Results of Proficiency Test
Aviation gasoline 100LL
April 2014

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Since 2011, the Institute for Interlaboratory Studies organized a proficiency scheme for Aviation Gasoline 100LL. During the annual proficiency testing program 2013/2014. It was decided to continue the round robin for the analysis of Aviation Gasoline 100LL.
In this 2014 interlaboratory study 17 laboratories in 12 different countries have participated. See appendix 2 for the number of participants per country. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. It was decided to evaluate the Aviation Gasoline according to the test scope of ASTM D910:13. The analyses for fit-for-use and homogeneity testing were subcontracted. In this proficiency test, the participants received one sample of Aviation Gasoline 100LL.
Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system on IEC/ISO17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 Organization, Statistics and Evaluation' (iis-protocol, version 3.3), which can be downloaded from www.iisnl.com.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The necessary sample was obtained from an European supplier. After homogenisation, the material was transferred into 55 brown glass bottles of 1 litre (#14025). The homogeneity of the subsamples #14025 was checked by determination of Density @15°C in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Density @ 15°C in kg/m^3
Sample #14025-1	711.08
Sample #14025-2	711.09
Sample #14025-3	711.08
Sample #14025-4	711.13
Sample #14025-5	711.01
Sample #14025-6	711.07
Sample #14025-7	711.01
Sample #14025-8	711.03

table 1: homogeneity test results of subsamples #14025

From the above test results, the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method which is in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density @ 15 °C in kg/m^3
r (sample #14025)	0.12
reference test	D4052:02e1
0.3*R (reference test)	0.15

table 2: evaluation of repeatability of the subsamples #14025

The calculated repeatability is less than 0.3 times the reproducibility of the corresponding reference method. Therefore, homogeneity of the subsamples #14025 was assumed.

To the participants one 1L bottle of sample #14025 was sent on March 12, 2014.

2.5 ANALYSIS

The participants were requested to determine on sample #14025: Colour, Copper Strip Corrosion 2 hrs/100 ˚C, Density, Distillation, Existent Gum, Freezing Point, Heat of Combustion (Net), Lead as Pb, Lead as TEL, Lead participate, MON, Potential Gum, Sulphur and Water reaction interface (volume change).
To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download from www.iisnl.com.
A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

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

Directly after the deadline, a reminder fax was sent to the laboratories that had not reported results at that moment. Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ (iis-protocol, version 3.3). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.
In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test (see appendix 3, no.15). Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05). Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

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

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

3.2 GRAPHICS

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

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

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

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

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

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

$\lvert z\rvert < 1$	good
$1 < \lvert z\rvert < 2$	satisfactory
$2 < \lvert z\rvert < 3$	questionable
$3 < \lvert z\rvert$	unsatisfactory

4 EVALUATION

No problems were encountered during the execution of this proficiency test. All laboratories reported test results. Two laboratories reported the test results after the final reporting date. Not all laboratories were able to perform all analyses requested. Finally, 17 laboratories did report 193 numerical test results. Observed were 9 outlying test results, which is 4.7%. In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section, the results are discussed per sample and per test. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences where possible and applicable. These methods are also in the tables together with the reported data. The abbreviations, used in these tables, are listed in appendix 3. In the iis PT reports, ASTM methods are referred to with a number (e.g. D2086) and an added designation for the year that the method was adopted or revised (e.g. D2086-08). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2086-08 (2013)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

The majority of the data sets proved to have a normal distribution. For many other tests the number of reported test results was too small to determine whether the data set was normally distributed.

Aromatics: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D1319:13. The large spread may (partly) be explained by the small number of reported test results.

Colour: The reporting was very divers. Three participants reported to have used ASTM 2392, but did not report conform this standard “acceptable or fail”.

Copper Corrosion: No conclusions were drawn. All participants agreed on result 1.

Density @15ºC: This determination was not problematic. Two statistical outliers were observed (=13%). However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D4052:02e1.

Distillation: This determination was not problematic. Three statistical outliers were observed for FBP. The calculated reproducibilities, after rejection of the statistical outliers, are all in agreement with the requirements of ASTM D86:12.

Existent Gum: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after the rejection of the statistical outlier is in good agreement with the requirements of ASTM D381:12.

Freezing Point: All reporting participants agreed on a result below -58˚C, except one. The value of -58˚C is the upper limit for freezing point according to the product specification ASTM D910:13.

Heat of Combustion: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all agreement with the requirements of ASTM D3338:09. The large spread may (partly) be explained by the small number of test results.

Lead as Pb: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D3341:05(2011).

Lead as TEL: This determination may be very problematic. Only two laboratories reported a test result.
In order to create a significant statistical estimation, *iis* calculated TEL values from Lead as Pb. The estimated reproducibility is not at all in agreement with the requirements of ASTM D3341:05(2011).

Lead precipitate: No significant conclusions were drawn. Seven laboratories agreed on a value “less 1 mg/100ml” and one laboratory reported a test result of 1.

MON: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D2700:13b.

Potential Gum: This determination was problematic. No significant conclusions were drawn because two ‘less than <1’ test results, four numerical test results below 1 and two numerical test results of 4 and 8.2 were reported.

Sulphur: No significant conclusions were drawn. One false positive test result was observed. Probably, interference of lead in the sample maybe an explanation for the false positive test result.
All other reporting laboratories agreed on a result below of near the application range (0.0003% M/M – 4.6% M/M) of ASTM D2622:10.

Water reaction: This determination maybe not problematic. Twelve of the fourteen participants reported a test result below 1.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average values, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM standards) are compared in the next table.

Parameter	Unit	n	mean	2.8 * sd	R (lit)
Aromatics	%V/V	11	13.3	2.6	2.2
Colour	-----	6	blue	n.a.	n.a.
Copper Corrosion 2 hrs/100 ˚C	-----	16	1	n.a.	n.a.
Density @15˚C	kg/m^3	15	711.1	0.4	0.5
Distillation @ 760 mm Hg	-				
- Initial Boiling Point	˚C	17	36.2	3.1	5.2
- 10% evaporated	˚C	17	63.5	2.9	3.2
- 40% evaporated	˚C	17	97.5	2.0	4.6
- 50% evaporated	˚C	17	104.0	1.0	1.9
- 90% evaporated	˚C	17	126.5	2.8	3.5
- Final Boiling Point	˚C	14	152.4	2.2	6.8
Existent Gum	mg/100ml	9	0.6	0.6	2.2
Freezing Point	˚C	13	<-58	n.a.	2.5
Heat of Combustion (Net)	MJ/kg	10	43.756	0.112	0.046
Lead as Pb	g/l	11	0.54	0.06	0.03
Lead as TEL	ml/l	2	0.51	n.a.	0.03
Lead precipitate content	mg/100ml	7	<1	n.a.	n.a.
MON (lean mixture)	-----	8	102.5	1.9	2.0
Potential Gum	mg/100ml	6	<1	n.a	n.a
Sulphur	%M/M	10	<0.0003	n.a.	n.a
Water reaction interface	ml	12	<1	n.a.	n.a.

table 3: performance evaluation sample #14025

Without further statistical calculations, it can be concluded that for several tests there is not a good compliance of the group of participants with the relevant standards. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF APRIL 2014 WITH PREVIOUS PT

	April 2014	April 2013	April 2012	May 2011
Number of reporting labs	17	17	18	13
Number of results reported	193	209	222	163
Statistical outliers	9	6	7	2
Percentage outliers	4.7%	2.9%	3.2%	1.2%

table 4: Comparison with previous proficiency tests

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

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

Parameter	April 2014	April 2013	April 2012	May 2011
Aromatics	-	--	--	n.e.
Density @15˚C	+	++	++	++
Distillation @ 760 mm Hg	+	+	+	++
Existent Gum	++	+/-	(++)	++
Heat of Combustion (Net)	--	--	--	--
Lead as Pb	--	--	++	--
Lead as Tel	--	--	++	--
MON (lean mixture)	+	--	--	n.e.
Potential Gum	n.a.	+	(++)	n.e.
Sulphur	n.a.	n.a.	n.a.	--

table 5: comparison determinations against the standard requirements

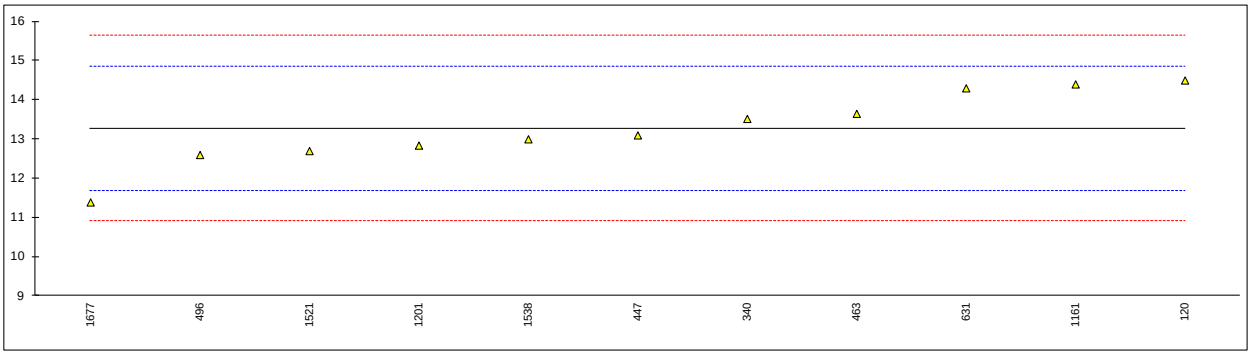
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

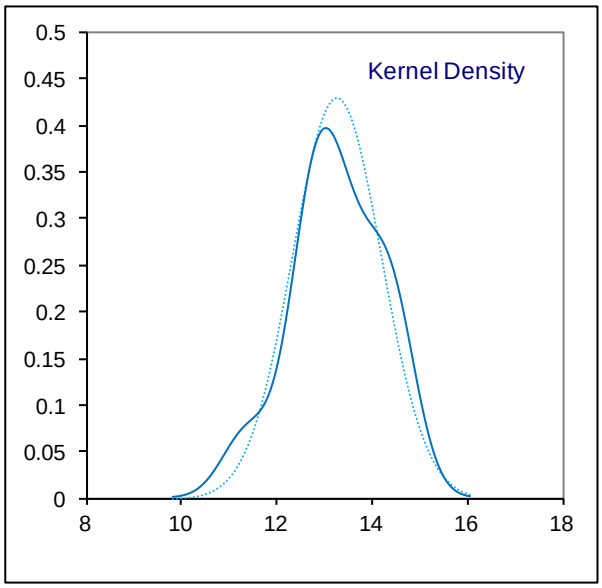
++: group performed much better than the standard
+ : group performed better than the standard
+/-: group performance equals the standard
- : group performed worse than the standard
-- : group performed much worse than the standard
n.e.: not evaluated

APPENDIX 1

Determination of Aromatics on sample #14025; results in %V/V

lab	method	value	mark	z(targ)	remarks
120	D5769	14.5		1.55	
334		-----		-----	
340	D1319	13.52		0.31	
353		-----		-----	
445		-----		-----	
447	D1319	13.1		-0.22	
463	D6379	13.65		0.48	
496	D1319	12.6		-0.85	
606		-----		-----	
631	D1319	14.3		1.30	
1016		-----		-----	
1161	D6379	14.4		1.43	
1201	D6379	12.84		-0.55	
1521	EN15553	12.7		-0.72	
1538	D1319	13.0		-0.35	
1677	D1319	11.39		-2.38	
1842		-----		-----	
					Only D1319 data
	normality	OK			suspect
	n	11			6
	outliers	0			0
	mean (n)	13.27			12.99
	st.dev. (n)	0.930			0.9717
	R(calc.)	2.60			2.72
	R(D1319:13)	2.21			2.16 Compare R(6379:11) = 1.56





Determination of Colour on sample #14025

lab	method	value	mark	z(targ)	remarks
120	Visual	Blue		-----	
334		-----		-----	
340		-----		-----	
353		-----		-----	
445	Visual	Blue		-----	
447	Visual	Blue		-----	
463		-----		-----	
496		-----		-----	
606		-----		-----	
631		-----		-----	
1016	D2392	Y1.3,B3.0		-----	
1161	D6045	>30		-----	
1201	D2392	Blue		-----	
1521	IP569	3.3 Blue		-----	
1538	Visual	Blue		-----	
1677	D2392	B3.0		-----	
1842	IP17	2.5		-----	
	normality	n.a.			
	n	10			
	outliers	n.a.			
	mean (n)	Acceptable / Blue			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D2392:96)	n.a.			

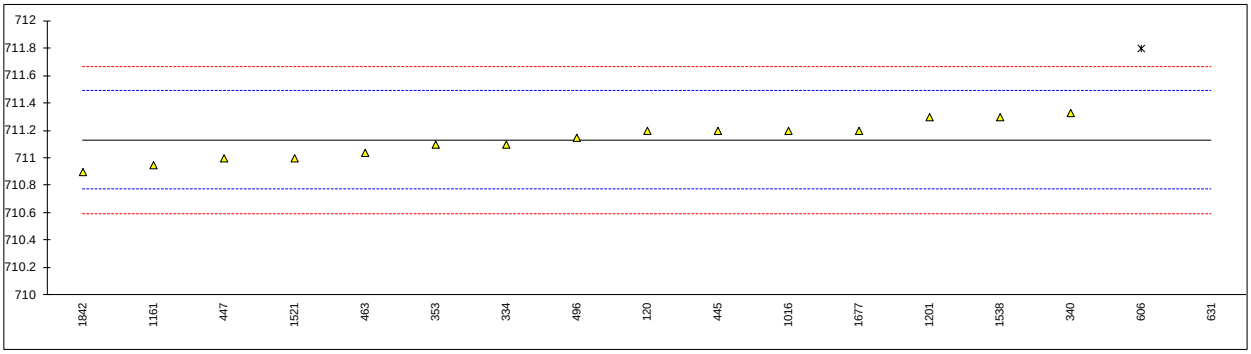
Determination of Copper Corrosion on sample #14025

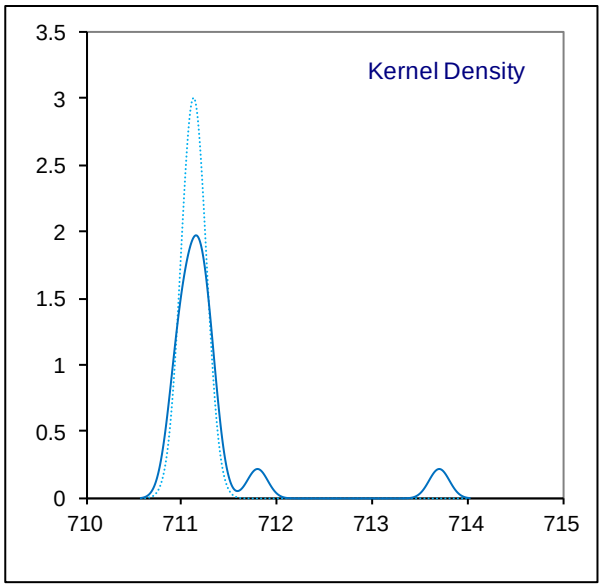
lab	method		value	mark	z(targ)	remarks
120	D130	1A			-----	
334	D130	1A			-----	
340	D130	1A			-----	
353	D130	1A			-----	
445	D130	1B			-----	
447	D130	1B			-----	
463	D130	1A			-----	
496	D130	1A			-----	
606	D130	1B			-----	
631	D130	1A			-----	
1016	D130	1A			-----	
1161	D130	1B			-----	
1201	D130	1A			-----	
1521	D130	1			-----	
1538		-----			-----	
1677	D130	1			-----	
1842	IP154	1A			-----	
	normality	n.a.				
	n	16				
	outliers	n.a.				
	mean (n)	1				
	st.dev. (n)	n.a.				
	R(calc.)	n.a.				
	R(D130:12)	n.a.				

Determination of Density @ 15°C on sample #14025; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
120	D4052	711.2		0.38	
334	D4052	711.1		-0.18	
340	D4052	711.33		1.11	
353	IP365	711.1		-0.18	
445	D4052	711.2		0.38	
447	D4052	711.0		-0.74	
463	D4052	711.04		-0.51	
496	D4052	711.15		0.10	
606	D4052	711.8	G(0.01)	3.74	
631	D4052	713.7	C,G(0.01)	14.38	first reported: 711.7
1016	D4052	711.2		0.38	
1161	ISO12185	710.95		-1.02	
1201	D4052	711.3		0.94	
1521	D4052	711.00		-0.74	
1538	D4052	711.30		0.94	
1677	D4052	711.2		0.38	
1842	D4052	710.9		-1.30	

normality	OK
n	15
outliers	2
mean (n)	711.13
st.dev. (n)	0.133
R(calc.)	0.37
R(D4052:02e1)	0.50



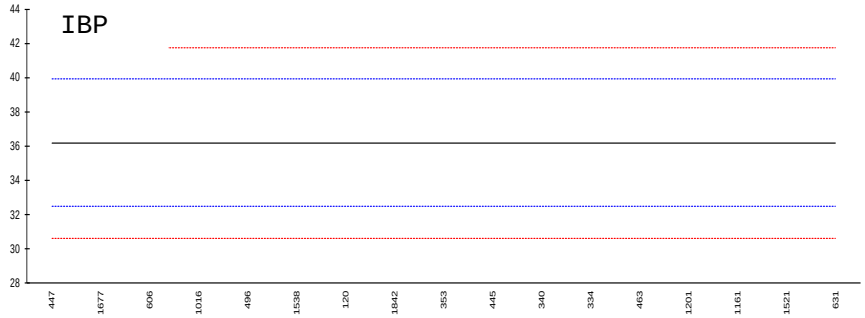


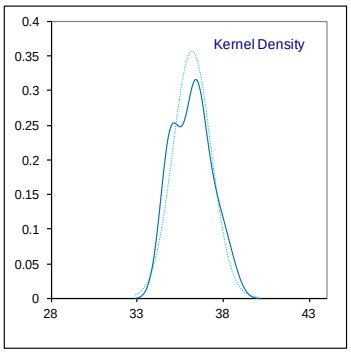
Determination of Distillation @ 760 mm Hg on sample #14025; results in °C.

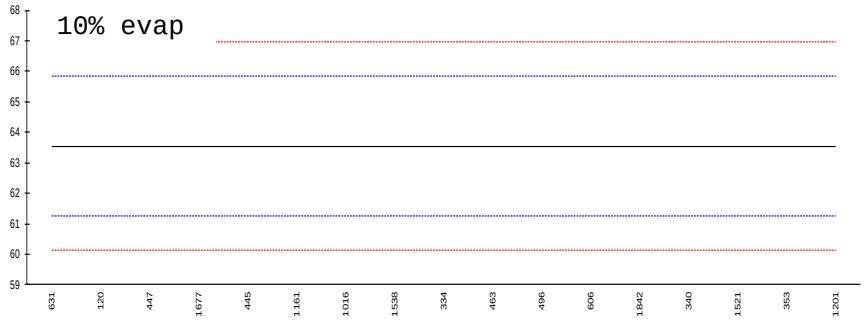
lab	method	IBP	mark	10%	mark	40%	mark	50%	mark	90%	mark	FBP	mark	residue
120	D86-A	36.2		62.1		97.1		103.8		126.2		152.2		0.8
334	D86-A	36.5		63.4		97.6		103.9		126.1		152.4		0.9
340	D86-A	36.5		64.3		97.1		103.6		125.6		153.3		0.8
353	IP123-A	36.3		65.2		98.7		104.5		129.0		153.0		0.8
445	D86-A	36.3		62.9		96.8		103.6		126.0		151.7		1.1
447	D86-A	34.6		62.3		98.1		104.5		126.4		152.4		1.0
463	D86-A	36.7		63.7		97.8		104.0		126.0		151.9		1.0
496	D86	35.1		64.0		97.2		103.7		125.7		151.8		0.8
606	D86-A	35.0		64.1		98.9		104.7		127.1		158.6	DG(0.05)	1.0
631	D86-M	38.4		62.0		96.0		103.5		125.5		153.5		0.6
1016	D86-A	35		63.2		98.1		104.2		125.8		154		1
1161	ISO3405-A	37.5		63.0		96.8		104.4		127.7		151.1		0.7
1201	D86-A	37.1		65.3		97.6		104.0		128.0		152.4		1.5
1521	D86-A	37.80		64.85		97.75		104.05		126.95		151.70		1.0
1538	D86-A	35.2		63.2		97.6		103.9		127.0		158.2	G(0.01)	0.6
1677	D86-A	34.8		62.6		96.8		103.9		125.7		152.5		0.8
1842	D86-A	36.2		64.2		97.6		104.1		125.9		159.5	DG(0.05)	0.6
	normality	OK		OK		OK		OK		not OK		OK		
	n	17		17		17		17		17		14		
	outliers	0		0		0		0		0		3		
	mean (n)	36.19		63.55		97.50		104.02		126.51		152.42		
	st.dev. (n)	1.117		1.029		0.726		0.347		0.983		0.797		
	R(calc.)	3.13		2.88		2.03		0.97		2.75		2.23		
	R(D86:12)	5.20		3.20		4.62		1.88		3.54		6.78		

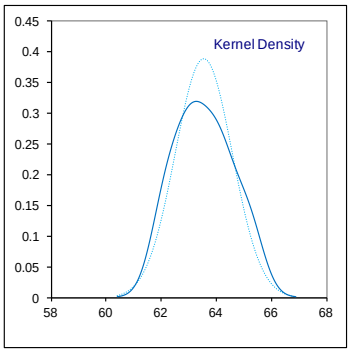
z-scores

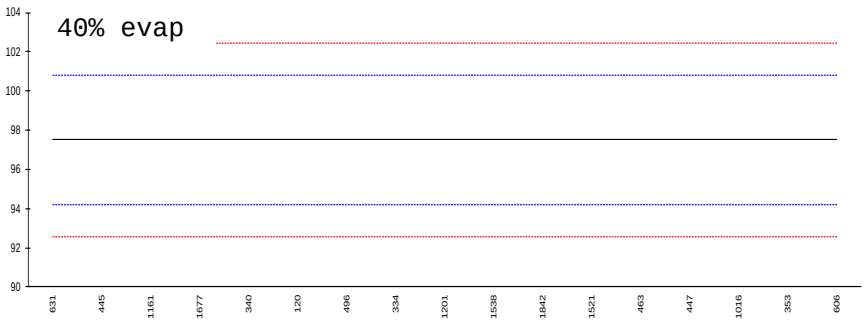
lab	method	IBP	10%	40%	50%	90%	FBP
120	D86-A	0.01	-1.27	-0.24	-0.33	-0.24	-0.09
334	D86-A	0.17	-0.13	0.06	-0.18	-0.32	-0.01
340	D86-A	0.17	0.66	-0.24	-0.63	-0.72	0.36
353	IP123-A	0.06	1.44	0.72	0.71	1.97	0.24
445	D86-A	0.06	-0.57	-0.43	-0.63	-0.40	-0.30
447	D86-A	-0.86	-1.09	0.36	0.71	-0.09	-0.01
463	D86-A	0.28	0.13	0.18	-0.03	-0.40	-0.22
496	D86	-0.59	0.39	-0.18	-0.48	-0.64	-0.26
606	D86-A	-0.64	0.48	0.85	1.01	0.47	2.55
631	D86-M	1.19	-1.36	-0.91	-0.78	-0.80	0.45
1016	D86-A	-0.64	-0.31	0.36	0.27	-0.56	0.65
1161	ISO3405-A	0.71	-0.48	-0.43	0.57	0.94	-0.55
1201	D86-A	0.49	1.53	0.06	-0.03	1.18	-0.01
1521	D86-A	0.87	1.14	0.15	0.04	0.35	-0.30
1538	D86-A	-0.53	-0.31	0.06	-0.18	0.39	2.39
1677	D86-A	-0.75	-0.83	-0.43	-0.18	-0.64	0.03
1842	D86-A	0.01	0.57	0.06	0.12	-0.48	2.92

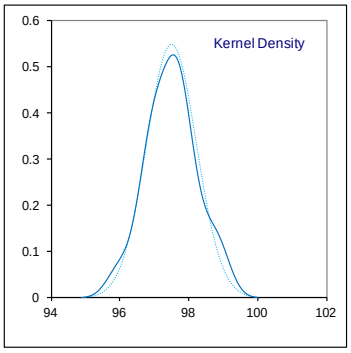

IBP
447 1677 606 1016 496 1538 120 1842 353 445 340 334 463 1201 1161 1521 631

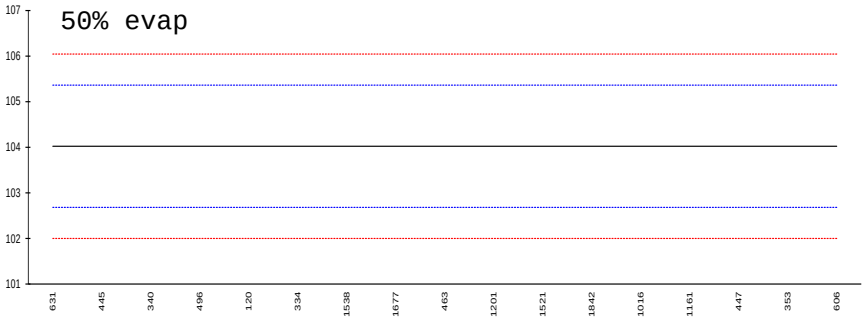

Kernel Density

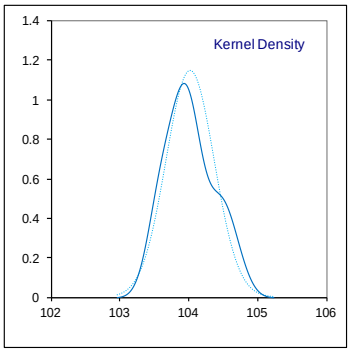

10% evap
631 120 447 1677 445 1161 1016 1538 334 463 496 606 1842 340 1521 353 1201

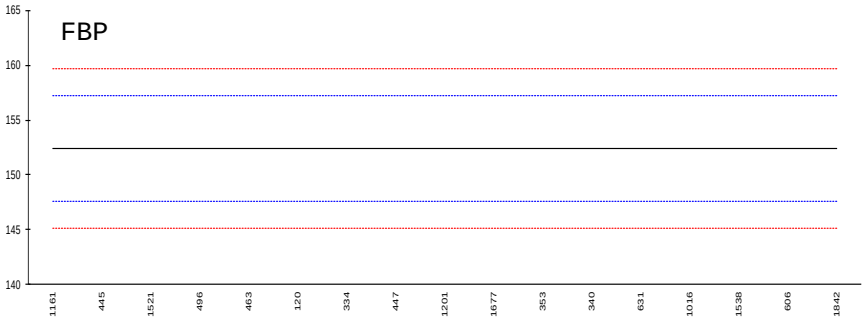

Kernel Density

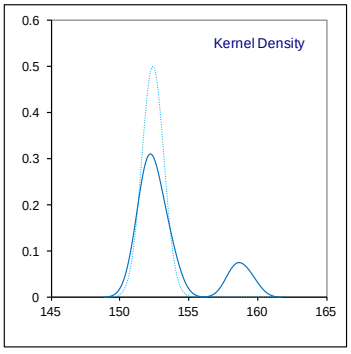

40% evap
631 445 1161 1677 340 120 496 334 1201 1538 1842 1521 463 447 1016 353 606


Kernel Density


50% evap
631 445 340 496 120 334 1538 1677 463 1201 1521 1842 1016 1161 447 353 606


Kernel Density

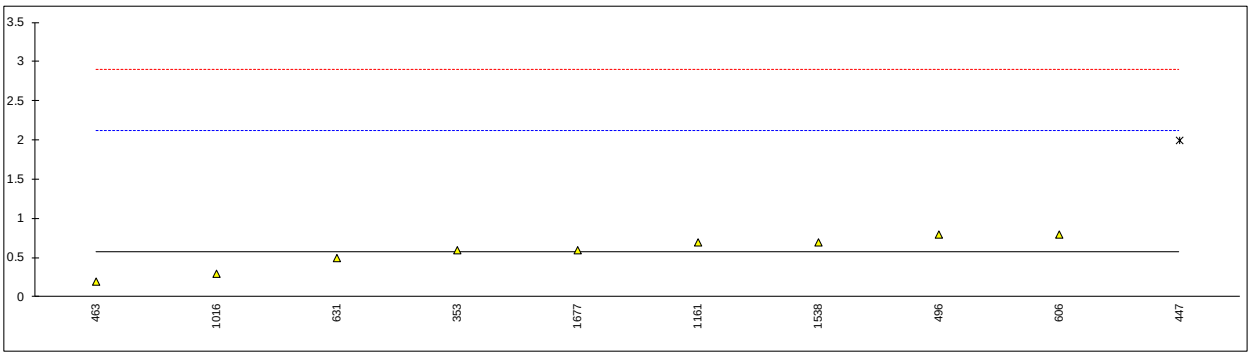

FBP
1161 445 1521 496 463 120 334 447 1201 1677 353 340 631 1016 1538 606 1842

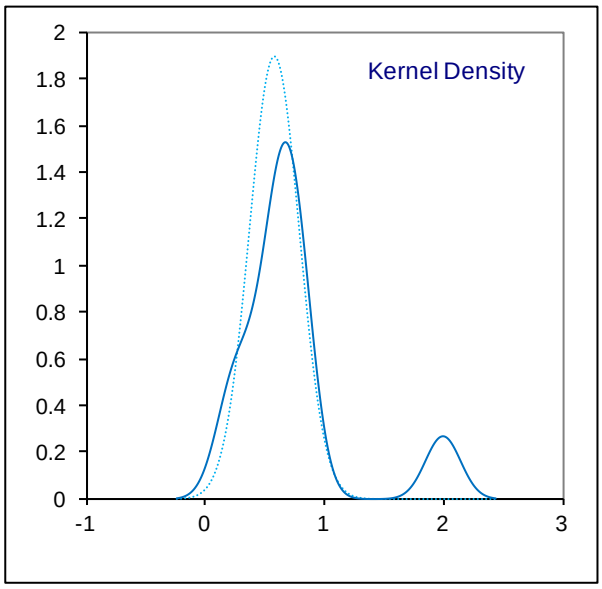

Kernel Density

Determination of Existent Gum on sample #14025; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
120	D381	<0.5		-----	
334		-----		-----	
340	D381	<1		-----	
353	IP131	0.6		0.03	
445	D381	<1		-----	
447	D381	2	G(0.01)	1.84	
463	D381	0.2		-0.49	
496	D381	0.8		0.29	
606	D381	0.80		0.29	
631	D381	0.5		-0.10	
1016	D381	0.3		-0.36	
1161	D381	0.7		0.16	
1201	D381	<1.0		-----	
1521	D381	<1		-----	
1538	D381	0.7		0.16	
1677	D381	0.6		0.03	
1842	IP131	<1		-----	

normality	OK
n	9
outliers	1
mean (n)	0.58
st.dev. (n)	0.211
R(calc.)	0.59
R(D381:12)	2.16





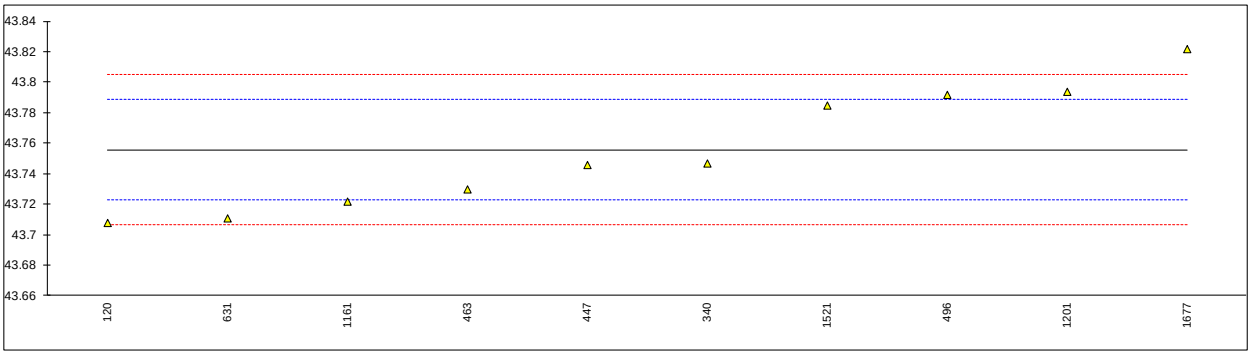
Determination of Freezing Point on sample #14025; results in °C

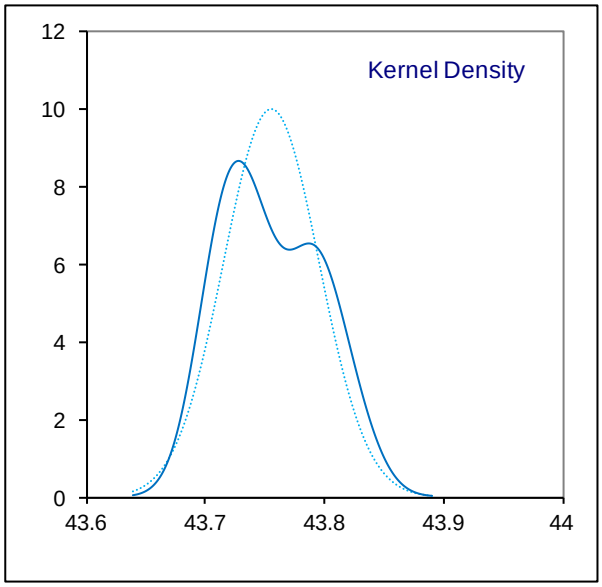
lab	method	value	mark	z(targ)	remarks
120	D2386	<-58.0		-----	
334		-----		-----	
340	D2386	<-70		-----	
353		-----		-----	
445	D2386	<-65		-----	
447	D2386	<-58		-----	
463	D2386	<-65		-----	
496	D2386	<-77.5		-----	
606		-----		-----	
631	D2386	<-58		-----	
1016	D2386	<-58		-----	
1161	D2386	<-55		-----	see §4.1
1201	D2386	<-67		-----	
1521	D7153	<-60		-----	
1538	D2386	<-80		-----	
1677	D2386	<-80		-----	
1842	IP16	<-80		-----	

normality	n.a
n	13
outliers	n.a
mean (n)	<-58
st.dev. (n)	n.a
R(calc.)	n.a
R(D2386:12)	2.5

Determination of Heat of Combustion (Net) on sample #14025; results in MJ/kg

lab	method	value	mark	z(targ)	remarks
120	D3338	43.708		-2.90	
334		-----		-----	
340	D3338	43.747		-0.53	
353		-----		-----	
445		-----		-----	
447	D3338	43.746	E	-0.59	iis calculated 43.764
463	D3338	43.73	E	-1.56	iis calculated 43.745
496	D3338	43.792	E	2.21	iis calculated 43.781
606		-----		-----	
631	D3338	43.711	E	-2.72	iis calculated 43.686
1016		-----		-----	
1161	D3338	43.722		-2.05	
1201	D3338	43.794	E	2.33	iis calculated 43.780
1521	D3338	43.785		1.78	
1538		-----		-----	
1677	D3338	43.822		4.04	
1842		-----		-----	
					calc. by iis
	normality	OK			OK
	n	10			11
	outliers	0			0
	mean (n)	43.7557			43.7552
	st.dev. (n)	0.03983			0.0390
	R(calc.)	0.1115			0.1093
	R(D3338:09)	0.0460			0.0460

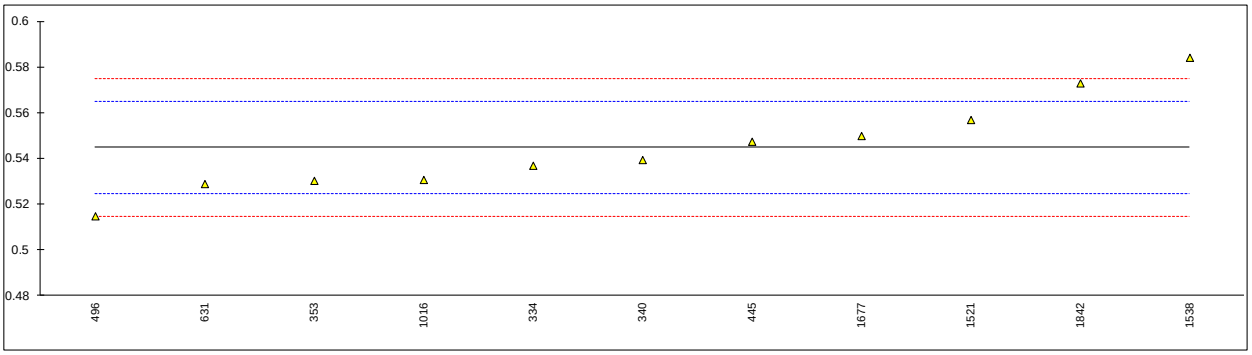


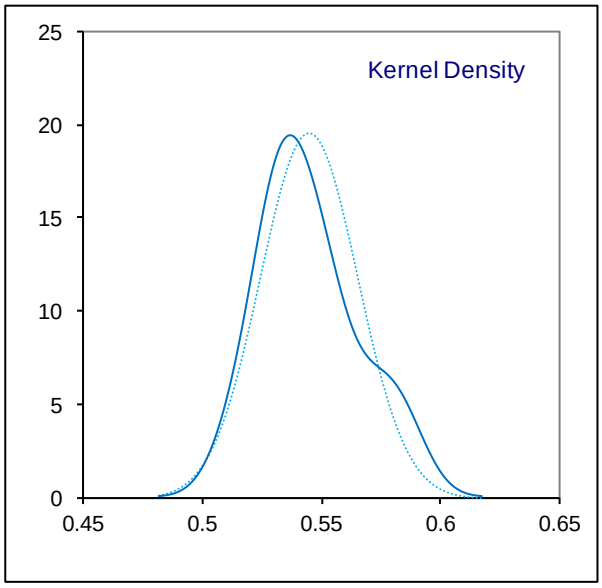


Determination of Lead as Pb on sample #14025; results in g/l

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
334	D3341	0.537		-0.78	
340	D3341	0.5395		-0.53	
353	IP270	0.5304		-1.43	
445	IP270	0.5475		0.26	
447		-----		-----	
463		-----		-----	
496	D3341	0.5149		-2.97	
606		-----		-----	
631	IP428	0.529	C	-1.57	first reported:0.469
1016	D5059	0.5308		-1.39	
1161		-----		-----	
1201		-----		-----	
1521	IP362	0.5570		1.21	
1538	D5059	0.5842		3.91	
1677	D3341	0.55		0.51	
1842	IP228	0.573		2.79	

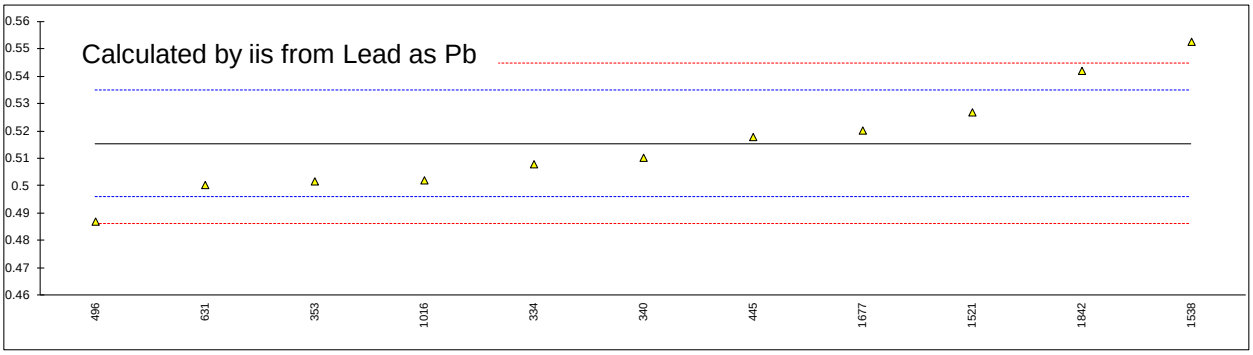
normality	OK
n	11
outliers	0
mean (n)	0.5448
st.dev. (n)	0.02041
R(calc.)	0.0572
R(D3341:05)	0.0282

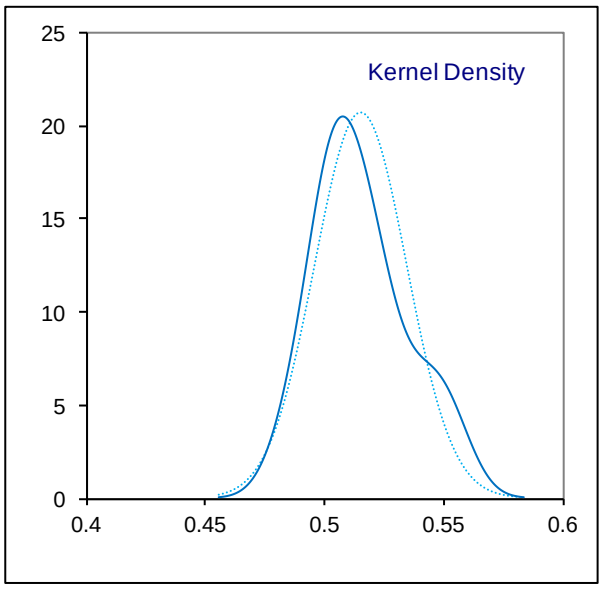




Determination of Lead as TEL on sample #14025; results in ml/l

lab	method	value	mark	z(targ)	calc. by iis, from Lead as Pb	remarks
120		-----		-----	-----	
334		-----		-----	0.5080	
340		-----		-----	0.5104	
353	IP270	0.5017		-1.40	0.5018	z-score calc .from Lead as Pb to Lead as TEL
445	IP270	0.5180		0.27	0.5179	z-score calc .from Lead as Pb to Lead as TEL
447		-----		-----	-----	
463		-----		-----	-----	
496		-----		-----	0.4871	
606		-----		-----	-----	
631		-----		-----	0.5004	
1016		-----		-----	0.5021	
1161		-----		-----	-----	
1201		-----		-----	-----	
1521		-----		-----	0.5269	
1538		-----		-----	0.5527	
1677		-----		-----	0.5203	
1842		-----		-----	0.5421	
	normality	unknown			OK	
	n	n			11	
	outliers	2			0	
	mean (n)	0.5100			0.5154	
	st.dev. (n)	n.a			0.01931	
	R(calc.)	n.a			0.0541	
	R(D3341:05)	n.a			0.0274	





Determination of Lead precipitate content on sample #14025; results in mg/100ml

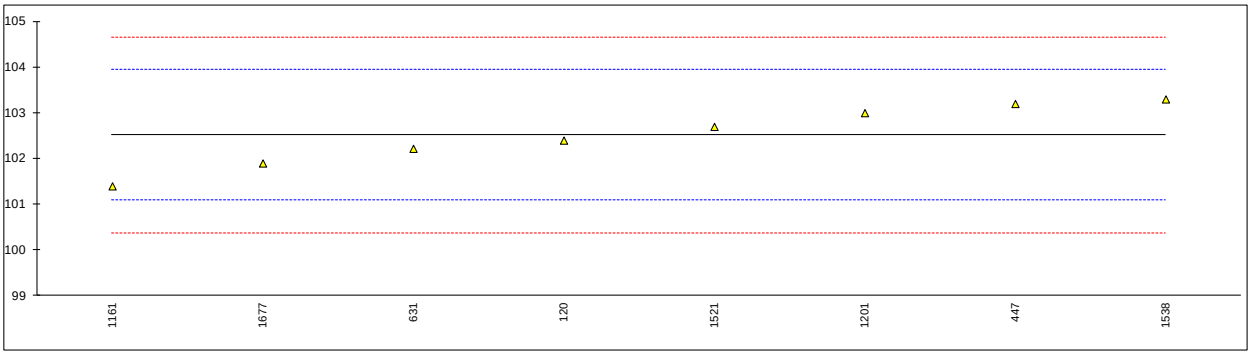
lab	method	value	mark	z(targ)	remarks
120	D873	<1.0		-----	
334		-----		-----	
340	D873	1		-----	
353		-----		-----	
445	D873	0.8		-----	
447		-----		-----	
463		-----		-----	
496		-----		-----	
606		-----		-----	
631	D873	<1		-----	
1016	D873	<1		-----	
1161		-----		-----	
1201		-----		-----	
1521	D873	<1		-----	
1538	D873	<1		-----	
1677	D873	<1		-----	
1842		-----		-----	

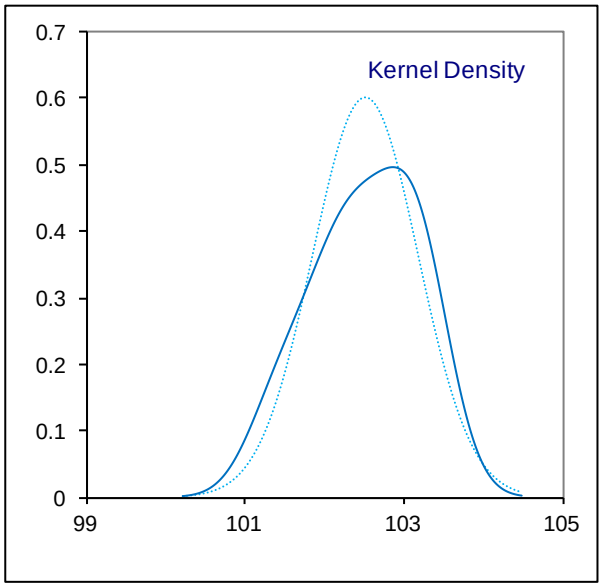
normality	n.a
n	7
outliers	n.a.
mean (n)	<1
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D873:12)	n.a.

Determination of MON (lean mixture) on sample #14025

lab	method	value	mark	z(targ)	remarks
120	D2700	102.4		-0.16	
334		-----		-----	
340		-----		-----	
353		-----		-----	
445		-----		-----	
447	D2700	103.2		0.96	
463	D2700	>100		-----	
496		-----		-----	
606		-----		-----	
631	D2700	102.22		-0.41	
1016		-----		-----	
1161	ISO5163	101.4		-1.56	
1201	D2700	103.0		0.68	
1521	D2700	102.7		0.26	
1538	D2700	103.3		1.10	
1677	D2700	101.9		-0.86	
1842		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	102.51
st.dev. (n)	0.663
R(calc.)	1.86
R(D2700:13b)	2.00





Determination of Potential Gum on sample #14025; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
120	D873	<1.0		-----	
334		-----		-----	
340	D873	4		-----	false positive result?
353		-----		-----	
445	D873	8.2		-----	false positive result?
447		-----		-----	
463		-----		-----	
496		-----		-----	
606		-----		-----	
631	D873	0.5		-----	
1016	D873	0.2		-----	
1161		-----		-----	
1201		-----		-----	
1521	D873	<1		-----	
1538	D873	0.5		-----	
1677	D873	0.3		-----	
1842		-----		-----	

normality	n.a
n	6
outliers	n.a
mean (n)	<1
st.dev. (n)	n.a
R(calc.)	n.a
R(D873:12)	n.a

Determination of Sulphur content on sample #14025; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D2622	<0.0003		-----	
334	D5453	0.00012		-----	
340	D2622	<0.0003		-----	
353		-----		-----	
445	D2622	<0.0003		-----	
447		-----		-----	
463		-----		-----	
496	D2622	0		-----	
606		-----		-----	
631		-----		-----	
1016	D2622	<0.0003		-----	
1161	ISO20846	0.0001		-----	
1201	D2622	0.0358		-----	false positive result?
1521	D2622	<0.0003		-----	
1538	D5453	<0.0001		-----	
1677	D2622	<1		-----	probably unit error?
1842	D2622	0.0001		-----	
	normality	n.a			
	n	10			
	outliers	n.a			
	mean (n)	<0.0003			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(D2622:10)	n.a			Application range: (0.0003 %M/M – 4.6 %M/M)

Determination of Water reaction interface on sample #14025; results in ml

lab	method	value	mark	z(targ)	remarks
120	D1094	1		-----	
334		-----		-----	
340	D1094	0		-----	
353		-----		-----	
445	D1094	0.5		-----	
447	D1094	<0.5		-----	
463	D1094	<0.5		-----	
496		-----		-----	
606	D1094	0.0		-----	
631	D1094	0.5		-----	
1016	D1094	<1		-----	
1161	D1094	<1		-----	
1201	D1094	<0.5		-----	
1521	D1094	0.5		-----	
1538	D1094	1		-----	
1677	D1094	0		-----	
1842	IP289	0		-----	

normality	OK
n	12
outliers	0
mean (n)	<1
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D1094:07)	n.a.

APPENDIX 2

Number of participants per country

1 lab in	ESTONIA
2 labs in	FRANCE
1 lab in	GERMANY
1 lab in	IRELAND
1 lab in	MALAYSIA
2 labs in	NETHERLANDS
1 lab in	PHILIPPINES
2 labs in	POLAND
1 lab in	SWEDEN
1 lab in	TURKEY
3 labs in	UNITED KINGDOM
1 lab in	UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
ex	= excluded from calculations
E	= error in calculations
n.e.	= not evaluated
W	= withdrawn
fr.	= first reported
U	= reported in different unit
SDS	= Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014

2 ASTM E178-02

3 ASTM E1301-03

4 ISO 5725-86

5 ISO 5725, parts 1-6, 1994

6 ISO13528-05

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

8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)

9 IP 367/84

10 DIN 38402 T41/42

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

12 J.N. Miller, Analyst, 118, 455, (1993)

13 Analytical Methods Committee Technical Brief, No4 January 2001

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

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