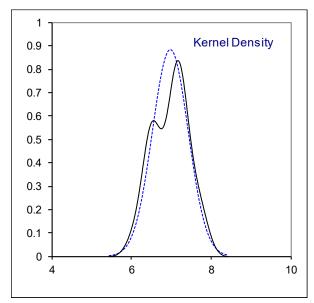




Determination of pHe (KCI) on sample #19250;

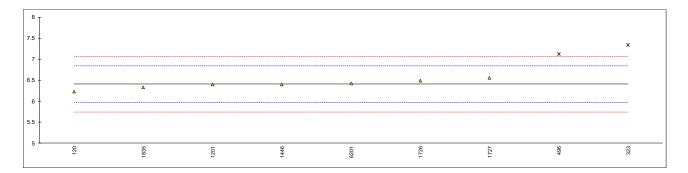
lab	method	value	mark	z(targ)	remarks
52	EN15490	6.8		-0.46	
120					
150	D6423	7.25		0.69	
169	D6423	7.33		0.89	
171	D6423	7.5	С	1.33	first reported 9; no details on which type of electrode was used
174	D6423	7.18		0.51	
175	D6423	6.5		-1.23	
194					
230 311	EN15490	6.42		 -1.44	
323	EN 13490	0.42		-1.44	
329					
333	EN15490	7.18		0.51	
334	EN15490	7.30		0.82	
337					
343					
357	D6423	6.5		-1.23	
360	EN145400	 7 7		4.04	
396	EN15490	7.7		1.84	
444 468					
496					
511					
541					
551	D6423	7.2		0.56	
554		6.96		-0.06	
558					
621	D6423	6.56		-1.08	
631					
633 657					
663					
823	D6423	6.1		-2.26	
913	20.20				
922	D6423	6.48		-1.28	
1201					
1213	D6423	6.58		-1.03	
1359		7.78		2.04	
1397					
1446 1523	EN15490	7.0825		0.26	
1656	LN 13490	7.0023		0.20	
1667					
1726					
1727					
1817					
1835					
1852					
1919					
6201 6214					
6224					
6262					
6292	D6423	7.11		0.33	
6297	D6423	7.1		0.30	
6303					
6305					
	normality.	OV			
	normality n	OK 21			
	n outliers	0			
	mean (n)	6.982			
	st.dev. (n)	0.4508			
	R(calc.)	1.262			
	st.dev.(D6423:19)	0.3907			
	R(D6423:19)	1.094			

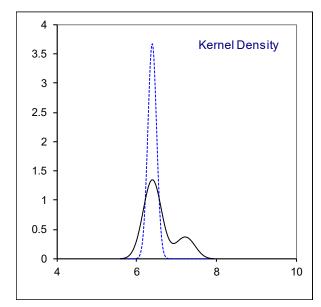




Determination of pHe (LiCl) on sample #19250;

lab	method	value	mark	z(targ)	remarks
52 120	D6423	6 221		-0.83	no details on which type of electrode week used
120 150	D0423	6.221		-0.83	no details on which type of electrode was used
169					
171					
174					
175 194					
230					
311					
323	EN15490	7.34	DG(0.01)	4.27	possibly used a KCl electrode?
329					
333 334					
337					
343					
357					
360 396					
444					
468					
496	EN15490	7.12	DG(0.01)	3.27	possibly used a KCl electrode?
511 541					
541 551					
554					
558					
621					
631 633					
657					
663					
823					
913 922					
1201	EN15490	6.4	С	-0.01	first reported 0.4; no details on which type of electrode was used
1213					71
1359					
1397	EN15400	6.4		0.01	
1446 1523	EN15490	6.4		-0.01 	
1656					
1667					
1726	EN15490	6.49		0.40 0.72	
1727 1817	EN15490	6.56 		0.72	
1835	EN15490	6.33		-0.33	
1852					
1919	EN45400	6.40		0.00	
6201 6214	EN15490	6.42		0.08	
6224					
6262					
6292					
6297 6303					
6305					
	normality	OK			
	n outliers	7 2			
	mean (n)	6.403			
	st.dev. (n)	0.1088			
	R(calc.)	0.305			
	st.dev.(EN15490:07) R(EN15490:07)	0.2195 0.615			
	11(LIN 10400.01)	0.013			





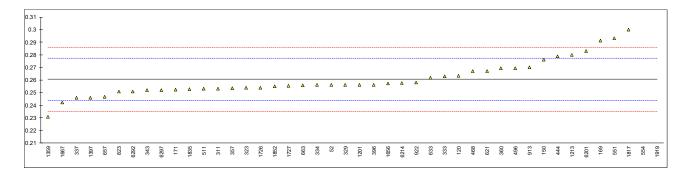
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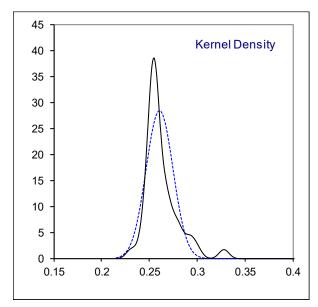
Determination of Phosphorus as P on sample #19250; results in mg/L

lab	method	value	mark	z(targ)	remarks
52	EN15487	<0.10			
120					
150					
169					
171					
174					
175					
194					
230					
311	EN15837	<0.13			
323	EN15487	<0.15			
329	EN15487	0.004			
333	EN15487	<0.15			
334					
337					
343	EN15487	<0.13			
357					
360	EN15837	< 0.10			
396					
444	EN15837	0.01			
468	EN15487	<0,15			
496	EN15487	<0.01			
511					
541					
551	INH-2047	<0.13			
554					
558					
621					
631					
633					
657					
663	LIODOGO				
823	UOP389	<0.11			
913					
922 1201	EN15407	 <0.15			
1213	EN15487				
1359	EN15487	 <0.01			
1397	EN15487	<0.01			
1446	LIV13401				
1523					
1656	EN15487	<0.01			
1667	LIVIOTOI				
1726	EN15487	0.005			
1727	EN15487	<0,01			
1817	21110101				
1835	EN15837	0.02			
1852					
1919					
6201					
6214	EN15487	0.1115			
6224					
6262					
6292					
6297					
6303					
6305					
	n	20			
	mean (n)	<0.15			
	application ran	ge test method EN15	5487:07: 0.	15 - 1.50 m	g/L

Determination of Water, Coulometric on sample #19250; results in %M/M

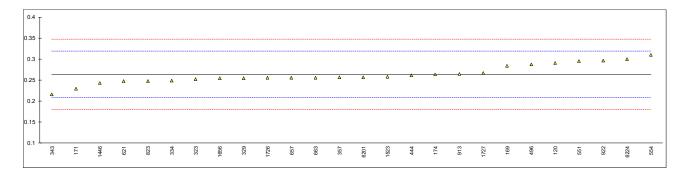
lab	method	value	mark	z(targ)	remarks
52	EN15489	0.256	mark	-0.54	Tomand
120	E1064	0.2635		0.35	
150	E1064	0.276		1.83	
169	E1064	0.2913	С	3.63	first reported 0.3423
171	EN15489	0.2523		-0.97	
174					
175					
194					
230	EN145400				
311	EN15489	0.253		-0.89	
323 329	EN15489 D6304	0.2540 0.2560		-0.77 -0.54	
333	EN15489	0.263		0.29	
334	EN15489	0.256		-0.54	
337	EN15489	0.246		-1.72	
343	EN15489	0.252		-1.01	
357	E1064	0.2537		-0.81	
360	EN15489	0.2693		1.03	
396	EN15489	0.2561		-0.53	
444	EN15489	0.2788		2.16	
468 496	EN15489	0.267 0.2695		0.76 1.06	
511	EN15489 E1064	0.253		-0.89	
541	L1004			-0.05	
551	E1064	0.293		3.83	
554	E1064	0.328	R(0.01)	7.97	
558			,		
621	D6304	0.267		0.76	
631			_		
633	D6304	0.262	С	0.17	reported 0.262 mg/kg
657	E1064	0.2466		-1.65	
663 823	E1064 E1064	0.2556 0.251		-0.58 -1.13	
913	E1064	0.231		1.15	
922	D6304	0.258	С	-0.30	first reported 0.298
1201	EN15489	0.256	Ü	-0.54	mot reported 0.200
1213	D6304	0.2799		2.29	
1359	EN15489	0.23085		-3.51	
1397	EN15489	0.246		-1.72	
1446					
1523	EN45400	0.0574		0.07	
1656	EN15489	0.2574		-0.37 -2.19	
1667 1726	EN15489 EN15489	0.242 0.254		-2.19 -0.77	
1727	EN15489	0.2553		-0.62	
1817	In house	0.30		4.66	
1835	EN15489	0.2527		-0.93	
1852	EN15489	0.2550		-0.66	
1919	EN15489	0.5262	R(0.01)	31.39	
6201	EN15489	0.28314	С	2.67	first reported 2831.4 without unit
6214	EN15489	0.25786		-0.32	
6224					
6262 6292	E1064	0.251		 -1.13	
6297	E1064	0.252		-1.13	
6303	L1004				
6305					
					EN15489:07 only
	normality	suspect			suspect
	n	42			24
	outliers	2 26055			1
	mean (n) st.dev. (n)	0.26055 0.014074			0.25680 0.011157
	R(calc.)	0.014074			0.03124
	st.dev.(EN15489:07)	0.00341			0.008439
	R(EN15489:07)	0.02370			0.02363
	compare				
	R(E1064:16)	0.04455			
	R(D6304:16e1)	0.18933			

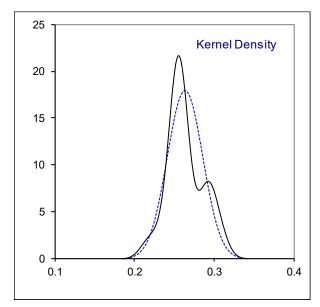




Determination of Water, Volumetric on sample #19250; results in %M/M

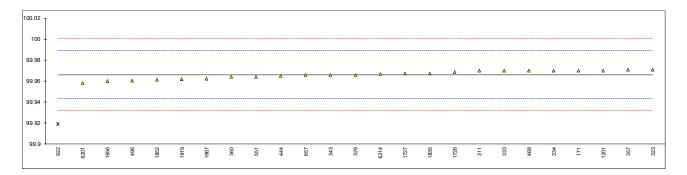
lab	method	value	mark	z(targ)	remarks
52					
120	E203	0.2902		0.96	
150					
169	E203	0.2838		0.73	
171	E203	0.2298		-1.21	
174	E203	0.2630		-0.02	
175					
194					
230					
311 323	E203	0.2520		-0.41	
323	E203	0.2520		-0.41	
333	L203	0.2341		-0.51	
334	E203	0.249		-0.52	
337	2200				
343	E203	0.216		-1.70	
357	E203	0.2562		-0.26	
360					
396					
444	E203	0.2617		-0.06	
468					
496	E203	0.2875		0.86	
511					
541					
551	E203	0.295		1.13	
554	E203	0.310		1.67	
558					
621	E203	0.248		-0.56	
631					
633	5000				
657	E203	0.2560		-0.27	
663	E203	0.2560		-0.27	
823 913	E203 E203	0.248 0.264		-0.56 0.02	
922	E203	0.204		1.17	
1201	E203	0.290		1.17	
1213					
1359					
1397					
1446	ISO760	0.2430		-0.73	
1523	E203	0.2575		-0.21	
1656	E203	0.2541		-0.34	
1667					
1726	EN15692	0.2551		-0.30	
1727	EN15692	0.2663		0.10	
1817					
1835					
1852					
1919	F000	0.05745	0		first and other CETA Finally 1
6201	E203	0.25715	С	-0.23	first reported 2571.5 without unit
6214	la lacusa	0.0		4.04	
6224	In house	0.3		1.31	
6262					
6292 6297					
6303					
6305					
0000					
	normality	OK			
	n	26			
	outliers	0			
	mean (n)	0.26346			
	st.dev. (n)	0.022243			
	R(calc.)	0.06228			
	st.dev.(E203:16)	0.027857			
	R(E203:16)	0.078			
	compare				
	R(EN15692:09)	0.09827			
	•				

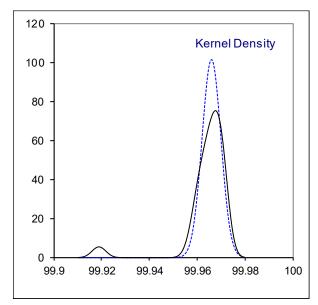




Determination of Ethanol incl. higher alcohols acc. to EN15721 on sample #19250 in %M/M

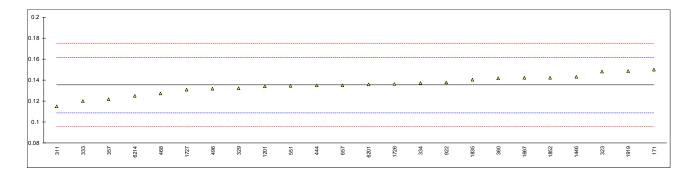
lab	method	value	mark	z(targ)	remarks
52					
120					
150					
169					
171	EN15721	99.97		0.35	
174					
175					
194					
230	EN145704			0.05	
311	EN15721	99.97		0.35	
323 329	EN15721 EN15721	99.971		0.43 0.00	
333	EN15721	99.966 99.970		0.00	
334	EN15721	99.970		0.35	
337					
343	EN15721	99.966	С	0.00	first reported 99.754
357	EN15721	99.971		0.43	
360	EN15721	99.9638		-0.19	
396					
444	EN15721	99.965		-0.09	
468	EN15721	99.97		0.35	
496 511	EN15721	99.9602		-0.51	
511 541					
551	INH-1313	99.964		-0.18	
554	11411-1010			-0.10	
558					
621					
631					
633					
657	INH-02	99.9658		-0.02	
663					
823					
913 922	INH-02	99.919	C,R(0.01)	-4.11	first reported 99.607
1201	EN15721	99.97	C,1X(0.01)	0.35	ilist reported 33:007
1213	LITIOIZI				
1359					
1397					
1446					
1523					
1656	EN15721	99.96	С	-0.53	first reported 99.74 (test result excl. higher alcohols)
1667	EN15721	99.962		-0.35	
1726	EN15721	99.9684		0.21 0.09	
1727 1817	EN15721	99.967 		0.09	
1835	EN15721	99.967		0.09	
1852	EN15721	99.9613		-0.41	
1919	EN15721	99.9616		-0.39	
6201	EN15721	99.958		-0.70	
6214	EN15721	99.9666		0.05	
6224					
6262					
6292					
6297					
6303 6305					
0303					
	normality	OK			
	n	24			
	outliers	1			
	mean (n)	99.96603			
	st.dev. (n)	0.003930			
	R(calc.)	0.01100			
	st.dev.(EN15721:13)	0.011455			
	R(EN15721:13)	0.03207			

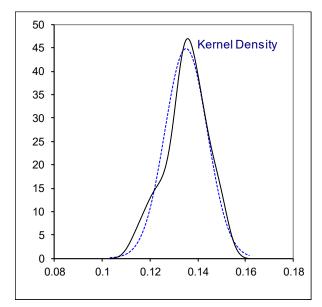




Determination of Higher alcohols acc. to EN15721 on sample #19250; results in %M/M

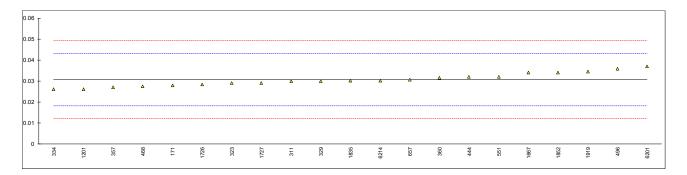
lab	method	value	mark	z(targ)	remarks
52					
120					
150 169					
171	EN15721	0.150		1.13	
174					
175 194					
230					
311	EN15721	0.115		-1.53	
323 329	EN15721 EN15721	0.148 0.1322		0.97 -0.23	
333	EN15721	0.120		-1.15	
334	EN15721	0.1371		0.15	
337 343					
357	EN15721	0.122		-1.00	
360	EN15721	0.1416		0.49	
396 444	EN15721	0.135		-0.01	
468	EN15721	0.1272		-0.61	
496	EN15721	0.1316		-0.27	
511 541					
551	INH-1313	0.1344		-0.06	
554					
558 621					
631					
633 657	INH-02	0.1351		-0.01	
663	11411-02				
823					
913 922	INH-02	0.1375		0.18	
1201	EN15721	0.134		-0.09	
1213 1359					
1339					
1446	EN15721	0.1429		0.59	
1523 1656			W		first reported 0.21
1667	EN15721	0.142	VV	0.52	ilist reported 0.21
1726	EN15721	0.1363		0.08	
1727 1817	EN15721	0.1308		-0.33 	
1835	EN15721	0.1403		0.39	
1852	EN15721	0.1423		0.54	
1919 6201	EN15721 EN15721	0.1484 0.136		1.00 0.06	
6214	EN15721	0.1248		-0.79	
6224					
6262 6292					
6297					
6303					
6305					
	normality	OK			
	n outliers	24 0			
	mean (n)	0.13519			
	st.dev. (n)	0.008895			
	R(calc.) st.dev.(EN15721:13)	0.02491 0.013165			
	R(EN15721:13)	0.03686			
	R(EN15721:13)	0.03686			

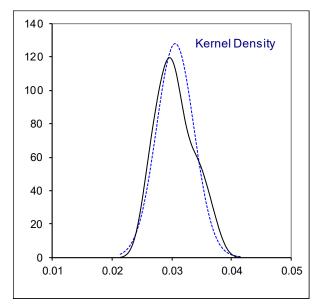




Determination of Impurities acc. to EN15721 on sample #19250; results in %M/M

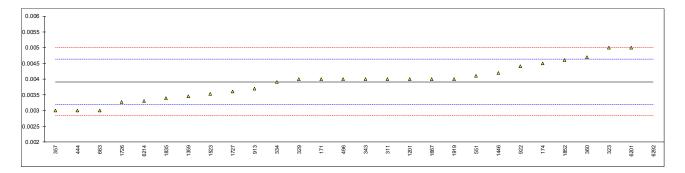
lab	method	value	mark	z(targ)	remarks
52	mourou		man		Tomano
120					
150					
169					
171	EN15721	0.028		-0.42	
174					
175					
194					
230					
311	EN15721	0.030		-0.10	
323	EN15721	0.029	С	-0.26	first reported 0.178
329	EN15721	0.030		-0.10	
333	EN15721	<0.100			
334	EN15721	0.026		-0.74	
337					
343	EN45704	0.007		0.50	
357	EN15721	0.027		-0.58 0.15	
360 396	EN15721	0.0315		0.15	
390 444	EN15721	0.032		0.23	
468	EN15721	0.032		-0.50	
496	EN15721	0.0273		0.84	
511					
541					
551	INH-1313	0.032		0.23	
554					
558					
621					
631					
633					
657	INH-02	0.0306		0.00	
663					
823					
913 922					
1201	EN15721	0.026		-0.74	
1213	LNISTZI	0.020		-0.74	
1359					
1397					
1446					
1523					
1656					
1667	EN15721	0.034		0.55	
1726	EN15721	0.0283		-0.37	
1727	EN15721	0.029		-0.26	
1817	EN45704				
1835	EN15721	0.0301		-0.08	
1852	EN15721	0.0341		0.57	
1919 6201	EN15721 EN15721	0.0344 0.037		0.61 1.03	
6214	EN15721 EN15721	0.037		-0.08	
6224	LINIOIZI	0.03011		-0.06	
6262					
6292					
6297					
6303					
6305					
	normality	OK			
	n	21			
	outliers	0			
	mean (n)	0.03059			
	st.dev. (n)	0.003116			
	R(calc.) st.dev.(Horwitz (n=9))	0.00873 0.006205			
	R(Horwitz (n=9))	0.006205			
	1.(1101WILZ (11-8))	0.01737			

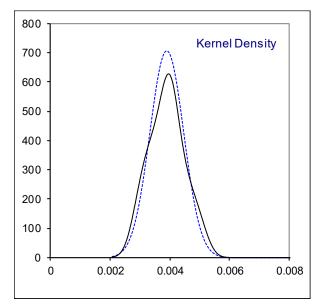




Determination of Methanol on sample #19250; results in %M/M

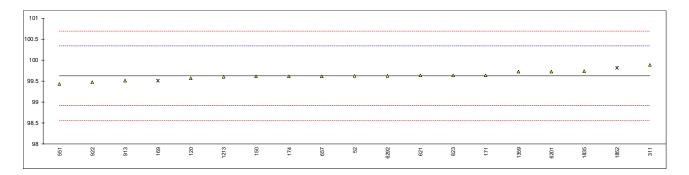
lab	method	value	mark	z(targ)	remarks
52					
120	D5501	<0.01	С		first reported 0.01
150					
169	=114==04				
171	EN15721	0.004		0.24	
174	D5501	0.0045		1.63	
175 194					
230					
311	EN15721	0.004		0.24	
323	EN15721	0.005		3.02	
329	EN15721	0.0040		0.24	
333	EN15721	<0.100			
334	EN15721	0.0039		-0.03	
337					
343	EN15721	0.004		0.24	
357	EN15721	0.003		-2.53 2.18	
360 396	EN15721	0.0047		2.10	
444	EN15721	0.003		-2.53	
468	EN15721	<0,01			
496	EN15721	0.0040		0.24	
511					
541					
551	INH-1313	0.0041		0.52	
554					
558 621	D5501	<0.01			
631	D3301				
633					
657	D5501	< 0.01			
663	INH-0002	0.003		-2.53	
823	D5501	<0.01			
913	D5501	0.0037		-0.59	
922	INH-02	0.0044		1.35	
1201	EN15721	0.004		0.24	
1213 1359	EN15721	0.003447		-1.29	
1397	LN13/21	0.003447		-1.29	
1446	EN15721	0.0042		0.80	
1523	D5501	0.003524		-1.08	
1656	EN15721	<0.01	С		first reported 0.01
1667	EN15721	0.004		0.24	
1726	EN15721	0.003265		-1.80	
1727	EN15721	0.0036		-0.87	
1817 1835	EN15721	0.0034		 -1.42	
1852	EN15721	0.0034		1.91	
1919	EN15721	0.0040		0.24	
6201	EN15721	0.005		3.02	
6214	EN15721	0.0033		-1.70	
6224					
6262	B==0.4		7(0.04)		
6292	D5501	0.02	R(0.01)	44.63	
6297 6303					
6305					
0303					
	normality	OK			
	n	27			
	outliers	1			
	mean (n)	0.00391			
	st.dev. (n)	0.000565			
	R(calc.)	0.00158			
	st.dev.(Horwitz) R(Horwitz)	0.000360 0.00101			
	compare	0.00101			
	R(D5501:12)	0.01301			application range: 0.01 – 0.6 %M/M
	R(EN15721:13)	-0.00499			application range: 0.1 – 3 %M/M
	-				

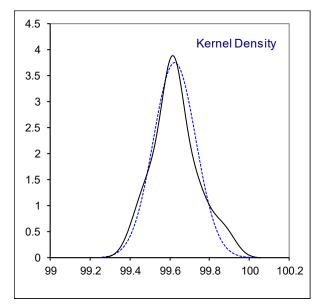




Determination of Ethanol acc. to ASTM D5501 on sample #19250; results in %M/M

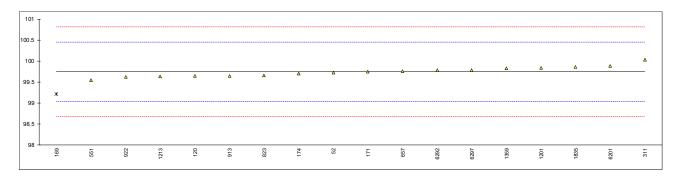
lab	method	value	mark	z(targ)	remarks
52	D5501	99.62		-0.02	
120	D5501	99.57		-0.16	
150	D5501	99.61		-0.04	
169	D5501	99.512	ex	-0.32	result excluded as Ethanol %M/M is > Ethanol %V/V which is not possible
171	D5501	99.634	CX	0.02	result excluded as Ethanor //////// is > Ethanor //// which is not possible
174	D5501	99.6103		-0.04	
	D330 I				
175					
194					
230	DEEOA			0.70	
311	D5501	99.886		0.73	
323					
329					
333					
334					
337					
343					
357					
360					
396					
444					
468					
496					
511					
541					
551	D5501	99.43		-0.55	
554					
558					
621	D5501	99.63		0.01	
631					
633					
657	D5501	99.6154		-0.03	
663					
823	D5501	99.63		0.01	
913	D5501	99.51		-0.33	
922	D5501	99.47		-0.44	
1201					
1213	D5501	99.605		-0.06	
1359	D5501	99.72		0.27	
1397					
1446					
1523					
1656					
1667					
1726					
1727					
1817					
1835	D5501	99.74		0.32	
1852		99.8190	ex	0.55	excl. as result reported in comments as pure Ethanol %M/M
1919					
6201	D5501	99.73		0.30	
6214					
6224					
6262					
6292	D5501	99.62		-0.02	
6297					
6303					
6305					
	normality	suspect			
	n	17			
	outliers	0 (+2 ex)			
	mean (n)	99.62533			
	st.dev. (n)	0.106304			
	R(calc.)	0.29765			
	st.dev.(D5501:12)	0.354765			
	R(D5501:12)	0.99334			

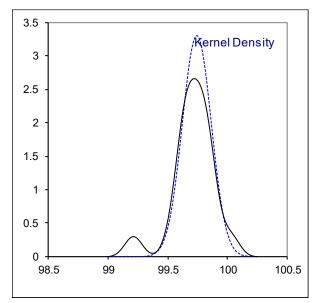




Determination of Ethanol acc. to ASTM D5501 on sample #19250; results in %V/V

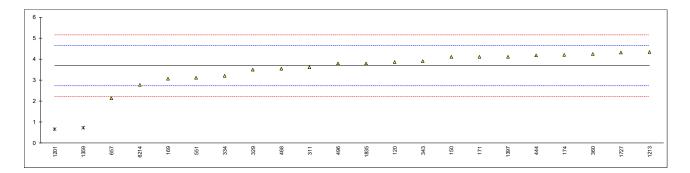
lab	method	value	mark	z(targ)	remarks
52	D5501	99.72		-0.07	
120	D5501	99.64		-0.29	
150					
169	D5501	99.212	ex	-1.50	result excluded as Ethanol %M/M is > Ethanol %V/V which is not possible
171	D5501	99.747		0.01	
174	D5501	99.6982		-0.13	
175					
194					
230	DEEOA	400.00		0.04	
311	D5501	100.03		0.81	
323					
329 333					
334					
337					
343					
357					
360					
396					
444					
468					
496					
511					
541	B==0.4				
551	D5501	99.54		-0.58	
554					
558					
621					
631 633					
657	D5501	99.7542		0.03	
663	D0001				
823	D5501	99.66		-0.24	
913	D5501	99.64		-0.29	
922	D5501	99.62		-0.35	
1201	D5501	99.84		0.27	
1213	D5501	99.630		-0.32	
1359	D5501	99.83		0.24	
1397					
1446					
1523					
1656					
1667 1726					
1726 1727					
1817					
1835	D5501	99.86		0.33	
1852					
1919					
6201	D5501	99.88		0.38	
6214					
6224					
6262					
6292	D5501	99.78		0.10	
6297	D5501	99.78		0.10	
6303					
6305					
	normality	OK			
	n	17			
	outliers	0 (+1 ex)			
	mean (n)	99.74408			
	st.dev. (n)	0.121040			
	R(calc.)	0.33891			
	st.dev.(D5501:12)	0.354512			
	R(D5501:12)	0.99263			

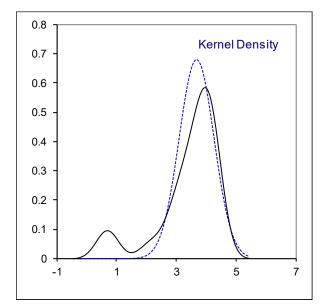




Determination of Inorganic Chloride as CI on sample #19251; results in mg/kg

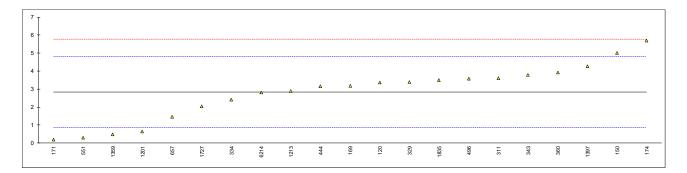
lab	method	value	mark	z(targ)	remarks
52	Motifou		mark	Z(tary)	- On Marito
120	D7319	3.85		0.33	
150	D7328	4.1		0.84	
169	D7319	3.061		-1.30	
171	D7319	4.1		0.84	
174	D7319	4.2		1.05	
175					
194					
230					reported: >2 but <3
311	EN15492	3.6		-0.19	
323					
329	EN15492	3.5		-0.39	
333	EN145400			4.04	
334	EN15492	3.2		-1.01 	
337 343	EN15492	3.9		0.43	
357	LIN 13432	J.J			
360	EN15492	4.24		1.13	
396					
444	EN15492	4.18		1.01	
468	EN15492	3.55		-0.29	
496	EN15492	3.78		0.18	
511					
541					
551	D7319	3.1		-1.22	
554					
558					
621 621					
631 633					
657	D7328	2.1343		-3.21	
663	D1020				
823					
913					
922					
1201	EN15492	0.67	R(0.01)	-6.23	
1213	D7328	4.332		1.32	
1359	EN15492	0.72	R(0.01)	-6.12	
1397	EN15492	4.11		0.86	
1446					
1523					
1656 1667					
1726					
1727	EN15492	4.32		1.30	
1817					
1835	EN15492	3.80		0.22	
1852					
1919					
6201					
6214	EN15492	2.7722		-1.89	
6224					
6262					
6292 6297					
6303					
6305					
0000					
	normality	suspect			
	n	20			
	outliers	2			
	mean (n)	3.691			
	st.dev. (n)	0.5860			
	R(calc.)	1.641			
	st.dev.(Horwitz)	0.4852			
	R(Horwitz)	1.359			
	compare	0 777			application range 4 20 mm/l/m
	R(EN15492:12) R(D7319:17)	0.777 1.041			application range: 1 – 30 mg/kg application range: 0.75 – 50 mg/kg
	1401010.11)	1.041			application range. 0.70 – 00 mg/kg

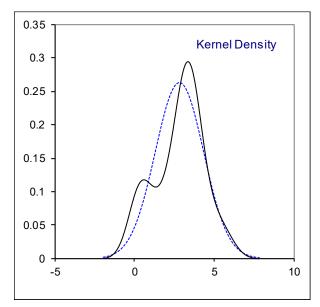




Determination of Sulfate as SO_4 on sample #19251; results in mg/kg

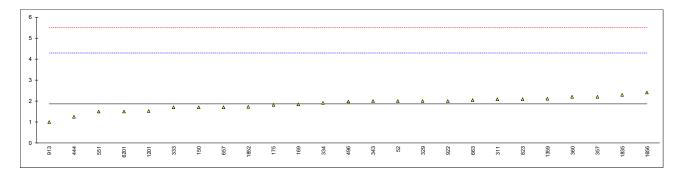
lab	method	value	mark z(targ)	remarks
52				
120	D7319	3.37	0.54	
150	D7328	5.0	2.21	
169	D7319	3.190	0.36	
171	D7319	0.2	-2.71	
174	D7319	5.7	2.93	
175				
194				
230	5 11.5100			
311	EN15492	3.6	0.78	
323	EN145400	2.4	 0.57	
329 333	EN15492	3.4	0.57	
334	EN15492	2.4	-0.45	
337	LIN 13432		-0.43	
343	EN15492	3.8	0.98	
357				
360	EN15492	3.92	1.10	
396				
444	EN15492	3.15	0.32	
468				
496	EN15492	3.57	0.75	
511				
541	D=0.40			
551	D7319	0.3	-2.60	
554 550				
558 621				
631				
633				
657	D7328	1.4627	-1.41	
663				
823				
913				
922				
1201	EN15492	0.65	-2.24	
1213	D7328	2.900	0.06	
1359	EN15492	0.47	-2.43	
1397	EN15492	4.27	1.46	
1446 1523				
1656				
1667				
1726				
1727	EN15492	2.03	-0.83	
1817				
1835	EN15492	3.50	0.67	
1852				
1919				
6201	EN45400			
6214	EN15492	2.7990	-0.04	
6224				
6262 6292				
6297				
6303				
6305				
-000				
	normality	OK		
	n	21		
	outliers	0		
	mean (n)	2.842		
	st.dev. (n)	1.5153		
	R(calc.)	4.243		
	st.dev.(D7319:17)	0.9766		application range, 1 20 mm/l/m
	R(D7319:17)	2.734		application range: 1 – 20 mg/kg
	compare R(EN15492:12)	0.741		application range: 1 - 20 mg/kg
	R(D7328:17)	1.595		application range: 0.55 – 20 mg/kg
	(= . == 0.11)			

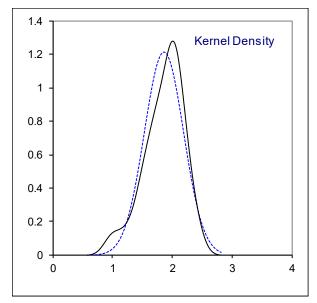




Determination of Sulfur on sample #19251; results in mg/kg

lab	method	value	mark z(targ)	remarks
	EN15486	2.0	0.11	TOTALINO
52 120	LIN 13400			
120	DE4E2	4.7		
150	D5453	1.7	-0.14	
169	D5453	1.85	-0.01	
171	D5453	<1		
174				
175	D5453	1.81	-0.05	
194				
230	In house			
311	D5453	2.1	0.19	
323	D0400			
	DE452			
329	D5453	2.0	0.11	
333	EN15486	1.7	-0.14	
334	ISO20846	1.9	0.03	
337	EN15486	<5		
343	D5453	2.0	0.11	
357	D5453	2.2	0.28	
360	EN15486	2.2	0.28	
396				
444	EN15485	1.26	-0.50	
468	EN15486	<2,0		
496	ISO20846	1.97	0.09	
511				
541				
551	D5453	1.5	-0.30	
554				
558				
621				
631				
633				
657	D5453	1.71	-0.13	
663	D5453	2.04	0.14	
823	D5453	2.1	0.19	
913	D5453	1.0	-0.71	
922	D5453	2.0	0.11	
1201	D5453	1.52	-0.28	
1213				
1359	In house	2.12	0.21	
1397	D2662	<3,0		
1446	DZ00Z			
1523				
	EN15406			
1656	EN15486	2.4	0.44	
1667				
1726				
1727				
1817				
1835	EN15486	2.3	0.36	
1852	ISO20846	1.734	-0.11	
1919				
6201	EN15486	1.503	-0.30	
6214		1.505	-0.50	
6224				
6262				
6292				
6297				
6303				
6305				
	normality	OK		
	n	25		
	outliers	0		
	mean (n)	1.865		
	st.dev. (n)	0.3284		
	R(calc.)	0.919		
	st.dev.(EN15485:07)	1.2109		
	R(EN15485:07)	3.391		application range: 7 – 20 mg/kg
	compare			
	R(EN15468:07)	1.968		application range: 5 – 20 mg/kg
	R(D5453:19a) [′]	0.925		at a concentration < 400 mg/kg; application range: 1 – 8000 mg/kg
	. ,			5 5. 11





APPENDIX 2

Number of participating laboratories per country:

- 1 lab in ARGENTINA
- 4 labs in BELGIUM
- 3 labs in BRAZIL
- 2 labs in BULGARIA
- 1 lab in CANADA
- 3 labs in COLOMBIA
- 1 lab in CROATIA
- 1 lab in CZECH REPUBLIC
- 1 lab in FINLAND
- 3 labs in FRANCE
- 2 labs in GERMANY
- 1 lab in HUNGARY
- 1 lab in INDIA
- 1 lab in INDONESIA
- 1 lab in ITALY
- 1 lab in MAURITIUS
- 5 labs in NETHERLANDS
- 1 lab in PAKISTAN
- 1 lab in PERU
- 2 labs in PHILIPPINES
- 1 lab in SINGAPORE
- 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

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

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

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

n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported

SDS = Safety Data Sheet

Literature

- 1 iis, Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- W. Horwitz and R. Albert, J. AOAC Int., <u>79</u>, <u>3</u>, 589, (1996)
- 3 ASTM E178:02
- 4 ASTM E1301:95(2003)
- 5 ISO 5725-86
- 6 ISO 5725, parts 1-6, 1994
- 7 ISO 13528:05
- 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, <u>118</u>, 455, (1993)
- 14 Analytical Methods Committee, Technical brief, No 4, January 2001
- P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 2002, <u>127</u>, 1359-1364 (2002).
- Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
- 17 M.A. Gonçalves et.al., Sensors and Actuators, <u>B158</u>, 327-332 (2011)