Results of Proficiency Test
Methanol
September 2004

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 1996, a proficiency test for Methanol was organised every year by The Institute for Interlaboratory Studies. During the annual proficiency testing program 2003/2004, it was decided to continue the round robin for the analysis of Methanol in accordance with the latest applicable version of the IMPCA specification and ASTM E346:03. In this international interlaboratory study 40 laboratories in 20 different countries have participated. See appendix 2 for a list of participants in alphabetical country order.
In this report the results of the proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory studies (i.i.s.) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. To get maximum information from this study it was decided to send 2 samples (1* 1000 mL, labelled 0450 and 1 * 500 mL, labelled 0451). Sample 0451 was spiked with sodium chloride (approx. 1.6 mg Cl/kg) and ethanol (18.7 mg/kg). Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluations.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO guide 43 and ILAC-G13:2000, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie)

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report ‘i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of November 2003 (iis-protocol, version 3.0).

2.3 SAMPLES

The necessary bulk material was purchased from a local trader. The approx. 75 litre bulk sample was homogenised in a pre-cleaned metal drum.
After homogenisation, 46 subsamples were transferred to 1000 mL brown glass bottles and labelled 0450. The remaining bulk material of 18.0 kg was spiked with 45.9 mg NaCl (Merck 1,06406,0050) and with 336.9 mg Ethanol; thus fortifying the Methanol with 1.55 mg Cl/kg and 18.7 mg Ethanol/kg. After homogenisation, 46 subsamples were transferred to 500 mL brown glass bottles and labelled 0451.

The homogeneity of subsample 0450 was checked by determination of Density, in accordance with ASTM D4052:02 and Water, in accordance with ASTM E203:01 on 5 stratified random selected samples. The homogeneity of subsample 0451 was checked by

determination of the Chloride content in accordance with IMPCA 002:1998 on 4 stratified random selected samples.

	Density at 20ºC in kg/L	*Water content in mg/kg*
sample 0450-1	0.7919	2400
sample 0450-2	0.7919	2420
sample 0450-3	0.7919	2440
sample 0450-4	0.7919	2400
sample 0450-5	0.7919	2450

table 1: homogeneity test of subsample 0450

	Chloride in mg/kg
sample 0451-1	1.5
sample 0451-2	1.5
sample 0451-3	1.5
sample 0451-4	1.5

table 2: homogeneity test of subsample 0451

From the results in table 1 and 2, the repeatability of the results were calculated by multiplication of the standard deviations by 2.8:

	Density at 15ºC in kg/L	*Water In %M/M*
r (sample 0450)	0.0000	0.0063
reference test	ASTM D4052:02	ASTM E203:01
r (reference test)	0.0001	0.0080

table 3: repeatabilities of subsamples 0450

	Chloride in mg/kg
r (sample 0451)	0.00
reference test	IMPC002:98
r (reference test)	0.01

table 4: repeatability of subsamples 0451

The repeatabilities of Density and Water for sample 0450 and the repeatability of Chloride for sample 0451 were in agreement with the respective methods. Therefore homogeneity of the subsamples was assumed.

Two samples of Methanol (1000 mL of sample 0450, 500 mL of sample 0451) were sent to the participating laboratories on August 25, 2004.

2.4 STABILITY OF THE SAMPLES

In order to be sure that the material, which was used in this proficiency test, was stable for the valid period, the stability of the material, packed in the brown glass bottles, has been checked prior to use.

2.5 ANALYSES

The participants were asked to determine Acidity, Anorganic Chloride, Appearance, Carbonisable substances Pt/Co, Density @ 20 °C, Distillation (IBP & DP), Acetone, Ethanol, Water Miscibility, Nonvolatile Matter, Purity (on dry basis), Permanganate Time Test, Specific Gravity 20°/20° C/C, Apparent Specific Gravity 20°/20°, Sulphur, Total Iron, Trimethylamine and Water (coulometric and titrimetric) on sample 0450. On sample 0451 participants were asked to determine Anorganic Chloride and Ethanol only.

To get comparable results, a detailed report form on which the units and the standard methods were printed, was sent together with each set of samples. Also a letter of instructions and a MSDS 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 ‘i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of November 2003 (iis-protocol, version 3.0).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<…' or '>…' were not used in the statistical evaluation.

First the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. After removal of outliers this check was repeated.

In case a data set does not have a normal distribution, the (results of the) statistical evaluation 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 and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

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

3.2 GRAPHICS

In order to visualise the results against the reproducibilities from literature, Gauss plots were made, using the sorted data for each determination (see appendix 1). On the Y-axis the analytical results are plotted. The corresponding laboratory numbers are under the X-axis. A straight line presents the average of the reported data. Two striped lines present the reproducibility limits of the selected standard, calculated as mean ± target reproducibility, parallel to the average line. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. 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:

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

As it was decided to evaluate the performance of the participants against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated according to:

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

4 EVALUATION

In this proficiency test some problems were encountered with despatch of the samples to Malaysia, Russia and Venezuela. The Russian laboratory didn't have an import license for Methanol and the samples were held by the Russian customs. Finally 17 participants reported after the deadline and 2 participants did not report any result at all.
Of the total of 40 participants, 38 did send in 481 numerical results. Observed were 18 outlying results, which is 3.7 % of the total of numerical results. 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.
Not normal distributions were found with the following determinations: Carbonisable Substances, Colour Pt/Co, Density @ 20 ^{o}C, IBP (automatic and manual), Ethanol #0450, Nonvolatile Matter, Permanganate Time Test, Purity on dry basis and Specific Gravity. In these cases the statistical evaluation should be used with due care.

Acidity: No analytical problems were observed. Only one result was outside the reproducibility limits and the observed reproducibility is, after rejection of the statistical outliers, in good agreement with the requirements of ASTM D1613:03.

Anorg. Chloride: This determination is problematic for some laboratories. For sample 0450 no results were outside the reproducibility limits and the calculated reproducibility is in agreement with IMPCA002:98. For the spiked sample 0451 five results were outside the reproducibility limits. The calculated reproducibility is, after rejection of the statistical outliers, not in agreement with the requirements of IMPCA002:98. The average recovery of the chloride (calculated from the difference between the original sample 0450 and the spiked sample 0451) and the theoretical increment of 1.55 mg Cl/kg is acceptable (80%). The individual recoveries vary over a large range. The same was found in previous proficiency tests. Experience has shown that very low levels of chloride are very difficult to determine with accuracy by means of turbidimetry. The best option for anorganic chloride with the highest accuracy is the IMPCA method using dedicated titration equipment.

Appearance: No analytical problems were observed. All labs, except one (1016), agreed about the appearance of the sample 0450.

Carb. subst.: This determination is not problematic. Only one result was outside the reproducibility limits and the observed reproducibility is, after recalculation of the "less then" results, in agreement with the requirements of ASTM E346:03.

Colour: No analytical problems were observed. No results were found outside the reproducibility limits. The observed reproducibility is in good agreement with the requirements of ASTM D1209:00.

Density @ 20°C: No analytical problems were observed. No results were outside the reproducibility limits. The observed reproducibility is, after rejection of the statistical outlier, in good agreement with the requirements of ASTM D4052:02.

Distillation: The automatic determination is only problematic for DP. In total two results were outside the reproducibility limits. The calculated reproducibility for IBP is in good agreement and for DP is not in agreement with the requirements of ASTM D1078:03 (automatic). The manual determination is not problematic. After rejection of two manual results (both results were excluded for IBP, as the IBP cannot be higher than the true boiling point) the calculated reproducibilities for IBP and DP as well are both in agreement with the requirements of ASTM D1078:03 (manual). The true boiling point for methanol is 64.5 °C (see table 3 of ASTM D1078:03), however also 64.6 °C is sometimes used as true boiling point.

Water Miscibility: No analytical problems were observed (former named as Hydrocarbons).

NVM: No analytical problems were observed. No results were outside the reproducibility limits. The reproducibility is in good agreement with the requirements of ASTM D1353:03.

PTT: No analytical problems were observed. Only one result was outside the reproducibility limits. The calculated reproducibility is, after rejection of the statistical outlier, in agreement with the requirements of ASTM D1363:01.

SG 20°C/20°C: No analytical problems were observed. Only two results were outside the reproducibility limits and the precision is, after rejection of the statistical outliers, in agreement with the requirements of ASTM D4052:02 and ASTM D891:00.
The Specific Gravity is defined as: "*the ratio of the weight in Vacuum of an unit volume of a material at stated temperature to the weight in Vacuum of an equal volume of gas-free distilled water at a stated temperature".*
SG 20˚/20˚C = (density material at 20˚ in vacuum) / (density water at 20˚ in vacuum).

ASG 20°C/20°C: Major analytical problems were observed. Seven laboratories found a significant lower result then the other reporting laboratories, probably due to a calculation error. Another eight results may be SG results and not ASG results. However, as calculations were not reported, it is not possible to check all calculations.
The Apparent Specific Gravity is defined as: "*the ratio of the weight in air of a unit volume of a material at stated temperature to the weight in air of equal density of an equal volume of gas-free distilled water at a stated temperature".*
SG Apparent 20˚/20˚C = (density material at 20˚in vacuum – 0.0012) / (density water at 20˚ in vacuum - 0.0012).

SG General: When the Specific Gravities and Apparent Specific Gravities were calculated from the reported densities, it was noticed that the reported results for the Specific Gravity 20˚/20˚C are in line with the calculated results for the majority of the group. However, when the reported results for the Apparent Specific Gravity 20˚/20˚C are compared with the calculated results, it is noticed that only three participants (311, 323 and 657) reported ASG results that are in full agreement with the respective reported SG and Density results.

Sulphur: Although many participants reported "less than" results the group of participants agreed about the sulphur content being less than 0.5 mg/kg. Unfortunately, in ASTM D3961:98 no reproducibility is mentioned. However, when the "less then" results are recalculated and the precision data of ASTM D5453 are used for comparison, four results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outlier, not in agreement with the requirements of ASTM D5453:03. Many different methods were used. Some methods are not applicable at this low concentration level.

Total Iron: This determination is rather problematic. Six results were outside the reproducibility limits. The calculated reproducibility is, after rejection of the statistical outliers, not in agreement with the reproducibility of ASTM E394:00.

Water (coul.): No analytical problems were observed. No results were outside the reproducibility limits. The calculated reproducibility is in agreement with the requirements of ASTM E1064:00.

Water (titr.): No analytical problems were observed. Only one result was outside the reproducibility limits. The calculated reproducibility is, after rejection of the statistical outlier, in agreement with the requirements of ASTM E203:01 and ASTM D1364:02.

Acetone: Many participants reported a “less then” result. Furthermore, there are precision data available. Therefore no significant conclusion can be drawn.

Ethanol: For Sample 0450, one result was outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outlier, in agreement with the requirements of ASTM E346:03. For the spiked sample 0451 no results were outside the reproducibility limits. The calculated reproducibility is again in agreement with the requirements of E346:03. The average recovery of the Ethanol (calculated from the difference between the original sample 0450 and the spiked sample 0451) and the theoretical increment of 18.7 mg Ethanol/kg is acceptable (85%). The individual recoveries vary over a large range.

Purity: Unfortunately, for this determination no precision data are available, therefore no significant conclusions can be drawn.

Purity on dry basis: Unfortunately, for this determination no precision data are available, therefore no significant conclusions can be drawn.

TMA: Only four participants reported a numerical result for this determination, therefore no meaningful conclusions could be drawn.

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 per sample, 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	34	11.13	5.91	14.00
Chloride as Cl on sample 0450	mg/kg	23	0.30	0.24	0.30
Chloride as Cl on sample 0451	mg/kg	23	1.54	0.45	0.30
Carbonisable substances	Pt/Co	25	3.70	4.04	5.00
Colour	Pt/Co	20	2.6	3.5	6.0
Density @ 20 °C	kg/L	33	0.79188	0.00029	0.00050
Initial Boiling Point (automatic)	°C	20	64.45	0.30	1.01
Dry Point (automatic)	°C	20	66.25	1.26	0.71
Initinal Boiling Point (manual)	°C	15	64.37	0.38	0.69
Dry Point (manual)	°C	12	66.09	0.87	0.86
Nonvolatile Matter	mg/100 mL	24	0.49	1.11	2.40
Permanganate Time Test	minutes	32	82.8	15.9	20.9
Specific Gravity 20°/20° C/C		18	0.79327	0.00036	0.00050
Apparent Specific Gravity 20°/20°		23	0.79290	0.00151	0.00050
Sulphur	mg/kg	7	0.22	0.37	0.19
Total Iron	mg/kg	10	0.0099	0.0076	0.0053
Water (coulometric)	mg/kg	28	2395.5	320.8	409.6
Water (titrimetric)	mg/kg	21	2400.3	163.0	270.0
Acetone	mg/kg	8	1.4	4.3	Unknown
Ethanol on sample 0450	mg/kg	15	1.9	0.6	0.8
Ethanol on sample 0451	mg/kg	23	17.44	7.17	7.56
Purity	%M/M	21	99.7617	0.0383	Unknown
Purity on dry basis	%M/M	22	99.9950	0.0124	Unknown

table 6: Reproducibilities for sample 0450 and 0451

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2004 WITH PREVIOUS PT'S

	September 2004	*September 2003*	*September 2002*	*September 2001*
Number of reporting labs	38	38	32	35
Number of results reported	481	475	441	433
Statistical outliers	18	34	17	19
Percentage outliers	3.7	7.2	3.9	4.4

table 7: 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	*September 2004*	*September 2003*	*September 2002*	*September 2001*
Acidity as acetic acid	++	++	++	+
Chloride as Cl, sample 0450	++	++	-	++
Chloride as Cl, sample 0451	--	--	--	-
Carbonisable substances	++	++	++	++
Colour	++	++	+	n.e.
Density @ 20 °C	++	++	++	++
Initial Boiling Point	++	++	++	++
Dry Point (automatic)	--	++	++	++
Initial Boiling Point (manual)	++	++	++	n.e.
Dry Point (manual)	+/-	++	++	n.e.
Nonvolatile Matter	++	++	++	++
Permanganate Time Test	++	-	--	+
Specific Gravity 20°/20° C/C	++	+	+	+
Sulphur	--	--	--	--
Total Iron	-	--	--	-
Water (coulometric)	++	--	--	--
Water (titrimetric)	++	++	++	++
Ethanol	++	--	--	-

table 8: 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:

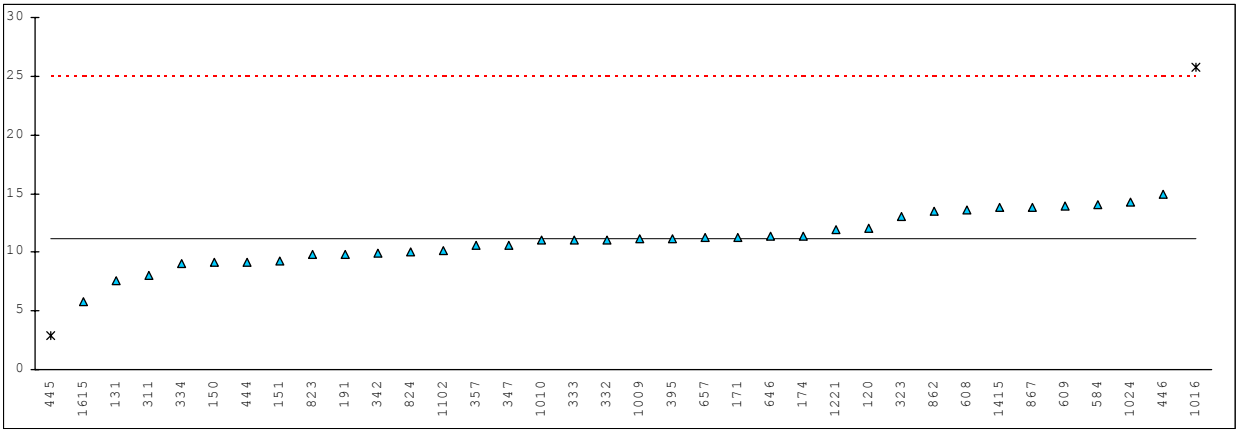
++: group performed much better than the standard
+ : group performed better than the standard
+/-: group performance equals the standard
- : group performed worse than the standard
-- : group performed much worse than the standard
n.e.: not evaluated

APPENDIX 1

Determination of Acidity on sample 0450; results in mg/kg

Lab	Method	value	mark	Z(targ)	remarks
120	D1613	12.1		0.19	
131	D1613	7.58		-0.71	
150	D1613	9.1		-0.41	
151	D1613	9.3		-0.37	
171	D1613	11.3		0.03	
174	D1613	11.4		0.05	
191	D1613	9.8		-0.27	
311	D1613	8.0		-0.63	
323	D1613	13	C	0.37	First reported 19
332	D1613	11		-0.03	
333	D1613	11		-0.03	
334	D1613	9		-0.43	
342	D1613	9.9		-0.25	
347	D1613	10.6		-0.11	
357	D1613	10.6		-0.11	
395	D1613	11.2		0.01	
444	D1613	9.1		-0.41	
445	D1613	2.9	G(0.05)	-1.65	
446	D1613	15		0.77	
584	D1613	14		0.57	
608	D1613	13.61		0.50	
609	D1613	13.93		0.56	
646	D1613	11.35		0.04	
657	D1613	11.3		0.03	
750		-----		-----	
823	D1613	9.8	C	-0.27	First reported 4.4
824	D1613	10		-0.23	
862	D1613	13.51		0.48	
867	D1613	13.8		0.53	
1009	D1613	11.1		-0.01	
1010	D1613	11		-0.03	
1016	D1613	25.8	G(0.01)	2.93	
1024	D1613	14.3		0.63	
1029		-----		-----	
1035		-----		-----	
1102	D1613	10.2		-0.19	
1108		-----		-----	
1221	D1613	11.93		0.16	
1415	D1613	13.8		0.53	
1615	D1613	5.8		-1.07	

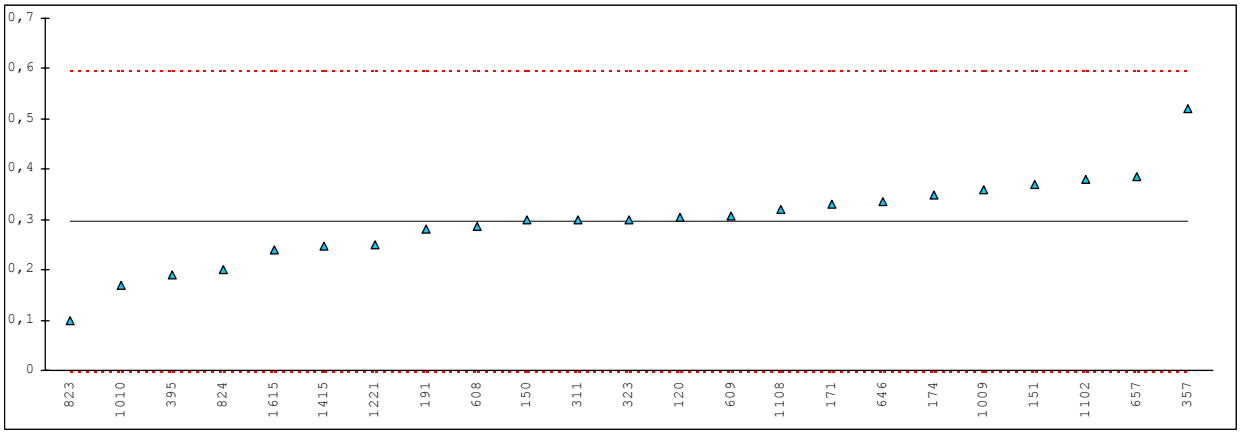
normality	OK
n	34
outliers	2
mean (n)	11.13
st.dev. (n)	2.112
R(calc.)	5.91
R(D1613:03)	14.00



Determination of (Anorganic) Chloride as Cl on sample 0450; results in mg/kg

lab	Method	value	mark	Z(targ)	remarks
120	IMPCA002	0.305		0.08	
131		-----		-----	
150	IMPCA002	0.3		0.03	
151	IMPCA002	0.37		0.68	
171	IMPCA002	0.33		0.31	
174	IMPCA002	0.35		0.50	
191	IMPCA002	0.28		-0.16	
311	IMPCA002	0.30		0.03	
323	In House	0.3		0.03	
332	Wickbold	<0.5		-----	
333	E291	<1		-----	
334		-----		-----	
342		-----		-----	
347		-----		-----	
357	IMPCA002	0.52		2.08	
395	In House	0.191		-0.99	
444	Turbidimetric	<0.5		-----	
445		-----		-----	
446		-----		-----	
584		-----		-----	
608	Turbidimetric	0.286		-0.10	
609	IMPCA002	0.307		0.10	
646	IMPCA002	0.335		0.36	
657	IMPCA002	0.384		0.82	
750		-----		-----	
823	Turbidimetric	0.1		-1.84	
824	Turbidimetric	0.2	C	-0.90	First reported 0.16
862	In house	<0.2		-----	
867	IMPCA002	<0.25		-----	
1009	In house	0.359		0.58	
1010	IMPCA002	0.17		-1.18	
1016		-----		-----	
1024		-----		-----	
1029		-----		-----	
1035		-----		-----	
1102	IMPCA002	0.38		0.78	
1108	IMPCA002	0.32		0.22	
1221	IMPCA002	0.25		-0.44	
1415	IMPCA002	0.246		-0.47	
1615	IMPCA002	0.24		-0.53	

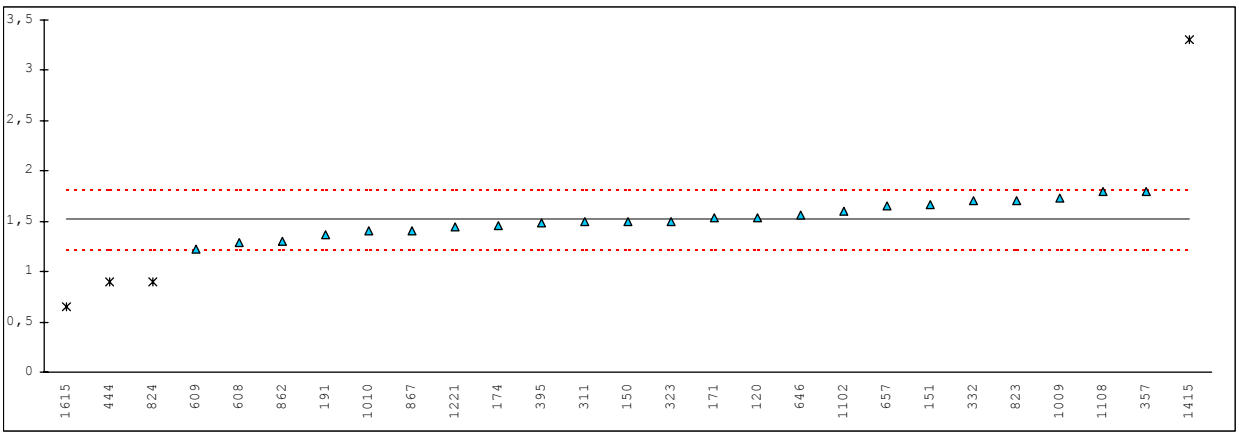
normality	OK
n	23
outliers	0
mean (n)	0.30
st.dev. (n)	0.086
R(calc.)	0.24
R(IMPCA002:98)	0.30



Determination of (Anorganic) Chloride as Cl on sample 0451; results in mg/kg

lab	method	value	mark	Z(targ)	Recovery (%)	remarks
120	In House	1.531		0.03	79	
131		-----		-----		
150	IMPCA002	1.5		-0.26	77	
151	IMPCA002	1.67		1.33	84	
171	IMPCA002	1.53		0.02	77	
174	IMPCA002	1.46		-0.63	72	
191	IMPCA002	1.37		-1.47	70	
311	IMPCA002	1.5		-0.26	77	
323	In House	1.5		-0.26	77	
332	Wickbold	1.7		1.61	90	
333	E291	<1		-----	<45	
334		-----		-----		
342		-----		-----		
347		-----		-----		
357	IMPCA002	1.80		2.54	83	
395	In House	1.486		-0.39	84	
444	Turbidimetric	0.9	DG(0.01)	-5.86	39	
445		-----		-----		
446		-----		-----		
584		-----		-----		
608	IMPCA002	1.286		-2.26	65	
609	IMPCA002	1.222		-2.85	59	
646	IMPCA002	1.565		0.35	79	
657	IMPCA002	1.649		1.13	82	
750		-----		-----		
823	Turbidimetric	1.7	C	1.61	103	First reported 0.8
824	Turbidimetric	0.9	C,DG(0.01)	-5.86	45	First reported 1.1
862	In house	1.3		-2.13	77	
867	IMPCA002	1.40		-1.19	82	
1009	In house	1.73		1.89	88	
1010	IMPCA002	1.40		-1.19	79	
1016	SMS2739	<1		-----	<45	
1024		-----		-----		
1029		-----		-----		
1035		-----		-----		
1102	IMPCA002	1.6		0.67	79	
1108	IMPCA002	1.79		2.45	95	
1221	IMPCA002	1.45		-0.73	77	
1415	IMPCA002	3.30	G(0.01)	16.54	197	
1615	In House	0.652	G(0.05)	-8.17	27	

normality	OK			
n	23			
outliers	4	Expected:	Recovery %:	
mean (n)	1.54	0.30 + 1.55 = 1.85	80	(= 1.54 / 1.85 *100%)
st.dev. (n)	0.160			
R(calc.)	0.45			
R(IMPCA002:98)	0.30			



Determination of Appearance on sample 0450.

lab	method	value	mark	Z(targ)	remarks
120	IMPCA003	cfsm		-----	
131		-----		-----	
150	Visual	cfsm		-----	
151	IMPCA003	B&C		-----	
171	IMPCA003	cfsm		-----	
174	IMPCA003	C&F		-----	
191	Visual	C&F		-----	
311	IMPCA003	cfsm		-----	
323	In house	cfsm		-----	
332	Visual	cfsm		-----	
333	IMPCA003	B&C		-----	
334	IMPCA003	Clear		-----	
342		-----		-----	
347	Visual	Cfsm		-----	
357	IMPCA003	Cfsm		-----	
395	IMCA003	Cfsm		-----	
444	Visual	C&F		-----	
445	IMPCA003	Cfsm		-----	
446	IMPCA003	Cfsm		-----	
584	Visual	B&C		-----	
608	IMPCA003	Cfsm		-----	
609	IMPCA003	Cfsm		-----	
646	IMPCA003	Cfsm		-----	
657	IMPCA003	Cfsm		-----	
750		-----		-----	
823	IMPCA003	Cfsm		-----	
824	IMPCA003	Cfsm		-----	
862	IMPCA003	Cfsm		-----	
867	IMPCA003	Cfsm		-----	
1009	In house	Pass		-----	
1010	IMPCA003	Cfsm		-----	
1016	LPM5037	Particles		-----	
1024	IMPCA003	Cfsm		-----	
1029		-----		-----	
1035		-----		-----	
1102	IMPCA003	Cfsm		-----	
1108		-----		-----	
1221	IMPCA003	Cfsm		-----	
1415	IMPCA003	Pass		-----	
1615	IMPCA003	Cfsm		-----	

Abbreviations:

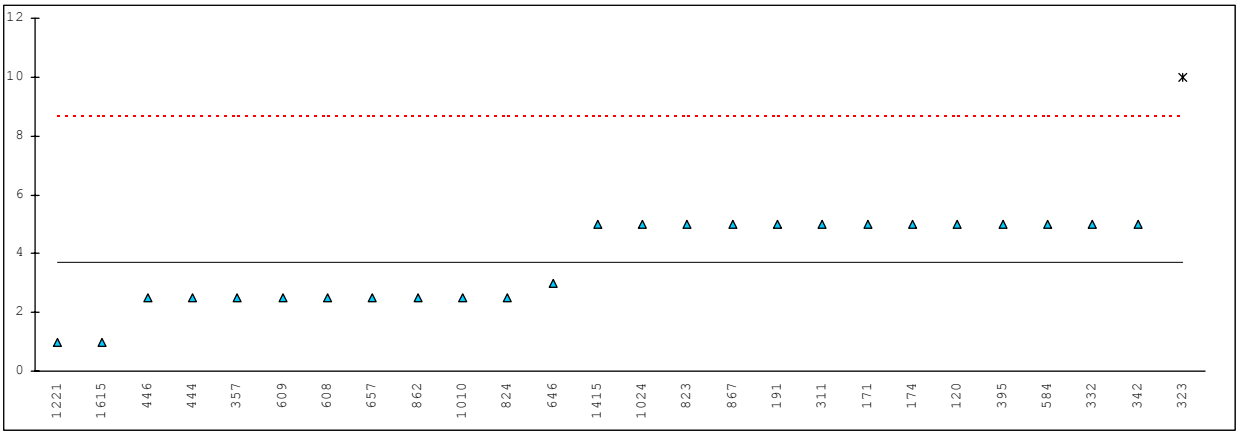
B&C: bright and clear
CFSM: clear and free of suspended matter
C&F: clear and free

Determination of Carbonisable substances Pt/Co on sample 0450

lab	method	value	mark	Z(targ)	remarks
120	E346	5		0.73	
131		-----		-----	
150		-----		-----	
151		-----		-----	
171	E346	5		0.73	
174	E346	5		0.73	
191	E346	<10		0.73	
311	E346	5		0.73	
323	E346	10	G(0.01)	3.53	First reported 15
332	E346	<10		0.73	
333	E346	<5		-----	
334		-----		-----	
342	E346	5		0.73	
347		-----		-----	
357	E346	<5		-0.67	
395	E346	5		0.73	
444	E346(Mod.)	<5		-0.67	
445		-----		-----	
446	E346	<5		-0.67	
584	E346	5		0.73	
608	E346	<5		-0.67	
609	E346	<5		-0.67	
646	E346	3		-0.39	
657	E346	<5		-0.67	
750		-----		-----	
823	E346	5		0.73	
824	E346	<5		-0.67	
862	E346	<5		-0.67	
867	E346	5		0.73	
1009	E346	pass		-----	
1010	E346	<5		-0.67	
1016		-----		-----	
1024	E346	5		0.73	
1029		-----		-----	
1035		-----		-----	
1102		-----		-----	
1108		-----		-----	
1221	E346	1.0		-1.51	
1415	E346	5		0.73	
1615	E346	1		-1.51	

			Only original reported data:
	normality	not OK	not OK
	n	25	15
	outliers	1	0
	mean (n)	3.70	4.67
	st.dev. (n)	1.443	2.059
	R(calc.)	4.04	5.76
	R(E346:03)	5.00	5.00

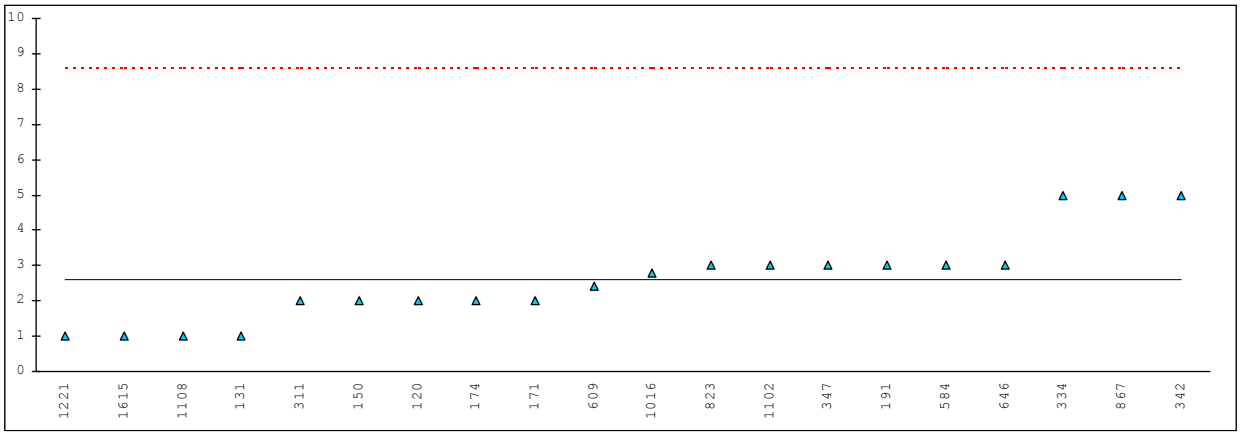
Nb.: In the calculation of the mean, standard deviation and the reproducibility and below graph a reported value of "<x' is changed into 'x/2' (for example <5 into 2.5)



Determination of Colour Pt/Co scale on sample 0450

lab	method	value	mark	Z(targ)	remarks
120	D1209	2		-0.28	
131	D1209	1		-0.75	
150	D1209	2		-0.28	
151	D1209	<5		-----	
171	D1209	2		-0.28	
174	D1209	2		-0.28	
191	D1209	3		0.18	
311	D1209	2		-0.28	
323	D1209	<5		-----	
332	D1209	<5		-----	
333	D1209	<5		-----	
334	D1209	5		1.12	
342	D1209	5		1.12	
347	D1209	3		0.18	
357	D1209	<5		-----	
395	D1209	<5		-----	
444	D5386	<1		-----	
445	D1209	<5		-----	
446	D1209	<5		-----	
584	D1209	3		0.18	
608	D1209	<5		-----	
609	D1209	2.4		-0.10	
646	D1209	3		0.18	
657	D1209	<5		-----	
750		-----		-----	
823	D1209	3		0.18	
824	D1209	<3		-----	
862	D1209	<5		-----	
867	E346	5		1.12	
1009	D1209	pass		-----	
1010	D1209	<5		-----	
1016	D1209	2.8		0.09	
1024	D1209	<5		-----	
1029		-----		-----	
1035		-----		-----	
1102	D1209	3		0.18	
1108	D1209	1		-0.75	
1221	D1209	1.0		-0.75	
1415	D1209	<5		-----	
1615	D1209	1		-0.75	

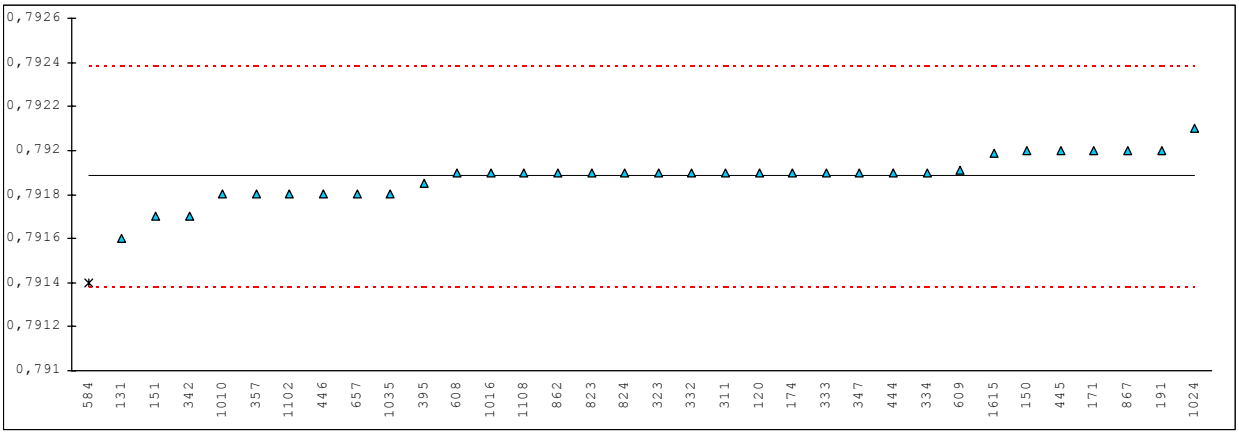
normality	not OK
n	20
outliers	0
mean (n)	2.6
st.dev. (n)	1.26
R(calc.)	3.5
R(D1209:00)	6.0



Determination of Density at 20 °C on sample 0450; results in kg/L

lab	method	Value	mark	Z(targ)	remarks
120	D4052	0.7919		0.10	
131	D4052	0.7916		-1.58	
150	D4052	0.7920		0.66	
151	D4052	0.7917		-1.02	
171	D4052	0.7920		0.66	
174	D4052	0.7919		0.10	
191	D4052	0.7920		0.66	
311	D4052	0.79190		0.10	
323	D4052	0.7919		0.10	
332	D4052	0.7919		0.10	
333	D4052	0.7919		0.10	
334	D4052	0.7919		0.10	
342	D4052	0.7917		-1.02	
347	D4052	0.7919		0.10	
357	D4052	0.7918		-0.46	
395	D4052	0.79185		-0.18	
444	D4052	0.7919		0.10	
445	D4052	0.7920		0.66	
446	D4052	0.7918		-0.46	
584	D3505	0.7914	G(0.01)	-2.70	
608	D4052	0.79190		0.10	
609	D4052	0.79191		0.15	
646		-----		-----	
657	D4052	0.7918		-0.46	
750		-----		-----	
823	D4052	0.7919		0.10	
824	D4052	0.7919		0.10	
862	D4052	0.7919		0.10	
867	D4052	0.7920		0.66	
1009		-----		-----	
1010	D4052	0.7918		-0.46	
1016	D4052	0.7919		0.10	
1024	D4052	0.7921	C	1.22	First reported 0.7924
1029		-----		-----	
1035	D4052	0.7918		-0.46	
1102	D4052	0.7918		-0.46	
1108	D4052	0.7919		0.10	
1221		-----		-----	
1415		-----		-----	
1615	D4052	0.79199		0.60	

normality	not OK
n	33
outliers	1
mean (n)	0.79188
st.dev. (n)	0.000102
R(calc.)	0.00029
R(D4052:02)	0.00050

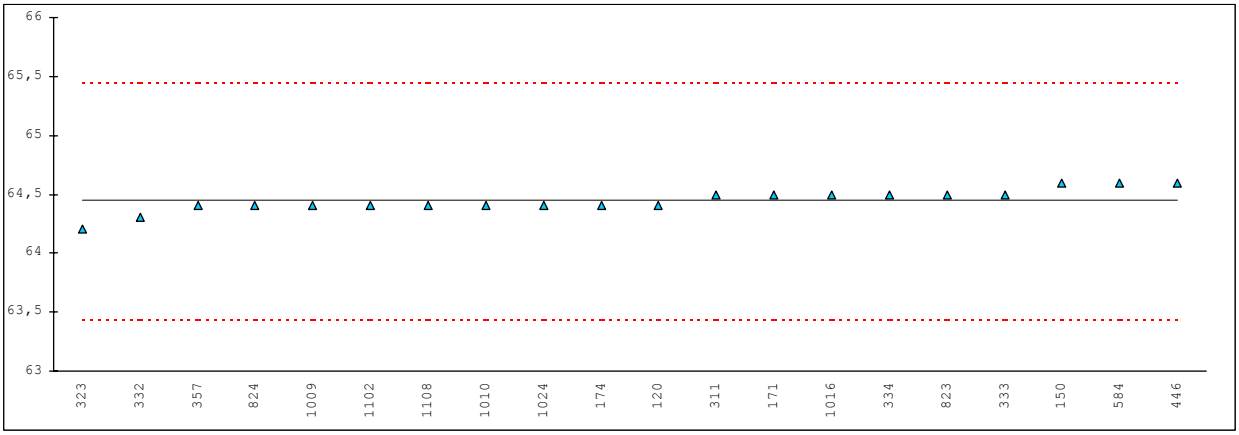


Determination of IBP and DP (automatic) @ 760 mmHg on sample 0450; results in °C

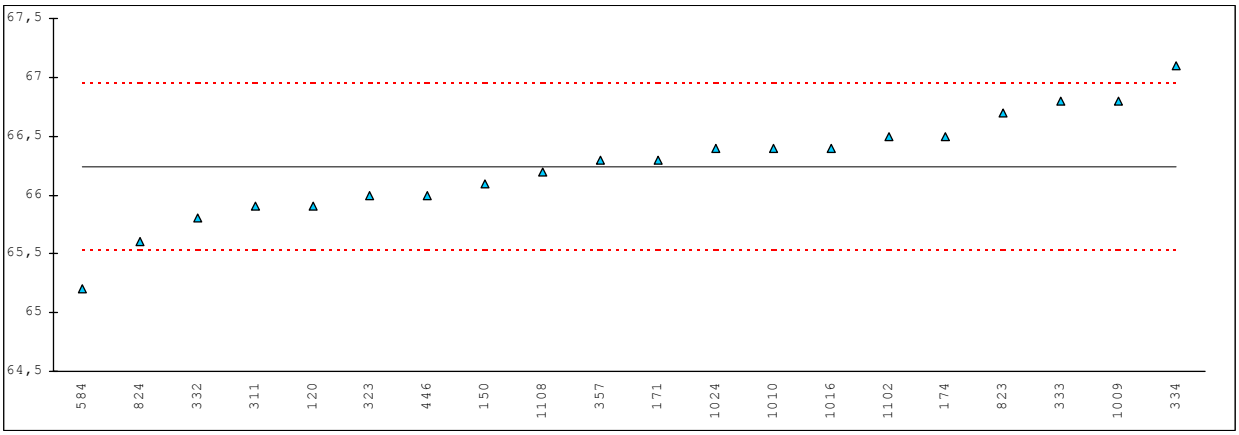
lab	method	IBP	mark	Z(targ)	DP	mark	Z(targ)	remarks
120	D1078-A	64.4		-0.13	65.9		-1.36	
131		-----		-----	-----		-----	
150	D1078-A	64.6		0.43	66.1		-0.57	
151		-----		-----	-----		-----	
171	D1078-A	64.5		0.15	66.3		0.22	
174	D1078-A	64.4		-0.13	66.5		1.01	
191		-----		-----	-----		-----	
311	D1078-A	64.5		0.15	65.9	C	-1.36	First reported 64.7
323	D1078-A	64.2		-0.68	66.0	C	-0.97	First reported 65.2
332	D1078-A	64.3		-0.40	65.8		-1.76	
333	D1078-A	64.5		0.15	66.8		2.19	
334	D1078-A	64.5		0.15	67.1		3.38	
342		-----		-----	-----		-----	
347		-----		-----	-----		-----	
357	D1078-A	64.4		-0.13	66.3		0.22	
395		-----		-----	-----		-----	
444		-----		-----	-----	C	-----	
445		-----		-----	-----		-----	
446	D1618-A	64.6		0.43	66.0		-0.97	
584	D1078-A	64.6		0.43	65.2		-4.13	
608		-----		-----	-----		-----	
609		-----		-----	-----		-----	
646		-----		-----	-----		-----	
657		-----		-----	-----		-----	
750		-----		-----	-----		-----	
823	D1078-A	64.5		0.15	66.7		1.80	
824	D1078-A	64.4		-0.13	65.6		-2.55	
862		-----		-----	-----		-----	
867		-----		-----	-----		-----	
1009	D1078-A	64.4		-0.13	66.8		2.19	
1010	D1078-A	64.4		-0.13	66.4		0.61	
1016	D1078-A	64.5		0.15	66.4		0.61	
1024	D1078-A	64.4		-0.13	66.4		0.61	
1029		-----		-----	-----		-----	
1035		-----		-----	-----		-----	
1102	D1078-A	64.4		-0.13	66.5		1.01	
1108	D1078-A	64.4		-0.13	66.2		-0.18	
1221		-----		-----	-----		-----	
1415		-----		-----	-----		-----	
1615		-----		-----	-----		-----	

normality	not OK	OK
n	20	20
outliers	0	0
mean (n)	64.45	66.25
st.dev. (n)	0.100	0.450
R(calc.)	0.30	1.26
R(D1078A:03)	1.01	0.71

Determination of IBP and DP (automatic) @ 760 mmHg on sample 0450; results in °C (continue)



Initial Boiling Point (automatic)



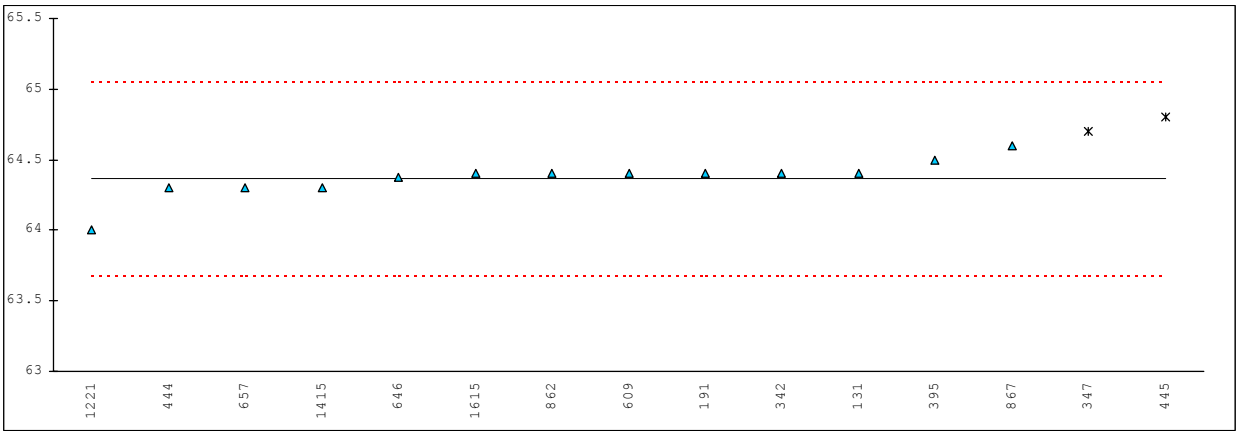
Dry Point (automatic)

Determination of IBP and DP (manual) @ 760 mmHg on sample 0450; results in °C

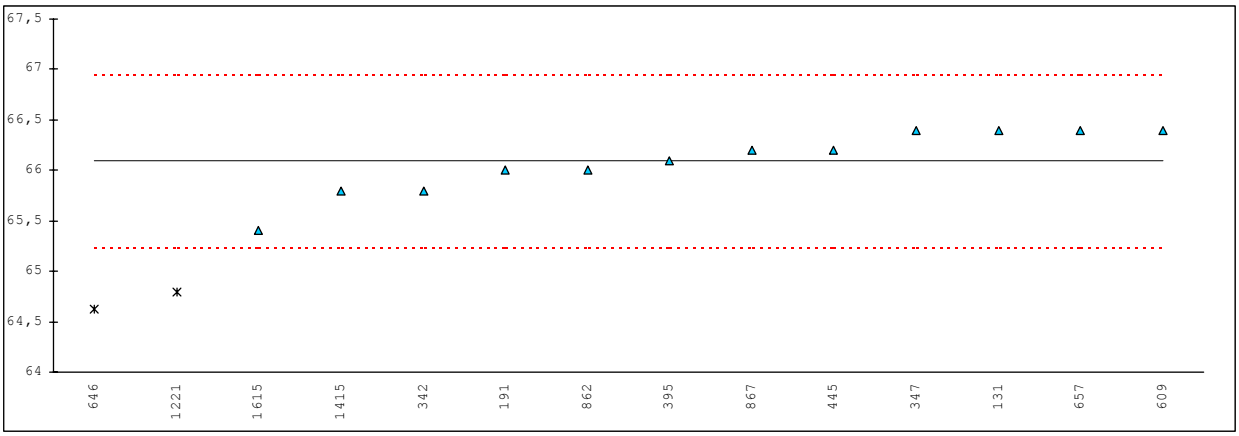
lab	method	IBP	mark	Z(targ)	DP	mark	Z(targ)	remarks
120		-----		-----	-----		-----	
131	D1078-M	64.4		0.13	66.4		1.00	
150		-----		-----	-----		-----	
151		-----		-----	-----		-----	
171		-----		-----	-----		-----	
174		-----		-----	-----		-----	
191	D1078-M	64.4		0.13	66.0		-0.30	
311		-----		-----	-----		-----	
323		-----		-----	-----		-----	
332		-----		-----	-----		-----	
333		-----		-----	-----		-----	
334		-----		-----	-----		-----	
342	D1078-M	64.4		0.13	65.8		-0.95	
347	D1078-M	64.70	ex	1.35	66.40		1.00	IBP > true boiling point (64.5)
357		-----		-----	-----		-----	
395	D1078-M	64.5		0.54	66.1		0.03	
444	D1078-M	64.3		-0.28	-----	C	-----	First reported 64.7
445	D1078-M	64.8	ex	1.76	66.2		0.35	IBP > true boiling point (64.5)
446		-----		-----	-----		-----	
584		-----		-----	-----		-----	
608		-----		-----	-----		-----	
609	D1078-M	64.4		0.13	66.4		1.00	
646	D1078-M	64.38		0.05	64.63	DG(0.01)	-4.76	
657	D1078-M	64.3		-0.28	66.4		1.00	
750		-----		-----	-----		-----	
823		-----		-----	-----		-----	
824		-----		-----	-----		-----	
862	D1078	64.4		0.13	66.0		-0.30	
867	D1078-M	64.6		0.94	66.2		0.35	
1009		-----		-----	-----		-----	
1010		-----		-----	-----		-----	
1016		-----		-----	-----		-----	
1024		-----		-----	-----		-----	
1029		-----		-----	-----		-----	
1035		-----		-----	-----		-----	
1102		-----		-----	-----		-----	
1108		-----		-----	-----		-----	
1221	D1078-M	64.0		-1.49	64.8	DG(0.01)	-4.21	
1415	D1078-M	64.3		-0.28	65.8		-0.95	
1615	D1078-M	64.4		0.13	65.4		-2.25	

normality	not OK	OK
n	13	12
outliers	0	2
mean (n)	64.37	66.09
st.dev. (n)	0.137	0.312
R(calc.)	0.38	0.87
R(D1078-M:03)	0.69	0.86

Determination of IBP and DP (manual) @ 760 mmHg on sample 0450; results in °C
(continue)



Initial Boiling Point (manual)



Dry Point (manual)

Determination of Water Miscibility on sample 0450

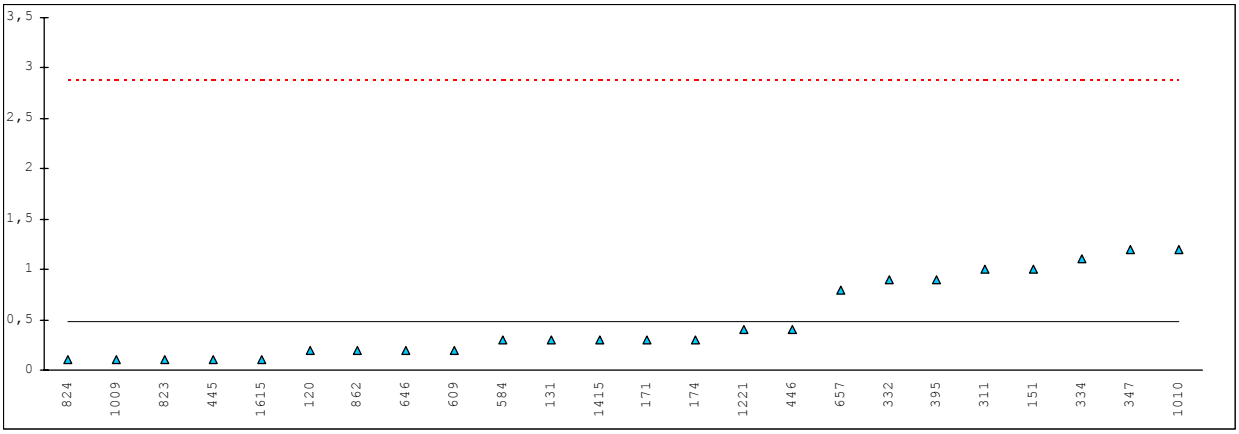
lab	method	value	mark	Z(targ)	remarks
120	D1722	pass		-----	
131	D1722	Pass		-----	
150	D1722	pass		-----	
151	D1722	Pass		-----	
171	D1722	pass		-----	
174	D1722	pass		-----	
191	D1722	pass		-----	
311	D1722	Passes test		-----	
323	D1722	passes		-----	
332	D1722	passes test		-----	
333		-----		-----	
334		-----		-----	
342	D1722	Passes test		-----	
347	D1722	pass		-----	
357	D1722	passes		-----	
395	D1722	Pass		-----	
444	D1722	Passes		-----	
445	D1722	passes test		-----	
446	D1722	Passes test		-----	
584	D1722	Pass test		-----	
608	D1722	pass		-----	
609	D1722	pass		-----	
646	D1722	pass		-----	
657	D1722	pass		-----	
750		-----		-----	
823	D1722	pass		-----	
824	D1722	pass		-----	
862	D1722	pass test		-----	
867	D1722	passes		-----	
1009	D1722	pass		-----	
1010	D1722	passes		-----	
1016	D1722	pass		-----	
1024	D1722	Passes test		-----	
1029		-----		-----	
1035		-----		-----	
1102	D1722	pass		-----	
1108		-----		-----	
1221	D1722	pass		-----	
1415	D1722	pass		-----	
1615	D1722	passes test		-----	

normality	n.a.
n	n.a.
outliers	n.a.
mean (n)	Pass
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D1722:04)	n.a.

Determination of Nonvolatile Matter on sample 0450; results in mg/100 mL

lab	method	value	mark	Z(targ)	remarks
120	D1353	0.2		-0.34	
131	D1353	0.3		-0.22	
150		-----		-----	
151	D1353	1.0		0.60	
171	D1353	0.30		-0.22	
174	D1353	0.3		-0.22	
191	D1353	<1		-----	
311	D1353	1		0.60	
323	D1353	<1		-----	
332	nft20606	0.9		0.48	
333		-----		-----	
334	D1353	1.1		0.71	
342		-----		-----	
347	D1353	1.2		0.83	
357	D1353	<1		-----	
395	D1353	0.9		0.48	
444	D1353	<0.5		-----	
445	D1353	0.10		-0.45	
446	D1353	0.4	C	-0.10	First reported 3.7
584	D1353	0.30		-0.22	
608		-----		-----	
609	D1353	0.2		-0.34	
646	D1353	0.20		-0.34	
657	D1353	0.8		0.36	
750		-----		-----	
823	D1353	0.1		-0.45	
824	D1353	0.1		-0.45	
862	D1353	0.2		-0.34	
867	D1353	<1		-----	
1009	D1353	0.1		-0.45	
1010	D1353	1.2	C	0.83	First reported 7
1016	D1353	<0.2		-----	
1024		-----		-----	
1029		-----		-----	
1035		-----		-----	
1102		-----		-----	
1108		-----		-----	
1221	D1353	0.4		-0.10	
1415	D1353	0.3		-0.22	
1615	D1353	0.1		-0.45	

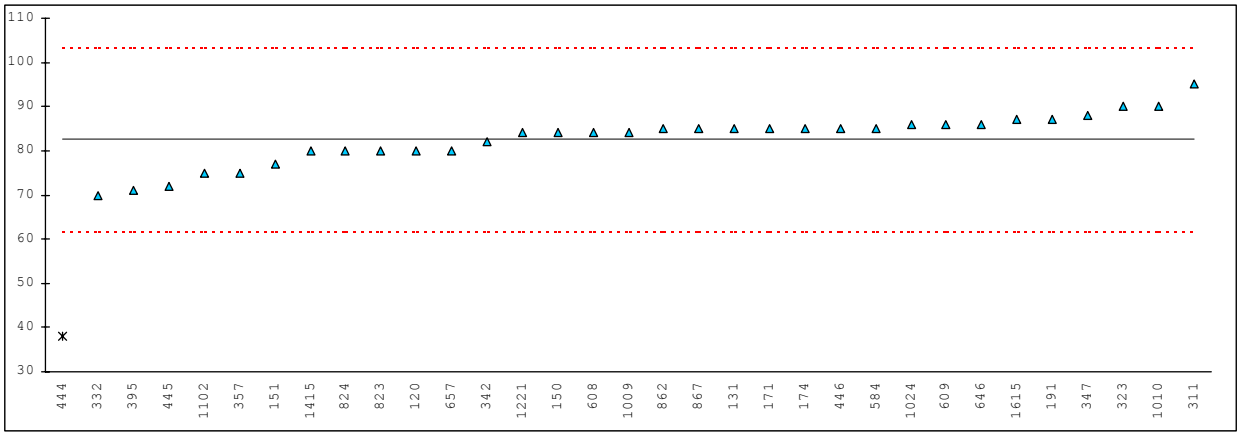
normality	not OK
n	24
outliers	0
mean (n)	0.49
st.dev. (n)	0.397
R(calc.)	1.11
R(D1353:03)	2.40



Determination of Permanganate Time Test @ 15°C on sample 0450; results in minutes

lab	method	value	mark	Z(targ)	remarks
120	D1363	80		-0.37	
131	D1363	85		0.30	
150	D1363	84		0.17	
151	D1363	77		-0.77	
171	D1363	85		0.30	
174	D1363	85		0.30	
191	D1363	87		0.57	
311	D1363	95		1.64	
323	D1363 mod	90		0.97	
332	D1363	70		-1.71	
333		-----		-----	
334		-----		-----	
342	D1363	82		-0.10	
347	D1363	88		0.70	
357	D1363	75		-1.04	
395	D1363	71		-1.58	
444	D1363	38	G(0.01)	-6.01	
445	D1363	72		-1.44	
446	D1363	85		0.30	
584	D1363	85		0.30	
608	D1363	84		0.17	
609	D1363	86		0.44	
646	D1363	86		0.44	
657	D1363	80		-0.37	
750		-----		-----	
823	D1363	80		-0.37	
824	D1363	80		-0.37	
862	D1363	85		0.30	
867	D1363	85		0.30	
1009	D1363	84		0.17	
1010	D1363	90		0.97	
1016	D1363	>50		-----	
1024	D1363	86		0.44	
1029		-----		-----	
1035		-----		-----	
1102	D1363	75		-1.04	
1108		-----		-----	
1221	D1363	84		0.17	
1415	D1363	80		-0.37	
1615	D1363	87		0.57	

Normality	not OK
n	32
outliers	1
mean (n)	82.8
st.dev. (n)	5.68
R(calc.)	15.9
R(D1363:01)	20.9

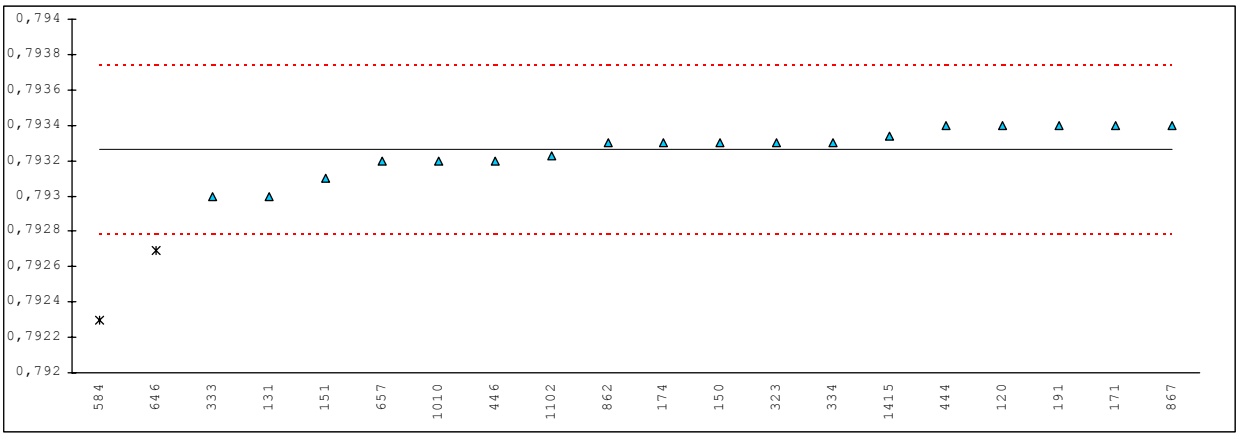


Determination of Specific Gravity 20°/20° on sample 0450

lab	method	value	mark	Z(targ)	remarks
120	D891	0.7934		0.79	
131	D891	0.7930		-1.55	
150	D4052	0.7933		0.20	
151	D4052	0.7931		-0.96	
171	D4052	0.7934		0.79	
174	D4052	0.7933		0.20	
191	D4052	0.7934		0.79	
311		-----		-----	
323	D891-A	0.7933		0.20	
332		-----		-----	
333	D1250	0.7930		-1.55	
334	D891	0.7933		0.20	
342		-----		-----	
347		-----		-----	
357		-----		-----	
395		-----		-----	
444	D4052	0.7934		0.79	
445		-----		-----	
446	D891	0.7932		-0.38	
584	D891	0.7923	G(0.01)	-5.63	
608		-----		-----	
609		-----		-----	
646	D891	0.79269	G(0.01)	-3.35	
657	D4052	0.7932		-0.38	
750		-----		-----	
823		-----		-----	
824		-----		-----	
862	D891	0.7933		0.20	
867	D891	0.7934		0.79	
1009		-----		-----	
1010	D891	0.7932		-0.38	
1016		-----		-----	
1024		-----		-----	
1029		-----		-----	
1035		-----		-----	
1102	D891	0.79323		-0.20	
1108		-----		-----	
1221		-----		-----	
1415	D4052	0.79334		0.44	
1615		-----		-----	

normality	not OK
n	18
outliers	2
mean (n)	0.79327
st.dev. (n)	0.000129
R(calc.)	0.00036
R(D4052:02)	0.00050

Compare R(D891:00) = 0.00048

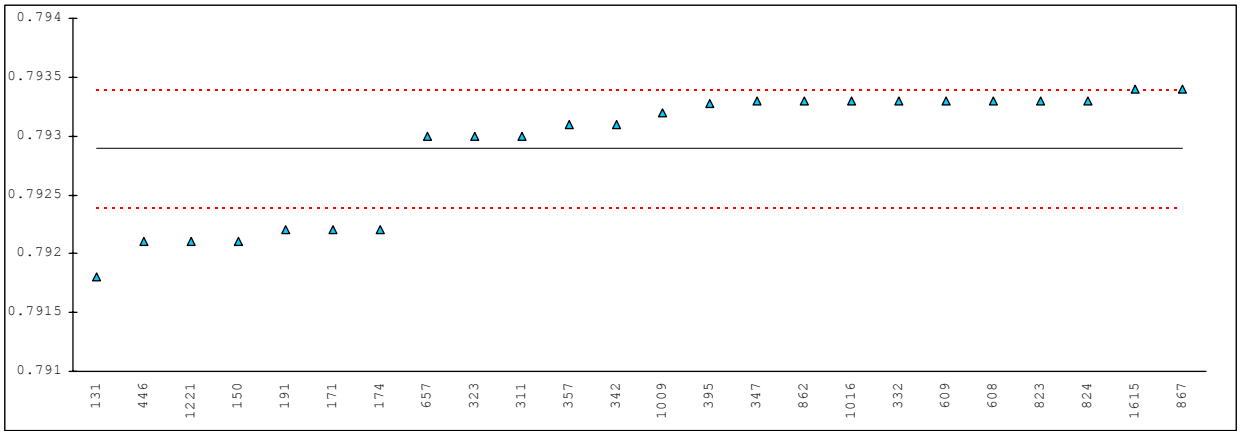


Determination of Specific Gravity, Apparent 20°/20° on sample 0450

lab	method	value	mark	Z(targ)	remarks
120		-----		-----	
131	D4052	0.7918		-----	result not in agreement with reported density
150	D4052	0.7921		-----	result not in agreement with reported density
151		-----		-----	
171	D4052	0.7922		-----	result not in agreement with reported density
174	D4052	0.7922		-----	result not in agreement with reported density
191	D4052	0.7922		-----	result not in agreement with reported density
311	D4052	0.7930		-----	
323	D4052	0.7930		-----	
332	D4052	0.7933		-----	
333		-----		-----	
334		-----		-----	
342	D4052	0.7931		-----	
347	D4052	0.7933		-----	
357	D4052	0.7931		-----	
395	D4052	0.79328		-----	
444		-----		-----	
445		-----		-----	
446	D4052	0.7921		-----	result not in agreement with reported density
584		-----		-----	
608	D4052	0.7933		-----	
609	D4052	0.7933		-----	
646		-----		-----	
657	D4052	0.793		-----	
750		-----		-----	
823	D4052	0.7933		-----	
824	D4052	0.7933		-----	
862	D4052	0.7933		-----	
867	D4052	0.7934		-----	
1009	D4052	0.7932		-----	
1010		-----		-----	
1016	D4052	0.7933		-----	
1024		-----		-----	
1029		-----		-----	
1035		-----		-----	
1102		-----		-----	
1108		-----		-----	
1221	D891	0.7921		-----	
1415		-----		-----	
1615	D4052	0.79340		-----	
				-----	

	normality	not OK
	n	24
	outliers	0
	mean (n)	0.79290
	st.dev. (n)	0.000541
	R(calc.)	0.00151
	R(D4052:02)	0.00050

Compare R(D891:00) = 0.00048



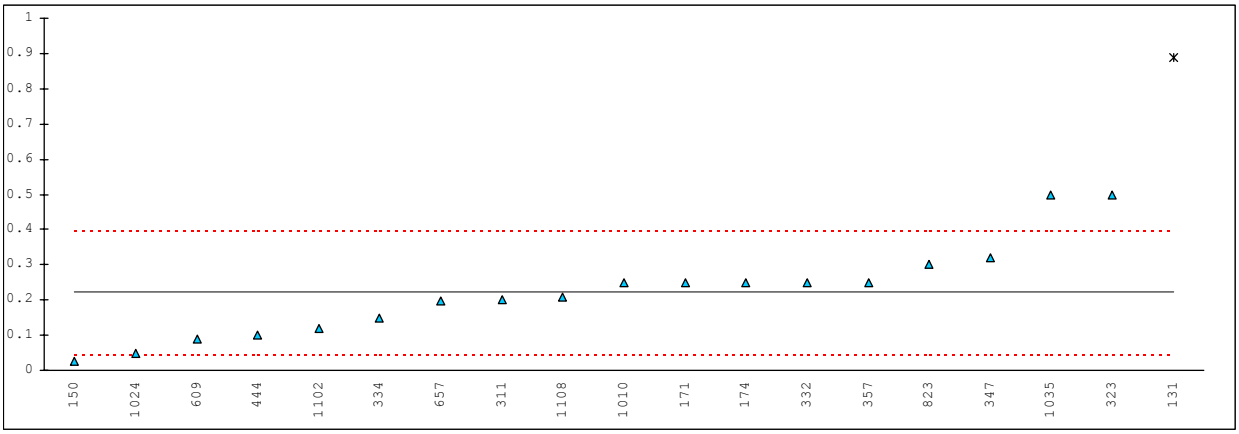
All reported results, see page 9 for discussion

Determination of Sulphur on sample 0450; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
120		-----		-----	
131	D5453	0.888	G(0.01)	9.91	
150	D5453	<0,05		-2.95	
151		-----		-----	
171	D3961	<0,5		0.40	
174	D3120	<0,5		0.40	
191		-----		-----	
311	D3961	0.2		-0.34	
323	D3961	<1		4.13	
332	NF24260	<0,5		0.40	
333		-----		-----	
334	D5453	<0.3		-1.08	
342		-----		-----	
347	D5453	0.32		1.45	
357	D3961	<0.5		0.40	
395		-----		-----	
444	D4488-mod	<0.2		-1.83	
445		-----		-----	
446		-----		-----	
584		-----		-----	
608		-----		-----	
609	D3961	0.088		-2.01	
646		-----		-----	
657	Microcolorimeter	0.198		-0.37	
750		-----		-----	
823	D3961	0.3		1.15	
824		-----		-----	
862		-----		-----	
867		-----		-----	
1009		-----		-----	
1010	D3961	<0.5		0.40	
1016		-----		-----	
1024	D6212	<0.1		-2.57	
1029		-----		-----	
1035	D3961	<1		4.13	
1102	D4629	0.12		-1.53	
1108	D5453	0.21		-0.19	
1221		-----		-----	
1415		-----		-----	
1615		-----		-----	

		Only original reported data:	
normality	OK	OK	
n	18	7	
outliers	1	1	
mean (n)	0.22	0.21	
st.dev. (n)	0.131	0.085	
R(calc.)	0.37	0.24	
R(D5453:03)	0.19	0.18	Compare R(D3961:98) = unknown

Nb.: In the calculation of the mean, standard deviation and the reproducibility and below graph a reported value of "<x' is changed into 'x/2' (for example <0.5 into 0.25)

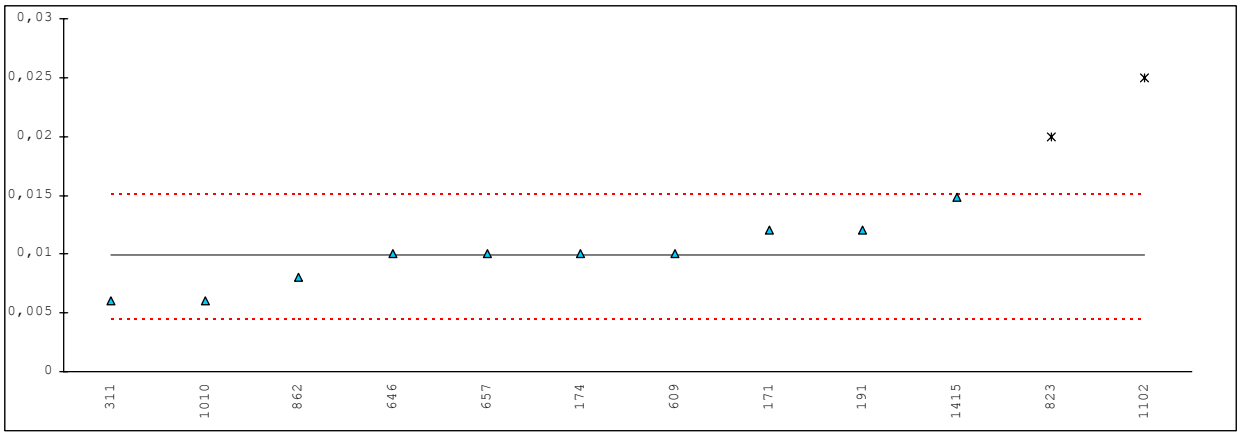


Determination of Total Iron content on sample 0450; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
120	E394	<0.01		-----	
131		-----		-----	
150		-----		-----	
151		-----		-----	
171	E394	0.012		1.11	
174	E394	0.01		0.06	
191	E394	0.012		1.11	
311	E394	0.006		-2.04	
323		-----		-----	
332	E394	<0.1		-----	
333		-----		-----	
334		-----		-----	
342		-----		-----	
347		-----		-----	
357	E394	<0.05		-----	
395	E394	<0.01		-----	
444	E394	<0.001		-----	
445		-----		-----	
446		-----		-----	
584		-----		-----	
608		-----		-----	
609	E394	0.01		0.06	
646	E394	0.010		0.06	
657	E394	0.01		0.06	
750		-----		-----	
823	E394	0.02	DG(0.05)	5.32	
824	E394	<0.01		-----	
862	E394	0.008		-0.99	
867	E394	<0.01		-----	
1009	E394	<0.010		-----	
1010	E394	0.006		-2.04	
1016	LPM5963	<0.2		-----	
1024		-----		-----	
1029		-----		-----	
1035		-----		-----	
1102	E394	0.025	DG(0.05)	7.94	
1108		-----		-----	
1221		-----		-----	
1415	E394	0.0148		2.58	
1615		-----		-----	

		After recalculation *)
normality	OK	OK
n	10	17
outliers	2	4
mean (n)	0.0099	0.0085
st.dev. (n)	0.00273	0.00463
R(calc.)	0.0076	0.0130
R(E394:00)	0.0053	0.0046

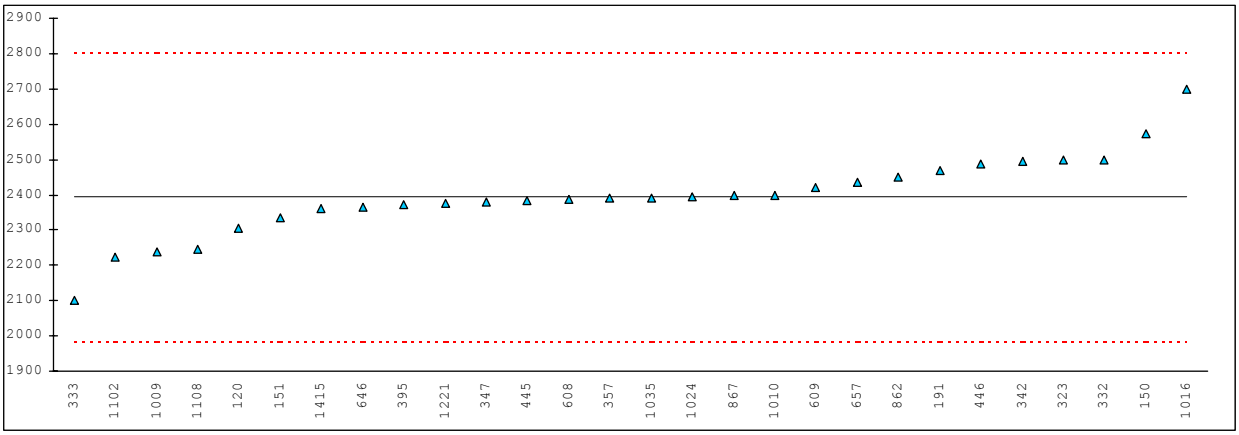
*) In the calculation of the mean. standard deviation and the reproducibility a reported value of '<x' is changed into 'x/2' (for example <0.1 into 0.05)



Determination of Water content (coulometric) on sample 0450; results in %M/M

lab	Method	Value	mark	Z(targ)	remarks
120	E1064	2305.1		-0.62	
131		-----		-----	
150	E1064	2574		1.22	
151	E1064	2334.1		-0.42	
171		-----		-----	
174		-----		-----	
191	E1064	2469		0.50	
311		-----		-----	
323	E1064	2500		0.71	
332	E1064	2500		0.71	
333	E1064	2100		-2.02	
334		-----		-----	
342	E1064	2493		0.67	
347	E1064	2379		-0.11	
357	E1064	2390		-0.04	
395	E1064	2371.3		-0.17	
444		-----		-----	
445	E1064	2383		-0.09	
446	E1064	2489		0.64	
584		-----		-----	
608	E1064	2387.0		-0.06	
609	E1064	2418.96		0.16	
646	E1064	2363		-0.22	
657	E1064	2434		0.26	
750		-----		-----	
823		-----		-----	
824		-----		-----	
862	E1064	2450		0.37	
867	E1064	2399		0.02	
1009	E1064	2239		-1.07	
1010	E1064	2400		0.03	
1016	D4672	2700		2.08	
1024	E1064	2396		0.00	
1029		-----		-----	
1035	E1064	2391.5		-0.03	
1102	E1064	2225		-1.17	
1108	E1064	2245		-1.03	
1221	E1064	2377		-0.13	
1415	E1064	2360		-0.24	
1615		-----		-----	

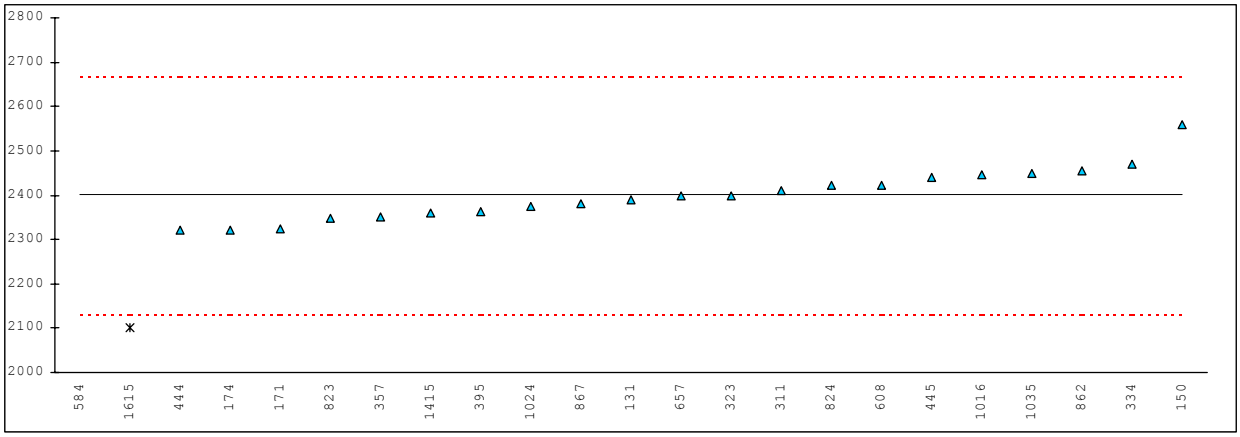
normality	OK
n	28
outliers	0
mean (n)	2395.5
st.dev. (n)	114.56
R(calc.)	320.8
R(E1064:00)	409.6



Determination of Water content (titrimetric) on sample 0450; results in %M/M

lab	method	value	mark	Z(targ)	remarks
120		-----		-----	
131	E203	2389.3		-0.11	
150	E203	2560		1.66	
151		-----		-----	
171	E203	2325		-0.78	
174	E203	2322	C	-0.81	First reported 2280
191		-----		-----	
311	E203	2410		0.10	
323	E203	2400		0.00	
332		-----		-----	
333		-----		-----	
334	E203	2470		0.72	
342		-----		-----	
347		-----		-----	
357	E203	2350		-0.52	
395	E203	2362.7		-0.39	
444	E203	2320		-0.83	
445	E203	2440		0.41	
446		-----		-----	
584	D1364	0.674	G(0.01)	-24.89	
608	E203	2421.0		0.21	
609		-----		-----	
646		-----		-----	
657	E203	2400		0.00	
750		-----		-----	
823	E203	2349	C	-0.53	First reported 2132
824	E203	2421		0.21	
862	D1364	2455		0.57	
867	E203	2382		-0.19	
1009		-----		-----	
1010		-----		-----	
1016	D1364	2445		0.46	
1024	DIN51777-1	2376		-0.25	
1029		-----		-----	
1035	E203	2449		0.50	
1102		-----		-----	
1108		-----		-----	
1221		-----		-----	
1415	E203	2360		-0.42	
1615	E203	2100	G(0.01)	-3.11	

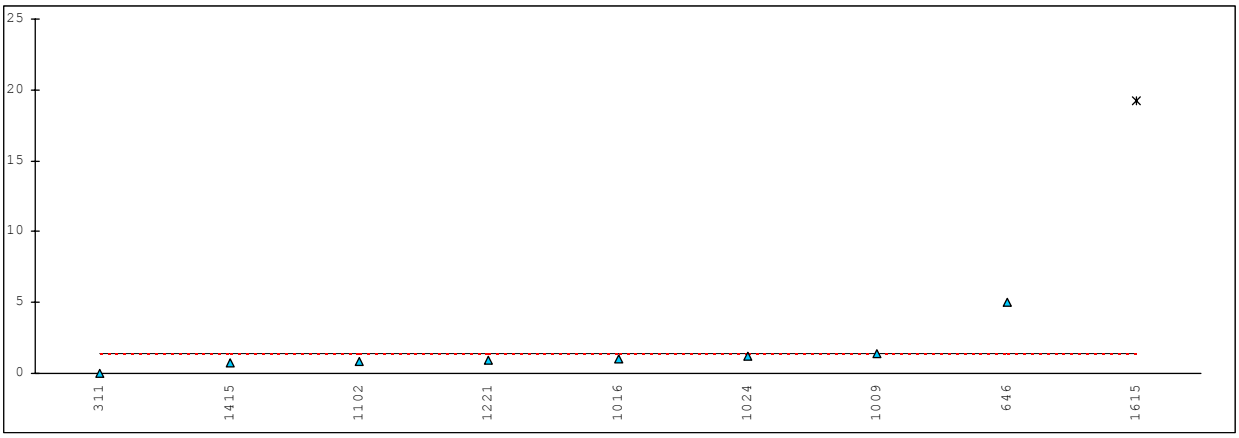
normality	OK
n	21
outliers	2
mean (n)	2400.3
st.dev. (n)	58.20
R(calc.)	163.0
R(E203:01)	270.0



Determination of Acetone content on sample 0450; results in mg/kg

lab	Method	value	mark	Z(targ)	remarks
120	IMPCA001	<1		-----	
131		-----		-----	
150	IMPCA001	<1		-----	
151	IMPCA001	<50		-----	
171	IMPCA001	<1		-----	
174	GC	<1		-----	
191	IMPCA001	<10		-----	
311	IMPCA001	0.0		-----	
323	In House	<5		-----	
332	D1612-mod	<30		-----	
333	D1612	<30		-----	
334	IMPCA001	<5		-----	
342		-----		-----	
347		-----		-----	
357	IMPCA001	<10		-----	
395	In House	<10		-----	
444	IMPCA001(Mod.)	<10		-----	
445		-----		-----	
446		-----		-----	
584		-----		-----	
608		-----		-----	
609	E346	<30		-----	
646	E346	5.05		-----	
657	In House	<5		-----	
750		-----		-----	
823	IMPCA001	<30	C	-----	First reported 14
824	E346	<30		-----	
862	E346	<30		-----	
867	IMPCA001	<10		-----	
1009	D1612-mod	1.4		-----	
1010	IMPCA001	<5		-----	
1016	LPM4627	1		-----	
1024	IMPCA001	1.17		-----	
1029		-----		-----	
1035		-----		-----	
1102	IMPCA001	0.85		-----	
1108		-----		-----	
1221	IMPCA001	0.9051		-----	
1415	IMPCA001	0.758		-----	
1615	IMPCA001	19.20	G(0.01)	-----	

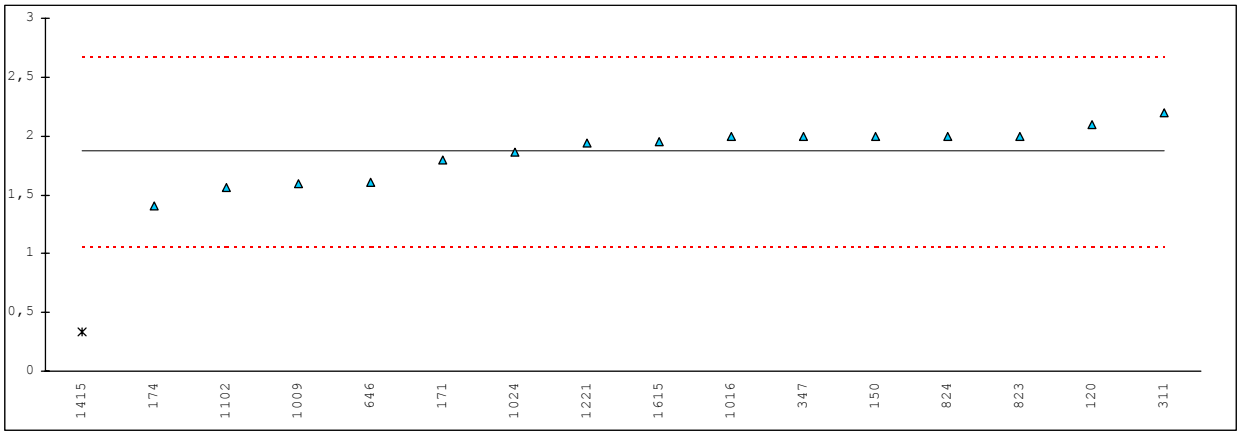
normality	not OK
n	8
outliers	1
mean (n)	1.39
st.dev. (n)	1.533
R(calc.)	4.29
R(lit)	unknown



Determination of Ethanol content on sample 0450; results in mg/kg

lab	method	value	mark	Z(targ)	Remarks
120	IMPCA001	2.1		0.80	
131		-----		-----	
150	IMPCA001	2		0.46	
151	IMPCA001	<50		-----	
171	IMPCA001	1.8		-0.24	
174	GC	1.4		-1.62	
191	IMPCA001	<10		-----	
311	IMPCA001	2.2		1.15	
323	E346 mod	<5		-----	
332	In house	<10		-----	
333	In house	<10		-----	
334	IMPCA001	<5		-----	
342		-----		-----	
347	GC	2		0.46	
357	IMPCA001	<5		-----	
395	IMPCA001	<5		-----	
444	IMPCA001(Mod.)	<5		-----	
445		-----		-----	
446		-----		-----	
584		-----		-----	
608		-----		-----	
609	IMPCA001	<17	C	-----	First reported 0.9842
646	E346	1.61		-0.89	
657	In house	<5		-----	
750		-----		-----	
823	IMPCA001	2	C	0.46	First reported 3
824	IMPCA001	2		0.46	
862	GLC	<10		-----	
867	IMPCA001	<10		-----	
1009	In house	1.6		-0.93	
1010	IMPCA001	<5		-----	
1016	LPM4627	2		0.46	
1024	IMPCA001	1.86		-0.03	
1029		-----		-----	
1035		-----		-----	
1102	IMPCA001	1.56		-1.07	
1108		-----		-----	
1221	IMPCA001	1.9430		0.26	
1415	IMPCA001	0.329	G(0.01)	-5.32	
1615	E346	1.95		0.28	

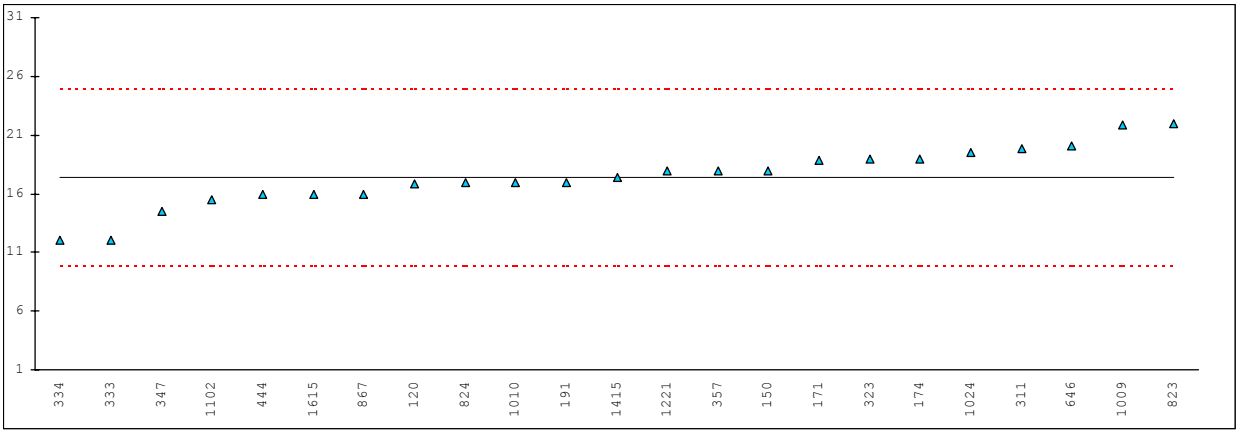
normality	not OK
n	15
outliers	1
mean (n)	1.87
st.dev. (n)	0.227
R(calc.)	0.64
R(E346:03)	0.81



Determination of Ethanol content on sample 0451; results in mg/kg

lab	method	value	mark	Z(targ)	Recovery (%)	remarks
120	IMPCA001	16.8		-0.24	79	
131		-----		-----		
150	IMPCA001	18		0.21	86	
151		-----		-----		
171	IMPCA001	18.8		0.50	91	
174	GC	19.0		0.58	94	
191	IMPCA001	17		-0.16	81	
311	IMPCA001	19.9		0.91	95	
323	E346 mod	19	C	0.58	92	First reported 12
332	In house	<20		-----	<105	
333	In house	12		-2.02	54	
334	IMPCA001	12		-2.02	54	
342		-----		-----		
347	GC	14.5		-1.09	67	
357	IMPCA001	18		0.21	86	
395	IMPCA001	<5		-----	<27	
444	IMPCA001(Mod.)	15.9		-0.57	75	
445		-----		-----		
446		-----		-----		
584		-----		-----		
608		-----		-----		
609	IMPCA001	<17	C	-----		First reported 7.3695
646	E346	20.125		1.00	99	
657	In house	<5		-----		
750		-----		-----		
823	IMPCA001	22		1.69	107	
824	IMPCA001	17		-0.16	80	
862	GLC	<10		-----	<53	
867	IMPCA001	16		-0.53	76	
1009	In house	21.8		1.62	108	
1010	IMPCA001	17		-0.16	81	
1016		-----		-----		
1024	IMPCA001	19.52		0.77	94	
1029		-----		-----		
1035		-----		-----		
1102	IMPCA001	15.49		-0.72	74	
1108		-----		-----		
1221	IMPCA001	17.9011		0.17	85	
1415	IMPCA001	17.4		-0.01	91	
1615	E346	15.94		-0.56	75	

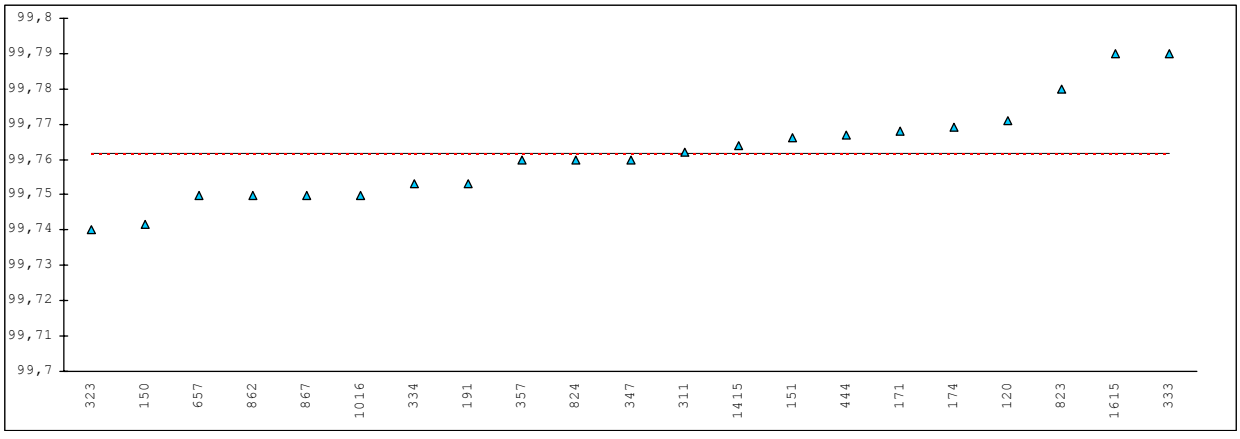
normality	OK			
n	23			
outliers	0	Expected:	Recovery %:	
mean (n)	17.44	1.9 + 18.7 = 20.6	85	(= 17.44 / 20.6 *100%)
st.dev. (n)	2.561			
R(calc.)	7.17			
R(E346:03)	7.56			



Determination Purity on sample 0450. results in %M/M

lab	method	value	mark	Z(targ)	remarks
120		99.771		-----	
131		-----		-----	
150	In house	99.7416		-----	
151	IMPCA001	99.766		-----	
171	IMPCA001	99.768		-----	
174	glc	99.769		-----	
191	In house	99.7531		-----	
311		99.762		-----	
323	In house	99.74		-----	
332		>99.7		-----	
333	In house	99.79		-----	
334	In house	99.7530		-----	
342		-----		-----	
347	GC	99.76		-----	
357	In house	99.76		-----	
395		-----		-----	
444		99.7671		-----	
445		-----		-----	
446		-----		-----	
584		-----		-----	
608		-----		-----	
609		-----		-----	
646	In house	>99.85	C	-----	First reported 99.99
657	In house	99.75		-----	
750		-----		-----	
823		99.78		-----	
824		99.76		-----	
862	glc	99.75	C	-----	First reported 99.99
867		99.75		-----	
1009		-----		-----	
1010		-----		-----	
1016	LPM4627	99.75	C	-----	First reported 99.99
1024		-----		-----	
1029		-----		-----	
1035		-----		-----	
1102		-----		-----	
1108		-----		-----	
1221		-----		-----	
1415		99.764		-----	
1615		99.79		-----	

normality	OK
n	21
outliers	0
mean (n)	99.7617
st.dev. (n)	0.01369
R(calc.)	0.0383
R(lit)	Unknown

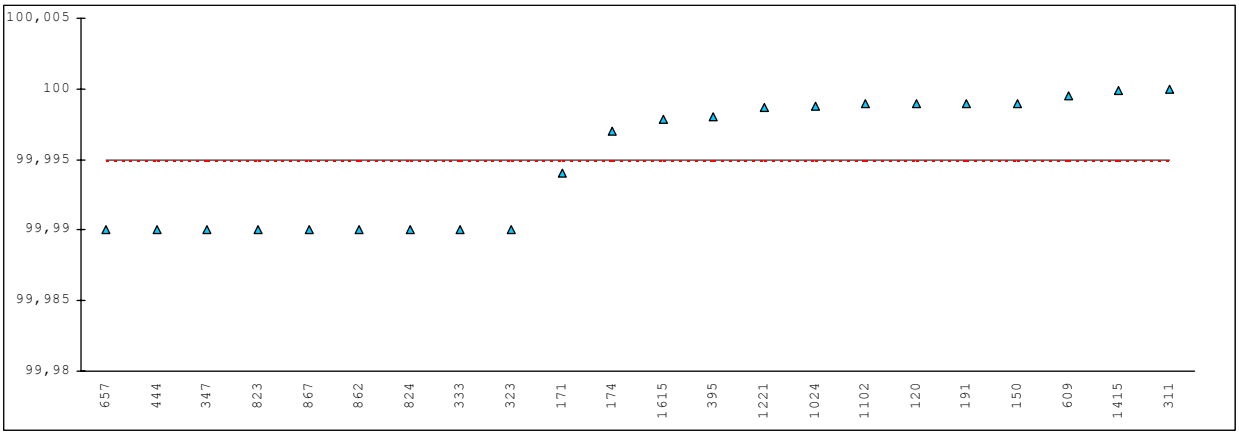


Determination Purity on dry basis on sample 0450. results in %M/M

lab	method	value	mark	Z(targ)	remarks
120	IMPCA001	99.999		-----	
131		-----		-----	
150	IMPCA001	99.999		-----	
151		-----		-----	
171	IMPCA001	99.994		-----	
174	GC	99.997		-----	
191	IMPCA001	99.999		-----	
311	IMPCA001	100.00		-----	
323	In house	99.99		-----	
332		-----		-----	
333	In house	99.99		-----	
334	IMPCA001	>99.99		-----	
342		-----		-----	
347	GC	99.99		-----	
357	IMPCA001	>99.99		-----	
395	IMPCA001	99.998		-----	
444	IMPCA001(Mod.)	99.99		-----	
445		-----		-----	
446		-----		-----	
584		-----		-----	
608		-----		-----	
609	IMPCA001	99.9995		-----	
646		-----		-----	
657	In house	99.99		-----	
750		-----		-----	
823	IMPCA001	99.99		-----	
824	IMPCA001	99.99		-----	
862	glc	99.99	C	-----	
867	IMPCA001	99.99		-----	
1009		-----		-----	
1010	IMPCA001	>99.99		-----	
1016		-----		-----	
1024	IMPCA001	99.9988		-----	
1029		-----		-----	
1035		-----		-----	
1102	IMPCA001	99.999		-----	
1108		-----		-----	
1221	IMPCA001	99.9987		-----	
1415	IMPCA001	99.9999		-----	
1615	IMPCA001	99.99788		-----	

	normality	not OK
	n	22
	outliers	0
	mean (n)	99.9950
	st.dev. (n)	0.00441
	R(calc.)	0.0124
	R(IMPCA001:98)	unknown

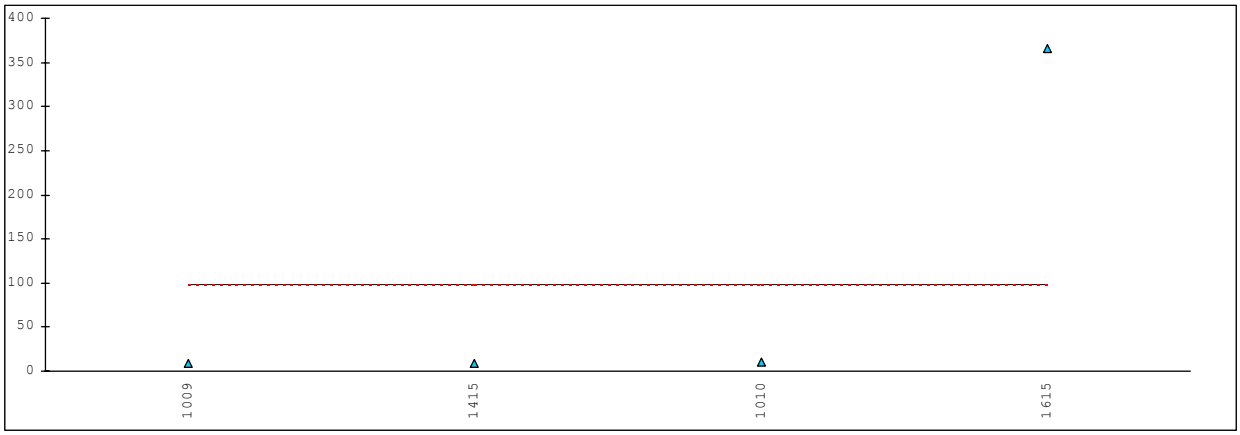
Compare R(iis03C07) = 0.0120



Determination of Trimethylamine on sample 0450; results in µg/kg

lab	method	value	mark	Z(targ)	remarks
120		-----		-----	
131		-----		-----	
150	E346	<5		-----	
151		-----		-----	
171		-----		-----	
174		-----		-----	
191		-----		-----	
311		-----		-----	
323	E346 mod	<1000		-----	
332		-----		-----	
333		-----		-----	
334		-----		-----	
342		-----		-----	
347		-----		-----	
357		-----		-----	
395		-----		-----	
444		-----		-----	
445		-----		-----	
446		-----		-----	
584		-----		-----	
608		-----		-----	
609		-----		-----	
646		-----		-----	
657		-----		-----	
750		-----		-----	
823		-----		-----	
824		-----		-----	
862		-----		-----	
867		-----		-----	
1009	E346	8.4		-----	
1010	E346	10		-----	
1016		-----		-----	
1024		-----		-----	
1029		-----		-----	
1035		-----		-----	
1102		-----		-----	
1108		-----		-----	
1221		-----		-----	
1415	E346	9.4		-----	
1615	E346	365.5		-----	

normality	n.a.
n	n.a.
outliers	n.a.
mean (n)	n.a.
st.dev. (n)	n.a.
R(calc.)	n.a.
R(E346:03)	unknown



APPENDIX 2

List of number of participants per country

Number of participating laboratories	Contry
1 lab in	AUSTRIA
1 lab in	BELGIUM
1 lab in	CANADA
1 lab in	FINLAND
2 labs in	FRANCE
2 labs in	GERMANY
1 lab in	GREECE
1 lab in	ITALY
2 labs in	KOREA
4 labs in	MALAYSIA
2 labs in	NEW ZEALAND
1 lab in	NORWAY
2 labs in	P.R. of CHINA
1 lab in	RUSSIA
1 lab in	SINGAPORE
2 labs in	SPAIN
2 labs in	THE NETHERLANDS
7 labs in	U.S.A.
3 labs in	UNITED KINGDOM
2 labs in	VENEZUELA

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
E	= error in calculations
ex	= excluded from calculations
n.a.	= not applicable

Literature:

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6 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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9 DIN 38402 T41/42
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11 J.N. Miller, Analyst, 118, 455, (1993)
12 IMPCA Methanol Reference Specifications, IMPCA, Brussels, April 1999.
13 ASTM E346-03