Results of Proficiency Test
Acetone
September 2015

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 1999, the Institute for Interlaboratory Studies organizes a proficiency scheme for the analysis of Acetone. During the annual proficiency testing program 2015/2016, it was decided to organize again a round robin for the analysis of Acetone.
In this interlaboratory study 24 laboratories in 15 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2015 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 send one sample Acetone (1*1L bottle, labelled #15162) to the participants. The analyses for fit-for-use and homogeneity determination were subcontracted. The 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/IEC 17043:2010. 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 a 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 is electronically available through the iis internet site 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, approximately 39 kg, for sample #15162 was obtained from a local supplier. The batch was spiked with 1.19 g Diacetone Alcohol and 0.89 g Mesityloxide. After homogenisation in a precleaned drum, 40 amber glass bottles of 1L were filled and labelled #15162. The homogeneity of the subsamples was checked by determination of Water in accordance with ASTM D1364 and Mesityloxide in accordance with an in house method on 4 stratified randomly selected samples.

	Water in mg/kg	Mesityloxide in mg/kg
sample #15162-1	1800	23
sample #15162-2	1800	23
sample #15162-3	1800	23
sample #15162-4	1810	23

table 1: homogeneity test results of subsamples #15162

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Water in mg/kg	Mesityloxide in mg/kg
r (sample #15162)	14	0
reference test method	ASTM D1364:07	Horwitz
0.3 x R(reference test)	81	2

table 2: evaluation of homogeneity of subsamples #15162

The calculated repeatabilities are each less than 0.3 times the reproducibility of the corresponding reference method. Therefore, homogeneity of the samples was assumed.

One 1L bottle, labelled #15162 was dispatched to each of the participating laboratories on August 19, 2015.

2.5 STABILITY OF THE SAMPLES

The stability of the Acetone, packed in the brown glass bottles, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine: Acidity, Aldehydes, Appearance, Chloride as Cl, Colour as Pt/Co, Density at 20^{o}C, Specific gravity 20/20^{o}C, Distillation (IBP, MBP and DP), Water Miscibility, Nonvolatile Matter, Permanganate Time Test at 25^{o}C, Purity on dry basis, Diacetonalcohol, Mesityloxide, Methanol, Refractive Index at 20^{o}C and Water on sample #15162 in accordance with specification ASTM D329:13.

It was explicitly requested to treat the samples as routine samples and to report the analytical results using the indicated units on the report form in the data entry portal and not to round the results, but report as much significant figures as possible. It was also requested not to report 'less than' results, which are above the detection limit, because such results can not be used for meaningful statistical calculations.

To get comparable results a detailed report form, on which the units were prescribed as well, as a letter of instructions were prepared and made available for download on the data entry portal www.kpmd.co.uk/sgs-iis/. A form to confirm receipt of the samples and a SDS were included into 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 represented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that did not report 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, see ref. 10) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. If appropriate, additional or corrected results are used for the data analysis and the 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 Organization, Statistics and Evaluation' of 2014 (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.

According to ISO 5725 the original results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the

Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test (ref. 18). Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. 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 significant 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 visualise 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 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms, see appendix 4, ref.13 and 14. 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, 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 target reproducibility (preferably taken from a standardized test method) by division with 2.8.
The z-scores were calculated in accordance with:

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

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

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. See also appendix 3; ref. 1.

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

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

4 EVALUATION

In this proficiency test, some problems were encountered with the dispatch of the samples to Brazil, India, Russia and Saudi Arabia due to custom clearance problems. From the total of 24 participants, three participants did not report any test result. In total 250 numerical results were reported. Observed were 10 outlying results, which is 4.0%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section, the results are discussed per test. The methods, which are used by the 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. When no suitable test method is available, the Horwitz equation was used.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D1613) and an added designation for the year that the method was adopted or revised (e.g. D1613:06). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1613:06(2012)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D1613:06 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.

Acidity: No analytical problems were observed. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D1613:06(2012).

Aldehydes: No conclusions could be drawn. Only two participants reported a numerical result in accordance with an in house test method. Three other laboratories used test method ASTM D329:07(2013) that describes a pass/fail test.

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #15162, which is bright and clear (or Pass).

Chloride, Inorganic: The Chloride content was near or below the detection limit. Therefore no significant conclusions were drawn.

Colour: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1209:05(2011).

Density at 20°C: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ISO12185:96.

Specific Gravity 20/20°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ISO12185:96.

Distillation: This determination was not problematic. No statistical outliers were observed. All three calculated reproducibilities (for IBP, MBP and DP) were in agreement with the requirements of ASTM D1078:11. From the reported results of the Mid Boiling Point (automated), it was concluded that all participating laboratories, except one may have corrected the results properly for barometric pressure and thermometer accuracy as described in ASTM D1078:11.

Water Miscibility: This determination was not problematic. All laboratories reported this test pass. The analytical method described in ASTM D1722:09 is a pass/fail test.

NVM: The NVM content was near or below the detection limit. Therefore no significant conclusions were drawn.

PTT: This determination was not problematic. No statistical outliers were observed. All participants agreed on a result far above 30 minutes. When a statistical evaluation is performed on the actually reported results, the calculated reproducibility is not at all in agreement with the requirements of ASTM D1363:06(2011). However, as it is unknown whether a Permanganate Time Test result of >100 minutes is in the applicability range, it is therefore difficult to draw any conclusions. Therefore, no z-scores were calculated.

Purity on DB: No statistical outliers were observed. The calculated reproducibility is in line with the calculated reproducibility of the 2013 PT on methanol iis13C07 (0.028 %MM vs 0.028 %M/M).

Diacetonalcohol: This determination may have been problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated requirements, calculated using the Horwitz equation. The average recovery of Diacetone alcohol (theoretical increment of 30.5 mg Diacetone alcohol/kg) may be good: “less than 168%” (the actual blank Diacetone alcohol content is unknown).

Mesityloxide: This determination may have been problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated requirements, calculated using the Horwitz equation. The average recovery of Mesityloxide (theoretical increment of 22.7 mg Mesityloxide/kg) may be good: “less than 103%” (the actual blank Mesityloxide content is unknown).

Methanol: This determination may have been problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the estimated requirements based on the Horwitz equation.

Refractive index: This test was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1218:12.

Water: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D1364:02(2012). A number of laboratories may have problems with the hydroscopic behaviour of acetone (see the Kernel density plot), which may (partly) explain the large variation.

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 results, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM standards) are compared in the next tables.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as acetic acid	mg/kg	19	12.2	9.5	14.0
Aldehydes	---	3	Pass	n.a.	n.a.
Appearance	---	18	Pass	n.a.	n.a.
Chloride as Cl	mg/kg	9	<1	n.a.	n.a.
Colour Pt/Co	---	18	2.5	4.0	7.0
Density at 20 °C	kg/L	18	0.7905	0.0002	0.0005
Specific Gravity 20/20ºC		15	0.7919	0.0002	0.0005
Initial Boiling Point	°C	19	56.0	0.2	0.9
Mid Boiling Point	°C	18	56.1	0.2	0.4
Dry Point	°C	19	56.4	0.5	0.6
Miscibility with water	---	16	Pass	n.a.	n.a.
Nonvolatile Matter	mg/100 mL	14	0.3	0.8	(0.1)
Permanganate Time Test	min	12	117	106	(30)
Purity on dry basis	%M/M	17	99.965	0.028	n.a.
Diacetone alcohol	mg/kg	10	51	15	13
Mesityloxide	mg/kg	9	23	8	7
Methanol	mg/kg	13	150	98	32
Refractive Index	---	15	1.3588	0.0005	0.0005
Water	mg/kg	19	1861	302	270

Table 3: Reproducibilities for sample #15162

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2015 WITH PREVIOUS PTS

	September 2015	September 2013	September 2011	September 2009
Number of reporting labs	21	21	18	19
Number of results reported	250	273	198	216
Statistical outliers	10	7	3	7
Percentage outliers	4.0%	2.6%	1.5%	3.2%

Table 4: comparison of summary data 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:

Determination	September 2013	September 2013	September 2011	September 2009
Acidity as acetic acid	++	++	+	++
Chloride as Cl	n.e.	n.e.	n.e.	n.e.
Colour Pt/Co	++	++	++	++
Density at 20 °C	++	++	++	++
Specific gravity 20/20ºC	++	+	n.e.	n.e.
Distillation	++	++	++	++
Nonvolatile Matter	(--)	++	++	++
Permanganate Time Test	(--)	(--)	n.e.	n.e.
Diacetonalcohol	-	--	--	--
Mesityloxide	-	+/-	n.e.	+
Methanol	--	-	+	++
Refractive Index	+/-	+	+/-	-
Water	-	+/-	--	++

Table 5: comparison determinations against the standard

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

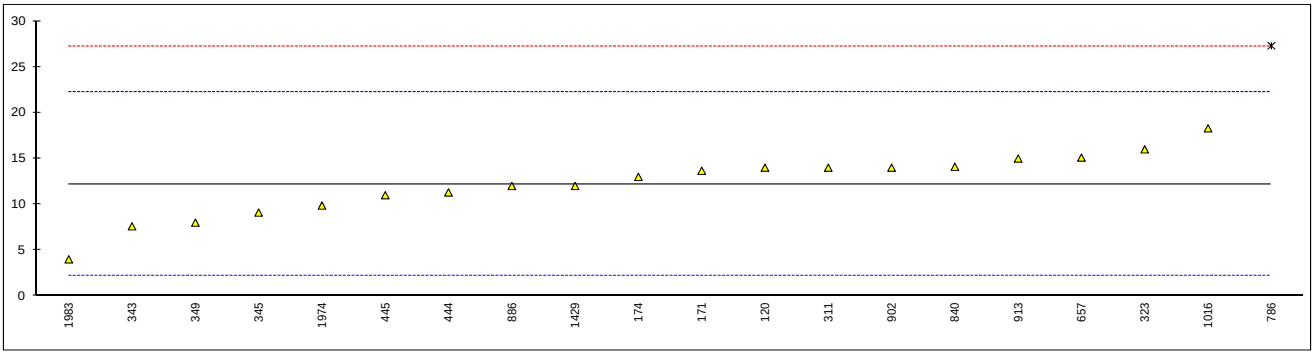
++: group performed much better than the standard
+ : group performed better than the standard
+/-: group performance equals the standard
- : group performed worse than the standard
-- : group performed much worse than the standard
n.e.: not evaluated

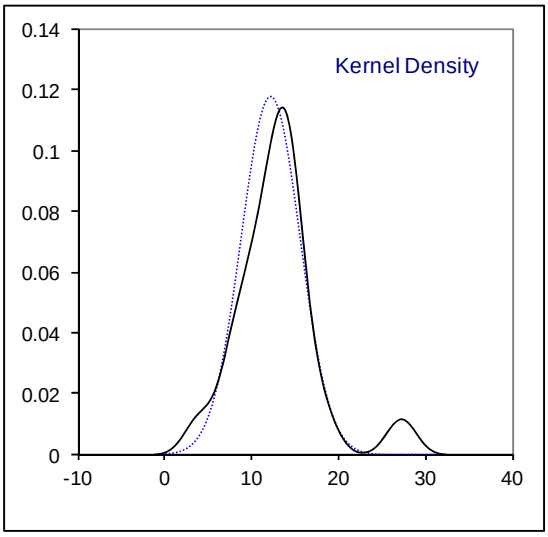
APPENDIX 1

Determination of Acidity on sample #15162; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	D1613	14		0.36	
171	D1613	13.6640		0.29	
174	D1613	13		0.16	
311	D1613	14		0.36	
323	D1613	16		0.76	
343	D1613	7.6		-0.92	
345	D1613	9.1		-0.62	
349	D1613	8		-0.84	
444	D1613	11.3		-0.18	
445	D1613	11		-0.24	
551		-----		-----	
557		-----		-----	
657	D1613	15.1		0.58	
786	D1613	27.3	R(0.01)	3.02	
840	D1613	14.1		0.38	
886	D1613	12	C	-0.04	First reported 0.0012 mg/kg (unit error!)
902	D1613	14	C	0.36	First reported 0.0014 mg/kg (unit error!)
913	D1613	15.0		0.56	
963		-----		-----	
1016	D1613	18.3		1.22	
1429	D1613	12		-0.04	
1438		-----		-----	
1974	D1613	9.87		-0.47	
1983	D1613	4		-1.64	

normality	OK
n	19
outliers	1
mean (n)	12.21
st.dev. (n)	3.395
R(calc.)	9.50
R(D1613:06)	14.00





Determination of Aldehydes on sample #15162;

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171		-----		-----	
174		-----		-----	
311		-----		-----	
323	D329	Pass		-----	
343	D329	Pass		-----	
345	INH-CQ	32.2		-----	
349		-----		-----	
444		-----		-----	
445		-----		-----	
551		-----		-----	
557		-----		-----	
657		-----		-----	
786		-----		-----	
840		-----		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	
963		-----		-----	
1016		-----		-----	
1429		-----		-----	
1438		-----		-----	
1974	D329	Pass		-----	
1983	INH236	42.5		-----	
	normality	n.a.			
	n	3			
	outliers	0			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D329:07)	n.a.			

Determination of Appearance on sample #15162;

lab	method	value	mark	z(targ)	remarks
120	Visual	Pass		-----	
171	E2680	Pass		-----	
174	E2680	Pass		-----	
311	E2680	Pass		-----	
323	Visual	C&B		-----	
343	Visual	C&B		-----	
345	INH-CQ	Pass		-----	
349	E2680	Pass		-----	
444	E2680	Pass		-----	
445	E2680	Pass		-----	
551		-----		-----	
557		-----		-----	
657	E2680	Pass		-----	
786	E2680	Pass		-----	
840	E2680	Pass		-----	
886		-----		-----	
902	E2680	Pass		-----	
913	E2680	Pass		-----	
963		-----		-----	
1016		-----		-----	
1429	E2680	C&B		-----	
1438		-----		-----	
1974	D4176	Pass		-----	
1983	Visual	Pass		-----	
	n	15			
	mean (n)	Pass			

Abbreviations:
C&B: clear and bright

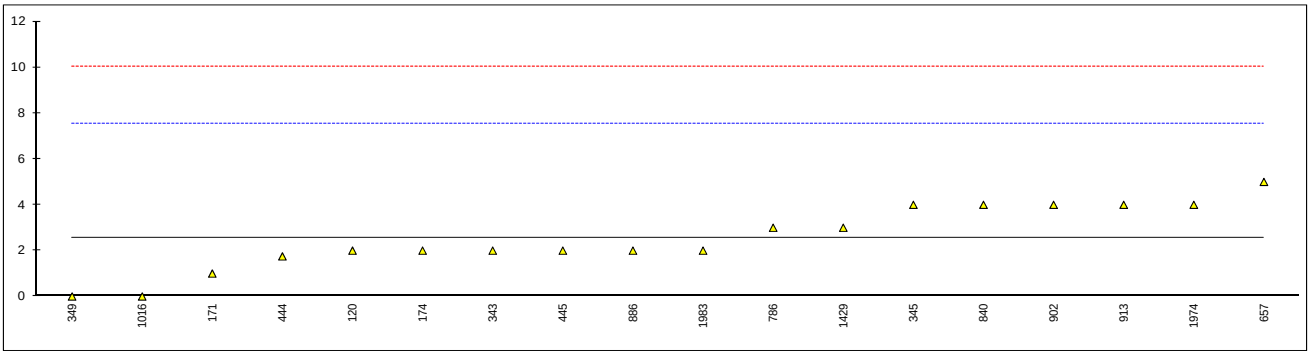
Determination of Chloride Inorganic as Cl on sample #15162; results in mg/kg

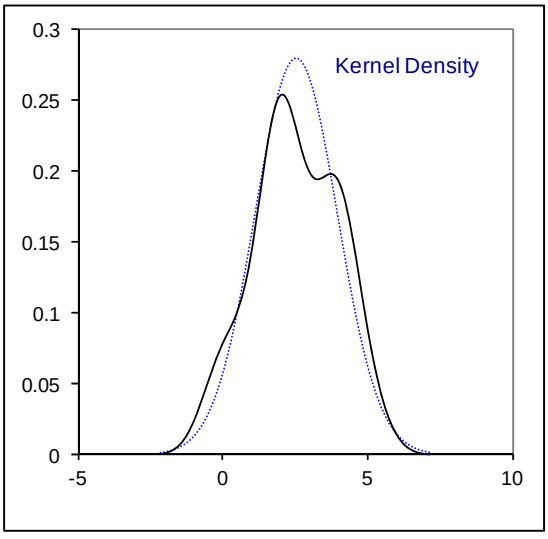
lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171	IMPCA002	0.15		-----	
174		0.15		-----	
311	INH-158	<0.2		-----	
323	INH-008	<1		-----	
343		-----		-----	
345		-----		-----	
349		-----		-----	
444	INH-009	<5		-----	
445		-----		-----	
551		-----		-----	
557		-----		-----	
657		-----		-----	
786	IMPCA002	<0.25		-----	
840	IMPCA002	0.03		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	
963		-----		-----	
1016		-----		-----	
1429	ISO6227	<0.5		-----	
1438		-----		-----	
1974		-----		-----	
1983	D5808	<0.5		-----	
	normality	n.a.			
	n	9			
	outliers	n.a.			
	mean (n)	<1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

Determination of Colour as Pt/Co on sample #15162;

lab	method	value	mark	z(targ)	remarks
120	D1209	2		-0.22	
171	D1209	1		-0.62	
174	D1209	2		-0.22	
311	D1209	<5		-----	
323	D1209	<5		-----	
343	D1209	2		-0.22	
345	D1209	4		0.58	
349	D5386	0		-1.02	
444	D5386	1.75		-0.32	
445	D1209	2		-0.22	
551		-----		-----	
557		-----		-----	
657	D1209	5		0.98	
786	D1209	3		0.18	
840	D1209	4		0.58	
886	D1209	2		-0.22	
902	D5386	4		0.58	
913	D5386	4		0.58	
963		-----		-----	
1016	D1209	0		-1.02	
1429	D6045	3		0.18	
1438		-----		-----	
1974	D1209	4		0.58	
1983	D1209	2		-0.22	

normality	OK
n	18
outliers	0
mean (n)	2.5
st.dev. (n)	1.43
R(calc.)	4.0
R(D1209:05)	7.0

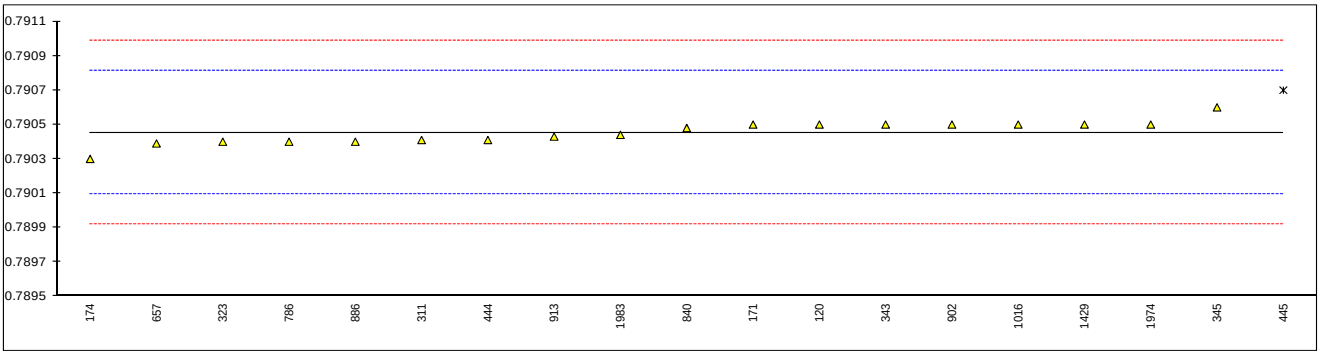


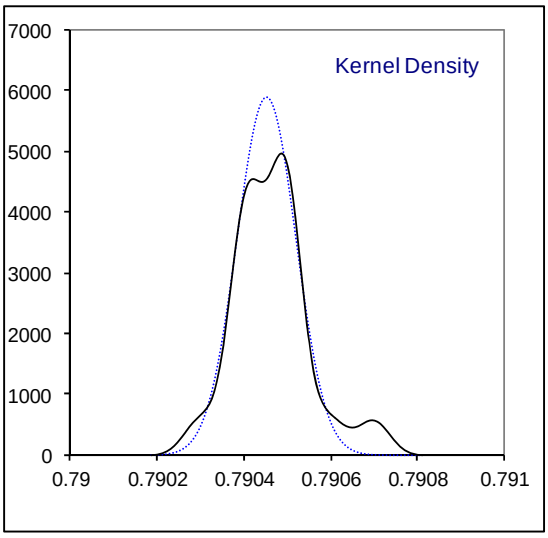


Determination of Density at 20°C on sample #15162; results in kg/L

lab	method	value	mark	z(targ)	remarks
120	D4052	0.7905		0.26	
171	D4052	0.7905		0.26	
174	D4052	0.7903		-0.86	
311	D4052	0.79041		-0.24	
323	D4052	0.7904		-0.30	
343	D4052	0.7905		0.26	
345	ISO12185	0.7906		0.82	
349		-----		-----	
444	D4052	0.79041		-0.24	
445	ISO12185	0.7907	G(0.05)	1.38	
551		-----		-----	
557		-----		-----	
657	D4052	0.79039		-0.35	
786	D4052	0.7904		-0.30	
840	D4052	0.79048		0.15	
886	D4052	0.7904		-0.30	
902	D4052	0.7905		0.26	
913	D4052	0.79043		-0.13	
963		-----		-----	
1016	ISO12185	0.7905		0.26	
1429	D4052	0.7905		0.26	
1438		-----		-----	
1974	D4052	0.7905		0.26	
1983	D4052	0.79044		-0.07	

normality	OK
n	18
outliers	1
mean (n)	0.79045
st.dev. (n)	0.000068
R(calc.)	0.00019
R(ISO12185:96)	0.00050

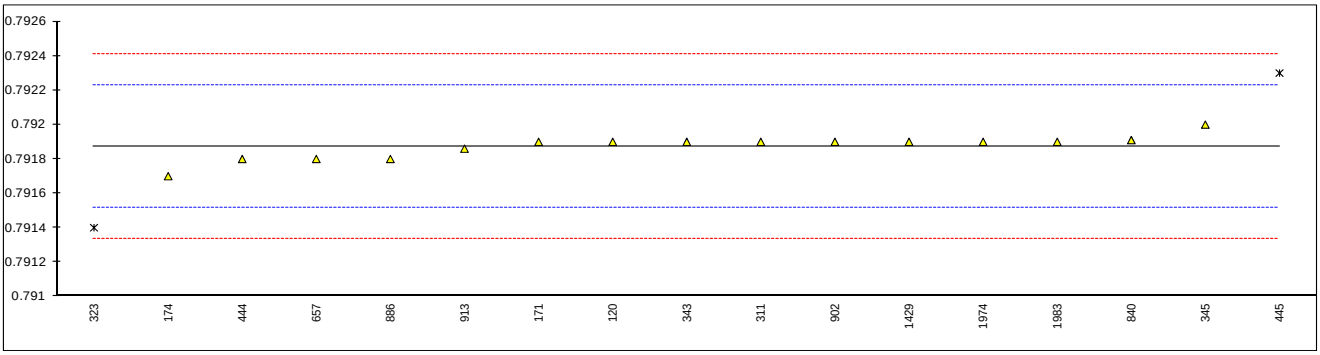


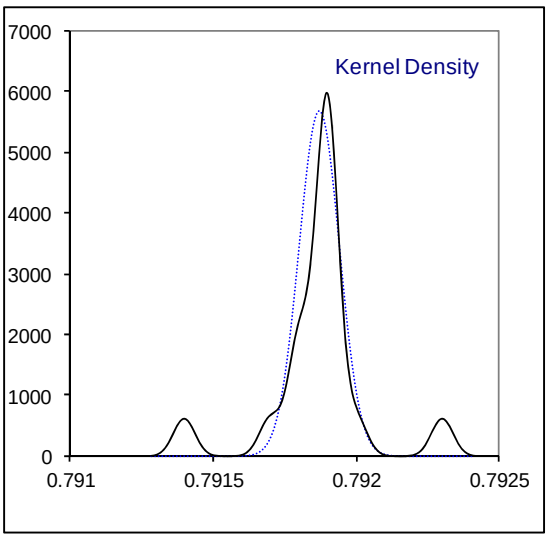


Determination of Specific Gravity 20/20 °C on sample #15162;

lab	method	value	mark	z(targ)	remarks
120	D4052	0.7919		0.16	
171	D4052	0.7919		0.16	
174	D4052	0.7917		-0.96	
311	D4052	0.7919		0.16	
323	D4052	0.7914	G(0.05)	-2.64	
343	D4052	0.7919		0.16	
345	ISO12185	0.7920		0.72	
349		-----		-----	
444	D4052	0.7918		-0.40	
445	ISO12185	0.7923	G(0.01)	2.40	
551		-----		-----	
557		-----		-----	
657	D4052	0.7918		-0.40	
786		-----		-----	
840	D4052	0.79191		0.22	
886	D4052	0.7918		-0.40	
902	D4052	0.7919		0.16	
913	D4052	0.79186		-0.06	
963		-----		-----	
1016		-----		-----	
1429	D4052	0.7919		0.16	
1438		-----		-----	
1974	D4052	0.7919		0.16	
1983	D4052	0.7919		0.16	

normality	suspect
n	15
outliers	2
mean (n)	0.79187
st.dev. (n)	0.000070
R(calc.)	0.00020
R(ISO12185:96)	0.00050

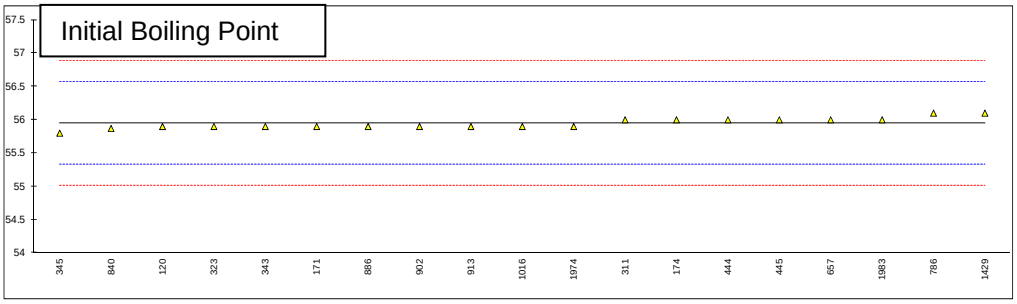


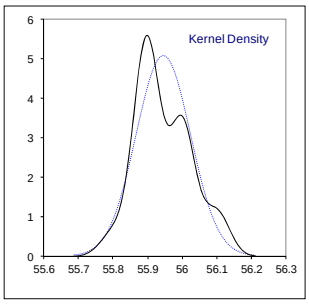


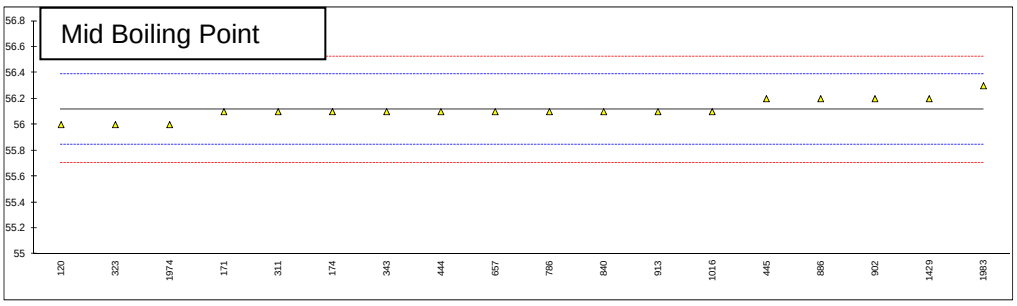
Determination of IBP, MBP and DP at 760 mmHg on sample #15162; results in °C

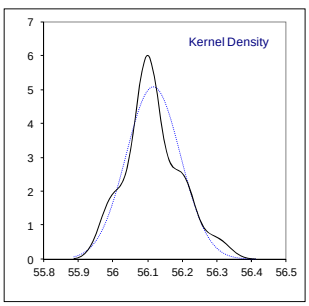
lab	method	mode	IBP	mark	z(targ)	MBP	mark	z(targ)	DP	mark	z(targ)
120	D1078	Automated	55.9		-0.15	56.0		-0.85	56.6		0.69
171	D1078	Automated	55.9		-0.15	56.1		-0.12	56.6		0.69
174	D1078	Automated	56.0		0.17	56.1		-0.12	56.7		1.15
311	D1078	Manual	56.0		0.17	56.1		-0.12	56.3		-0.71
323	D1078	Manual	55.9		-0.15	56.0		-0.85	56.3		-0.71
343	D1078	Automated	55.9		-0.15	56.1		-0.12	56.2		-1.17
345	D1078	Automated	55.8		-0.47	-----		-----	56.4		-0.24
349			-----		-----	-----		-----	-----		-----
444	D1078	Manual	56.0	C	0.17	56.1	C	-0.12	56.5	C	0.22
445	D1078	Manual	56.0		0.17	56.2		0.61	56.8		1.61
551			-----		-----	-----		-----	-----		-----
557			-----		-----	-----		-----	-----		-----
657	D1078	Manual	56.0		0.17	56.1		-0.12	56.5		0.22
786	D1078	Manual	56.1		0.49	56.1		-0.12	56.3		-0.71
840	D1078	Automated	55.87		-0.24	56.10		-0.12	56.29		-0.75
886	D1078		55.9		-0.15	56.2		0.61	56.3		-0.71
902	D1078	Manual	55.9		-0.15	56.2		0.61	56.5		0.22
913	D1078	Automated	55.9		-0.15	56.1		-0.12	56.7		1.15
963			-----		-----	-----		-----	-----		-----
1016	D1078	Automated	55.9		-0.15	56.1		-0.12	56.4		-0.24
1429	D1078	Automated	56.1		0.49	56.2		0.61	56.4		-0.24
1438			-----		-----	-----		-----	-----		-----
1974	D1078	Automated	55.9		-0.15	56.0		-0.85	56.1		-1.63
1983	D1078	Automated	56.0		0.17	56.3		1.34	56.7		1.15
	normality		OK			OK			OK		
	n		19			18			19		
	outliers		0			0			0		
	mean (n)		55.95			56.12			56.45		
	st.dev. (n)		0.079			0.079			0.193		
	R(calc.)		0.22			0.22			0.54		
	R(D1078:11-A)		0.87			0.38			0.60		
Comp.	R(D1078:11-B)		0.60			0.36			0.73		

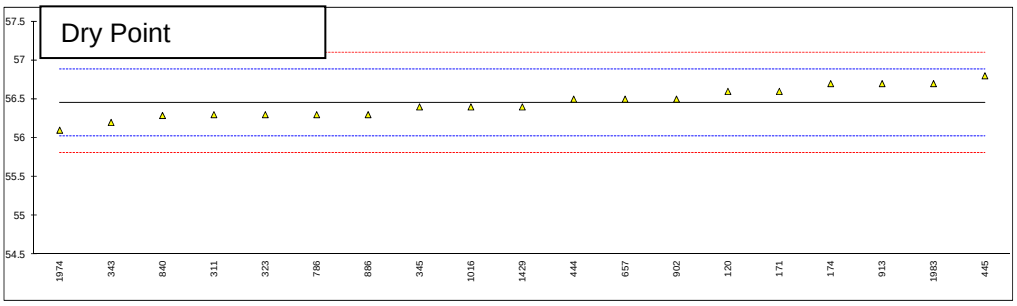
Lab 444: first reported 55.3; 55.4; 55.8

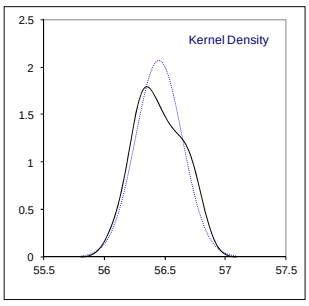










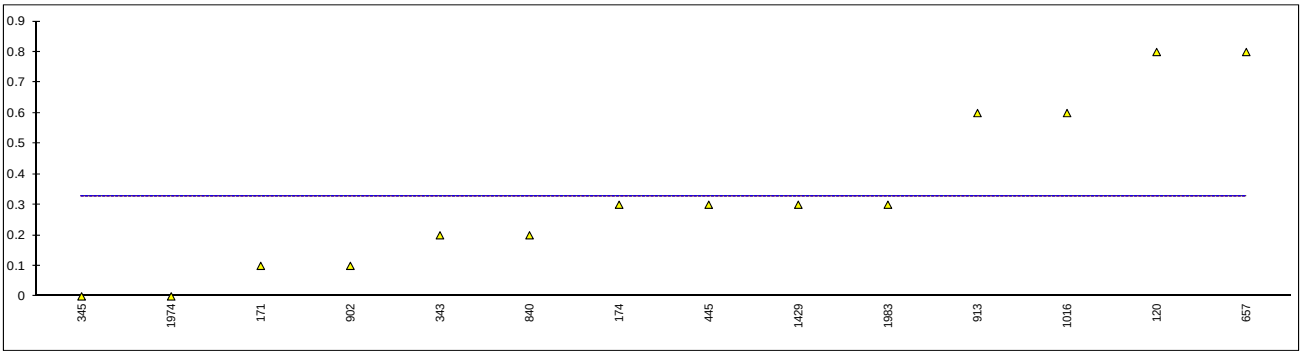


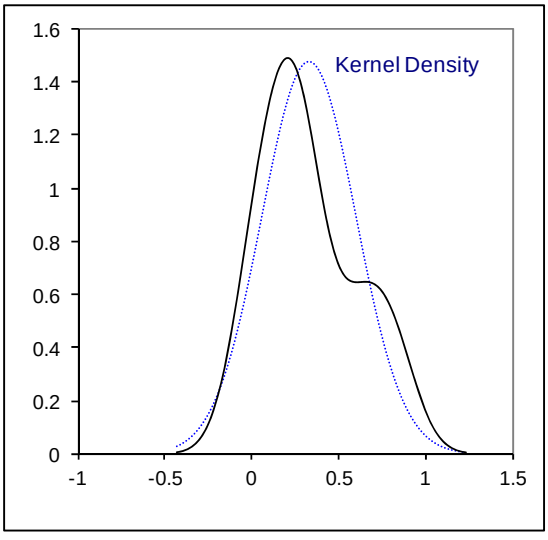
Determination of Miscibility with Water on sample #15162;

lab	method	value	mark	z(targ)	remarks
120	D1722	Pass		-----	
171	D1722	Pass		-----	
174	D1722	Pass		-----	
311	D1722	Pass		-----	
323	D1722	Pass		-----	
343		-----		-----	
345		-----		-----	
349		-----		-----	
444	D1722	Pass		-----	
445	D1722	Pass		-----	
551		-----		-----	
557		-----		-----	
657	D1722	Pass		-----	
786	D1722	Pass		-----	
840	D1722	Pass		-----	
886		-----		-----	
902	D1722	Pass		-----	
913	D1722	Pass		-----	
963		-----		-----	
1016	D1722	Pass		-----	
1429	D1722	Pass		-----	
1438		-----		-----	
1974	D1722	Pass		-----	
1983	D1722	Pass		-----	
	normality	n.a.			
	n	16			
	outliers	0			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

Determination of Nonvolatile Matter on sample #15162; results in mg/100 mL

lab	method	value	mark	z(targ)	remarks
120	D1353	0.8		-----	
171	D1353	0.1		-----	
174	D1353	0.3		-----	
311	D1353	<1.0		-----	
323	D1353	<1		-----	
343	D1353	0.2		-----	
345	D1353	0		-----	
349		-----		-----	
444	D1353	<0.1	C	-----	First reported 7.9
445	D1353	0.3		-----	
551		-----		-----	
557		-----		-----	
657	D1353	0.8		-----	
786		-----		-----	
840	D1353	0.2		-----	
886		-----		-----	
902	D1353	0.1		-----	
913	D1353	0.6		-----	
963		-----		-----	
1016	D1353	0.600		-----	
1429	D1353	0.3		-----	
1438		-----		-----	
1974	D1353	0.0000		-----	
1983	D1353	0.3	C	-----	First reported 2.3
	normality	OK			
	n	14			
	outliers	0			
	mean (n)	0.33			
	st.dev. (n)	0.270			
	R(calc.)	0.76			
	R(D1353:13)	(0.13)			

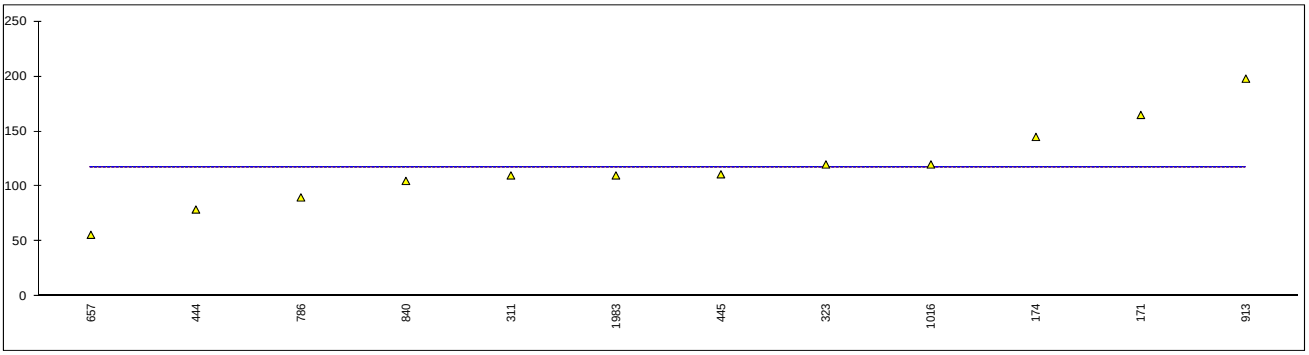


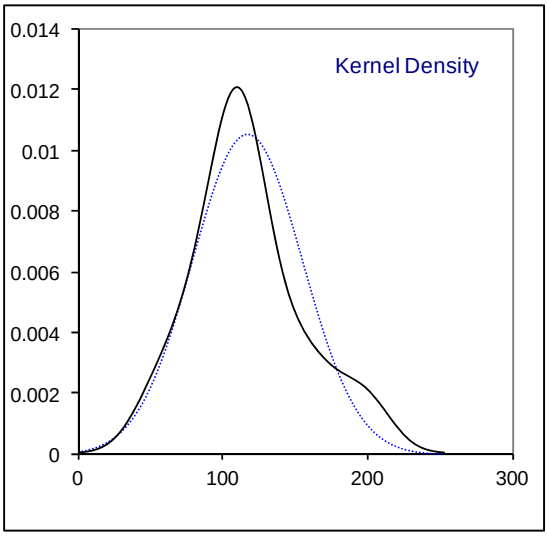


Determination of Permanganate Time Test at 25°C on sample #15162; results in minutes

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171	D1363	165		-----	
174	D1363	145		-----	
311	D1363	110		-----	
323	D1363	120		-----	
343	D1363	>150		-----	
345	INH-CQ	>150		-----	
349		-----		-----	
444	D1363	79		-----	
445	D1363	111		-----	
551		-----		-----	
557		-----		-----	
657	D1363	56		-----	
786	D1363	90		-----	
840	D1363	105		-----	
886		-----		-----	
902	D1363	>30		-----	
913	D1363	198		-----	
963		-----		-----	
1016	D1363	120		-----	
1429	D1363	>60		-----	
1438		-----		-----	
1974	D1363	>120		-----	
1983	D1363	110		-----	

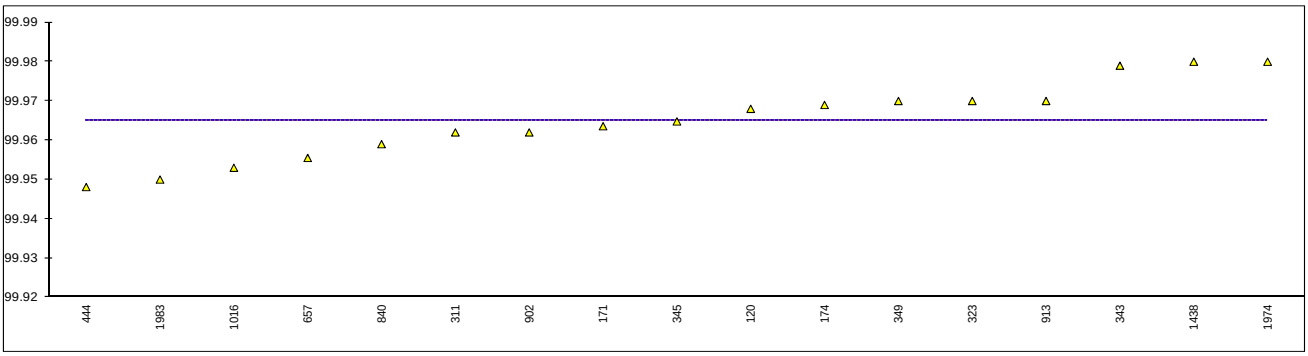
normality	OK
n	12
outliers	0
mean (n)	117
st.dev. (n)	37.9
R(calc.)	106
R(D1363:06)	(30)

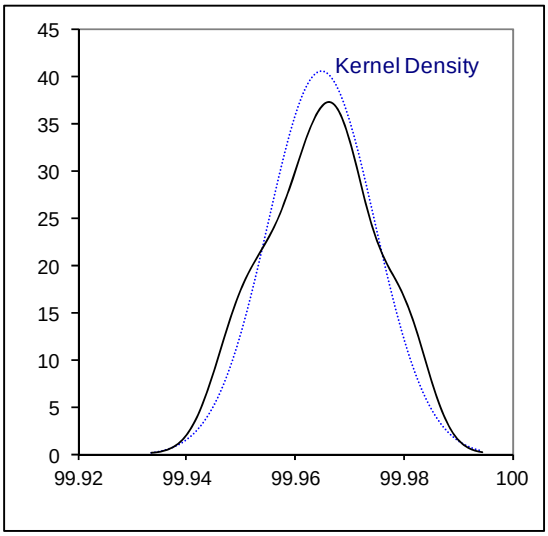




Determination of Purity on Dry Basis on sample #15162, results in %M/M

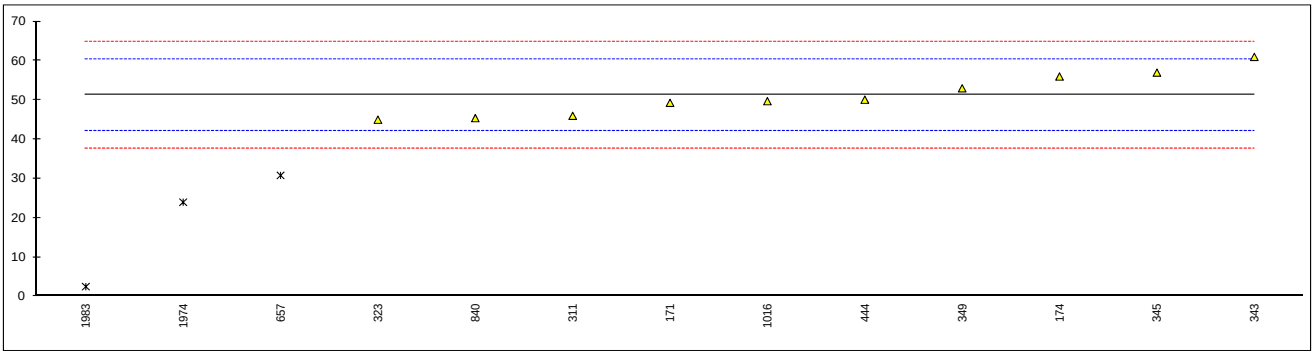
lab	method	value	mark	z(targ)	remarks
120		99.968		-----	
171	INH-001	99.96358		-----	
174	INH-ACS	99.969		-----	
311	INH-394	99.962		-----	
323	INH-020	99.97		-----	
343	INH-CM	99.979		-----	
345	INH-CQ	99.9648		-----	
349	INH-034	99.97		-----	
444	INH-001	99.9481		-----	
445		-----		-----	
551		-----		-----	
557		-----		-----	
657	INH-009	99.9555		-----	
786		-----		-----	
840	INH-005	99.959		-----	
886		-----		-----	
902	INH-125	99.962		-----	
913	D2804	99.970		-----	
963		-----		-----	
1016	DIN55687	99.953		-----	
1429		-----		-----	
1438	in house	99.98		-----	
1974	D2017	99.98		-----	
1983	INH-236	99.95		-----	
	normality	OK			
	n	17			
	outliers	0			
	mean (n)	99.9649			
	st.dev. (n)	0.00983			
	R(calc.)	0.0275			
	R(lit)	unknown			Compare R(iis13C07) = 0.0283

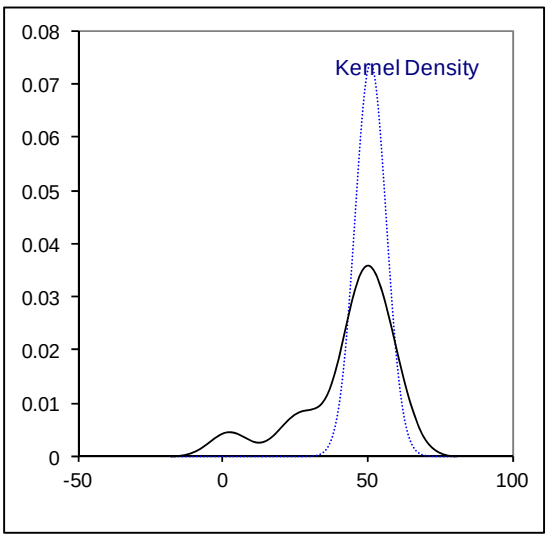




Determination of Diacetonalcohol on sample #15162, results in mg/kg

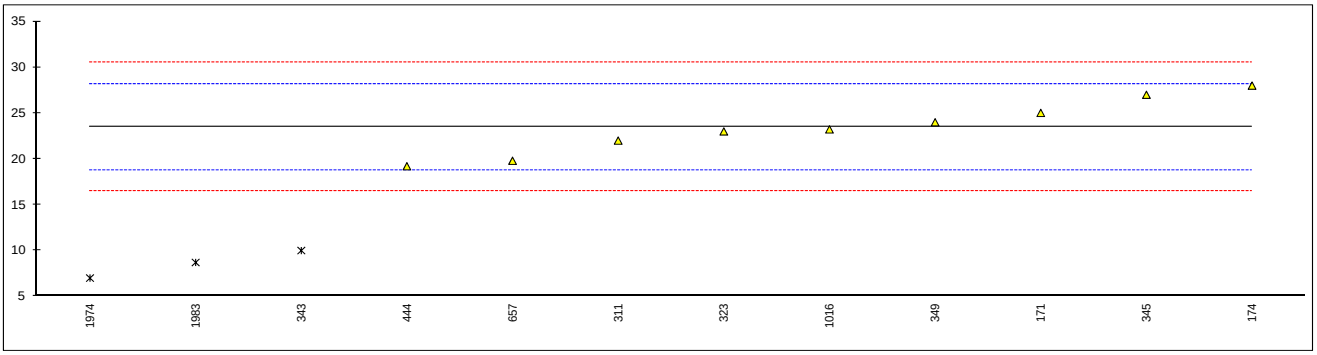
lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171	INH-001	49.3		-0.43	
174	INH-ACS	56		1.05	
311	INH-394	46		-1.16	
323	INH-020	45		-1.38	
343	INH-CM	61		2.15	
345	INH-CQ	57		1.27	
349	INH-034	53		0.39	
444	INH-001	50.1		-0.25	
445		-----		-----	
551		-----		-----	
557		-----		-----	
657	INH-009	30.8192	DG(0.05)	-4.51	
786		-----		-----	
840	INH-005	45.4		-1.29	
886		-----		-----	
902		-----		-----	
913		-----		-----	
963		-----		-----	
1016	DIN55687	49.722		-0.34	
1429		-----		-----	
1438		-----		-----	
1974	D2017	24	DG(0.05)	-6.01	
1983	INH-236	2.5	C,G(0.05)	-10.75	First reported 0.0
	normality	OK			
	n	10			
	outliers	3	Spike:		
	mean (n)	51.25	30.5		Recovery <168%
	st.dev. (n)	5.396			
	R(calc.)	15.11			
	R(Horwitz)	12.70			

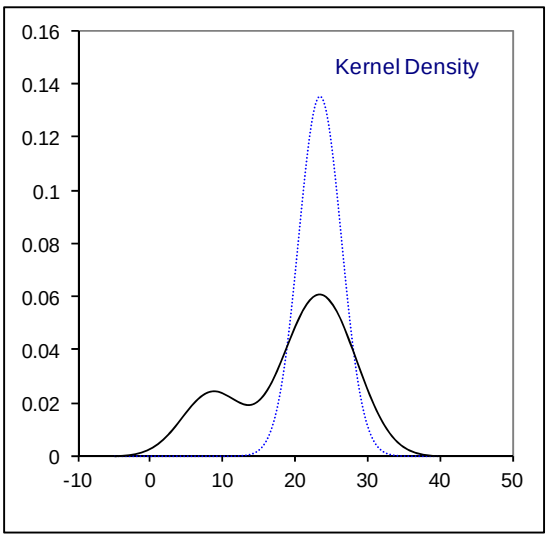




Determination of Mesityloxide on sample #15162, results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171	INH-001	25.02		0.66	
174	INH-ACS	28		1.94	
311	INH-394	22		-0.63	
323	INH-020	23		-0.20	
343	INH-CM	10	DG(0.05)	-5.77	
345	INH-CQ	27		1.51	
349	INH-034	24		0.23	
444	INH-001	19.2		-1.83	
445		-----		-----	
551		-----		-----	
557		-----		-----	
657	INH-009	19.8	C	-1.57	First reported 96
786		-----		-----	
840		-----		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	
963		-----		-----	
1016	DIN55687	23.221		-0.11	
1429		-----		-----	
1438		-----		-----	
1974	D2017	7	G(0.05)	-7.05	
1983	INH-236	8.7	DG(0.05)	-6.32	
	normality	OK			
	n	9			
	outliers	3	Spike:		
	mean (n)	23.47	22.7		Recovery <103%
	st.dev. (n)	2.954			
	R(calc.)	8.27			
	R(Horwitz)	6.54			

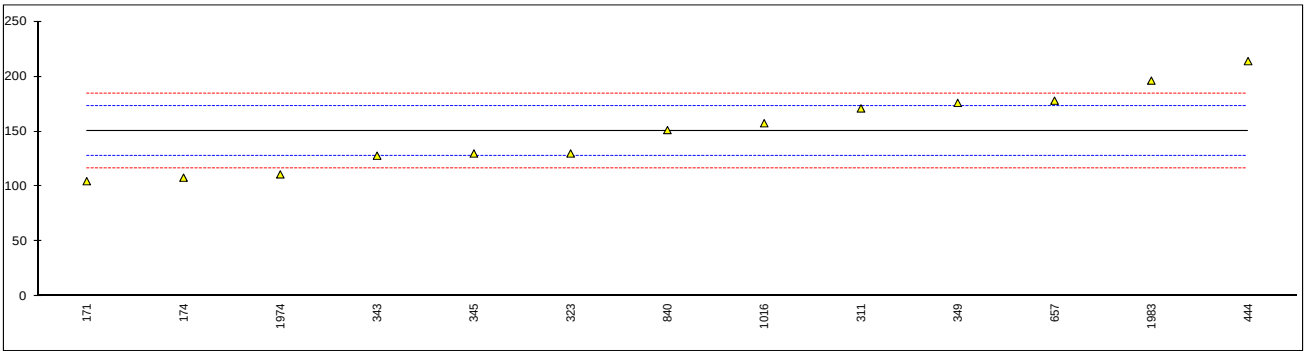


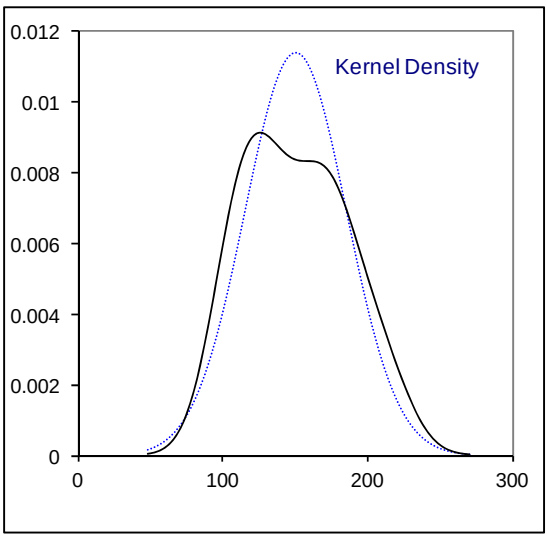


Determination of Methanol on sample #15162, results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171	INH-001	104.8		-4.03	
174	INH-ACS	108		-3.75	
311	INH-394	171		1.82	
323	INH-020	130		-1.81	
343	INH-CM	128		-1.98	
345	INH-CQ	130		-1.81	
349	INH-034	176		2.26	
444	INH-001	214.0		5.61	
445		-----		-----	
551		-----		-----	
557		-----		-----	
657	INH-009	178		2.43	
786		-----		-----	
840	INH-005	151.3		0.07	
886		-----		-----	
902		-----		-----	
913		-----		-----	
963		-----		-----	
1016	DIN55687	157.511		0.62	
1429		-----		-----	
1438		-----		-----	
1974	D2017	111		-3.49	
1983	INH-236	196.3		4.05	

normality	OK
n	13
outliers	0
mean (n)	150.45
st.dev. (n)	35.129
R(calc.)	98.36
R(Horwitz)	31.69

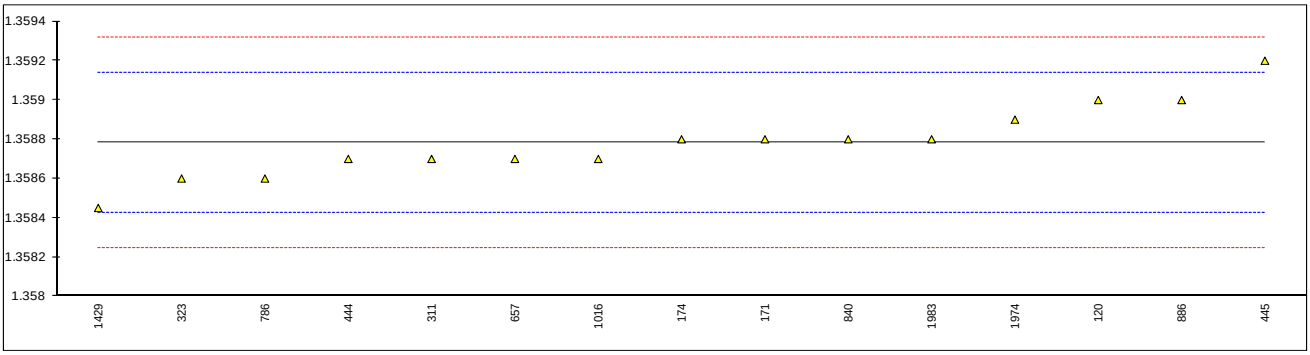


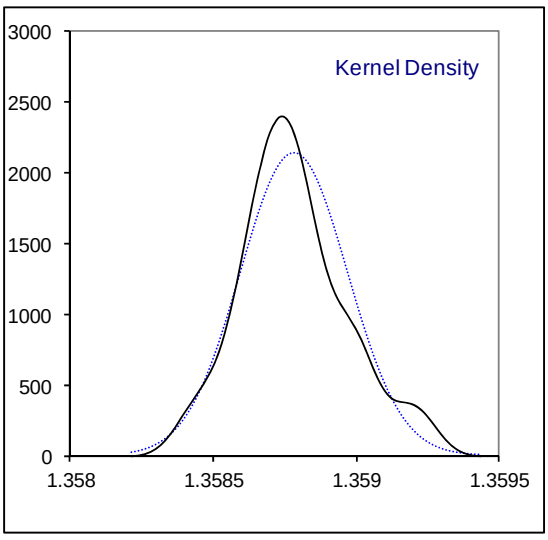


Determination of Refractive Index at 20°C on sample #15162;

lab	method	value	mark	z(targ)	remarks
120	D1218	1.359		1.21	
171	D1218	1.3588		0.09	
174	D1218	1.3588		0.09	
311	D1218	1.3587		-0.47	
323	D1218	1.3586		-1.03	
343		-----		-----	
345		-----		-----	
349		-----		-----	
444	D1218	1.3587		-0.47	
445	D1218	1.3592		2.33	
551		-----		-----	
557		-----		-----	
657	D1218	1.3587		-0.47	
786	D1218	1.3586		-1.03	
840	D1218	1.3588		0.09	
886	D1218	1.3590		1.21	
902		-----		-----	
913		-----		-----	
963		-----		-----	
1016	D1218	1.3587		-0.47	
1429	D1218	1.35845		-1.87	
1438		-----		-----	
1974	D1218	1.3589		0.65	
1983	D1218	1.3588		0.09	

normality	OK
n	15
outliers	0
mean (n)	1.35878
st.dev. (n)	0.000187
R(calc.)	0.00052
R(D1218:12)	0.00050

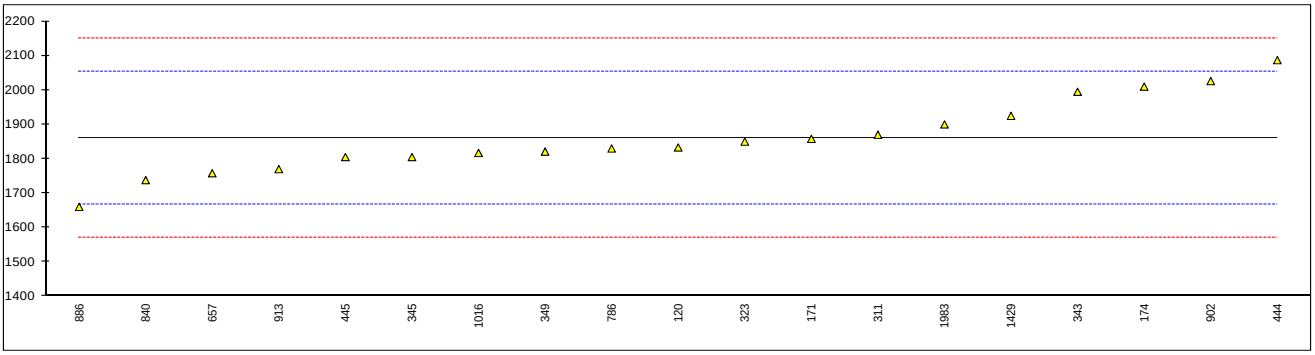


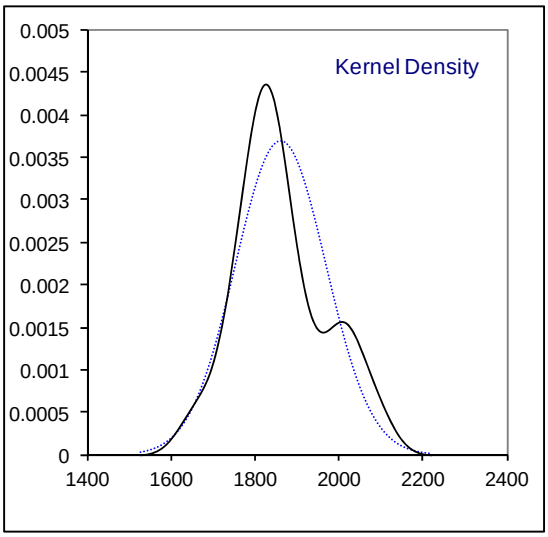


Determination of Water, titrimetric on sample #15162; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	E203	1833		-0.29	
171	D1364	1858		-0.03	
174	D1364	2010		1.55	
311	D1364	1870		0.09	
323	D1364	1850		-0.11	
343	E203	1995		1.39	
345	D1364	1805		-0.58	
349	D1364	1821	C	-0.41	First reported 2621
444	E203	2087		2.34	
445	E203	1805		-0.58	
551		-----		-----	
557		-----		-----	
657	E1064	1758		-1.07	
786	D1364	1830		-0.32	
840	D1364	1738		-1.28	
886	E203	1660		-2.08	
902	D1364	2026		1.71	
913	D1364	1770		-0.94	
963		-----		-----	
1016	D1364	181.7	C,G(0.01)	-0.46	First reported 0.1817 mg/kg (unit error!)
1429	D1364	1925		0.66	
1438		-----		-----	
1974		-----		-----	
1983	D1364	1900		0.40	

normality	OK
n	19
outliers	0
mean (n)	1861
st.dev. (n)	108.1
R(calc.)	302
R(D1364:02)	270





APPENDIX 2

Number of participants per country

1 lab in BELGIUM
2 labs in BRAZIL
1 lab in INDIA
1 lab in ISRAEL
2 labs in NETHERLANDS
1 lab in RUSSIAN FEDERATION
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
3 labs in SPAIN
1 lab in TAIWAN R.O.C.
1 lab in THAILAND
1 lab in TURKEY
4 labs in U.S.A.
3 labs in UNITED KINGDOM
1 lab in VIETNAM

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
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner outlier test
E	= error in calculations
ex	= excluded from calculations
n.a.	= not available / not applicable
n.e.	= not evaluated
wd	= withdrawn method

Literature:

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