Results of Proficiency Test
Aviation gasoline
April 2016

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2011, the Institute for Interlaboratory Studies (iis) organizes proficiency tests for the analysis of Aviation Gasoline 100LL (AvGas) once every two years. In the annual proficiency testing program of 2015/2016, it was decided to continue the proficiency test for the analysis of Aviation Gasoline 100LL in accordance with the latest applicable specifications for AvGas: ASTM D 910 and UK DEF STAN 91-90. In the interlaboratory study for AvGas 20 laboratories from 16 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2016 proficiency test on AvGas are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. The analyses of the samples for fit-for-use and homogeneity determination were subcontracted to an accredited laboratory. It was decided to send two identical samples of AvGas (2 times 1 litre bottle, labelled #16038) to the participants. Participants were requested to report rounded and unrounded test results. The unrounded test 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 IEC/ISO17043:2010. 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 organisation 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 April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from 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

The necessary bulk material of AvGas was obtained from an European supplier. From the approximately 160 litre, after homogenisation, 100 amber glass bottles of 1 litre were filled and labelled #16038. The homogeneity of the subsamples #16038 was checked by determination of Density at 15°C in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Density at 15°C in kg/L
sample #16038-1	0.71359
sample #16038-2	0.71362
sample #16038-3	0.71362
sample #16038-4	0.71362
sample #16038-5	0.71360
sample #16038-6	0.71361
sample #16038-7	0.71361
sample #16038-8	0.71360

Table 1: homogeneity test results of Benzene sub samples #16038

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

	Density at 15°C in kg/L
r (observed)	0.00003
Reference test method	D4052:15
0.3*R (reference test method)	0.00095

Table 2: evaluation of repeatabilities of subsamples #16038

The calculated repeatability for sample #16038 was in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the samples was assumed.

To the participants two 1L bottles of sample #16038 were sent on March 16, 2016.

2.5 STABILITY OF THE SAMPLES

The stability of Aviation Gasoline 100LL, 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 requested to determine on the sample #16038: Aromatics by HPLC, Color, Copper Strip 2hrs/100°C, Density at 15°C, Distillation, Existent Gum, Freezing Point, Heat of Combustion (Net), Lead as Pb, Lead as TEL, Lead Precipitate, Potential Gum, Sulphur, Water Reaction and MON.

To get comparable test results a detailed report form, on which the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. A SDS and a form to confirm receipt of the samples was added to the sample package.

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 raw data of these tests (no reanalysis). Additional or corrected test results are used for data analysis and the original test results are placed under 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 Organization, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). For the statistical evaluation the *unrounded* (when available) test results 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 judgment 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 test results should be used with due care.

In accordance with ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon 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 test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty

failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as an “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 reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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

The z-scores were calculated in accordance with:

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

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

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

	$\lvert z \rvert < 1$	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, 20 laboratories did report 211 numerical test results. Observed were 3 outlying test results, which is 1.4%. In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

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

In the iis PT reports, ASTM methods are referred to with a number (e.g. D3338) and an added designation for the year that the method was adopted or revised (e.g. D3338:09). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D3338:09(2014e1)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D3338:09 will be used.

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.

Aromatics: This determination was very problematic. No statistical outliers were observed. However, one result was excluded as it was reported as %V/V. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ASTM D6379:11. The large spread may (partly) be explained by the small number of reported test results.

Colour: The reporting was very divers. Four participants reported to have used ASTM 2392, but did not report conform this standard “acceptable or fail”. All participants, except one, reported the color as “Blue”.
One test result was excluded because the test method used was not compatible to ASTM D2392.

Copper Corrosion: All participants agreed on result 1 (1a or 1b).

Density at 15ºC: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D4052:15.

Distillation: This determination was not problematic. Only one statistical outlier was observed for FBP. All calculated reproducibilities, after rejection of the statistical outlier, are in agreement with the requirements of ASTM D86:15.

Existent Gum: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility 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 problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D3338:09(2014e1).

Lead as Pb: This determination was problematic. The data appear to be bimodally divided. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D3341:05(2011).

Lead as TEL: This determination may be very problematic. Five laboratories reported a test result. No statistical outliers were observed and the calculated reproducibility is not in agreement with the requirements of ASTM D3341:05(2011).
In order to create a significant statistical estimation, *iis* calculated TEL values from the test results reported for Lead as Pb. The estimated reproducibility is again not in agreement with the requirements of ASTM D3341:05(2011).

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

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

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 (3 mg/kg – 4.6% M/M) of ASTM D2622:10.

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

MON: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D2700:16.

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 by HPLC	%M/M	5	17.4	5.9	1.9
Colour	-----	8	blue	n.a.	n.a.
Copper Corrosion 2 hrs/100°C	-----	19	1	n.a.	n.a.
Density at 15°C	kg/m^3	19	713.7	0.7	3.2
Distillation at 760 mm Hg					
- Initial Boiling Point	°C	19	36.4	4.0	4.7
- 10% evaporated	°C	19	62.7	4.2	4.4
- 40% evaporated	°C	19	96.8	2.0	3.9
- 50% evaporated	°C	19	103.8	1.6	3.4
- 90% evaporated	°C	19	131.0	3.6	4.8
- Final Boiling Point	°C	18	158.5	4.6	7.1
Existent Gum	mg/100ml	8	0.4	0.7	2.1
Freezing Point	°C	13	<-58	n.a.	n.a.
Heat of Combustion (Net)	MJ/kg	10	43.69	0.08	0.05
Lead as Pb	g/l	13	0.54	0.04	0.03
Lead as TEL	ml/l	5	0.51	0.04	0.03
Lead precipitate content	mg/100ml	6	<1	n.a.	n.a.
Potential Gum	mg/100ml	6	1.6	3.1	3.0
Sulphur	mg/kg	7	<3	n.a.	n.a
Water reaction interface	ml	16	<0.5	n.a.	n.a.
MON (lean mixture)	-----	8	102.5	2.6	2.0

Table 3: performance evaluation sample #16038

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 2016 WITH PREVIOUS PT

	April 2016	April 2014	April 2013	April 2012
Number of reporting labs	20	17	17	18
Number of results reported	211	193	209	222
Statistical outliers	3	9	6	7
Percentage outliers	1.4%	4.7%	2.9%	3.2%

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 2016	April 2014	April 2013	April 2012
Aromatics by HPLC	--	-	--	--
Density at 15˚C	++	+	++	++
Distillation at 760 mm Hg	++	+	+	+
Existent Gum	++	++	+/-	(++)
Heat of Combustion (Net)	--	--	--	--
Lead as Pb	-	--	--	++
Lead as Tel	-	--	--	++
Potential Gum	+/-	n.a.	+	(++)
Sulphur	n.a.	n.a.	n.a.	n.a.
MON (lean mixture)	-	+	--	--

Table 5: comparison determinations against the standard requirements

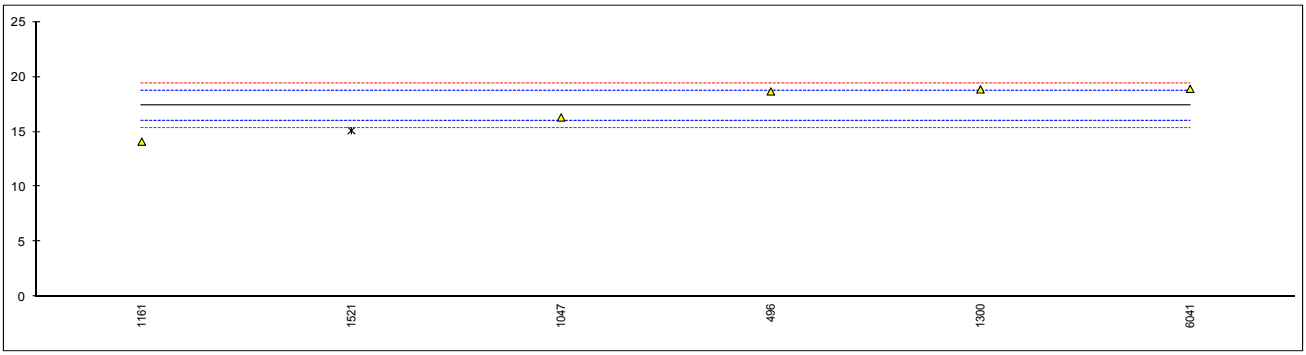
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

++: 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 by HPLC on sample #16038; results in %M/M

lab	method	value	mark	z(targ)	remarks
62		-----		-----	
150		-----		-----	
334		-----		-----	
445		-----		-----	
447		-----		-----	
496	D6379	18.67		1.96	
606		-----		-----	
631		-----		-----	
823		-----		-----	
970		-----		-----	
1016		-----		-----	
1047	D1319	16.3		-1.60	
1161	EN12916	14.1		-4.90	
1300	D6379	18.85		2.23	
1521	EN15553	15.1	ex	-3.40	Result excluded, reported test result in %V/V
1538		-----		-----	
1613		-----		-----	
1755		-----		-----	
1757		-----		-----	
6041	D6379	18.9		2.30	
	normality	unknown			
	n	5			
	outliers	0 (+ 1 excl)			
	mean (n)	17.364			
	st.dev. (n)	2.1248			
	R(calc.)	5.949			
	R(D6379:11)	1.866			Compare R(D1319:15) = 2.894



Determination of Colour on sample #16038

lab	method	value	mark	z(targ)	remarks
62		-----		-----	
150	D2392	Blue		-----	
334		-----		-----	
445		-----		-----	
447	IP569	3.08		-----	
496		-----		-----	
606		-----		-----	
631		-----		-----	
823	D2392	Blue		-----	
970		-----		-----	
1016	D2392	3.0		-----	
1047		-----		-----	
1161	D6045	>+30	ex	-----	Result excluded, method is not compatible with D2392
1300	IP17	0R:0,9Y:3,0B:0,8N		-----	
1521	D2392	blue		-----	
1538		-----		-----	
1613	VISUAL	BLUE		-----	
1755		-----		-----	
1757		-----		-----	
6041		-----		-----	

normality	n.a.
n	7 (+ 1 excl)
outliers	n.a.
mean (n)	Acceptable/Blue
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D2392:15)	n.a.

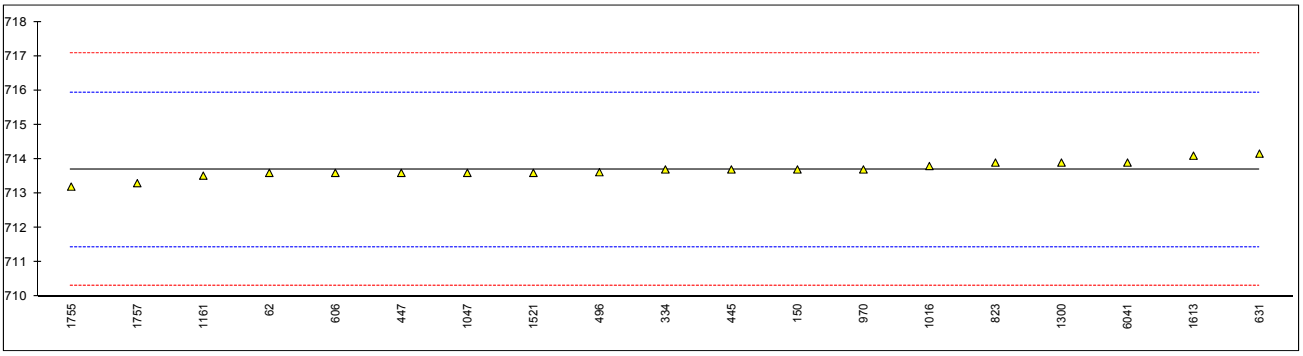
Determination of Copper Corrosion 2hrs/100°C on sample #16038

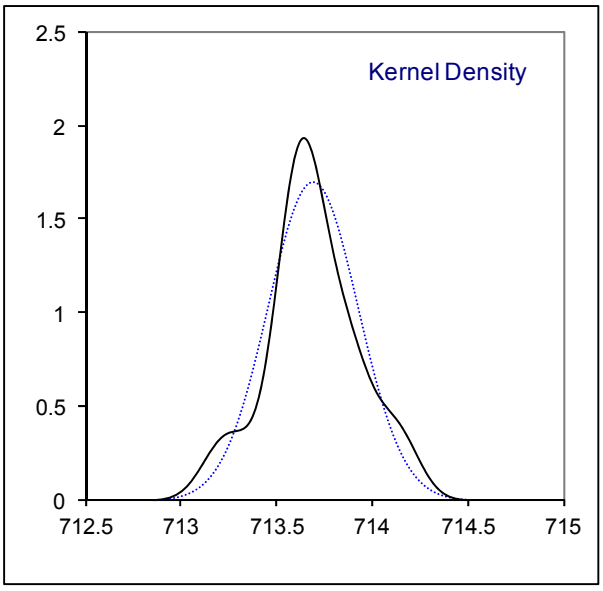
lab	method	value	mark	z(targ)	Remarks
62	D130	1b		-----	
150	D130	1a		-----	
334	D130	1a		-----	
445	IP154	1b		-----	
447	D130	1b		-----	
496	D130	1b		-----	
606	D130	1b		-----	
631	D130	1A		-----	
823	D130	1		-----	
970	D130	1a		-----	
1016	D130	1a		-----	
1047	D130	1		-----	
1161	ISO2160	1A		-----	
1300	D130	1a		-----	
1521	D130	1		-----	
1538		-----		-----	
1613	D130	1A		-----	
1755	D130	1A		-----	
1757	D130	1a		-----	
6041	D130	1a		-----	
	normality	n.a.			
	n	19			
	outliers	n.a.			
	mean (n)	1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D130:12)	n.a.			

Determination of Density at 15°C on sample #16038; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
62	D4052	713.6		-0.08	
150	D4052	713.7		0.00	
334	D4052	713.7		0.00	
445	IP365	713.7		0.00	
447	D4052	713.6		-0.08	
496	D4052	713.62		-0.07	
606	D4052	713.6		-0.08	
631	D4052	714.16		0.41	
823	D4052	713.9		0.18	
970	D4052	713.7		0.00	
1016	D4052	713.8		0.09	
1047	D4052	713.6		-0.08	
1161	ISO12185	713.52		-0.16	
1300	D4052	713.9		0.18	
1521	D4052	713.6		-0.08	
1538		-----		-----	
1613	D4052	714.1		0.36	
1755	D4052	713.2		-0.44	
1757	D7777	713.3		-0.35	
6041	D1298	713.9		0.18	

normality	OK
n	19
outliers	0
mean (n)	713.69
st.dev. (n)	0.236
R(calc.)	0.66
R(D4052:15)	3.15





Determination of Distillation at 760 mm Hg on sample #16038; results in °C.

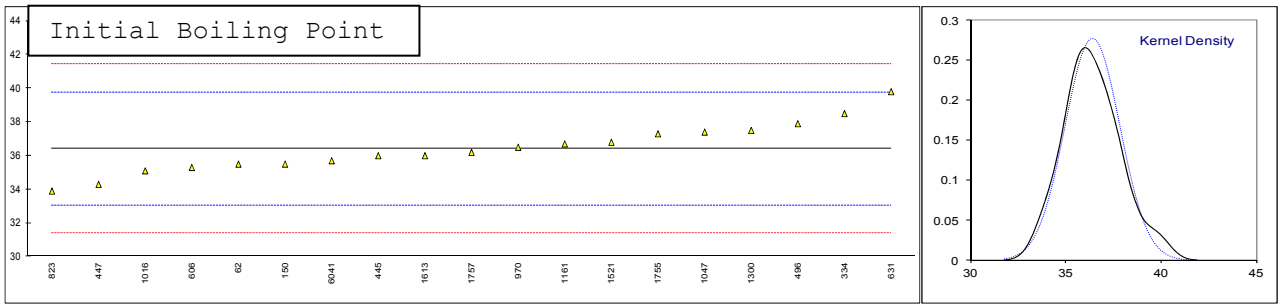
lab	method	mode	IBP	mark	10%	mark	40%	mark	50%	mark	90%	mark	FBP	mark	res.
62	D86	Auto	35.5		63.7		98.5		104.7		132.0		158.5		1.0
150	D86	Auto	35.5		64.1		97.1		104.1		130.9		158.0		0.9
334	D86	Auto	38.5		61.8		96.6		103.6		130.7		157.6		1.4
445	IP123	Auto	36.0		61.6	C	95.9	C	103.4	C	130.6	C	157.3		1.1
447	D86	Auto	34.3		60.8		96.5		103.8		131.3		161.5		1.0
496	D86	Auto	37.9		64.1		96.7		103.8		130.8		157.9		1.1
606	D86	Auto	35.3		62.5		96.4		103.5		130.8		157.5		1.0
631	D86	Man	39.8		59.2	C	95.8		102.3		129.0	C	173.0	C,G(1)	0.8
823	D86	Auto	33.9		61.5		96.5		103.5		129.7		157.3		0.6
970	D86	Auto	36.5		64.2		97.2		104.6		131.0		157.5		1.0
1016	D86	Auto	35.1		62.8		96.8		103.8		130.7		161.3		0.8
1047	D86	Auto	37.4		63.8		96.7		103.7		130.9		157.9		1.0
1161	ISO3405	Auto	36.7		61.1		95.9		103.3		128.8		158.4		1.1
1300	D86	Auto	37.5		63.3		96.8		103.8		131.6		158.2		1.0
1521	D86	Auto	36.8		62.2		96.6		103.6		130.9		157.2		0.9
1538			-----		-----		-----		-----		-----		-----		-----
1613	D86	Auto	36.0		64.5		97.5		104.3		132.1		162.8		0.8
1755	D86	Auto	37.3		64.6		97.8		104.7		134.0		158.7		0.9
1757	D86	Auto	36.2		64.0		98.1		104.3		133.5		157.8		0.9
6041	D86	Auto	35.7		62.1		96.6		103.7		129.9		157.7		0.7
	normality		OK		OK		OK		suspect		suspect		not OK		
	n		19		19		19		19		19		18		
	outliers		0		0		0		0		0		1		
	mean (n)		36.42		62.73		96.84		103.82		131.01		158.51		
	st.dev. (n)		1.444		1.487		0.722		0.567		1.293		1.626		
	R(calc.)		4.04		4.16		2.02		1.59		3.62		4.55		
	R(D86:15)		4.70		4.38		3.91		3.39		4.80		7.10		

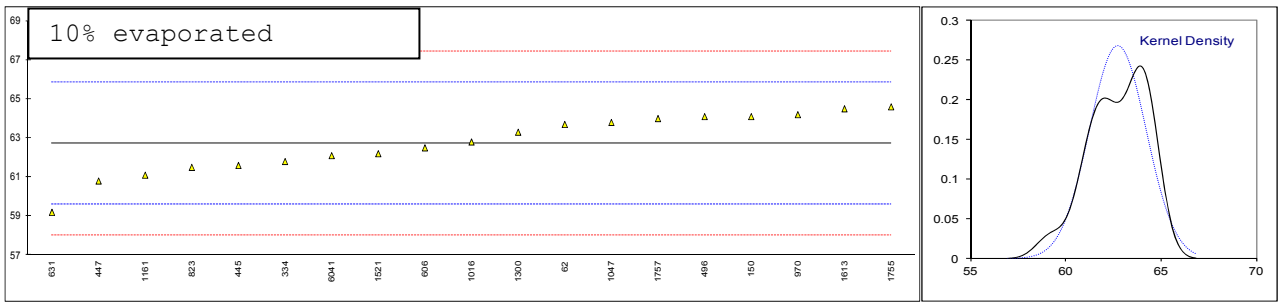
z-scores

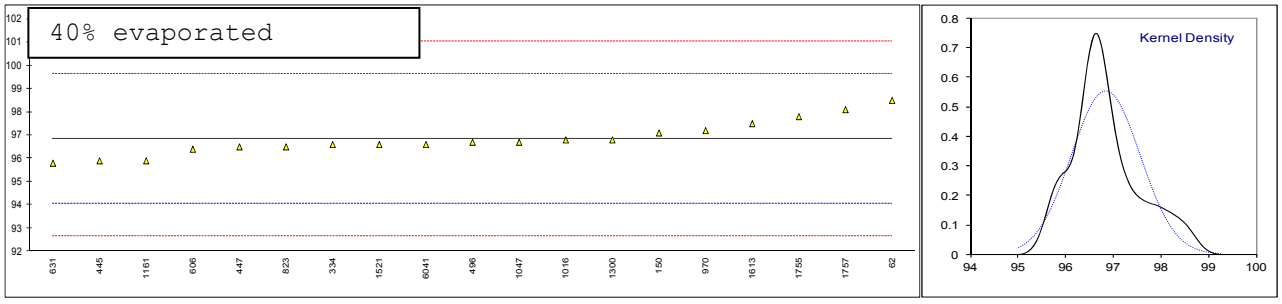
lab	method	IBP	10%	40%	50%	90%	FBP
62	D86	-0.55	0.62	1.19	0.73	0.58	0.00
150	D86	-0.55	0.87	0.18	0.23	-0.06	-0.20
334	D86	1.24	-0.60	-0.17	-0.18	-0.18	-0.36
445	IP123	-0.25	-0.72	-0.67	-0.34	-0.24	-0.48
447	D86	-1.26	-1.23	-0.25	-0.01	0.17	1.18
496	D86	0.88	0.87	-0.10	-0.01	-0.12	-0.24
606	D86	-0.66	-0.15	-0.32	-0.26	-0.12	-0.40
631	D86	2.02	-2.26	-0.75	-1.25	-1.17	5.72
823	D86	-1.50	-0.79	-0.25	-0.26	-0.76	-0.48
970	D86	0.05	0.94	0.26	0.65	-0.01	-0.40
1016	D86	-0.78	0.04	-0.03	-0.01	-0.18	1.10
1047	D86	0.59	0.68	-0.10	-0.10	-0.06	-0.24
1161	ISO3405	0.17	-1.04	-0.67	-0.43	-1.29	-0.04
1300	D86	0.65	0.36	-0.03	-0.01	0.34	-0.12
1521	D86	0.23	-0.34	-0.17	-0.18	-0.06	-0.51
1538		-----	-----	-----	-----	-----	-----
1613	D86	-0.25	1.13	0.47	0.40	0.63	1.69
1755	D86	0.53	1.19	0.69	0.73	1.74	0.08
1757	D86	-0.13	0.81	0.90	0.40	1.45	-0.28
6041	D86	-0.43	-0.40	-0.17	-0.10	-0.65	-0.32

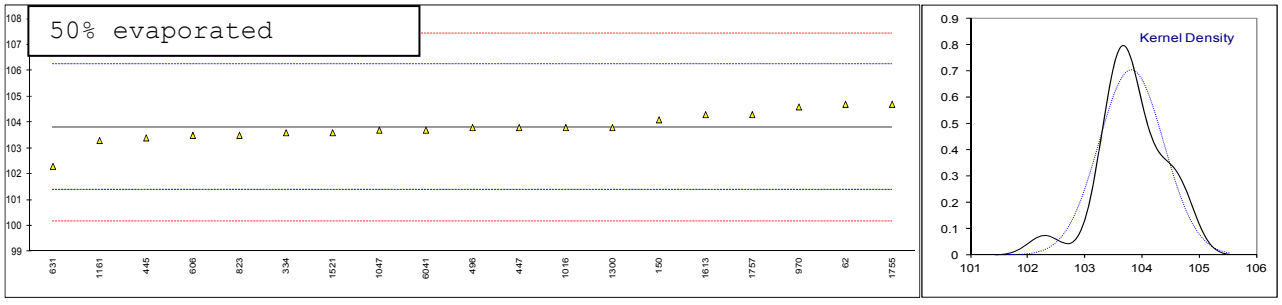
Lab 445: first reported 64.1; 97.9, 104.3, 136.2

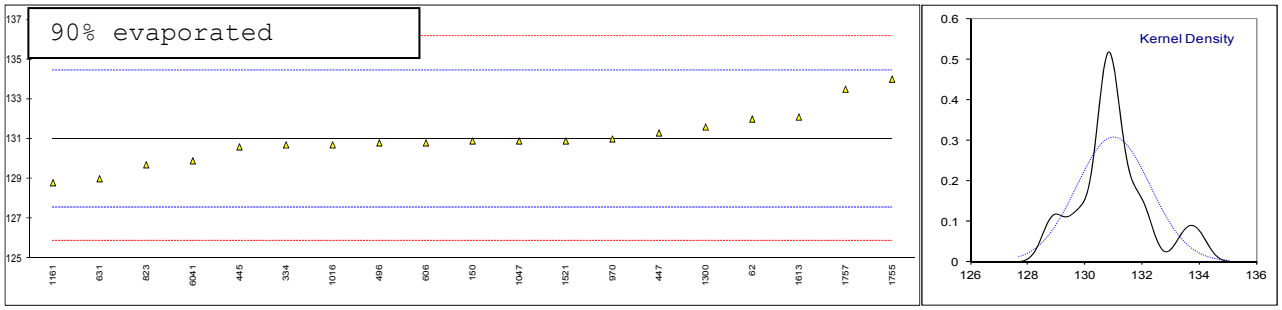
Lab 631: first reported 57.8, 135.4, 161.3

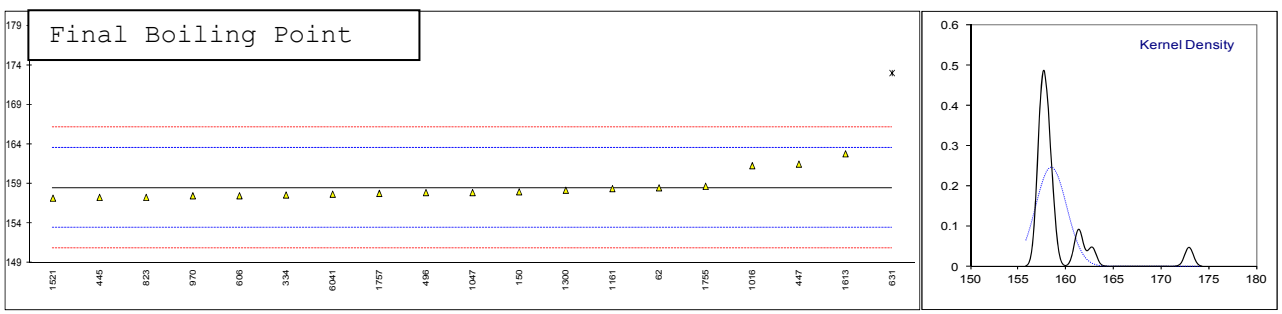

Initial Boiling Point
Kernel Density


10% evaporated
Kernel Density


40% evaporated
Kernel Density


50% evaporated
Kernel Density

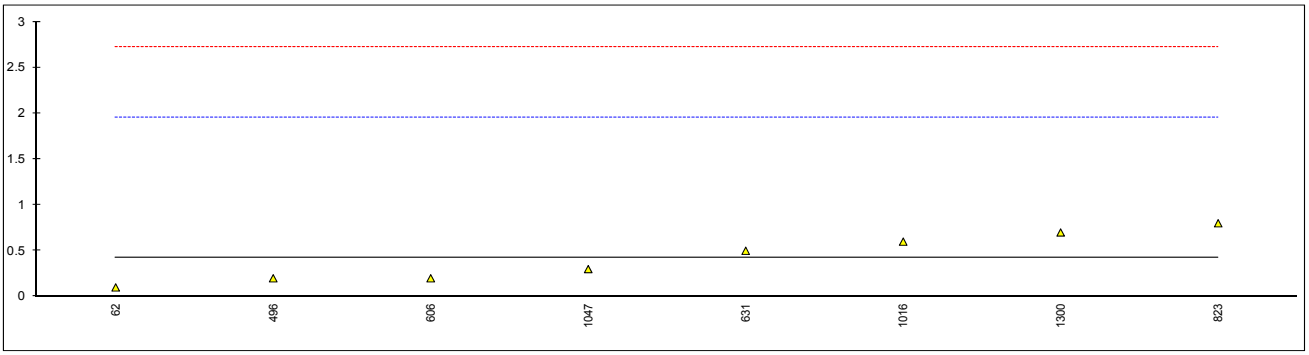

90% evaporated
Kernel Density


Final Boiling Point
Kernel Density

Determination of Existent Gum, solvent washed on sample #16038; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
62	D381	0.1		-0.42	
150	D381	<1		-----	
334	D381	<0.5		-----	
445	IP131	<1		-----	
447	D381	<1		-----	
496	D381	0.2		-0.29	
606	D381	0.2		-0.29	
631	IP540	0.5		0.10	
823	D381	0.8		0.49	
970		-----		-----	
1016	D381	0.6		0.23	
1047	ISO6246	0.3		-0.16	
1161		-----		-----	
1300	D381	0.7		0.36	
1521	D381	<1		-----	
1538		-----		-----	
1613	D381	<1.0		-----	
1755		-----		-----	
1757		-----		-----	
6041	D381	<0.5		-----	

normality	unknown
n	8
outliers	0
mean (n)	0.42
st.dev. (n)	0.260
R(calc.)	0.73
R(D381:12)	2.14



Determination of Freezing Point on sample #16038; results in °C

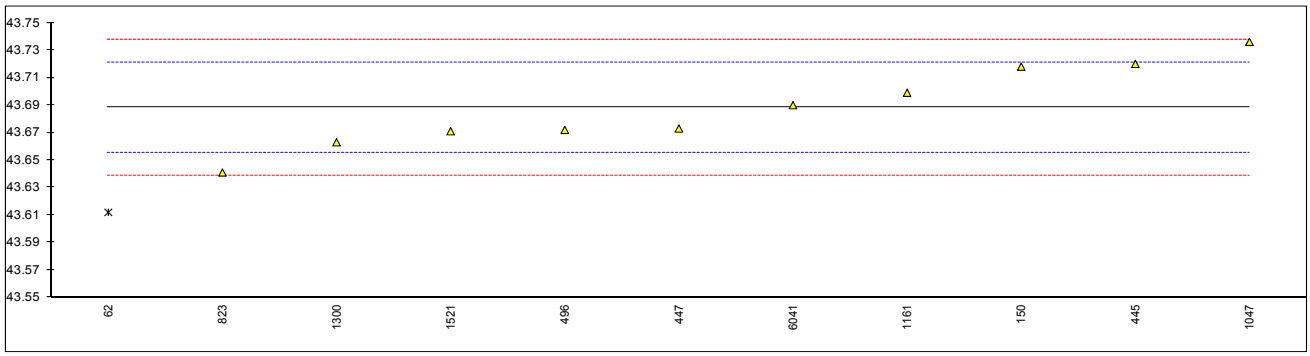
lab	method	value	mark	z(targ)	remarks
62		-----		-----	
150	D7153	<-80.0		-----	
334		-----		-----	
445	D2386	<-70		-----	
447	D2386	<-58		-----	
496	D2386	<-77.5		-----	
606		-----		-----	
631	D2386	<-58.0		-----	
823	D2386	<-60		-----	
970		-----		-----	
1016	D2386	-58		-----	
1047	D7153	< -100		-----	
1161		-----		-----	
1300	D2386	<-65		-----	
1521	D2386	< -60		-----	
1538		-----		-----	
1613	D7153	<-80		-----	
1755	D2386	< -70		-----	
1757		-----		-----	
6041	D2386	<-58		-----	

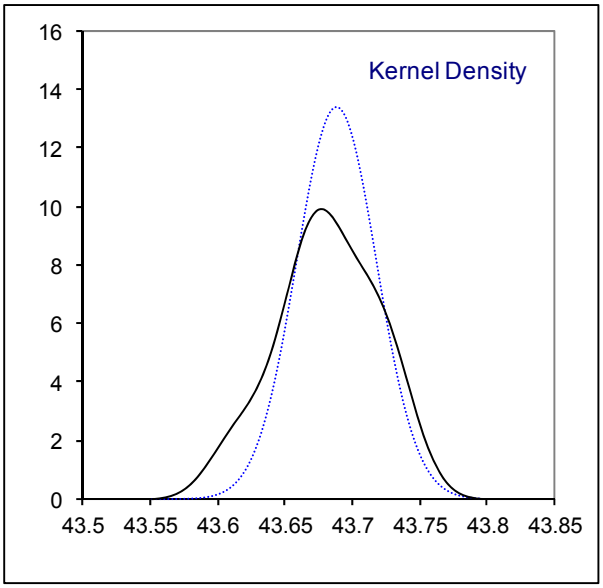
normality	unknown
n	13
outliers	n.a.
mean (n)	<-58
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D2386:15e1)	n.a.

Determination of Heat of Combustion (Net) on sample #16038; results in MJ/kg

lab	method	value	mark	z(targ)	remarks
62	D3338	43.612	G(0.05)	-4.64	
150	D3338	43.718		1.81	
334		-----		-----	
445	D3338	43.720		1.93	
447	D3338	43.673		-0.93	
496	D3338	43.672	C	-0.99	First reported 43.546
606		-----		-----	
631		-----		-----	
823	D3338	43.641		-2.88	
970		-----		-----	
1016		-----		-----	
1047	D3338	43.736		2.90	
1161	D3338	43.699		0.65	
1300	D3338	43.663		-1.54	
1521	D3338	43.671		-1.05	
1538		-----		-----	
1613		-----		-----	
1755		-----		-----	
1757		-----	W	-----	Reported first 43.844 by ASTM D4529
6041	D3338	43.690		0.10	

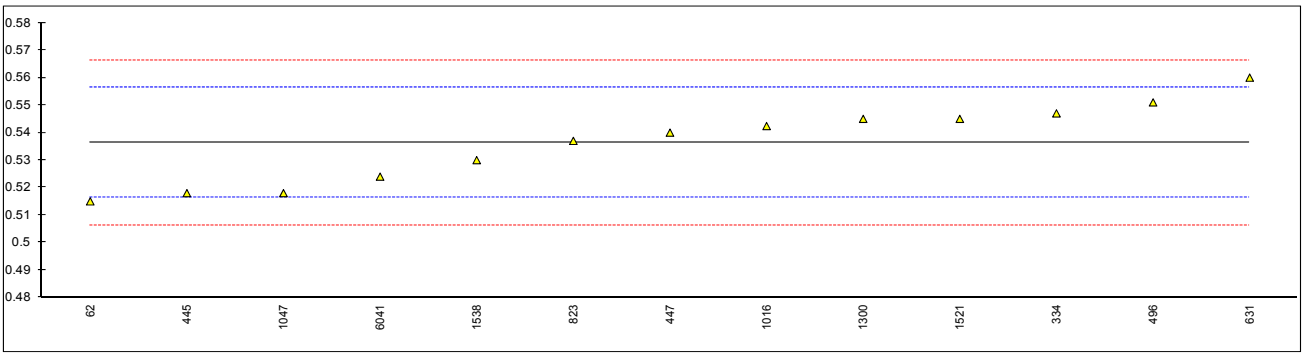
normality	OK
n	10
outliers	1
mean (n)	43.6883
st.dev. (n)	0.02973
R(calc.)	0.0833
R(D3338:09)	0.0460

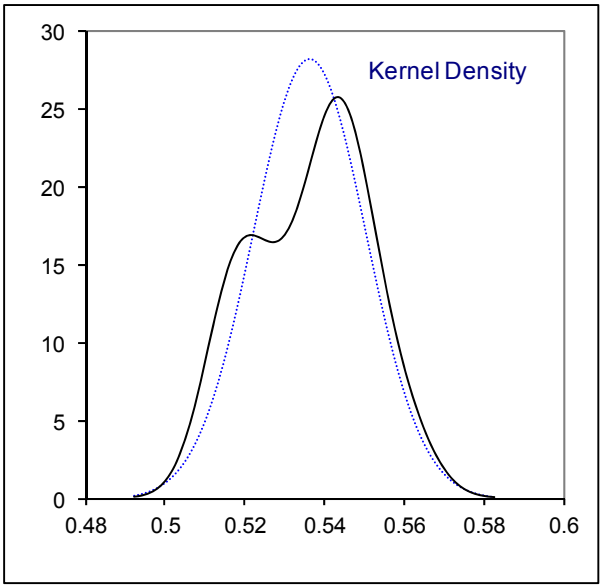




Determination of Lead as Pb on sample #16038; results in g Pb/l

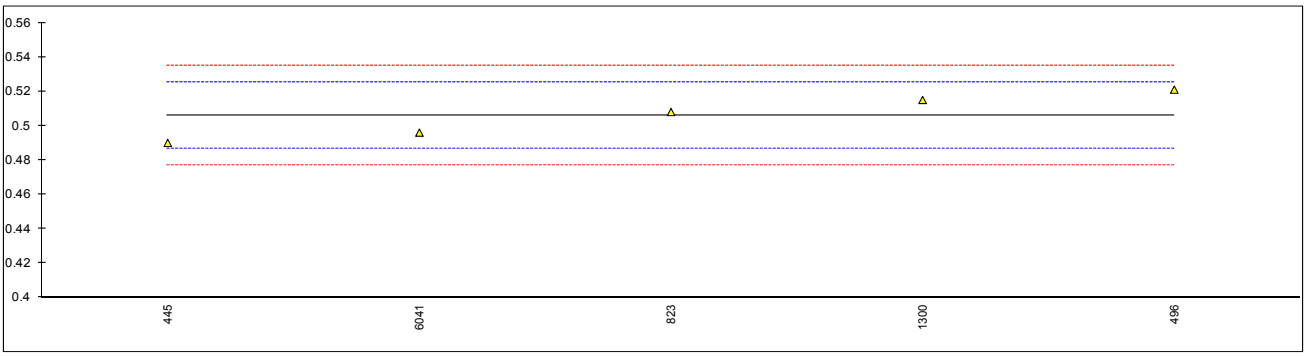
lab	method	value	mark	z(targ)	remarks
62	D3341	0.515		-2.14	
150		-----		-----	
334	D3341	0.547		1.07	
445	IP270	0.518		-1.84	
447	IP362	0.54		0.37	
496	D3341	0.551		1.47	
606		-----		-----	
631	IP428	0.56	C	2.37	First reported 0.67
823	D3341	0.537		0.07	
970		-----		-----	
1016	D5059-meth C	0.5424		0.61	
1047	D3341	0.518		-1.84	
1161		-----		-----	
1300	D3341	0.545		0.87	
1521	D5059-meth A	0.545		0.87	
1538	D5059-meth A	0.53		-0.63	
1613		-----		-----	
1755		-----		-----	
1757		-----		-----	
6041	D3341	0.524		-1.23	
	normality	OK			
	n	13			
	outliers	0			
	mean (n)	0.5363			
	st.dev. (n)	0.01417			
	R(calc.)	0.0397			
	R(D3341:05)	0.0280			





Determination of Lead as Tetra Ethyl Lead on sample #16038; results in ml/l

lab	method	value	mark	z(targ)	Calc by iis	remarks
62		-----		-----	0.487	
150		-----		-----	-----	
334		-----		-----	0.517	
445	D3341	0.490		-1.65	0.490	
447		-----		-----	0.511	
496	D3341	0.521		1.55	0.521	
606		-----		-----	-----	
631		-----		-----	0.530	
823	D3341	0.508		0.21	0.508	
970		-----		-----	-----	
1016		-----		-----	0.513	
1047		-----		-----	0.490	
1161		-----		-----	-----	
1300	D3341	0.515		0.93	0.516	
1521		-----		-----	0.516	
1538		-----		-----	0.501	
1613		-----		-----	-----	
1755		-----		-----	-----	
1757		-----		-----	-----	
6041	D3341	0.496		-1.03	0.496	
	normality	unknown			OK	
	n	5			13	
	outliers	0			0	
	mean (n)	0.5060			0.5074	
	st.dev. (n)	0.01290			0.01340	
	R(calc.)	0.0361			0.0375	
	R(D3341:05)	0.0272			0.0272	



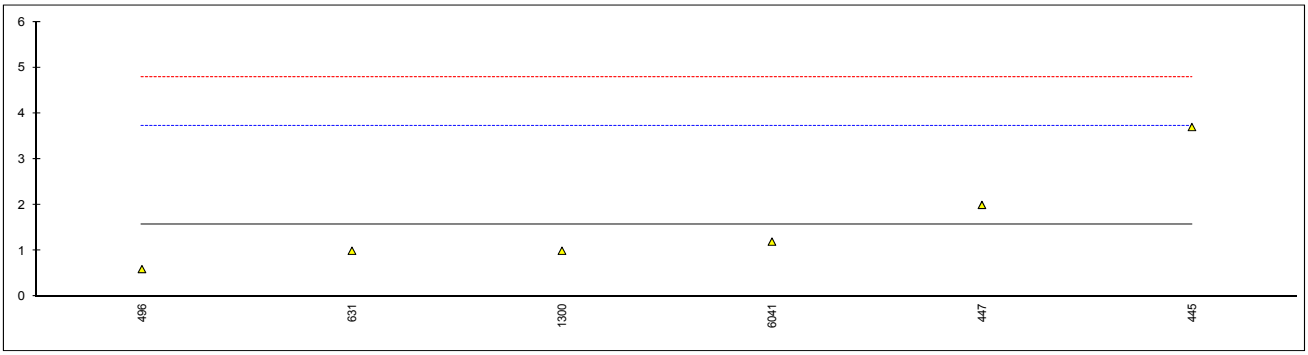
Determination of Lead precipitate content on sample #16038; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
62		-----		-----	
150		-----		-----	
334		-----		-----	
445	D873	0.3		-----	
447		-----		-----	
496		-----		-----	
606		-----		-----	
631	D873	0		-----	
823		-----		-----	
970		-----		-----	
1016	D873	0		-----	
1047	D873	<1		-----	
1161		-----		-----	
1300		-----		-----	
1521	D873	<1		-----	
1538		-----		-----	
1613		-----		-----	
1755		-----		-----	
1757		-----		-----	
6041	D873	<1		-----	
	normality	unknown			
	n	6			
	outliers	n.a.			
	mean (n)	<1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D873:12)	n.a.			

Determination of Potential Gum on sample #16038; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
62		-----		-----	
150	D873	<1		-----	
334		-----		-----	
445	D873	3.7		1.98	
447	IP138	2		0.39	
496	D873	0.6		-0.92	
606		-----		-----	
631	D873	1		-0.54	
823		-----		-----	
970		-----		-----	
1016	D873	<1		-----	
1047	D873	<1		-----	
1161		-----		-----	
1300	D873	1.0		-0.54	
1521	D873	<1		-----	
1538		-----		-----	
1613		-----		-----	
1755		-----		-----	
1757		-----		-----	
6041	D873	1.2		-0.36	

normality	unknown
n	6
outliers	0
mean (n)	1.58
st.dev. (n)	1.136
R(calc.)	3.18
R(D873:12)	3.00



Determination of Sulphur content on sample #16038; results in mg/kg

lab	method	value	mark	z(targ)	remarks
62	D5453	0		-----	
150	D2622	<3		-----	
334	D5453	0.2		-----	
445	D2622	<3		-----	
447		-----		-----	
496	D5453	0.34		-----	
606		-----		-----	
631	D5453	0.04		-----	
823	D5453	<1		-----	
970	D5453	<1.0		-----	
1016	D2622	<3		-----	
1047	ISO20846	< 3		-----	
1161	ISO20846	<0.5		-----	
1300		-----		-----	
1521	D2622	<3.0		-----	
1538		-----		-----	
1613	D5453	1		-----	
1755	D2622	27.63	C	-----	False positive test result? First reported 0.0042%M/M
1757		-----		-----	
6041	D5453	1		-----	
	normality	unknown			
	n	7			
	outliers	n.a.			
	mean (n)	<3			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D2622:10)	n.a.			

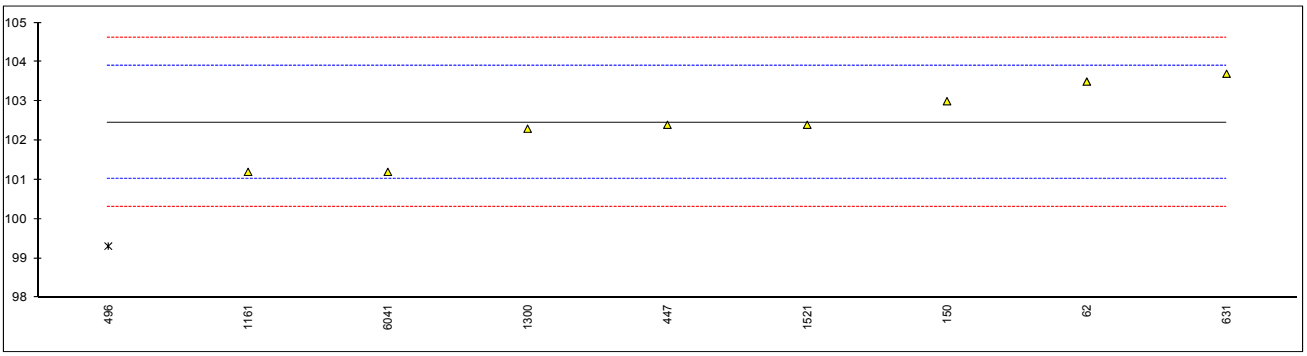
Determination of Water reaction, volume change on sample #16038; results in ml

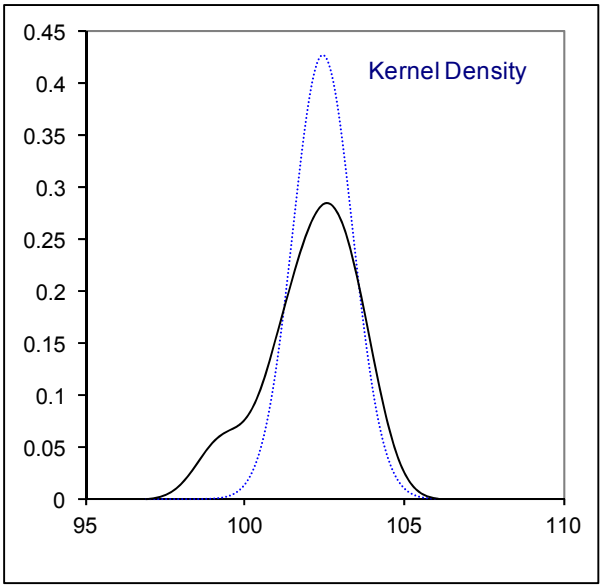
lab	method	value	mark	z(targ)	remarks
62		-----		-----	
150	D1094	0.0		-----	
334		-----		-----	
445	D1094	<0.5		-----	
447	D1094	<0.5		-----	
496	D1094	0		-----	
606	D1094	0.0		-----	
631	D1094	<0.5		-----	
823	D1094	<1		-----	
970		-----		-----	
1016	D1094	<0.5		-----	
1047	D1094	0		-----	
1161		-----		-----	
1300	D1094	<0.5		-----	
1521	D1094	0.0		-----	
1538	D1094	<0.5		-----	
1613	D1094	0.0		-----	
1755	D1094	0.5		-----	
1757	D1094	0.5		-----	
6041	D1094	-0.5		-----	
	normality	suspect			
	n	16			
	outliers	n.a.			
	mean (n)	<0.5			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D1094:07)	n.a.			

Determination of MON (lean mixture) on sample #16038

lab	method	value	mark	z(targ)	remarks
62	D2700	103.5		1.45	
150	D2700	103.0		0.75	
334		-----		-----	
445	D2700	>100		-----	
447	D2700	102.4		-0.09	
496	D2700	99.31	G(0.05)	-4.41	
606		-----		-----	
631	D2700	103.7		1.73	
823		-----		-----	
970		-----		-----	
1016		-----		-----	
1047		-----		-----	
1161	ISO5163	101.2		-1.77	
1300	D2700	102.3		-0.23	
1521	D2700	102.4		-0.09	
1538		-----		-----	
1613		-----		-----	
1755		-----		-----	
1757		-----		-----	
6041	D2700	101.2		-1.77	

normality	OK
n	8
outliers	1
mean (n)	102.46
st.dev. (n)	0.935
R(calc.)	2.62
R(D2700:16)	2.00





APPENDIX 2

Number of participants per country

2 labs in BULGARIA
1 lab in CANADA
1 lab in CHINA, People's Republic of
1 lab in ESTONIA
1 lab in FRANCE
1 lab in GERMANY
1 lab in JORDAN
1 lab in MALAYSIA
1 lab in NETHERLANDS
1 lab in OMAN
1 lab in PHILIPPINES
3 labs in POLAND
1 lab in SOUTH KOREA
1 lab in TURKEY
2 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 Man 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).