Results of Proficiency Test
Aviation gasoline 100LL
April 2013

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2011, the Institute for Interlaboratory Studies organized a proficiency scheme for Aviation Gasoline 100LL. During the annual proficiency testing program 2012/2013, it was decided to continue the round robin for the analysis of Aviation Gasoline 100LL.
In this 2013 interlaboratory study 19 laboratories in 15 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2013 Aviation Gasoline proficiency test are presented and discussed. 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 based on ISO/IEC17043:2010 and ILAC-G13:2007(R007). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Also customer's satisfaction is measured on a 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' of January 2010 (iis-protocol, version 3.2), 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 (#13050). The homogeneity of the subsamples #13050 was checked by determination of Density @15°C in accordance with ASTM D4052:11 on 8 stratified randomly selected samples.

	Density @ 15°C in kg/m^3
Sample #13050-1	713.53
Sample #13050-2	713.58
Sample #13050-3	713.59
Sample #13050-4	713.58
Sample #13050-5	713.60
Sample #13050-6	713.60
Sample #13050-7	713.60
Sample #13050-8	713.61

table 1: homogeneity test results of subsamples #13050

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 #13050)	0.07
reference test	D4052:02e1
0.3*R (reference test)	0.15

table 2: evaluation of repeatability of the subsamples #13050

The calculated repeatability is less than 0.3 times the reproducibility of the corresponding reference method. Therefore, homogeneity of the subsamples #13050 was assumed.

To the participants one 1L bottle of sample #13050 was sent on April 10, 2013.

2.5 ANALYSIS

The participants were requested to determine on sample #13050: 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' of January 2010 (*iis*-protocol, version 3.2).

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

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

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported 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).

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. 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 the fit-for-useness of the reported test result.

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 is as follows:

	| z | < 1	good
1 <	| z | < 2	satisfactory
2 <	| z | < 3	questionable
3 <	| z |	unsatisfactory

4 EVALUATION

No problems were encountered during the execution of this proficiency test. One laboratory reported the test results after the final reporting date. Two laboratories reported no test results. And not all laboratories were able to perform all analyses requested. Finally, 17 laboratories did report 209 numerical test results. Observed were 6 outlying test results, which is 2.9%. 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 test. For the Density determination and for Lead as Pb, not normal distributions were found. 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 not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is almost in agreement with the requirements of ASTM D1319:13.

Colour: Seven laboratories reported a test result of “Blue”, which is a positive identification of Avgas grade 100LL. No further conclusions were drawn.

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

Density @15ºC: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D4052:02e1.

Distillation: This determination was not problematic. Two statistical outliers were observed, one for 10% and one for 50% evaporated. The calculated reproducibilities, after rejection of the statistical outliers, are all in good agreement with the requirements of ASTM D86:12a.

Existent Gum: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with the requirements of ASTM D381:12.

Freezing Point: In this determination no problems have been observed. All reporting participants agreed on a result below -58˚C. This value is the upper limit for freezing point according to the product specification ASTM D910:13.

Heat of Combustion: This determination was very problematic. Two test results were excluded for statistical evaluation, because of calculation errors. In order to create a more significant estimation, *iis* recalculated the excluded test results and re-evaluated the statistical data.
Both the initial and the re-evaluated reproducibilities are not at all in agreement with the requirements of ASTM D3338:09.

Lead as Pb: This determination was very problematic. One statistical outlier was observed.
The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D3341:11.

Lead as TEL: This determination may be very problematic. The low numbers of reported test results may (partly) explain the large spread.
In order to create a more 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 D3341:11.

Lead precipitate: No significant conclusions were drawn. The eight reporting laboratories agreed on a value “less 1 mg/100ml”.

MON: 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 D2700:12.

Potential Gum: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D873:12.

Sulphur: This determination may be problematic for some laboratories. Two statistical outliers (false positive test results) were observed. Probably, interference of lead in the sample may be an explanation for the false positive test results.
All other reporting laboratories agreed on a result below of near the detection limit of ASTM D2622 (0.0003%).

Water reaction: This determination may be not problematic. All participants reported 1 or 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	9	14.6	2.8	2.5
Colour	-----	7	blue	n.a.	n.a.
Copper Corrosion 2 hrs/100 ˚C	-----	17	1	n.a.	n.a.
Density @15˚C	kg/m^3	17	713.7	0.7	0.5
Distillation @ 760 mm Hg	-				
- Initial Boiling Point	˚C	17	34.7	4.5	5.1
- 10% evaporated	˚C	16	61.0	3.2	3.2
- 40% evaporated	˚C	17	96.1	1.5	4.1
- 50% evaporated	˚C	16	103.4	1.6	1.9
- 90% evaporated	˚C	17	128.7	2.0	3.1
- Final Boiling Point	˚C	17	157.2	2.8	6.8
Existent Gum	mg/100ml	13	1.3	2.4	2.3
Freezing Point	˚C	12	< -58	n.a.	2.5
Heat of Combustion (Net)	MJ/kg	9	43.682	0.116	0.046
Lead as Pb	g/l	9	0.49	0.10	0.03
Lead as TEL	ml/l	4	0.47	n.a.	0.03
Lead precipitate content	mg/100ml	8	<1	n.a.	n.a.
MON (lean mixture)	-----	9	101.5	3.1	0.9
Potential Gum	mg/100ml	8	1.9	2.7	3.0
Sulphur	%M/M	7	<0.0003	n.a.	n.a.
Water reaction interface	ml	9	1 or <1	n.a.	n.a.

table 3: performance evaluation sample #13050

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 2013 WITH PREVIOUS PT

	April 2013	April 2012	May 2011
Number of reporting labs	17	18	13
Number of results reported	209	222	163
Statistical outliers	6	7	2
Percentage outliers	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 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.e.
Sulphur	n.a.	n.a.	--

table 5: comparison determinations against the standard requirements

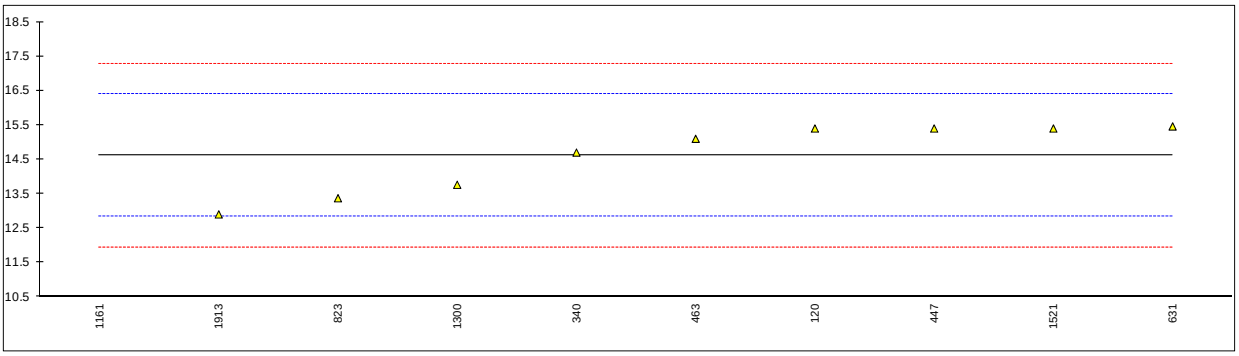
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

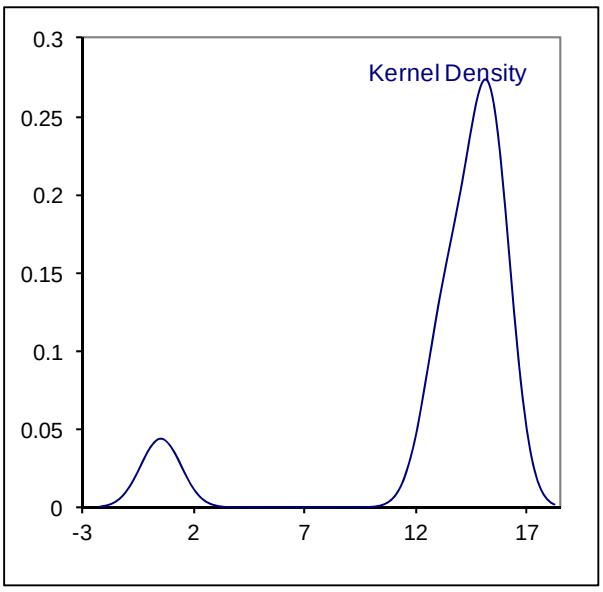
++: group performed much better than the standard
+ : group performed better than the standard
+/-: group performance equals the standard
- : group performed worse than the standard
-- : group performed much worse than the standard
n.e.: not evaluated

APPENDIX 1

Determination of Aromatics on sample #13050; results in %V/V

lab	method	value	mark	z(targ)	remarks
120	D1319	15.4		0.88	
334		-----		-----	
340	D1319	14.7		0.10	
353		-----		-----	
445		-----		-----	
447	D1319	15.4		0.88	
463	D1319	15.1		0.55	
496		-----		-----	
606		-----		-----	
631	D1319	15.46		0.95	
823	D6379	13.37		-1.39	
962		-----		-----	
1017		-----		-----	
1161	D6379	0.5	G(0.01)	-15.80	
1300	D6379	13.7620		-0.95	
1428		-----		-----	
1521	EN15553	15.4		0.88	
1677		-----		-----	
1913	D1319	12.9		-1.92	
	normality	OK			
	n	9			
	outliers	1			
	mean (n)	14.61			
	st.dev. (n)	1.002			
	R(calc.)	2.80			
	R(D1319:13)	2.50			Compare R(D6379:11) = 1.66





Determination of Colour on sample #13050

lab	method	value	mark	z(targ)	remarks
120	Visual	Blue		-----	
334		-----		-----	
340	Visual	Blue		-----	
353		-----		-----	
445	Visual	Blue		-----	
447	Visual	Blue		-----	
463		-----		-----	
496	D2392	Blue		-----	
606		-----		-----	
631		-----		-----	
823		-----		-----	
962		-----		-----	
1017		-----		-----	
1161	D2392	Acceptable		-----	
1300	D2392	Green		-----	
1428		-----		-----	
1521	IP17	3.0 Blue		-----	
1677	IP569	0.0R 1.9Y **2.9B** 0.8N		-----	
1913		-----		-----	

normality	n.a.
n	7
outliers	n.a.
mean (n)	blue
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D2392:11)	n.a.

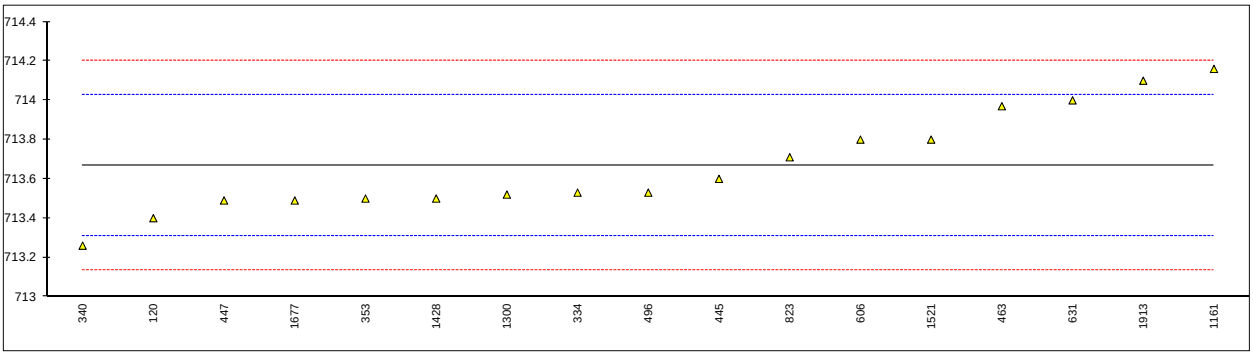
Determination of Copper Corrosion on sample #13050

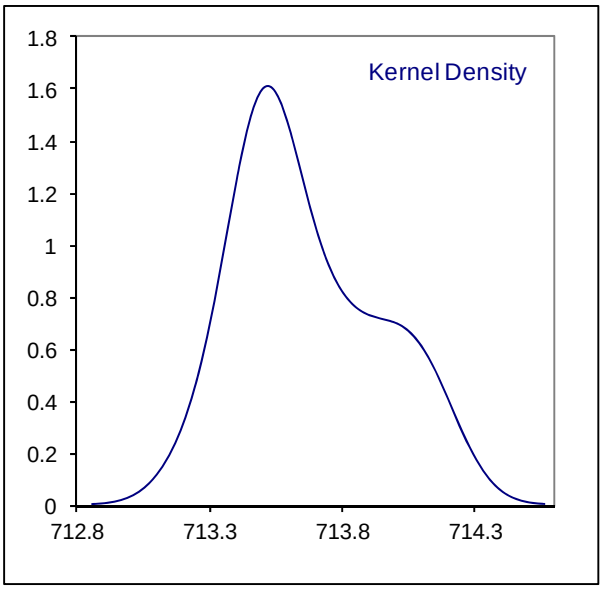
lab	method	value	mark	z(targ)	remarks
120	D130	1A		-----	
334	D130	1A		-----	
340	D130	1A		-----	
353	D130	1A		-----	
445	IP154	1A		-----	
447	D130	1B		-----	
463	D130	1A		-----	
496	D130	1A		-----	
606	D130	1A		-----	
631	D130	1B		-----	
823	D130	1A		-----	
962		-----		-----	
1017		-----		-----	
1161	D130	1A		-----	
1300	D130	1A		-----	
1428	ISO2160	1A		-----	
1521	D130	1		-----	
1677	D130	1B		-----	
1913	D130	1B		-----	
	normality	n.a.			
	n	17			
	outliers	0			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D130:12)	n.a.			

Determination of Density @ 15°C on sample #13050; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
120	D4052	713.4		-1.50	
334	D4052	713.53		-0.77	
340	D4052	713.26		-2.29	
353	IP365	713.5		-0.94	
445	D4052	713.6		-0.38	
447	D4052	713.49		-1.00	
463	D4052	713.97		1.69	
496	D4052	713.53		-0.77	
606	D4052	713.8		0.74	
631	D4052	714.0		1.86	
823	D4052	713.71	C	0.23	first reported 714.97
962		-----		-----	
1017		-----		-----	
1161	D4052	714.16		2.75	
1300	D4052	713.52		-0.83	
1428	ISO12185	713.5		-0.94	
1521	D4052	713.8		0.74	
1677	D4052	713.49		-1.00	
1913	D4052	714.1		2.42	

normality	not OK
n	17
outliers	0
mean (n)	713.67
st.dev. (n)	0.261
R(calc.)	0.73
R(D4052:02e1)	0.50





Distillation @ 760 mm Hg (automated + manual) on sample #13050 results in °C.

lab	method	IBP	mark	10%	mark	40%	mark	50%	mark	90%	mark	FBP	mark
120	D86-A	33.3		59.4		95.7		103.1		128.2		157.7	
334	D86-A	33.3		61.1		95.9		102.8		128.2		155.4	
340	D86-A	34.4		61.5		96.4		103.7		129.6		157.3	
353	IP123-A	34.2		60.5		96.7		103.9		129.1		158.3	
445	IP123-A	34.3		61.6		95.5		103.2		128.2		158.0	
447	D86-A	31.9		59.4		95.9		103.2		128.2		156.4	
463	D86-A	33.2		65.5	C, G(0.05)	95.6		102.9		130.5		155.9	
496	D86-A	33.7		60.6		96.1		103.4		128.7		158.0	
606	D86-A	33.9		61.2		96.4		103.6		128.8		157.2	
631	D86-M	37.0		60.0		97	C	105		129.5		157.0	
823	D86-A	37.1		61.9		96.2		103.3		129.0		158.0	
962		-----		-----		-----		-----		-----		-----	
1017		-----		-----		-----		-----		-----		-----	
1161	D86-A	36.3		62.6		97.0		105.7	G(0.05)	128.0		155.7	
1300	D86-A	37.0		58.9		94.9	C	102.5	C	128.5		156.4	
1428	ISO3405-A	35.9		62.9		95.8		103.1		128.7		157.1	
1521	D86-A	36.1		61.5		96.1		103.4		129.1		157.7	
1677	D86-A	33.0		61.5		95.7		103.5		128.7		159.0	
1913	D86-A	34.8		62.0		95.9		103.2		127.4	C	157.6	
	normality	OK		OK		OK		OK		OK		OK	
	n	17		16		17		16		17		17	
	outliers	0		1		0		1		0		0	
	mean (n)	34.67		61.03		96.05		103.36		128.73		157.22	
	st.dev. (n)	1.607		1.156		0.542		0.557		0.721		0.986	
	R(calc.)	4.50		3.24		1.52		1.56		2.02		2.76	
	R(D86:12a)	5.11		3.20		(4.06)*		1.88		3.10		6.78	

*Calculated from D86:07a

Lab 463 first reported 67.1

Lab 631 first reported 94.5

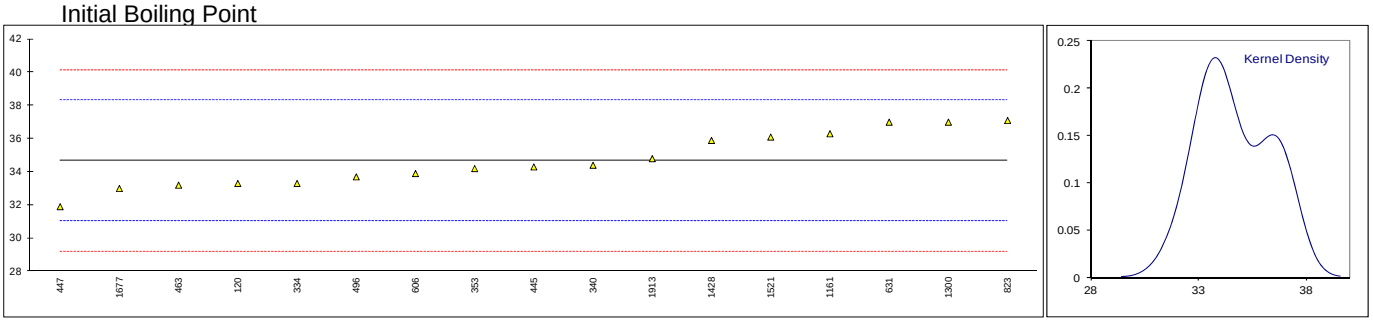
Lab 1300 first reported for 40%: 93.5 ; for 50%: 101.8

Lab 1913 first reported 131.2

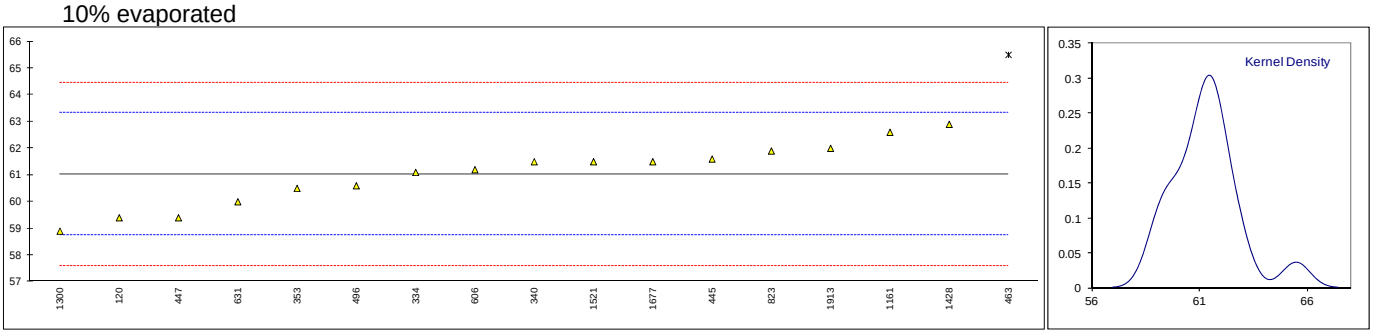
Z-scores

lab	IBP	10%	40%	50%	90%	FBP
120	-0.75	-1.43	-0.17	-0.39	-0.48	0.20
334	-0.75	0.05	-0.07	-0.84	-0.48	-0.75
340	-0.15	0.40	0.17	0.50	0.79	0.03
353	-0.26	-0.47	0.31	0.80	0.33	0.45
445	-0.20	0.49	-0.26	-0.24	-0.48	0.32
447	-1.52	-1.43	-0.07	-0.24	-0.48	-0.34
463	-0.81	3.90	-0.22	-0.69	1.60	-0.54
496	-0.53	-0.38	0.03	0.06	-0.03	0.32
606	-0.42	0.14	0.17	0.35	0.06	-0.01
631	1.28	-0.91	0.46	2.44	0.70	-0.09
823	1.33	0.75	0.07	-0.09	0.24	0.32
962	-----	-----	-----	-----	-----	-----
1017	-----	-----	-----	-----	-----	-----
1161	0.89	1.37	0.46	3.48	-0.66	-0.63
1300	1.28	-1.87	-0.55	-1.28	-0.21	-0.34
1428	0.67	1.63	-0.12	-0.39	-0.03	-0.05
1521	0.78	0.40	0.03	0.06	0.33	0.20
1677	-0.92	0.40	-0.17	0.20	-0.03	0.74
1913	0.07	0.84	-0.07	-0.24	-1.20	0.16

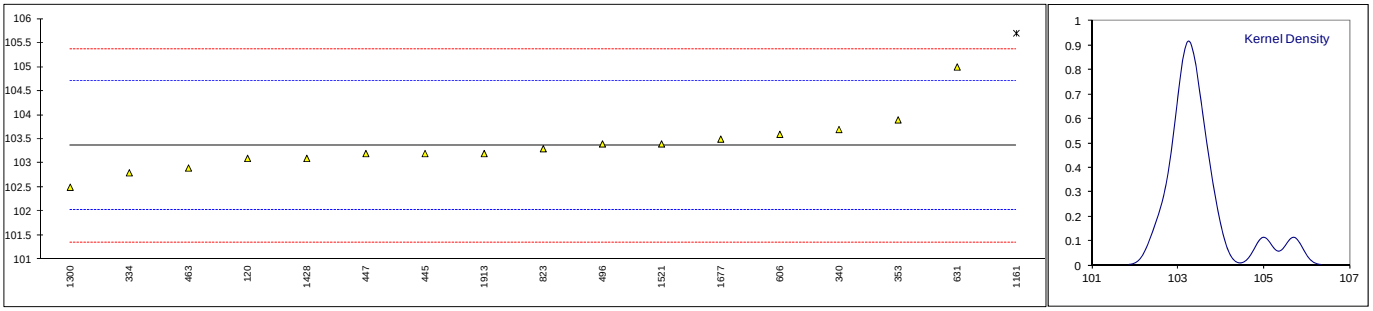
Initial Boiling Point



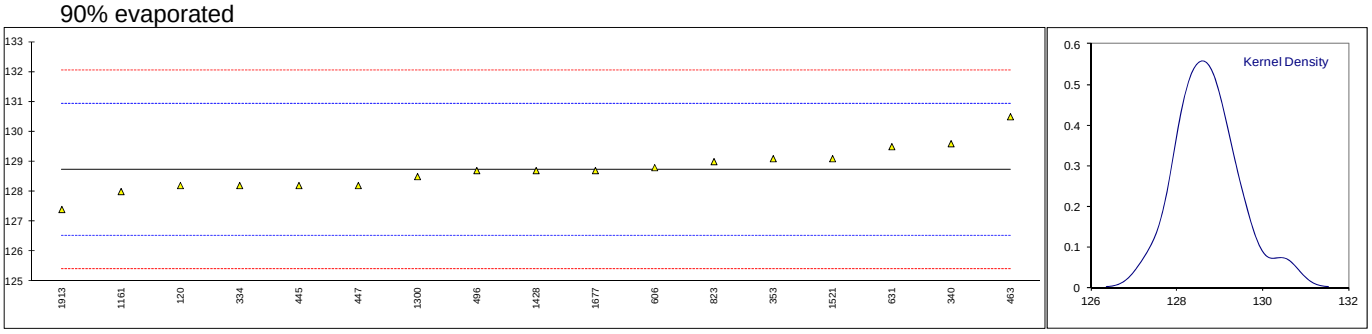
10% evaporated



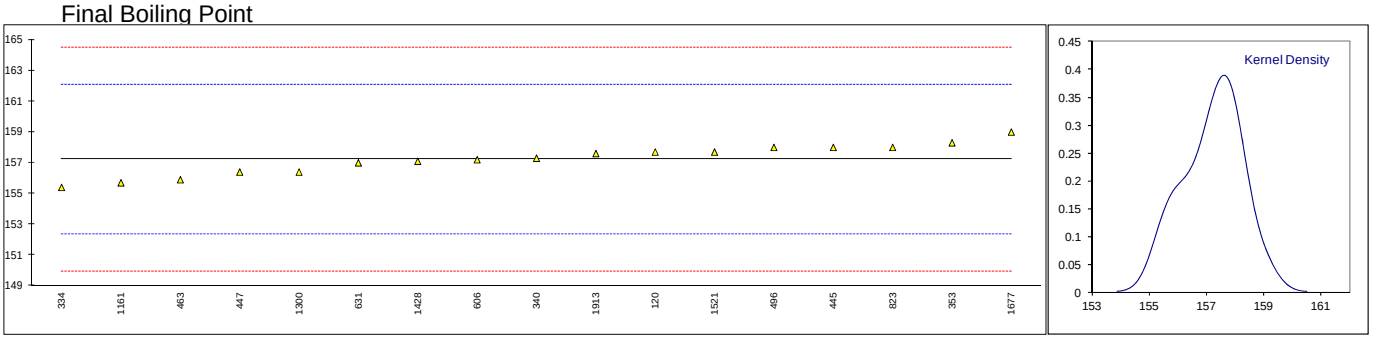
50% evaporated



90% evaporated



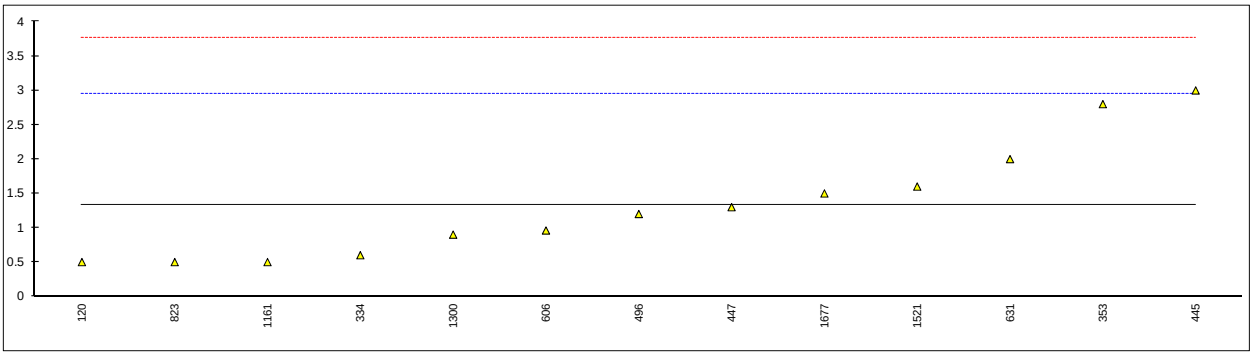
Final Boiling Point

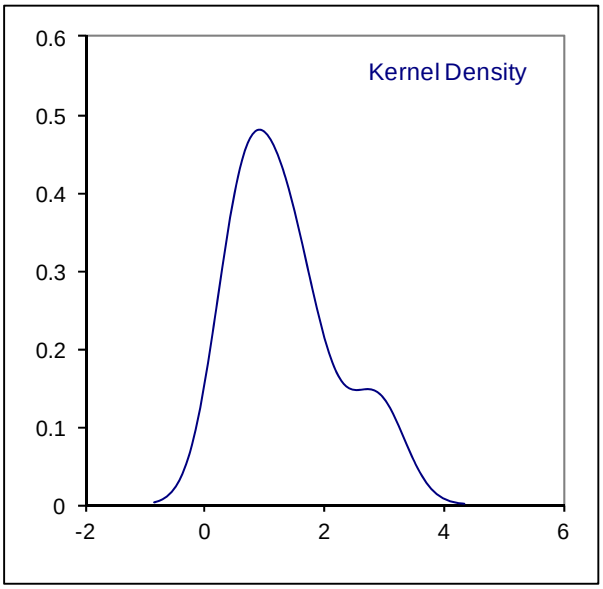


Determination of Existent Gum on sample #13050; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
120	D381	0.5		-1.04	
334	D381	0.6		-0.91	
340		-----		-----	
353	D381	2.8		1.82	
445	IP131	3		2.06	
447	D381	1.3		-0.04	
463	D381	<0.5		-----	
496	D381	1.2		-0.17	
606	D381	0.96		-0.47	
631	D381	2		0.82	
823	D381	0.5		-1.04	
962		-----		-----	
1017		-----		-----	
1161	D381	0.5		-1.04	
1300	D381	0.9		-0.54	
1428		-----		-----	
1521	D381	1.6		0.33	
1677	D381	1.5		0.20	
1913		-----		-----	

normality	OK
n	13
outliers	0
mean (n)	1.34
st.dev. (n)	0.839
R(calc.)	2.35
R(D381:12)	2.26





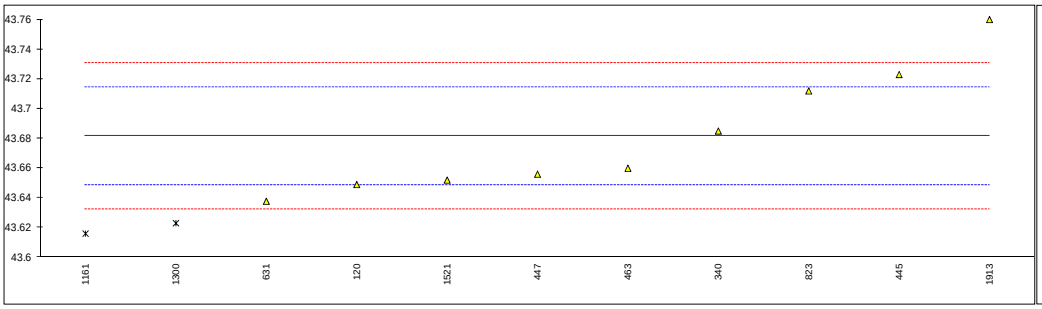
Determination of Freezing Point on sample #13050; results in °C

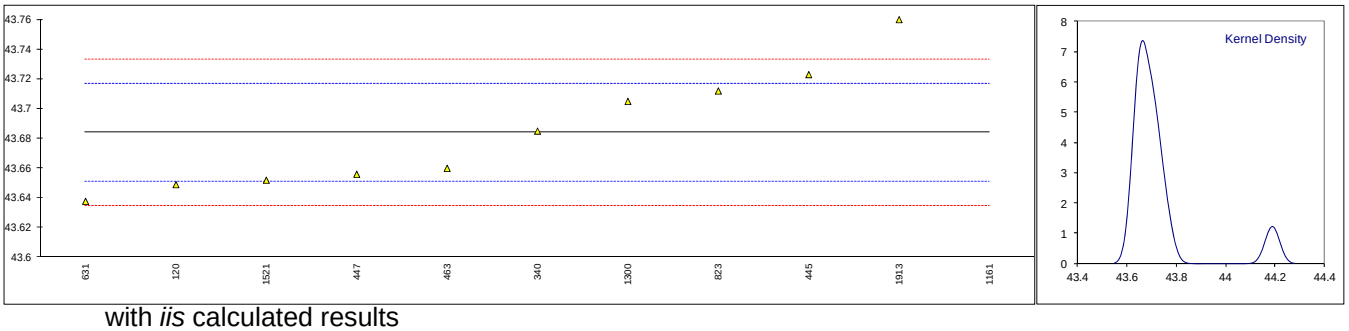
lab	method	value	mark	z(targ)	remarks
120	D2386	<-58.0		-----	
334		-----		-----	
340	D2386	<-65		-----	
353		-----		-----	
445	IP16	<-65.0		-----	
447	D2386	<-58		-----	
463	D2386	<-65	C	-----	first reported <-0.65
496	D2386	<-77.5		-----	
606		-----		-----	
631	D2386	<-58		-----	
823	D2386	<-60.0		-----	
962		-----		-----	
1017		-----		-----	
1161		-----		-----	
1300	D2386	<-70		-----	
1428		-----		-----	
1521	D2386	<-60		-----	
1677	D2386	<-80		-----	
1913	D2386	<-64		-----	
	normality	n.a.			
	n	12			
	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 #13050; results in MJ/kg

lab	method	value	mark	z(targ)	remarks
120	D3338	43.649		-1.99	
334		-----		-----	
340	D3338	43.685		0.20	
353		-----		-----	
445	D3338	43.723		2.52	
447	D3338	43.656		-1.56	
463	D3338	43.66		-1.32	
496		-----		-----	
606		-----		-----	
631	D3338	43.6378		-2.67	
823	D3338	43.712		1.85	
962		-----		-----	
1017		-----		-----	
1161	D3338	43.616	E, ex	-4.00	*iis* calculated result: 44.189, G(0.01)
1300	D3338	43.623	E, ex	-3.57	*iis* calculated result: 43.705
1428		-----		-----	
1521	D3338	43.652		-1.80	
1677		-----		-----	
1913	D3338	43.76		4.77	

			with *iis* calculated results
	normality	OK	OK
	n	9	10
	outliers	0	1
	mean (n)	43.6816	43.6840
	st.dev. (n)	0.04150	0.03981
	R(calc.)	0.1162	0.1115
	R(D3338:09)	0.0460	0.0460



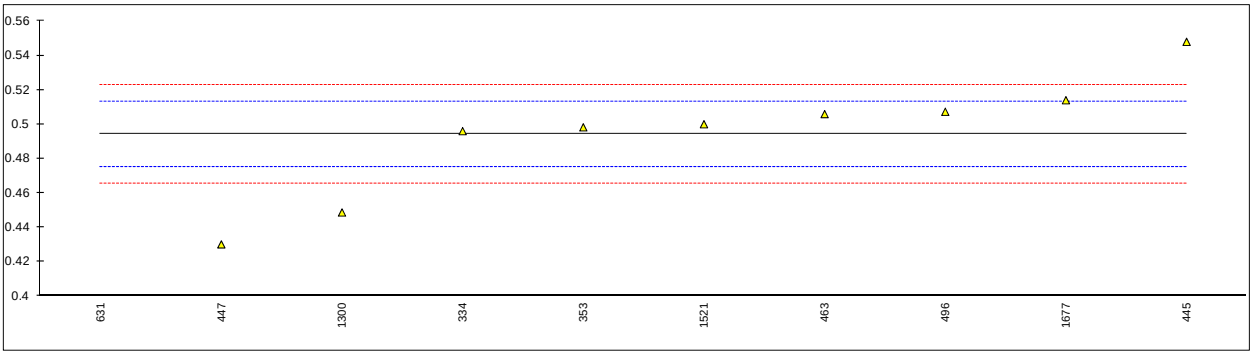


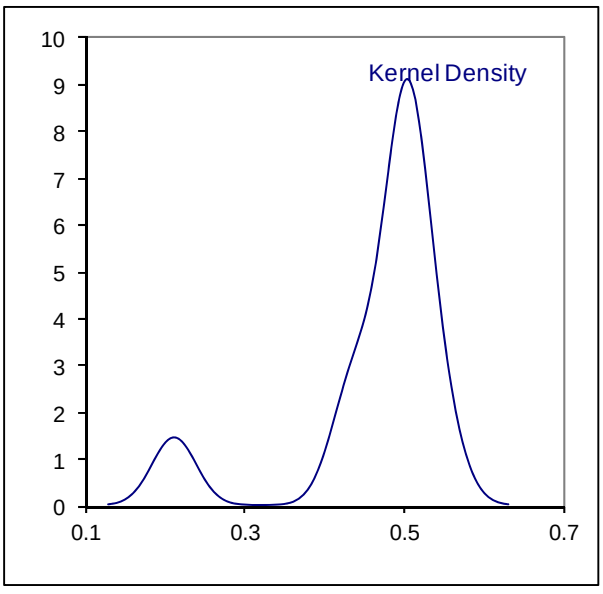
with *iis* calculated results

Determination of Lead as Pb on sample #13050; results in g/l

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
334	D3341	0.496		0.19	
340		-----		-----	
353	IP270	0.4982		0.42	
445	IP270	0.548		5.61	
447	IP367	0.430		-6.70	
463	D3341	0.5059		1.22	
496	D3341	0.50726		1.36	
606		-----		-----	
631	D3237	0.212	C, G(0.01)	-29.44	first reported 0.3418
823		-----		-----	
962		-----		-----	
1017		-----		-----	
1161		-----		-----	
1300	D3341	0.4486	C	-4.76	first reported 0.4217
1428		-----		-----	
1521	D5059-A	0.500		0.60	
1677	D3341	0.514		2.06	
1913		-----		-----	

normality	not OK
n	9
outliers	1
mean (n)	0.4942
st.dev. (n)	0.03509
R(calc.)	0.0982
R(D3341:11)	0.0268

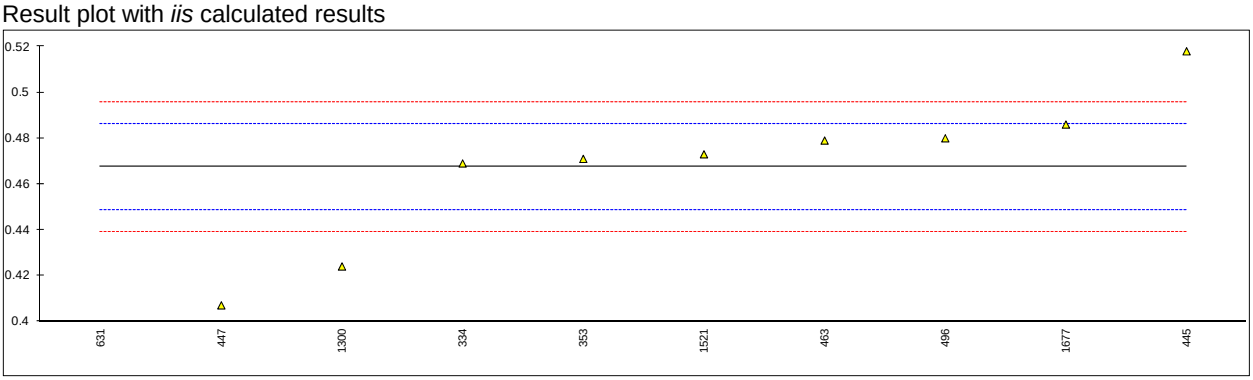




Determination of Lead as TEL on sample #13050; results in ml/l

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
334		-----		-----	
340		-----		-----	
353	IP270	0.471		-----	
445	IP270	0.519		-----	
447		-----		-----	
463	D3341	0.4786		-----	
496		-----		-----	
606		-----		-----	
631		-----		-----	
823		-----		-----	
962		-----		-----	
1017		-----		-----	
1161		-----		-----	
1300	D3341	0.4243	C	-----	first reported 0.3989
1428		-----		-----	
1521		-----		-----	
1677		-----		-----	
1913		-----		-----	
					iis calculated results from Lead as Pb
	normality	n.a.			not OK
	n	4			9
	outliers	0			1
	mean (n)	0.4733			0.4674
	st.dev. (n)	n.a.			0.03313
	R(calc.)	n.a.			0.0928
	R(D3341:11)	0.0263			0.0261

Result plot with *iis* calculated results



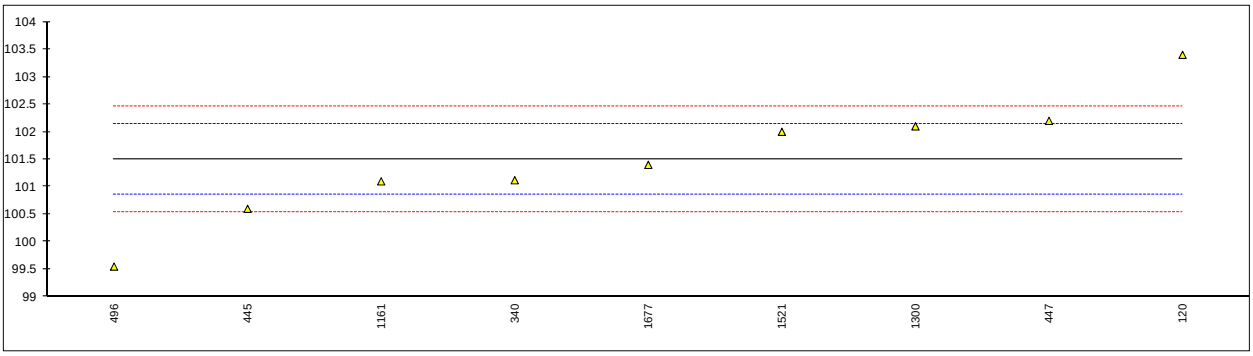
Determination of Lead precipitate content on sample #13050; results in mg/100ml

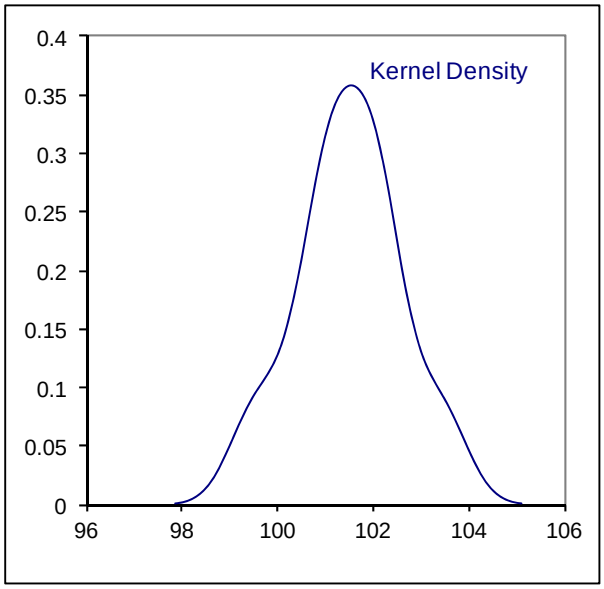
lab	method	value	mark	z(targ)	remarks
120	D873	<1.0		-----	
334		-----		-----	
340		-----		-----	
353		-----		-----	
445	IP138	none visible		-----	
447	IP138	<1		-----	
463	D873	<1		-----	
496		-----		-----	
606		-----		-----	
631		-----		-----	
823	D873	<1		-----	
962		-----		-----	
1017		-----		-----	
1161		-----		-----	
1300	D873	0.3		-----	
1428		-----		-----	
1521	D873	<1		-----	
1677	D873	<1		-----	
1913		-----		-----	
	normality	n.a			
	n	8			
	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 #13050

lab	method	value	mark	z(targ)	remarks
120	D2700	103.4		5.92	
334		-----		-----	
340	D2700	101.12		-1.17	
353		-----		-----	
445	IP236	100.6		-2.79	
447	D2700	102.2		2.19	
463	D2700	>100		-----	
496	D2700	99.55		-6.06	
606		-----		-----	
631		-----		-----	
823		-----		-----	
962		-----		-----	
1017		-----		-----	
1161	D2700	101.1		-1.23	
1300	D2700	102.1		1.88	
1428		-----		-----	
1521	D2700	102.0		1.57	
1677	D2700	101.4		-0.30	
1913		-----		-----	

normality	OK
n	9
outliers	0
mean (n)	101.5
st.dev. (n)	1.10
R(calc.)	3.1
R(D2700:12)	0.9

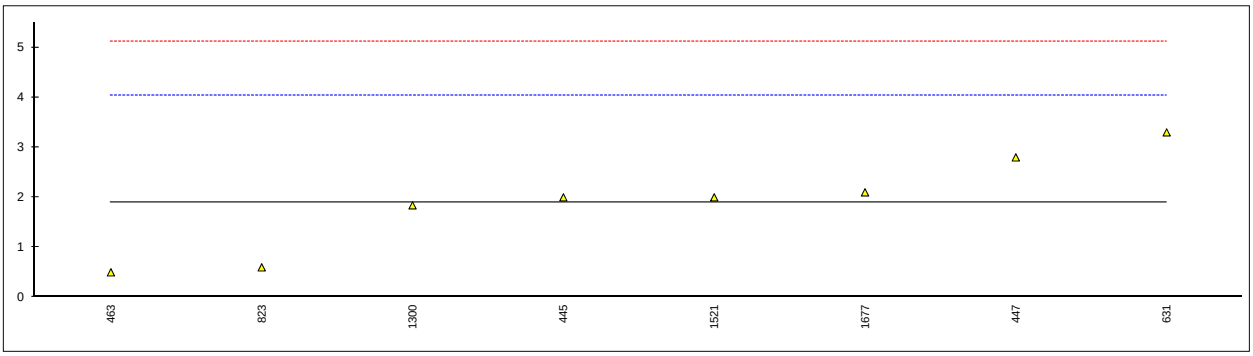


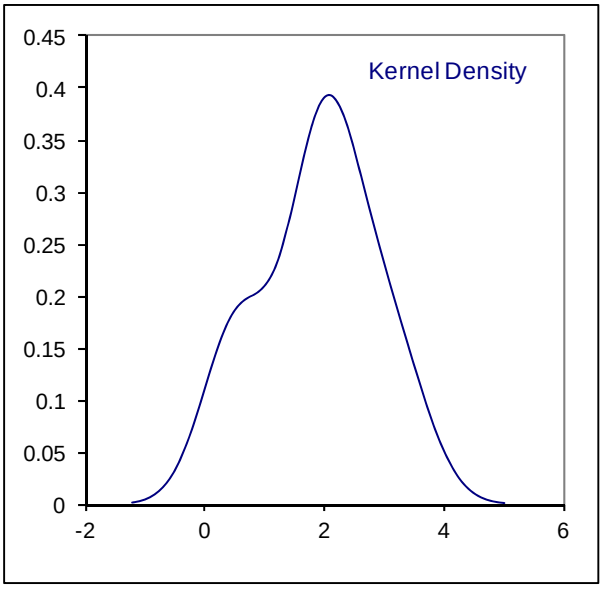


Determination of Potential Gum on sample #13050; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
120	D873	<1.0		-----	
334		-----		-----	
340		-----		-----	
353		-----		-----	
445	IP138	2		0.10	
447	IP138	2.8		0.85	
463	D873	0.5		-1.30	
496		-----		-----	
606		-----		-----	
631	D873	3.3		1.31	
823	D873	0.6		-1.21	
962		-----		-----	
1017		-----		-----	
1161		-----		-----	
1300	D873	1.84		-0.05	
1428		-----		-----	
1521	D873	2		0.10	
1677	D873	2.1		0.19	
1913		-----		-----	

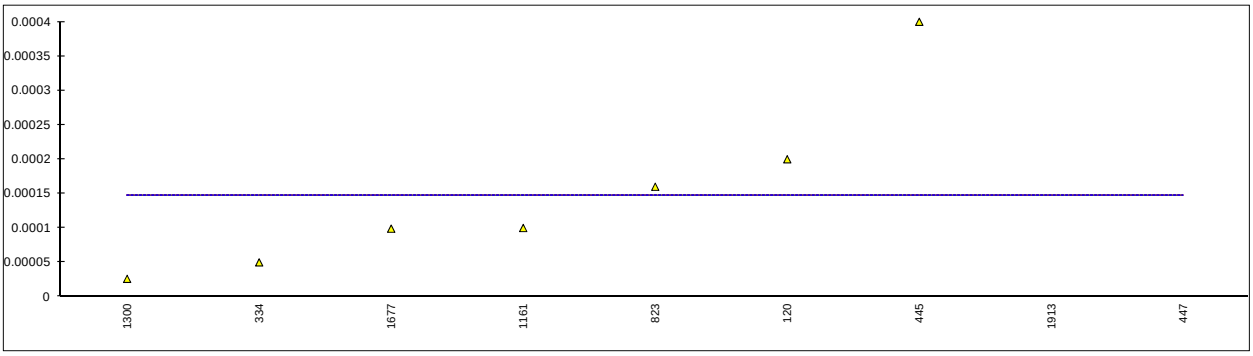
normality	OK
n	8
outliers	0
mean (n)	1.89
st.dev. (n)	0.962
R(calc.)	2.69
R(D873:12)	3.00

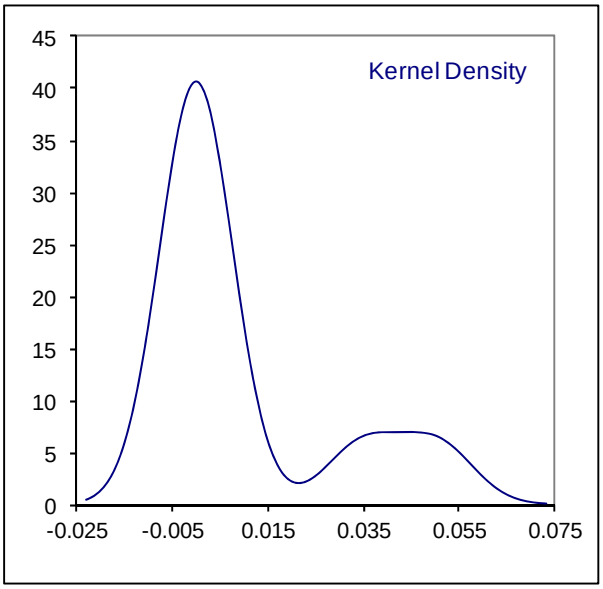




Determination of Sulphur content on sample #13050; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D2622	0.0002		-----	
334	EN20846	0.00005		-----	reported in mg/kg; *iis* converted to %M/M
340	D2622	<0.00001		-----	
353		-----		-----	
445	D5453	0.0004		-----	
447	IP336	0.0505	DG(0.01)	-----	false positive test result?
463		-----		-----	
496	D2622	<0.0005		-----	
606		-----		-----	
631	D4294	<0.015	C	-----	first reported 0.0328
823	D5453	0.00016		-----	
962		-----		-----	
1017		-----		-----	
1161	D2622	0.0001		-----	
1300	D5453	0.000026		-----	
1428		-----		-----	
1521	D2622	<0.0003		-----	
1677	D5453	0.000099		-----	reported in mg/kg; *iis* converted to %M/M
1913	D1266	0.035	C, DG(0.01)	-----	first reported 0.012: false positive test result?
	normality	OK			
	n	7			
	outliers	2			
	mean (n)	<0.0003			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D2622:10)	n.a.			application range >0.0003 %M/M





Determination of Water reaction interface on sample #13050; results in ml

lab	method	value	mark	z(targ)	remarks
120	D1094	1		-----	
334		-----		-----	
340	D1094	0		-----	
353		-----		-----	
445	D1094	NIL		-----	
447	D1094	0		-----	
463	D1094	<0.5		-----	
496	D1094	1.0		-----	
606		-----		-----	
631	D1094	0		-----	
823	D1094	0		-----	
962		-----		-----	
1017		-----		-----	
1161	D1094	<0.5		-----	
1300	D1094	<0.5		-----	
1428		-----		-----	
1521	D1094	0.0		-----	
1677	D1094	1		-----	
1913	D1094	0.0		-----	
	normality	n.a.			
	n	9			
	outliers	n.a.			
	mean (n)	1 or <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	U.S.A.
2 labs in	FRANCE
1 lab in	IRELAND
2 labs in	UNITED KINGDOM
1 lab in	SWEDEN
1 lab in	GERMANY
1 lab in	MALAYSIA
1 lab in	PHILIPPINES
1 lab in	KOREA
1 lab in	SAUDI ARABIA
1 lab in	BELGIUM
2 labs in	TURKEY
2 labs in	ESTONIA
1 lab in	POLAND
1 lab in	P.R. of CHINA

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
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, January 2010

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/)

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