

Results of Proficiency Test
Isopropanol
(Isopropyl alcohol)
November 2013

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2003, the Institute for Interlaboratory Studies organises a proficiency test for the analysis of Isopropanol. As part of the annual proficiency test program of 2013/2014 the Institute decided to continue this proficiency test on Isopropanol. The proficiency test of Isopropanol has been organised in accordance with the latest applicable version of the ASTM D770:11 specifications and a number of additional tests requested by some participants. In this interlaboratory study, 17 laboratories out of 12 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2013 proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. Analyses for fit-for-use and homogeneity testing were subcontracted. It was decided to send one 500 ml bottle with Isopropanol, labelled #13225 to the participants. 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 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' (iis-protocol, version 3.2) of January 2010.

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

Approximately 25 litre of high purity Isopropanol was obtained from a local chemical supplier. After homogenisation, 39 amber glass bottles of 500 ml with inner and outer caps were filled and labelled #13225. The homogeneity of subsample #13225 was checked by determination of the Density in accordance with ASTM D4052:11 and Water in accordance with ASTM D1364:12 on resp. 8 and 7 stratified randomly selected samples.

	Density at 20°C in kg/L	Water content in %M/M
sample #13225-1	0.78504	0.0134
sample #13225-2	0.78503	0.0134
sample #13225-3	0.78504	0.0146
sample #13225-4	0.78504	0.0133
sample #13225-5	0.78504	0.0131
sample #13225-6	0.78503	0.0138
sample #13225-7	0.78502	0.0147
sample #13225-8	0.78505	--

Table 1: homogeneity test of subsample #13225

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

	Density at 20°C in kg/l	Water in %M/M
r (Observed)	0.00003	0.0018
reference method	ISO12185:96	ASTM D1364:12
0.3 * R (ref method)	0.00015	0.0021

Table 2: repeatabilities of subsamples #13225

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the target method. Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories 1* 0.5 litre bottle, labelled #13225 was sent on October 30, 2013.

2.5 STABILITY OF THE SAMPLES

The stability of Isopropanol, packed in a amber glass bottle, 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, Anorganic Chloride, Appearance, Colour Pt/Co, Density 20°C, Distillation (IBP, 50% evaporated & DP), Nonvolatile Matter, Peroxide, Specific Gravity 20/20°C, Water, Purity (both "as received" and on dry basis), Ethanol, n-Propanol, n-Butanol, Methyleneethylketone and Other Impurities on sample #13225. To get

comparable results, a detailed report form on which the units and the preferred test methods were printed, was sent together with each of the samples. Also a letter of instructions and a SDS were added to the 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). 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 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 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 cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This method is for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nr.12 and 13).

3.3 Z-SCORES

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

The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated in accordance with:

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

The $Z_{(\text{target})}$ 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.

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:

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

4 EVALUATION

In this proficiency test, some problems were encountered with despatch of the samples. The participant in Brazil received the sample after the final reporting date. From the 16 reporting participants, four participants did report the results after the final reporting date. The 13 reporting laboratories submitted 168 numerical results. Observed were 7 outlying results, which is 4.2%. 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. When no literature reproducibility was available, other target values were used. In these cases, the Horwitz equation is used to estimate the target reproducibility.

Not normal distributions were found for the following determinations: Colour Pt/Co, Distillation (MBP and DP) and Purity on dry basis. In these cases the statistical evaluation should be used with due care.

Acidity: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1613:12.

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #13225, which is bright, clear and free of suspended matter (= pass).

Anorg. Chloride: Only three participants reported a numerical test result near or below the detection limit. Therefore, no significant conclusions were drawn.

Colour Pt/Co: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1209:11.

Density @ 20 °C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ISO12185:96.

Specific Gravity @ 20/20 °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.

- Distillation: This determination was not problematic. No statistical outliers were observed and all three calculated reproducibilities are in good agreement with the requirements of ASTM D1078:11 for the automated and the manual mode. Two laboratories probably did not correct the results for barometric pressure and/or thermometric correction properly.
- NVM: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1353:09.
- Water: This determination was problematic for a number of laboratories. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D1364:12.
- Purity "as received": Regretfully, the methods used do not provide any reproducibility limit. Therefore no significant conclusions were drawn. However, in comparison with the previous proficiency test, the calculated reproducibility is much smaller than the calculated reproducibility found in the previous proficiency test (iis11C14) of November 2011 (0.028 vs 0.087)
- Purity on dry basis: Regretfully, the methods used do not provide any reproducibility limit. Therefore no significant conclusions were drawn. However, in comparison with the previous proficiency test, the calculated reproducibility is smaller than the calculated reproducibility found in the previous proficiency test (iis11C14) of November 2011 (0.017 vs 0.025)
- Ethanol: No participant reported a numerical test result. Therefore, no significant conclusions were drawn.
- n-Propanol: This determination may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility calculated using the Horwitz equation.
- n-Butanol: Only one participant reported a numerical test result. Therefore, no significant conclusions were drawn.
- MEK: No participant reported a numerical test result. Therefore, no significant conclusions were drawn.
- Other Imp.: This determination may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility calculated using the Horwitz equation (for 4 components). One should note that the

actual amount of so called “other impurities” depends on a number of parameters, like: definition, GLC column, GLC conditions, Stop time, detection limits, etc.

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 that participated. The reproducibilities derived from literature standards (in casu ASTM standards) and the calculated reproducibilities of sample #13225 are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as Acetic Acid	%M/M	15	0.0008	0.0005	0.0014
Appearance		14	Pass	n.a.	n.a.
Anorganic Chloride	mg/kg	6	<0.2	n.a.	n.a.
Colour	Pt/Co	12	3.2	3.7	7.0
Density @ 20°C	kg/L	15	0.7851	0.0004	0.0005
Specific Gravity @ 20/20°C		14	0.7865	0.0004	0.0005
Initial Boiling Point	°C	13	82.2	0.3	1.3
50% evaporated	°C	13	82.3	0.3	0.6
Dry Point	°C	13	82.4	0.3	0.9
Nonvolatile Matter	mg/100mL	10	0.5	0.7	2.4
Water	%M/M	14	0.014	0.007	0.007
Purity	%M/M	9	99.955	0.028	unknown
Purity on dry basis	%M/M	13	99.969	0.017	unknown
Ethanol	mg/kg	6	<5	n.a.	n.a.
n-Propanol	mg/kg	10	298	72	57
n-Butanol	mg/kg	8	<5	n.a.	n.a.
Methylethylketone	mg/kg	7	<5	n.a.	n.a.
Other Impurities	mg/kg	6	16.4	16.1	9.7

Table 3: Reproducibilities for sample #13225

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2013 WITH THE PREVIOUS PTS.

	<i>November 2013</i>	<i>November 2011</i>	<i>November 2009</i>	<i>November 2007</i>
Number of reporting labs	16	13	15	15
Number of results reported	168	143	165	148
Statistical outliers	7	10	5	10
Percentage outliers	4.2%	7.0%	3.0%	6.8%

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:

Determination	<i>November 2013</i>	<i>November 2011</i>	<i>November 2009</i>	<i>November 2007</i>
Acidity as acetic acid	++	++	++	++
Anorganic Chlorides as Cl	n.e.	n.e.	-	++
Colour Pt/Co	++	++	++	++
Density @ 20°C	+	++	++	++
Specific Gravity 20/20 °C	+	++	++	++
Initial Boiling Point	++	++	++	++
50% evaporated	++	++	++	n.e.
Dry Point	++	++	++	++
Nonvolatile Matter	++	++	++	++
Water	+/-	--	++	++
Purity	(++)	(--)	(+)	(--)
Purity on dry basis	(++)	(++)	(-)	(+)
Ethanol	n.e.	-	(--)	n.e.
n-Propanol	-	+/-	+/-	+/-
n-Butanol	n.e.	n.e.	--	++
Methylethylketone	n.e.	--	--	++
Other impurities	--	+	n.e.	n.e.

Table 5: comparison determinations against the standard requirements

Results between brackets are compared with reproducibility of the previous round robin, due to the lack of target data.

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

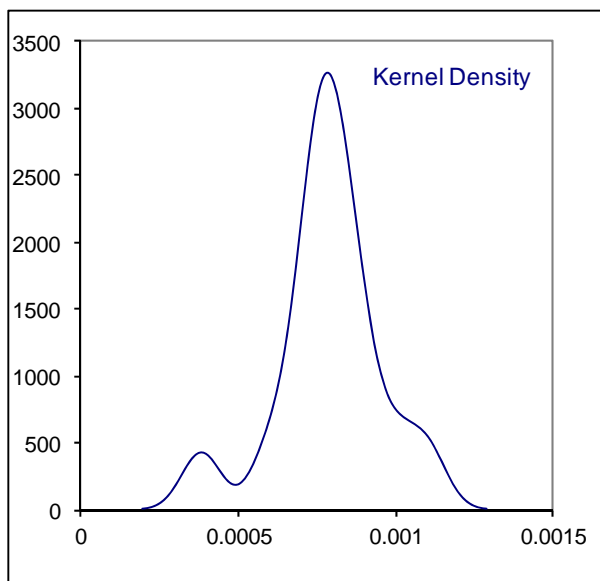
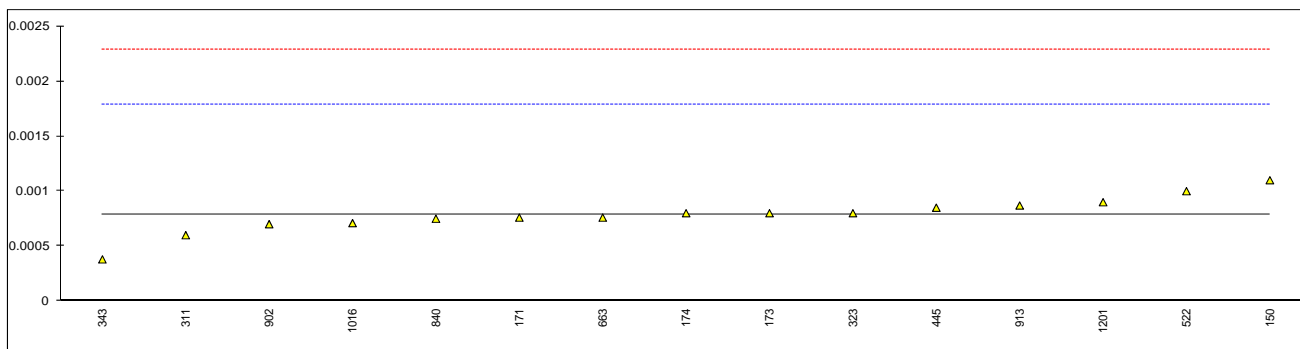
++:	group performed much better than the standard
+	group performed better than the standard
+/-:	group performance equals the standard
- :	group performed worse than the standard
-- :	group performed much worse than the standard
n.e.:	not evaluated

APPENDIX 1

Determination of Acidity as Acetic Acid on sample #13225; results in %M/M.

lab	method	value	mark	z(targ)	remarks
150	D1613	0.0011		0.63	
171	D1613	0.00076		-0.05	
173	D1613	0.0008		0.03	
174	D1613	0.0008		0.03	
311	D1613	0.0006		-0.37	
323	D1613	0.0008		0.03	
343	D1613	0.00038		-0.81	
445	D1613	0.00085		0.13	
522	D1613	0.001		0.43	
551		-----		-----	
663	D1613	0.00076		-0.05	
840	D1613	0.00075		-0.07	
902	D1613	0.0007		-0.17	
913	D1613	0.00087		0.17	
1016	D1613	0.00071		-0.15	
1201	D1613	0.0009		0.23	
1438		-----		-----	

normality OK
n 15
outliers 0
mean (n) 0.00079
st.dev. (n) 0.000166
R(calc.) 0.00046
R(D1613:12) 0.00140



Determination of Appearance on sample #13225;

lab	method	value	mark	z(targ)	remarks
150	E2680	Pass		----	
171	E2680	Pass		----	
173	E2680	Pass		----	
174	E2680	Pass		----	
311	E2680	Pass		----	
323	E2680	Pass		----	
343	E2680	Pass		----	
445	E2680	Pass		----	
522	INH-6	Pass		----	
551		----		----	
663	E2680	Pass		----	
840	E2680	Pass		----	
902	E2680	Pass		----	
913	E2680	Pass		----	
1016	in house	Pass		----	
1201	E2680	B&C		----	
1438		----		----	

C&B = Clear and Bright
CFFSM = Clear and free from Suspended Matter
SM = Suspended Matter

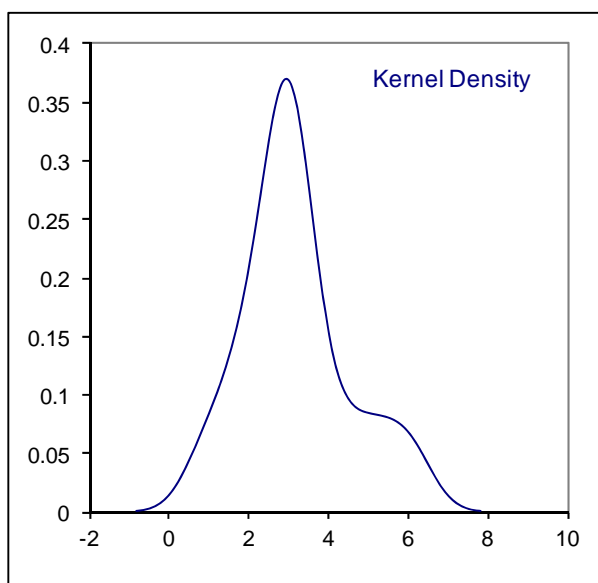
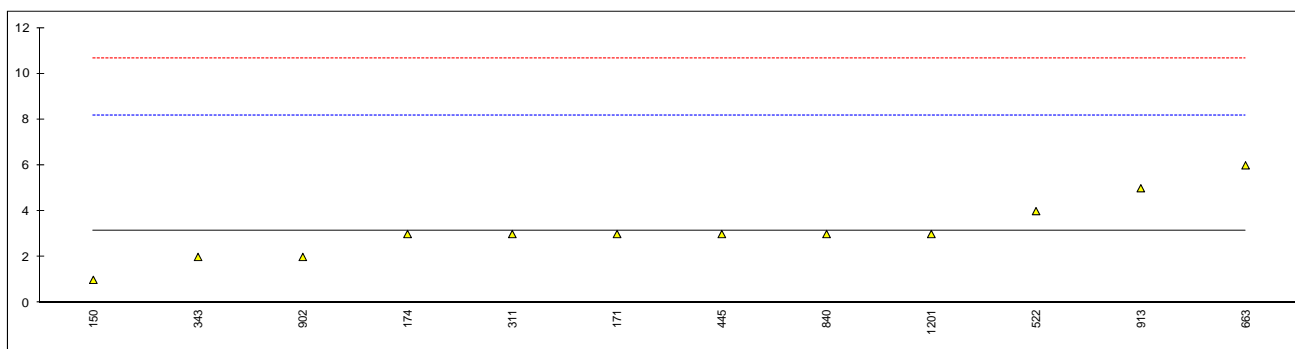
Determination of Anorganic Chloride as Cl on sample #13225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	D7359	<0.1		----	
171	D7359	0.107		----	
173	INH-221	<1		----	
174	E2369	0.08		----	
311	INH-158	<0.2		----	
323		----		----	
343		----		----	
445		----		----	
522		----		----	
551		----		----	
663	D5808	<1		----	
840	IMPCA002	0.12		----	
902		----		----	
913		----		----	
1016		----		----	
1201		<0.1		----	
1438		----		----	
	normality	n.a.			
	n	6			
	outliers	0			
	mean (n)	<0.2			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit.)	n.a.			

Determination of Colour Pt/Co scale on sample #13225;

lab	method	value	mark	z(targ)	remarks
150	D5386	1		-0.87	
171	D1209	3		-0.07	
173	D1209	<5		----	
174	D1209	3		-0.07	
311	D1209	3		-0.07	
323	D1209	<5		----	
343	D5386	2		-0.47	
445	D1209	3		-0.07	
522	D1209	4		0.33	
551		----		----	
663	D1209	6		1.13	
840	D1209	3		-0.07	
902	D5386	2		-0.47	
913	D5386	5		0.73	
1016		----		----	
1201	D1209	3		-0.07	
1438		----		----	

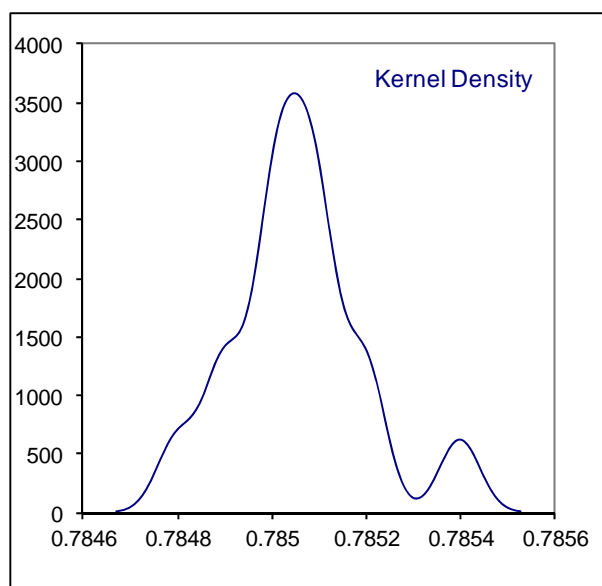
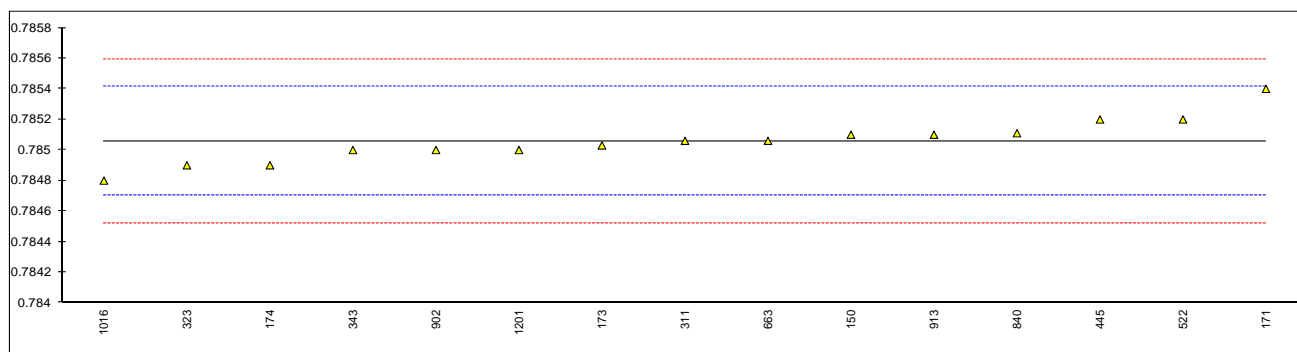
normality not OK
n 12
outliers 0
mean (n) 3.17
st.dev. (n) 1.337
R(calc.) 3.74
R(D1209:11) 7.00



Determination of Density 20 °C on sample #13225; results in kg/L.

lab	method	value	mark	z(targ)	remarks
150	D4052	0.7851		0.24	
171	D4052	0.7854		1.92	
173	D4052	0.78503		-0.15	
174	D4052	0.7849		-0.88	
311	D4052	0.78506		0.02	
323	D4052	0.7849		-0.88	
343	D4052	0.7850		-0.32	
445	D4052	0.7852		0.80	
522	D4052	0.7852		0.80	
551		-----		-----	
663	D4052	0.78506		0.02	
840	D4052	0.78511		0.30	
902	D4052	0.7850		-0.32	
913	D4052	0.7851		0.24	
1016	D4052	0.7848		-1.44	
1201	D4052	0.7850		-0.32	
1438		-----		-----	

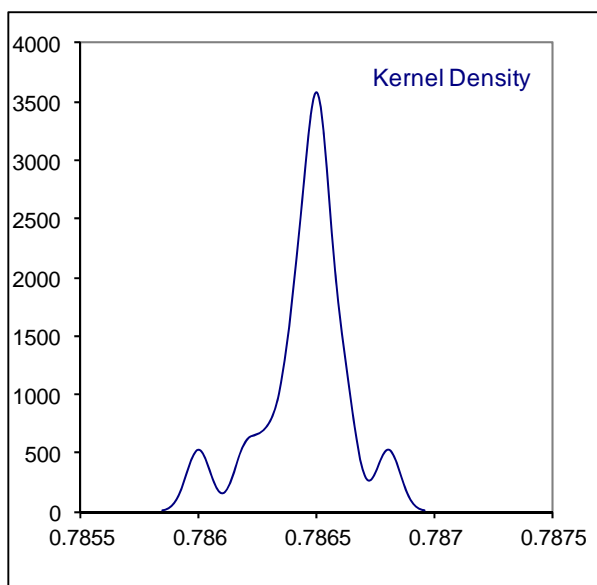
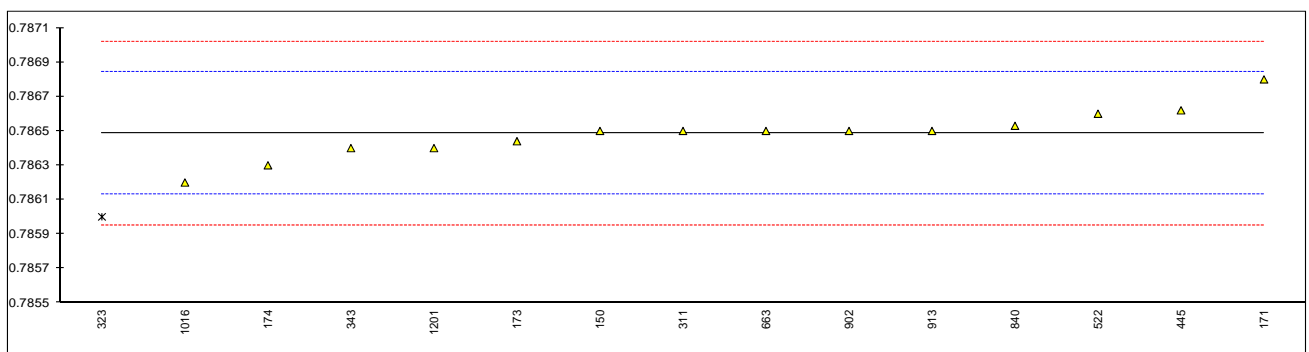
normality OK
n 15
outliers 0
mean (n) 0.78506
st.dev. (n) 0.000144
R(calc.) 0.00040
R(ISO12185:96) 0.00050



Determination of Specific Gravity 20/20°C on sample #13225;

lab	method	value	mark	z(targ)	remarks
150	D4052	0.7865		0.08	
171	D4052	0.7868		1.76	
173	D4052	0.78644		-0.25	
174	D4052	0.7863		-1.04	
311	D4052	0.7865		0.08	
323	D4052	0.7860	G(0.05)	-2.72	
343		0.7864		-0.48	
445		0.78662		0.76	
522	D4052	0.7866		0.64	
551		-----		-----	
663	D4052	0.7865		0.08	
840	D4052	0.78653		0.25	
902	D4052	0.7865		0.08	
913	D4052	0.7865		0.08	
1016		0.7862		-1.60	
1201		0.7864		-0.48	
1438		-----		-----	

normality OK
n 14
outliers 1
mean (n) 0.78649
st.dev. (n) 0.000143
R(calc.) 0.00040
R(ISO12185:96) 0.00050



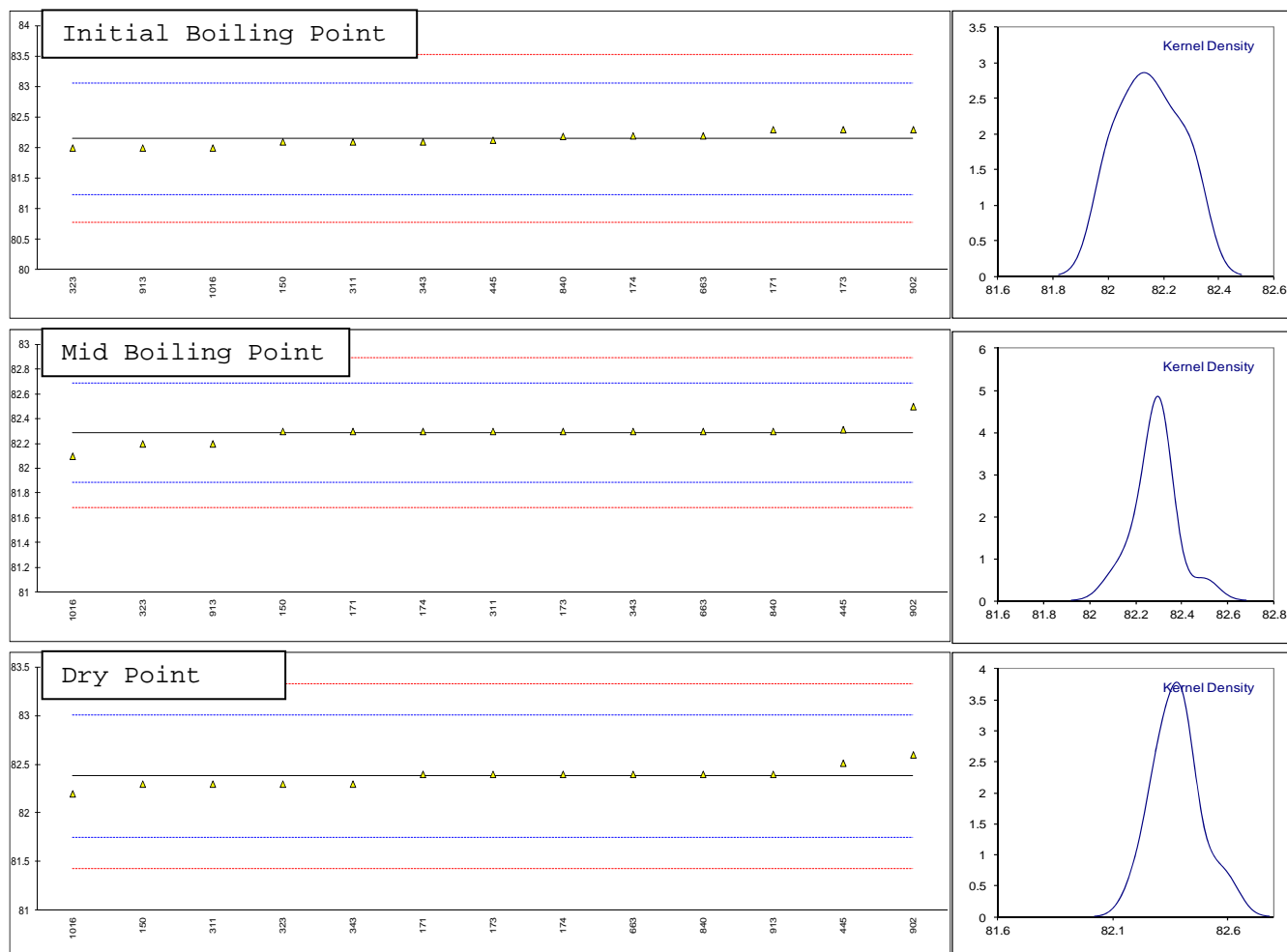
Determination of Distillation @ 760 mmHg on sample #13225; results in °C.

lab	method	IBP	mark	z(targ)	MBP	mark	z(targ)	DP	mark	z(targ)
150	D1078-A	82.1		-0.10	82.3		0.07	82.3		-0.25
171	D1078-A	82.3		0.33	82.3		0.07	82.4		0.07
173	D1078-A	82.3		0.33	82.3		0.07	82.4		0.07
174	D1078-A	82.2		0.11	82.3		0.07	82.4		0.07
311	D1078-A	82.1		-0.10	82.3		0.07	82.3		-0.25
323	D1078-M	82.0		-0.32	82.2		-0.43	82.3		-0.25
343	D1078-A	82.1	C	-0.10	82.3		0.07	82.3		-0.25
445	D1078-M	82.128		-0.04	82.314		0.14	82.514		0.43
522		----		----	----		----	----		----
551		----		----	----		----	----		----
663	D1078-A	82.2		0.11	82.3		0.07	82.4		0.07
840	D1078-A	82.19		0.09	82.30		0.07	82.40		0.07
902	D1078-M	82.3	n.c.	0.33	82.5	n.c.	1.07	82.6	n.c.	0.71
913	D1078-M	82.0		-0.32	82.2		-0.43	82.4		0.07
1016	D1078	82.0	n.c.	-0.32	82.1	n.c.	-0.93	82.2	n.c.	-0.57
1201		----		----	----		----	----		----
1438		----		----	----		----	----		----
	normality	OK			not OK			not OK		
	n	13			13			13		
	outliers	0			0			0		
	mean (n)	82.15			82.29			82.38		
	st.dev. (n)	0.112			0.090			0.103		
	R(calc.)	0.31			0.25			0.29		
	R(D1078:11-A)	1.28			0.56			0.88		
	R(D1078:11-M)	0.88			0.53			1.07		

Lab 343: first reported 81.2

Lab 902: n.c.: results probably not corrected for barometer pressure, see also §4.1

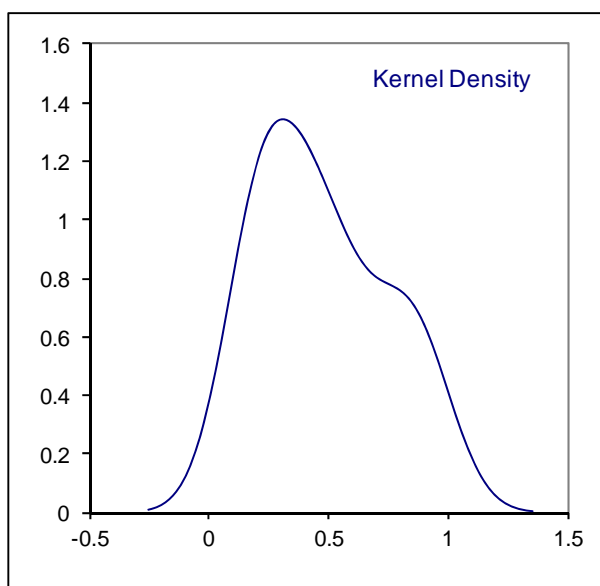
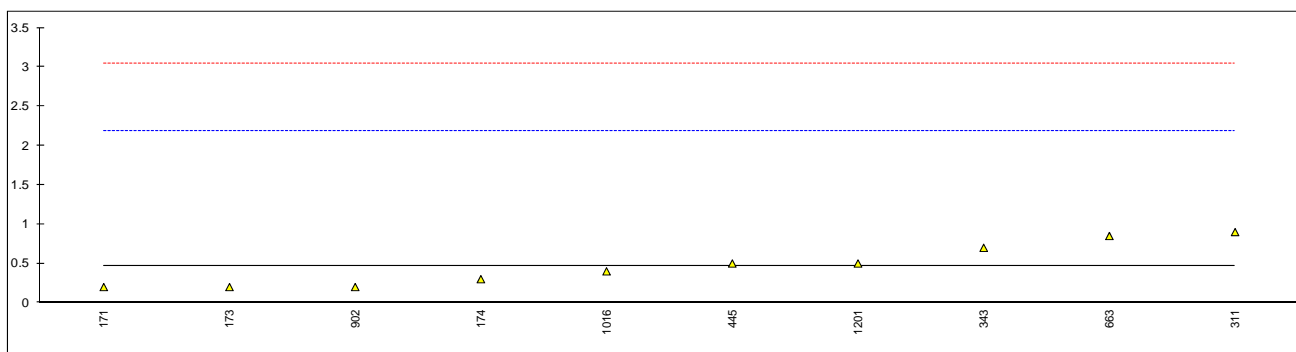
Lab 1016: n.c.: results probably not corrected for barometer pressure, see also §4.1.



Determination of Nonvolatile Matter on sample #13225; results in mg/100 mL.

lab	method	value	mark	z(targ)	remarks
150	D1353	<0.1		-----	
171	D1353	0.2		-0.32	
173	D1353	0.2		-0.32	
174	D1353	0.3		-0.20	
311	D1353	0.9		0.50	
323	D1353	<1.0		-----	
343	D1353	0.7	C	0.26	First reported 2.2
445	D1353	0.50		0.03	
522		-----		-----	
551		-----		-----	
663	D1353	0.85		0.44	
840	D1353	<1		-----	
902	D1353	0.2		-0.32	
913		-----		-----	
1016	D1353	0.4		-0.09	
1201	D1353	0.5		0.03	
1438		-----		-----	

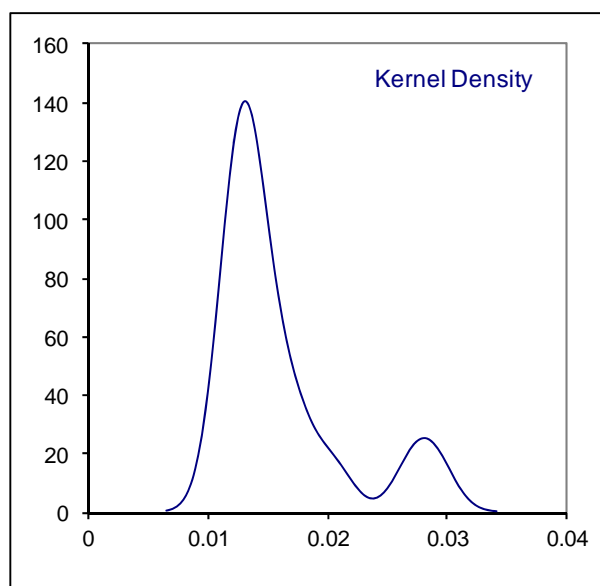
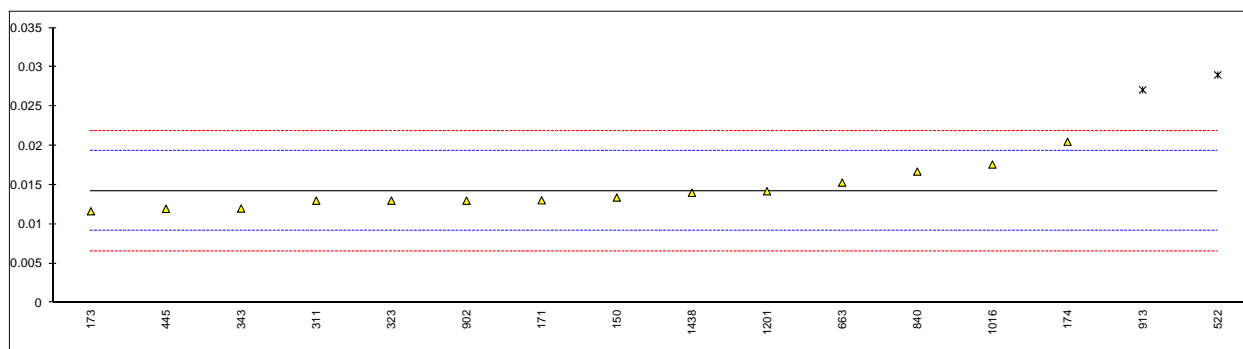
normality OK
n 10
outliers 0
mean (n) 0.48
st.dev. (n) 0.266
R(calc.) 0.74
R(D1353:09) 2.40



Determination of Water content on sample #13225; results in %M/M.

lab	method	value	mark	z(targ)	remarks
150	E1064	0.0134		-0.33	
171	D1364	0.01305		-0.47	
173	D1364	0.01165		-1.01	
174	D1364	0.0205	C	2.45	First reported 0.0285
311	D1364	0.013		-0.48	
323	D1364	0.013		-0.48	
343	D1364	0.012		-0.88	
445	E1064	0.01196		-0.89	
522	E203	0.029	DG(0.01)	5.77	
551		-----		-----	
663	D1364	0.0153		0.41	
840	E1064	0.0167		0.96	
902	D1364	0.013		-0.48	
913	D1364	0.0271	DG(0.01)	5.03	
1016	D1364	0.0176		1.31	
1201	D1364	0.0142		-0.02	
1438	D1364	0.014		-0.09	

normality OK
n 14
outliers 2
mean (n) 0.0142
st.dev. (n) 0.00250
R(calc.) 0.0070
R(D1364:12) 0.0072

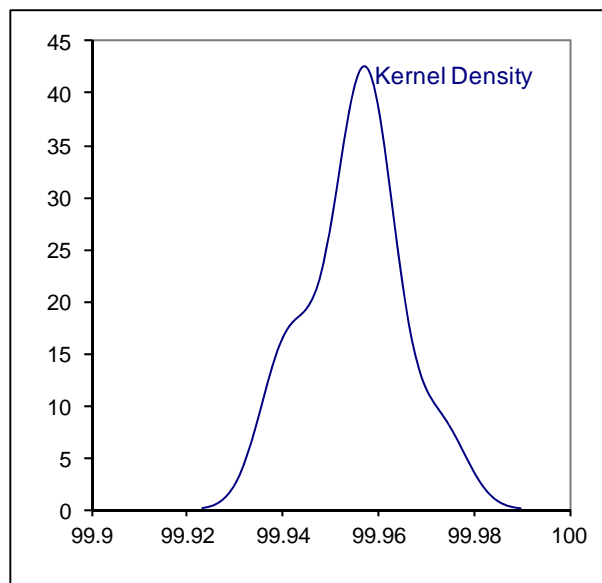
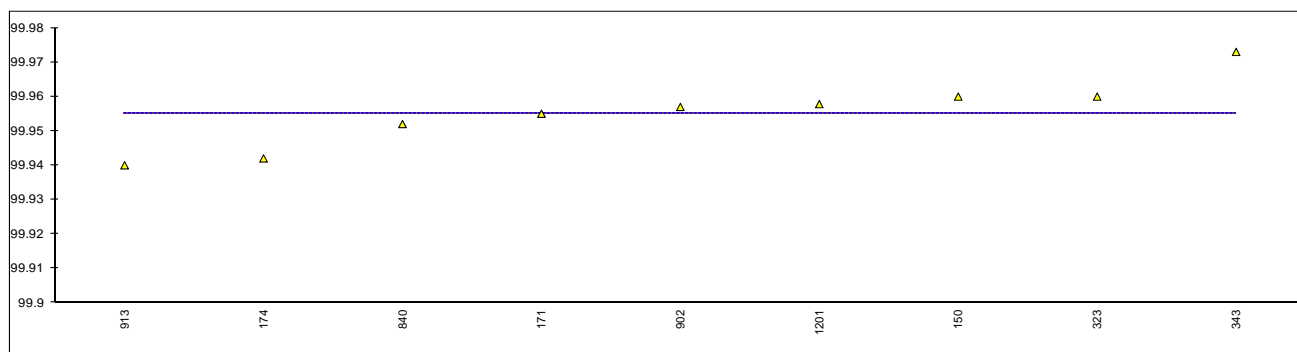


Determination of Purity “as received” on sample #13225; results in %M/M.

lab	method	value	mark	z(targ)	remarks
150	DIN55635	99.96		----	
171	INH-IA	99.955		----	
173		----		----	
174	INH-582	99.942		----	
311		----		----	
323	INH-060	99.96		----	
343	DIN55685	99.973		----	
445		----		----	
522		----		----	
551		----		----	
663		----		----	
840	INH-015	99.952		----	
902	INH-129	99.957		----	
913	D5501	99.94		----	
1016		----		----	
1201		99.9578		----	
1438		----		----	

normality OK
n 9
outliers 0
mean (n) 99.9552
st.dev. (n) 0.00993
R(calc.) 0.0278
R(lit.) unknown

Compare R(iis11C14) = 0.0868

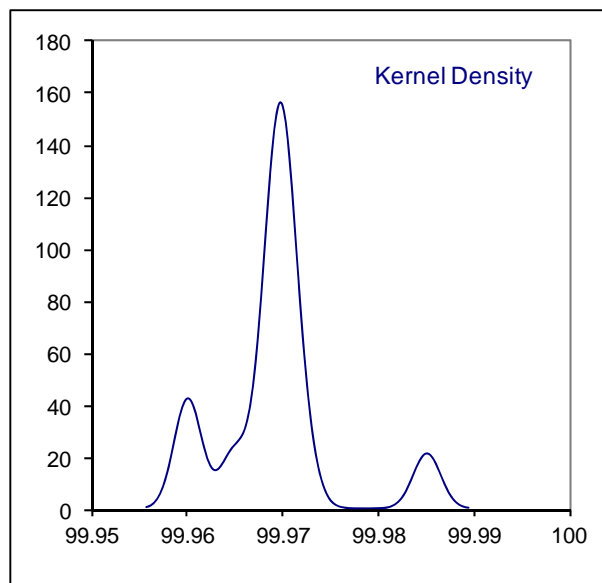
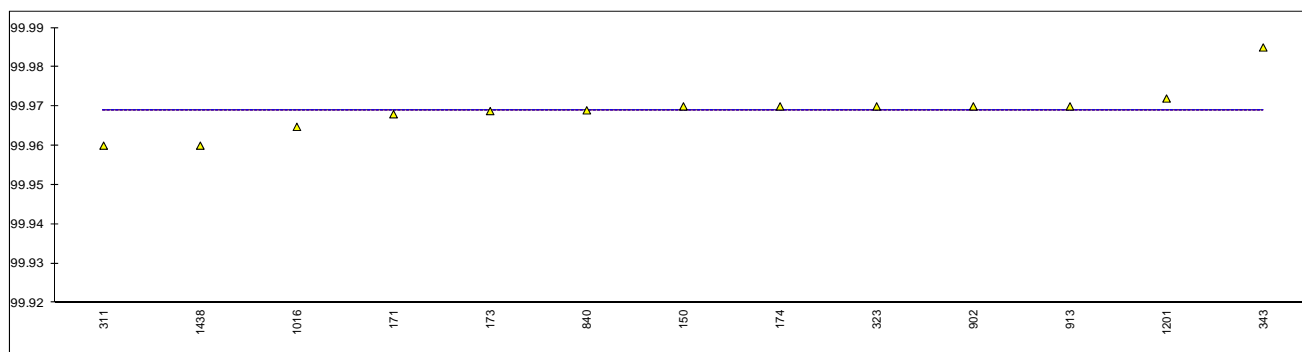


Determination of Purity on dry basis on sample #13225, results in %M/M.

lab	method	value	mark	z(targ)	remarks
150	DIN55635	99.97		----	
171	INH-IA	99.968		----	
173	INH-6012	99.9688		----	
174	INH-582	99.970		----	
311		99.96		----	
323	INH-060	99.97		----	
343	DIN55685	99.985		----	
445		----		----	
522		----		----	
551		----		----	
663		----		----	
840	INH-015	99.969		----	
902	INH-129	99.97		----	
913	D5501	99.97		----	
1016		99.9648		----	
1201		99.972		----	
1438	in house	99.96		----	

normality not OK
n 13
outliers 0
mean (n) 99.9690
st.dev. (n) 0.00613
R(calc.) 0.0172
R(lit.) unknown

Compare R(iis11C14) = 0.0250



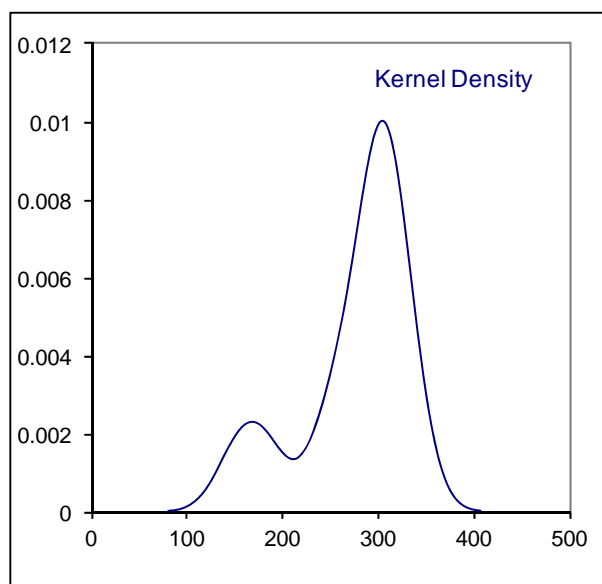
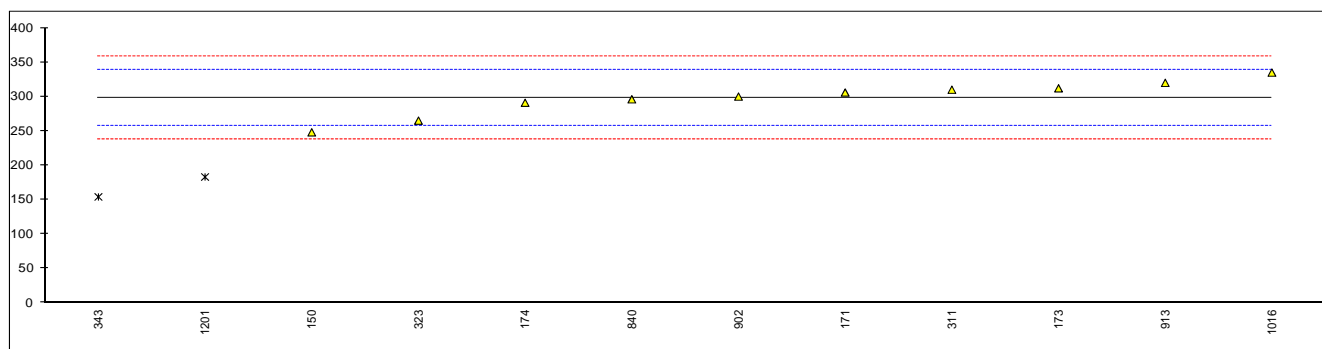
Determination of Ethanol content on sample #13225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	DIN55635	<5		-----	
171	INH-IA	<1		-----	
173	INH-6012	n.d.		-----	
174	INH-582	<10		-----	
311		<5		-----	
323	INH-060	<10		-----	
343	DIN55685	<10	C	-----	First reported 5.6
445		-----		-----	
522		-----		-----	
551		-----		-----	
663		-----		-----	
840	INH-015	<5		-----	
902		-----		-----	
913	D5501	<5		-----	
1016		-----		-----	
1201		<5		-----	
1438		-----		-----	
	normality	n.a.			
	n	6			
	outliers	n.a.			
	mean (n)	<5			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

Determination of n-Propanol on sample #13225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	DIN55635	248		-2.48	
171	INH-IA	305.9		0.38	
173	INH-6012	312		0.68	
174	INH-582	291		-0.36	
311		310		0.58	
323	INH-060	265		-1.64	
343	DIN55685	154	C,DG(0.01)	-7.13	First reported 134
445		----		----	
522		----		----	
551		----		----	
663		----		----	
840	INH-015	296		-0.11	
902	INH-129	300		0.08	
913	D5501	320		1.07	
1016		334.9		1.81	
1201		183	DG(0.01)	-5.69	
1438		----		----	

normality OK
n 10
outliers 2
mean (n) 298.28
st.dev. (n) 25.589
R(calc.) 71.65
R(Horwitz) 56.68



Determination of n-Butanol on sample #13225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	DIN55635	<5		----	
171	INH-IA	<1		----	
173	INH-6012	n.d.		----	
174	INH-582	<10		----	
311		<5		----	
323	INH-060	<5		----	
343	DIN55685	<10		----	
445		----		----	
522		----		----	
551		----		----	
663		----		----	
840	INH-015	<5		----	
902		----		----	
913	D5501	<5		----	
1016		0.4		----	
1201		<5		----	
1438		----		----	
	normality	n.a.			
	n	8			
	outliers	n.a.			
	mean (n)	<5			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

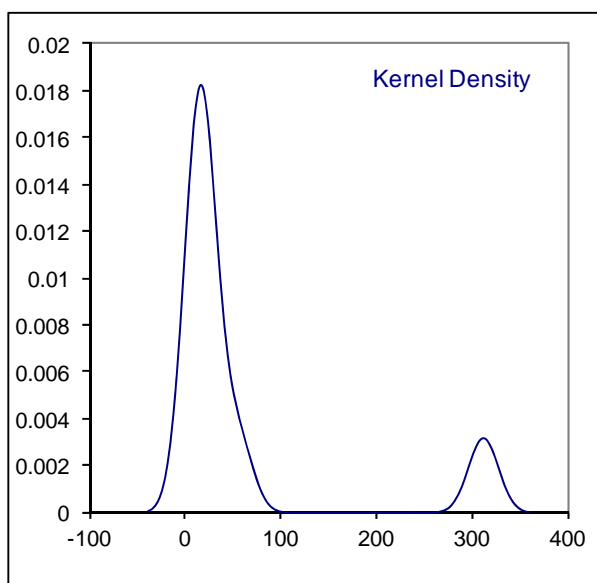
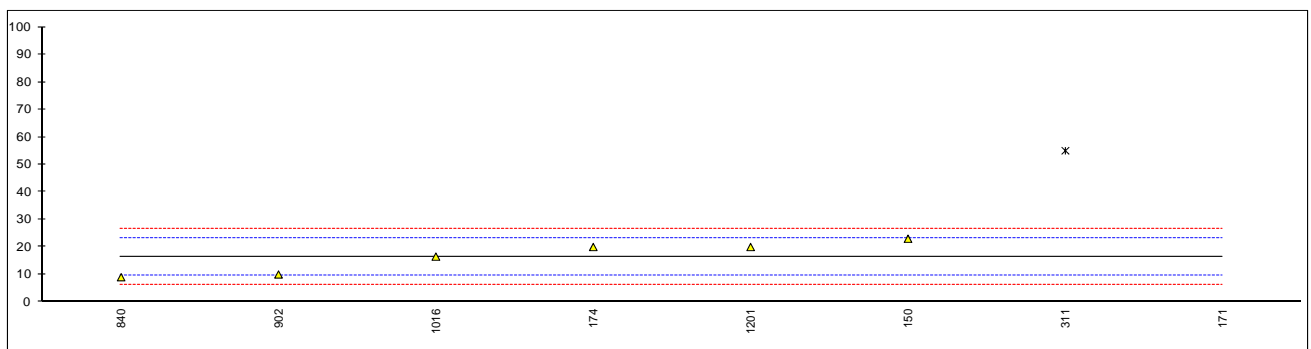
Determination of Methylketone on sample #13225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	DIN55635	<5		----	
171	INH-IA	<1		----	
173	INH-6012	n.d.		----	
174	INH-582	<10		----	
311		<5		----	
323	INH-060	<5		----	
343	DIN55685	<10		----	
445		----		----	
522		----		----	
551		----		----	
663		----		----	
840	INH-015	<5		----	
902	INH-029	<10		----	
913		----		----	
1016		<5		----	
1201		<5		----	
1438		----		----	
	normality	n.a.			
	n	7			
	outliers	n.a.			
	mean (n)	<5			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(Horwitz)	n.a.			

Determination of Other Impurities on sample #13225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
150	DIN55635	23		1.91	
171	INH-IA	312	G(0.01)	85.74	
173		----		----	
174	INH-582	20		1.04	
311		55	G(0.05)	11.19	
323		----		----	
343		----		----	
445		----		----	
522		----		----	
551		----		----	
663		----		----	
840	INH-015	9		-2.15	
902	INH-129	10		-1.86	
913		----		----	
1016		16.5		0.02	
1201		20		1.04	
1438		----		----	

normality OK
n 6
outliers 2
mean (n) 16.42
st.dev. (n) 5.748
R(calc.) 16.09
R(Horwitz) 9.65



APPENDIX 2

Number of participants per country

1 laboratory in BELGIUM
1 laboratory in BRAZIL
1 laboratory in INDIA
1 laboratory in ISRAEL
1 laboratory in MEXICO
1 laboratory in SPAIN
1 laboratory in THAILAND
3 laboratories in THE NETHERLANDS
1 laboratory in TURKEY
4 laboratories in U.S.A.
1 laboratory in UNITED KINGDOM
1 laboratory in VIETNAM

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect result
U	= reported in wrong unit
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 statistical calculations
E	= error in calculations
n.a.	= not applicable
W	= withdrawn on request participant
SDS	= Safety Data Sheet

Literature:

- 1 i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
- 2 ASTM E178-02
- 3 ASTM E1301-03
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- 6 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 7 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 8 IP 367/84
- 9 DIN 38402 T41/42
- 10 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 11 J.N. Miller, Analyst, 118, 455, (1993)
- 12 Analytical Methods Committee Technical Brief, No4 January 2001
- 13 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/>)