Results of Proficiency Test
MTBE
February 2017

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

Author: ing. A.S. Noordman – de Neef
Correctors: dr. R.G. Visser and ing. R.J. Starink

Report no. iis17C03

March 2017

CONTENTS

1 INTRODUCTION

Since 1995, the Institute for Interlaboratory Studies (iis) organizes a proficiency test for the analysis of Methyl Tertiary Butyl Ether (MTBE). During the annual proficiency testing program 2016/2017, it was decided to continue the round robin for the analyses of MTBE.
In this interlaboratory study for MTBE 19 laboratories in 12 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2017 proficiency test (PT) are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample of MTBE (0.5 litre bottle, labelled #17003) to the participants. Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for the 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). The protocol can be downloaded from iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

The necessary 25 litres of MTBE bulk material were obtained from a local supplier. After homogenisation, the bulk material was transferred into 50 brown glass bottles of 500 mL and labelled #17003. The homogeneity of the subsamples was checked by determination of Density at 20˚C in accordance with ASTM D4052 and Water in accordance with ASTM D1364 on 8 stratified randomly selected samples.

	Density at 20°C in kg/L	Water in mg/kg
sample #17003-1	0.74105	137
sample #17003-2	0.74104	120
sample #17003-3	0.74104	128
sample #17003-4	0.74104	109
sample #17003-5	0.74106	132
sample #17003-6	0.74103	121
sample #17003-7	0.74104	122
sample #17003-8	0.74104	132

Table 1: homogeneity test results of subsamples #17003

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

	Density at 20°C in kg/L	Water in mg/kg
r (observed)	0.00002	25.0
reference test method	ASTM D4052:16	ASTM D1364:12
0.3xR (reference test method)	0.00015	20.1
r (reference test method)	0.00011	33.6

Table 2: evaluation of the repeatabilities of the subsamples #17003

The calculated repeatability of the Density determination was in agreement with 0.3 times the corresponding reproducibility of the reference test method and of the Water determination with the repeatability of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one bottle of 500 ml, labelled #17003 was sent on January 25, 2017. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Methyl Tertiary Butyl Ether, packed in the brown glass bottles was checked. The material has been found stable for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine on sample #17003 one to all of the following parameters: Appearance, Carbonyls, Density at 15°C, Refractive index at 20°C, Water, Purity (on dry basis), Methanol, and some impurities (Sum of 5 Diisobutylenes [2,4,4-Trimethyl-1-pentene; 2,4,4-Trimethyl-2-pentene; 2,3,4-Trimethyl-2-pentene; 3,4,4-Trimethyl-1-pentene and 3,5-Dimethyl-1-hexene], tert-Butylalcohol, Hydrocarbons (C4- and C5) and other impurities.

It was explicitly requested to treat the samples as if they were routine samples. Therefore, each laboratory is advised to perform only those analyses that normally are done in daily routine (but the laboratories are allowed to do all analyses). Furthermore, it was requested to report the test results using the indicated units on the report form and not to round the test results more, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon'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 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 test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as 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. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

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

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other targets values were used. In some cases a reproducibility based on former iis proficiency tests could be used.

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

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

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

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

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

4 EVALUATION

In this proficiency test, some problems were encountered with the dispatch of the samples. Participants in Brazil and Saudi Arabia received the samples late or not at all due to problems at customs. From the 19 participants 3 participants did not report any test results at all. Not all laboratories were able to report all analyses requested.
The 16 reporting laboratories submitted 132 numerical test results. Observed were 10 outlying test results, which is 7.6%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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.

4.1 EVALUATION PER TEST

In this section, the reported test results are discussed per test.
The test methods, which are used by the different laboratories, are taken into account for explaining the observed differences when possible and applicable. These test methods are also listed in the tables together with the reported test results. The abbreviations, used in these tables, are listed in appendix 3.
Unfortunately, a suitable reference test method providing the precision data is not available for all determinations. For the tests without precision data the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

In the iis PT reports, ASTM test methods are referred to with a number (e.g. D1218) and an added designation for the year that the test method was adopted or revised (e.g. D1218:12). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1218:12 (2016)). In the results tables of appendix 1 only the test method number and year of adoption or revision (D1218:12) will be used.

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #17003, which is pass or bright and clear.

Carbonyls: Only two laboratories reported a test result, therefore, no conclusions were drawn.

Density at 15°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 ASTM D4052:16.

Refractive Index: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1218:12(2016).

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1364:02(2012).

Purity: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5441:98(2013).

Methanol: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5441:98(2013).

Diisobutylene (=sum 5 DIB): This determination is not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D5441:98(2013).

2,4,4-Trime-1-pent.: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5441:98(2013).

2,4,4-Trime-2-pent.: This determination was problematic for three participants. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated reproducibility using the Horwitz equation.

Other DIB.'s: It should be noticed that for the other DIB's, test method D5441:98(2013) is applicable for concentrations >0.02 %M/M. Only few numerical test results were reported. Therefore, no significant conclusions were drawn.

Tert-Butanol: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D5441:98(2013).

C4-hydrocarbons: 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 ASTM D5441:98(2013).

C5-hydrocarbons: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D5441:98(2013).

Other Impurities: Other impurities may be all components listed in table 3 of D5441:98(2013) and not evaluated separately in this proficiency test.
ASTM D5441:98(2013) does not specify requirements for the reproducibility for the sum of these components. Therefore, the estimated reproducibility using the Horwitz equation based on 9 components is used.
This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility using the Horwitz equation (9 components). The higher variation may be due to that each laboratory makes a different decision in what to add to “other impurities”.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and these parameters as found for the group of participating laboratories. The average results and the calculated reproducibilities (2.8 * sd) are compared in the next tables with the reproducibilities, derived from reference test methods (in casu the ASTM standards, see tables in appendix 1).

	unit	n	average	2.8 * sd	R (lit)
Appearance		12	pass	n.a.	n.a.
Carbonyls	µg/g	2	n.a.	n.a.	n.a.
Density at 15°C	kg/L	13	0.7463	0.0003	0.0005
Refractive Index at 20°C		7	1.3694	0.0004	0.0005
Water	mg/kg	14	158.9	47.3	75.6
Purity	%M/M	14	98.141	0.378	0.411
Methanol	%M/M	13	0.491	0.071	0.083
Diisobutylene (=sum of 5)	% M/M	7	0.191	0.057	0.059
2,4,4-Trimethyl-1-pentene	%M/M	8	0.145	0.029	0.047
2,4,4-Trimethyl-2-pentene	%M/M	5	0.040	0.003	0.007
tert-Butanol	%M/M	13	0.363	0.077	0.132
C4 – hydrocarbons	%M/M	8	0.187	0.035	0.062
C5 – hydrocarbons	%M/M	8	0.090	0.034	0.033
Other impurities	%M/M	6	0.405	0.520	0.156

Table 3: reproducibilities of tests on sample #17003

Without further statistical calculations, it can be concluded that for number of tests for MTBE 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 FEBRUARY 2017 WITH PREVIOUS PTS

	February 2017	February 2015	February 2013	February 2012	March 2010
Number of reporting labs	16	17	16	17	33
Number of results reported	132	159	154	178	305
Statistical outliers	10	13	16	16	22
Percentage outliers	7.6%	8.2%	10.4%	9.0%	7.2%

Table 4: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations for the MTBE sample of the proficiency test was compared against the requirements of the respective reference test methods. The conclusions are given the following table:

Determination	February 2017	February 2015	February 2013	February 2012	March 2010
Carbonyls	n.e.	n.e.	--	n.e	n.e
Density at 15°C	++	++	++	+	+
Refractive Index at 20°C	+	+	+	+/-	+
Water	+	++	--	+/-	++
Purity	+	--	+	--	+
Methanol	+	+/-	--	-	+
2,4,4-Trimethyl-1-pentene	+	+	+/-	++	++
2,4,4-Trimethyl-2-pentene	++	+	-	+	--
tert-Butanol	++	++	++	++	++
C4 – hydrocarbons	++	--	-	+/-	--
C5 – hydrocarbons	+/-	+	++	--	++

Table 5: comparison determinations against the reference test methods

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

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

APPENDIX 1

Determination of Appearance on sample #17003;

lab	method	value	mark	z(targ)	remarks
171	E2680	Clear and Free		-----	
311	E2680	pass		-----	
312	Visual	Br&Cl		-----	
323	E2680	C&B		-----	
334		-----		-----	
343	E2680	PASS		-----	
347	E2680	PASS		-----	
555		-----		-----	
657	E2680	PASS		-----	
963		-----		-----	
1201		-----		-----	
1221		-----		-----	
1251	Visual	Bright and Clear		-----	
1530	Visual	c & b		-----	
1728	Visual	Clear and bright		-----	
1788	Visual	CLEAR		-----	
1866		-----		-----	
1940		-----		-----	
3166	Visual	Clear colorless liquid		-----	
	normality	n.a.			
	n	12			
	outliers	0			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit.)	n.a.			

Determination of Carbonyls as CO on sample #17003; results in µg/g

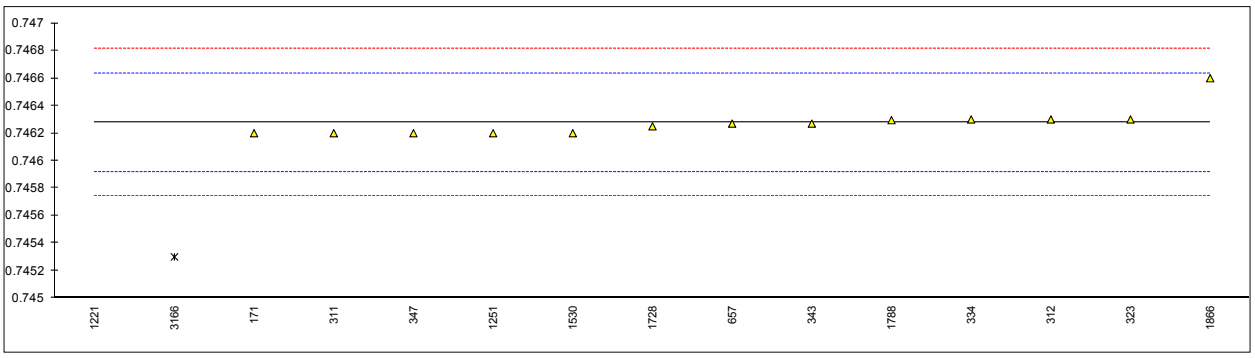
lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343		-----		-----	
347		-----		-----	
555		-----		-----	
657	E411	579.96		-----	
963		-----		-----	
1201		-----		-----	
1221		-----		-----	
1251		-----		-----	
1530		-----		-----	
1728		-----		-----	
1788		-----		-----	
1866	E411	68		-----	
1940		-----		-----	
3166		-----		-----	

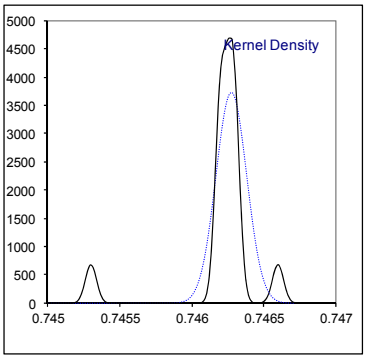
normality	n.a.
n	2
outliers	n.a.
mean (n)	n.a.
st.dev. (n)	n.a.
R(calc.)	n.a.
R(lit.)	n.a.

Determination of Density at 15°C on sample #17003; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.7462		-0.42	
311	D4052	0.7462		-0.42	
312	D4052	0.7463		0.14	
323	D4052	0.7463		0.14	
334	D4052	0.7463	C	0.14	reported: 746.3 kg/L
343	D4052	0.74627		-0.03	
347	D4052	0.7462		-0.42	
555		-----		-----	
657	D4052	0.74627		-0.03	
963		-----		-----	
1201		-----		-----	
1221	D4052	0.74098	C,D(0.01)	-29.66	first reported: 0.74466
1251	D4052	0.7462		-0.42	
1530	D4052	0.7462		-0.42	
1728	D4052	0.74625		-0.14	
1788	D4052	0.746295		0.11	
1866	D4052	0.7466		1.82	
1940		-----		-----	
3166	D1475	0.7453	D(0.01)	-5.46	

normality	not OK
n	13
outliers	2
mean (n)	0.74628
st.dev. (n)	0.000107
R(calc.)	0.00030
R(D4052:16)	0.00050

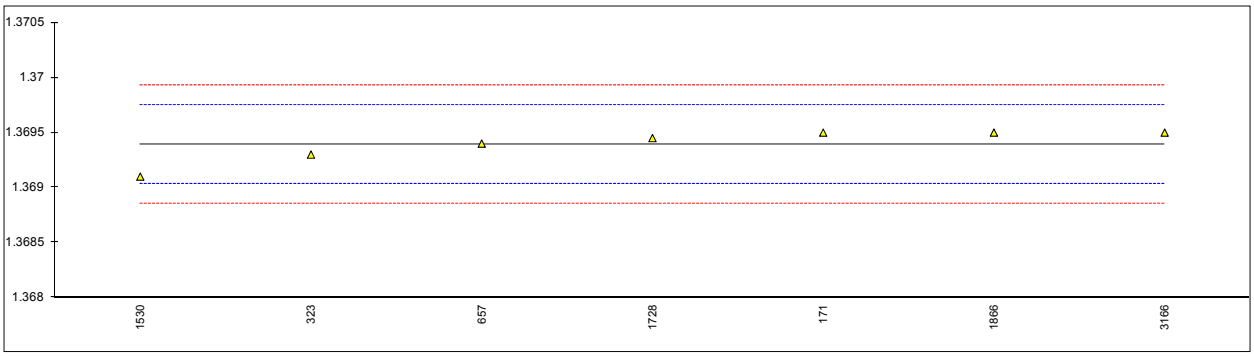




Determination of Refractive Index at 20°C on sample #17003;

lab	method	value	mark	z(targ)	remarks
171	D1218	1.3695	C	0.60	first reported: 1.3706
311		-----		-----	
312		-----		-----	
323	D1218	1.36930		-0.52	
334		-----		-----	
343		-----		-----	
347		-----		-----	
555		-----		-----	
657	D1218	1.3694		0.04	
963		-----		-----	
1201		-----		-----	
1221		-----		-----	
1251		-----		-----	
1530	D1218	1.3691		-1.64	
1728		1.36945		0.32	
1788		-----		-----	
1866	D1218	1.3695		0.60	
1940		-----		-----	
3166	USP831	1.3695		0.60	

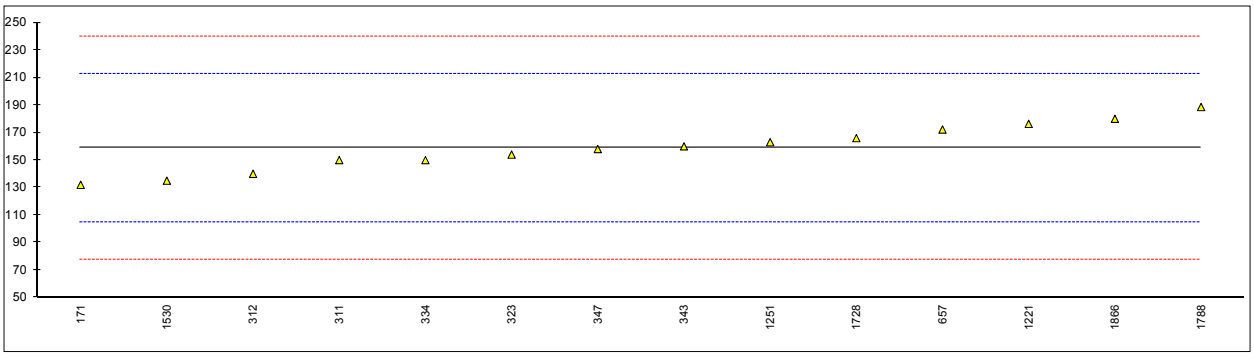
normality	unknown
n	7
outliers	0
mean (n)	1.36939
st.dev. (n)	0.000148
R(calc.)	0.00042
R(D1218:12)	0.00050

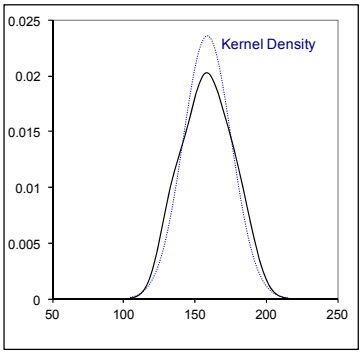


Determination of Water, titrimetric on sample #17003; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	132		-1.00	
311	D1364	150		-0.33	
312	ISO12937	140		-0.70	
323	D1364	154		-0.18	
334	D1364	150		-0.33	
343	E1064	160	C	0.04	first reported: 0.016 mg/kg
347	E1064	158		-0.03	
555		-----		-----	
657	E1064	172.25		0.49	
963		-----		-----	
1201		-----		-----	
1221	E1064	176.35		0.64	
1251	D1364	163		0.15	
1530	ISO12937	135		-0.89	
1728	E203	166		0.26	
1788	D6304	188.6		1.10	
1866	D1364	180		0.78	
1940		-----		-----	
3166		-----		-----	

normality	OK
n	14
outliers	0
mean (n)	158.94
st.dev. (n)	16.902
R(calc.)	47.32
R(D1364:02)	75.64

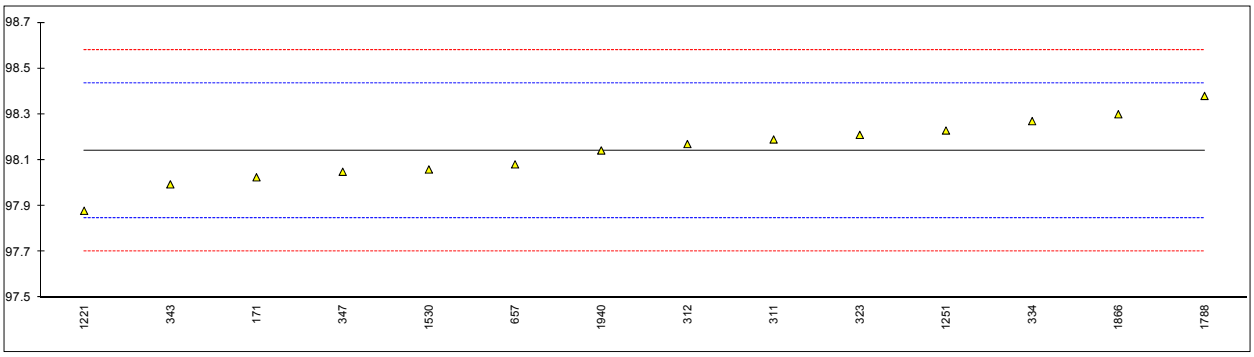


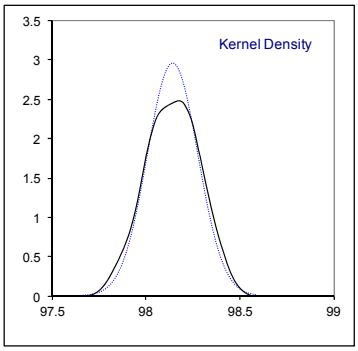


Determination of Purity by GC on dry basis on sample #17003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	98.025		-0.79	
311	D5441	98.19		0.33	
312	D5441	98.17		0.20	
323	D5441	98.21		0.47	
334	D5441	98.27		0.88	
343	D5441	97.994		-1.00	
347	D5441	98.049		-0.63	
555		-----		-----	
657	D5441	98.0813		-0.41	
963		-----		-----	
1201		-----		-----	
1221	D5441	97.8788		-1.79	
1251	D5441	98.229		0.60	
1530	D5441	98.059		-0.56	
1728		-----		-----	
1788	D5441	98.38		1.63	
1866	D5441	98.30		1.08	
1940	D5441	98.142		0.00	
3166		-----		-----	

normality	OK
n	14
outliers	0
mean (n)	98.1413
st.dev. (n)	0.13512
R(calc.)	0.3783
R(D5441:98)	0.4110

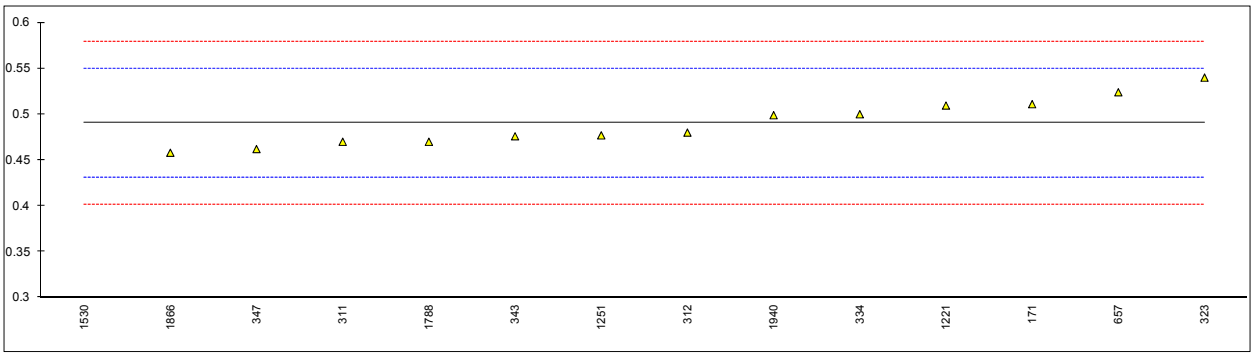


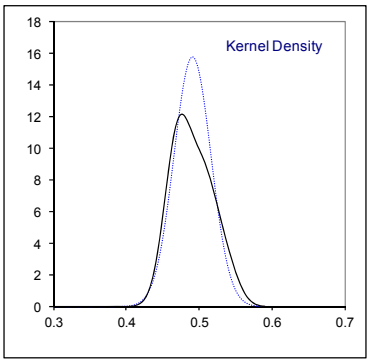


Determination of Methanol on sample #17003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.511		0.69	
311	D5441	0.47		-0.69	
312	D5441	0.48		-0.35	
323	D5441	0.54		1.67	
334	D5441	0.50		0.32	
343	D5441	0.476		-0.49	
347	D5441	0.4620		-0.96	
555		-----		-----	
657	D5441	0.524		1.13	
963		-----		-----	
1201		-----		-----	
1221	D5441	0.5095		0.64	
1251	D5441	0.477		-0.45	
1530	D5441	0.223	G(0.01)	-9.00	
1728		-----		-----	
1788	D5441	0.47		-0.69	
1866	D5441	0.458		-1.09	
1940	D5441	0.499		0.29	
3166		-----		-----	

normality	OK
n	13
outliers	1
mean (n)	0.4905
st.dev. (n)	0.02538
R(calc.)	0.0711
R(D5441:98)	0.0832



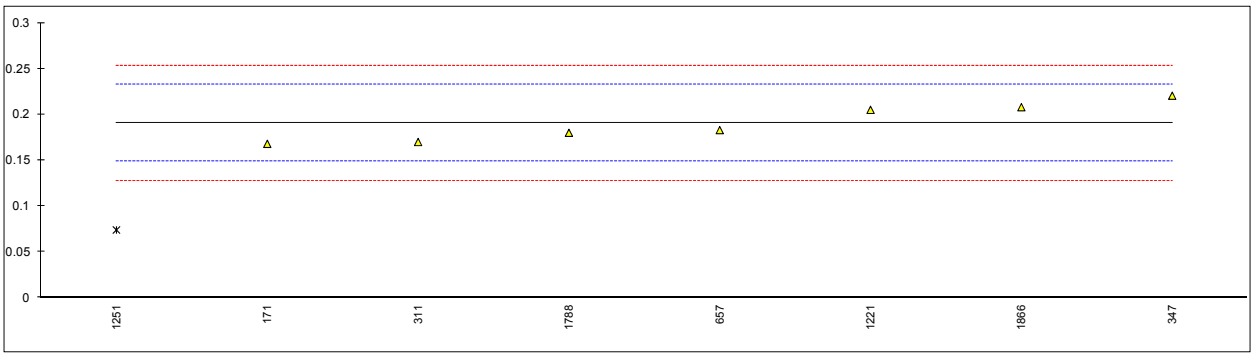


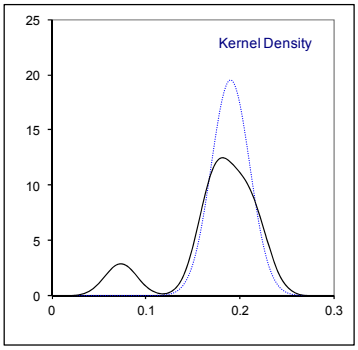
Determination of Diisobutylene (=sum 5 DIB*) on sample #17003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.168		-1.08	
311	D5441	0.17		-0.99	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343		-----		-----	
347	D5441	0.2205		1.43	
555		-----		-----	
657	D5441	0.183		-0.37	
963		-----		-----	
1201		-----		-----	
1221	D5441	0.2051		0.69	
1251	D5441	0.074	G(0.05)	-5.58	
1530		-----		-----	
1728		-----		-----	
1788	D5441	0.18		-0.51	
1866	D5441	0.208		0.83	
1940		-----		-----	
3166		-----		-----	

normality	unknown
n	7
outliers	1
mean (n)	0.1907
st.dev. (n)	0.02046
R(calc.)	0.0573
R(D5441:98)	0.0586

* Sum of 5 DIB: 2,4,4-Trimethyl-1-pentene; 2,4,4-Trimethyl-2-pentene; 2,3,4-Trimethyl-2-pentene; 3,4,4-Trimethyl-1-pentene and 3,5-Dimethyl-1-hexene.

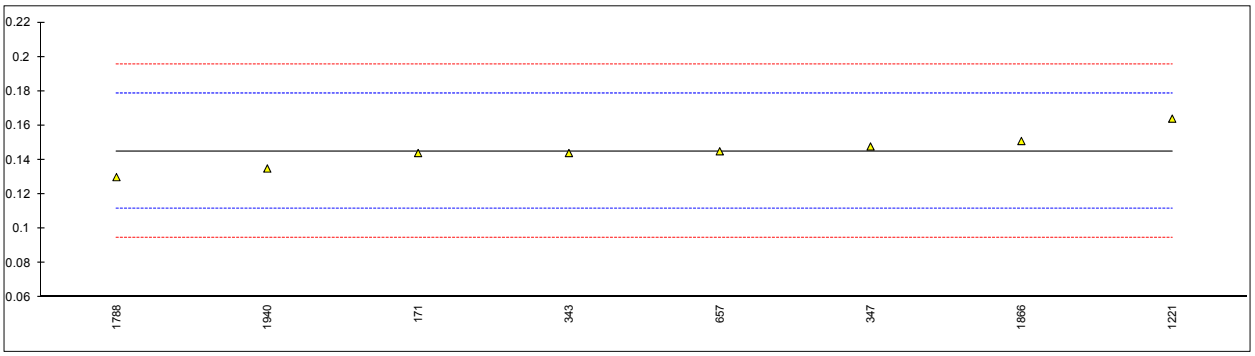


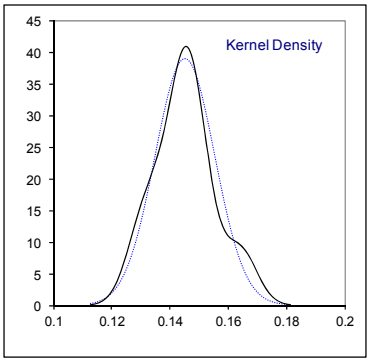


Determination of 2,4,4-Trimethyl-1-pentene on sample #17003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.144		-0.07	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	0.144		-0.07	
347	D5441	0.1478		0.16	
555		-----		-----	
657	D5441	0.145		-0.01	
963		-----		-----	
1201		-----		-----	
1221	D5441	0.1640		1.12	
1251		-----		-----	
1530		-----		-----	
1728		-----		-----	
1788	D5441	0.13		-0.90	
1866	D5441	0.151		0.35	
1940	D5441	0.135		-0.60	
3166		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	0.1451
st.dev. (n)	0.01022
R(calc.)	0.0286
R(D5441:98)	0.0471

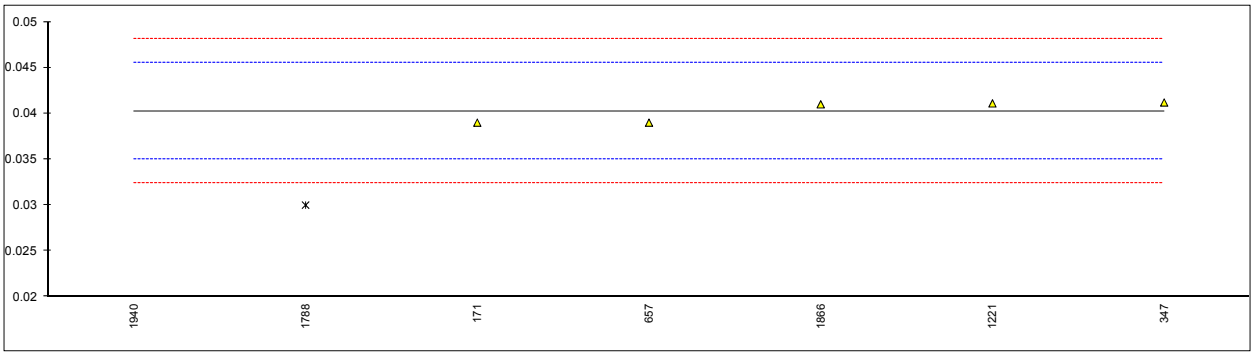




Determination of 2,4,4-Trimethyl-2-pentene on sample #17003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.039		-0.48	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	<0.01		<-11.59	Possibly a false negative test result?
347	D5441	0.0412		0.36	
555		-----		-----	
657	D5441	0.039		-0.48	
963		-----		-----	
1201		-----		-----	
1221	D5441	0.0411		0.32	
1251		-----		-----	
1530		-----		-----	
1728		-----		-----	
1788	D5441	0.03	G(0.01)	-3.93	
1866	D5441	0.041		0.28	
1940	D5441	0.0073	G(0.01)	-12.62	
3166		-----		-----	

normality	unknown
n	5
outliers	2
mean (n)	0.0403
st.dev. (n)	0.00115
R(calc.)	0.0032
R(Horwitz)	0.0073



Determination of other individual diisobutylenes on sample #17003; results in %M/M

lab	method	243T2P *)	mark	z(targ)	344T1P *)	mark	z(targ)	35D1H *)	mark	z(targ)
171	D5441	<0.02		-----	<0.02		-----	<0.02		-----
311		-----		-----	-----		-----	-----		-----
312		-----		-----	-----		-----	-----		-----
323		-----		-----	-----		-----	-----		-----
334		-----		-----	-----		-----	-----		-----
343	D5441	0.052	**f+?**	-----	-----		-----	-----		-----
347	D5441	<0.02		-----	<0.02		-----	<0.02		-----
555		-----		-----	-----		-----	-----		-----
657	D5441	<0.02		-----	<0.02		-----	<0.02		-----
963		-----		-----	-----		-----	-----		-----
1201		-----		-----	-----		-----	-----		-----
1221		-----		-----	-----		-----	-----		-----
1251		-----		-----	-----		-----	-----		-----
1530		-----		-----	-----		-----	-----		-----
1728		-----		-----	-----		-----	-----		-----
1788	D5441	0.00		-----	0.02		-----	0.00		-----
1866		-----		-----	-----		-----	-----		-----
1940		-----		-----	-----		-----	-----		-----
3166		-----		-----	-----		-----	-----		-----
	normality	n.a.			n.a.			n.a.		
	n	4			4			4		
	outliers	0			0			0		
	mean (n)	<0.02			<0.02			<0.02		
	st.dev. (n)	n.a.			n.a.			n.a.		
	R(calc.)	n.a.			n.a.			n.a.		
	R(lit.)	n.a.			n.a.			n.a.		

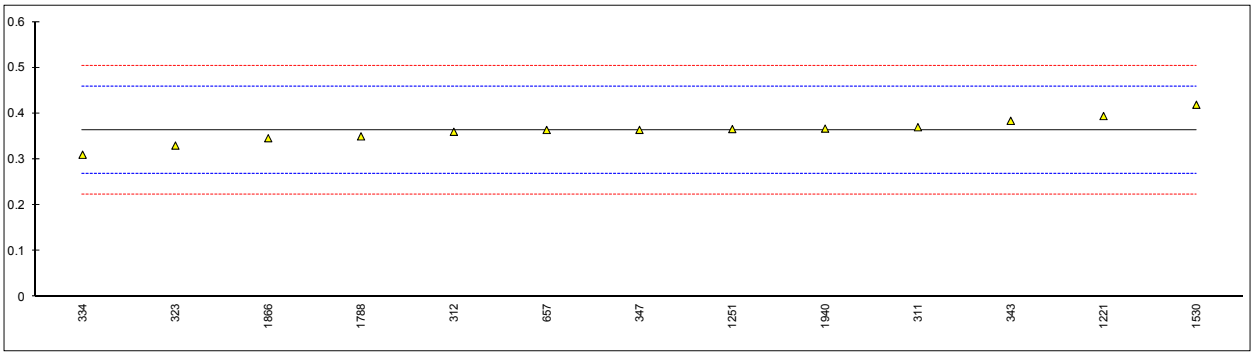
F+?= Possibly a false positive test result?

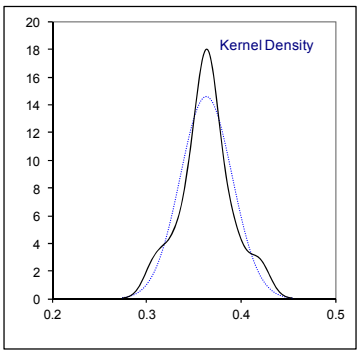
*) 243T2P = 2,4,3-Trimethyl-2-pentene
*) 344T1P = 3,4,4-Trimethyl-1-pentene
*) 35D1H = 3,5-Dimethyl-1-hexene

Determination of Tert-Butanol on sample #17003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	<0.02		<-7.28	Possibly a false negative test result?
311	D5441	0.37		0.14	
312	D5441	0.36		-0.07	
323	D5441	0.33		-0.71	
334	D5441	0.31		-1.13	
343	D5441	0.384		0.44	
347	D5441	0.3642		0.02	
555		-----		-----	
657	D5441	0.364		0.01	
963		-----		-----	
1201		-----		-----	
1221	D5441	0.3944		0.66	
1251	D5441	0.366		0.05	
1530	D5441	0.419		1.18	
1728		-----		-----	
1788	D5441	0.35		-0.28	
1866	D5441	0.346		-0.37	
1940	D5441	0.367		0.08	
3166		-----		-----	

normality	suspect
n	13
outliers	0
mean (n)	0.3634
st.dev. (n)	0.02739
R(calc.)	0.0767
R(D5441:98)	0.1320

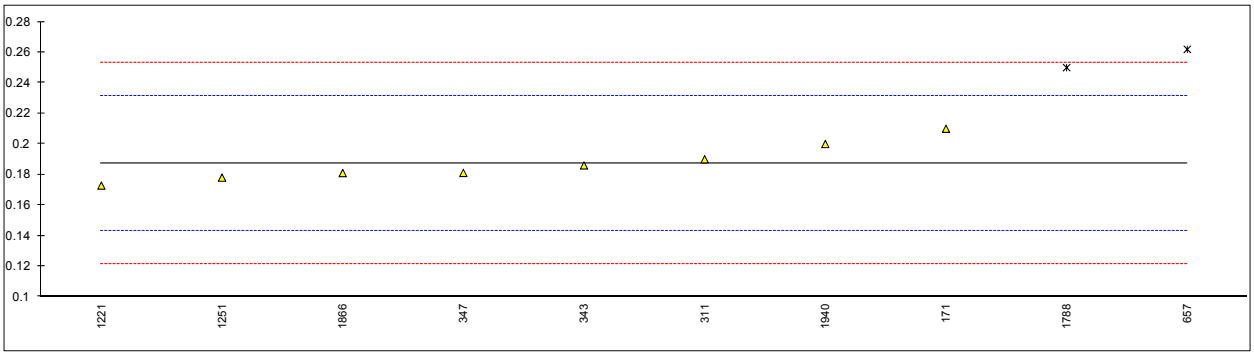


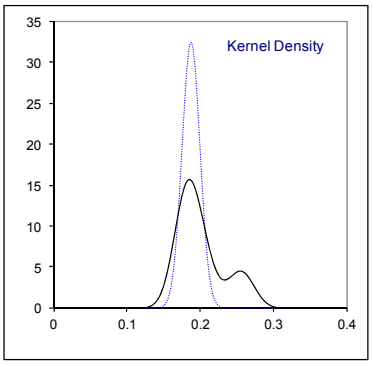


Determination of Hydrocarbons C4 on sample #17003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.210		1.03	
311	D5441	0.19		0.12	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	0.186		-0.06	
347	D5441	0.1811		-0.29	
555		-----		-----	
657	D5441	0.262	DG(0.05)	3.40	
963		-----		-----	
1201		-----		-----	
1221	D5441	0.1728		-0.66	
1251	D5441	0.178		-0.43	
1530		-----		-----	
1728		-----		-----	
1788	D5441	0.25	DG(0.05)	2.85	
1866	D5441	0.181		-0.29	
1940	D5441	0.20		0.58	
3166		-----		-----	

normality	OK
n	8
outliers	2
mean (n)	0.1874
st.dev. (n)	0.01231
R(calc.)	0.0345
R(D5441:98)	0.0615

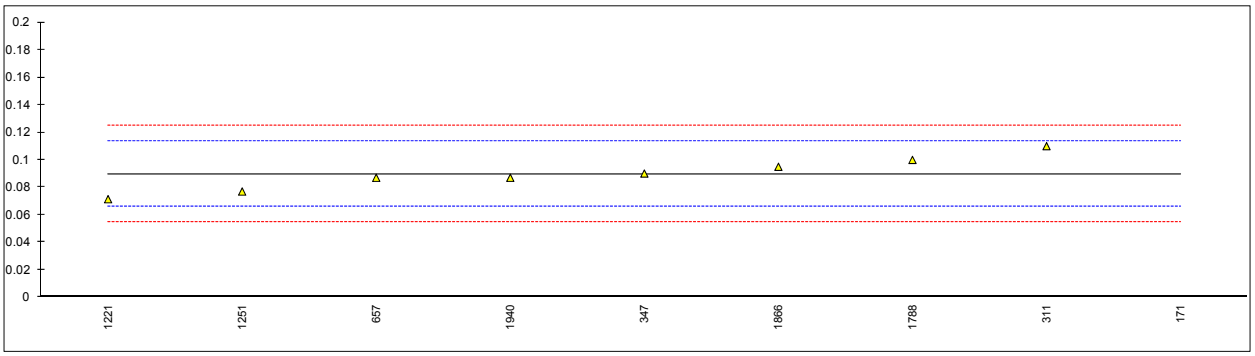


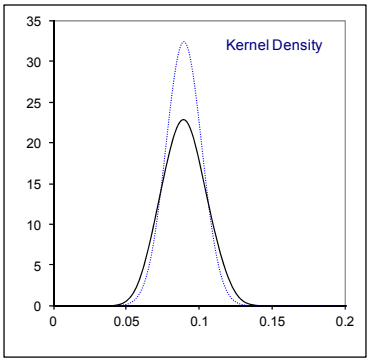


Determination of Hydrocarbons C5 on sample #17003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.368	C,G(0.01)	23.63	first reported: 98.60
311	D5441	0.11		1.72	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	<0.01		<-6.77	Possibly a false negative test result?
347	D5441	0.0901		0.04	
555		-----		-----	
657	D5441	0.087		-0.23	
963		-----		-----	
1201		-----		-----	
1221	D5441	0.0714		-1.55	
1251	D5441	0.077		-1.08	
1530		-----		-----	
1728		-----		-----	
1788	D5441	0.10		0.88	
1866	D5441	0.095		0.45	
1940	D5441	0.087		-0.23	
3166		-----		-----	

normality	OK
n	8
outliers	1
mean (n)	0.0897
st.dev. (n)	0.01229
R(calc.)	0.0344
R(D5441:98)	0.0330

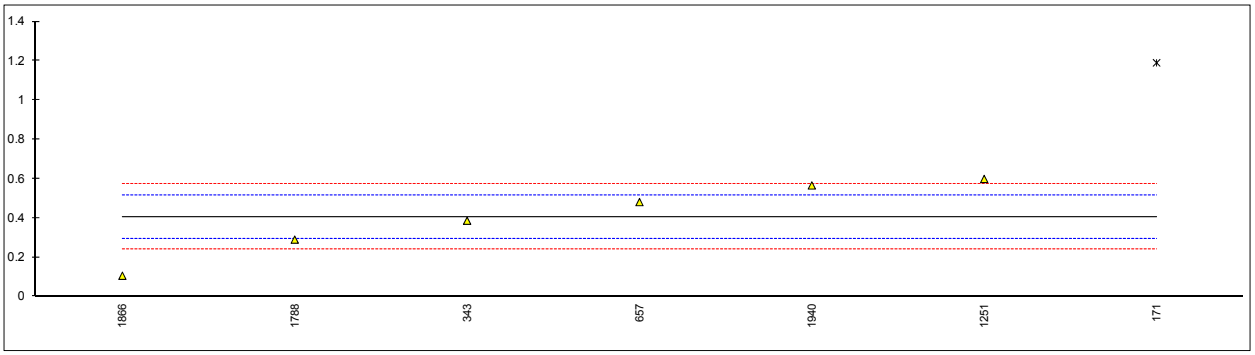




Determination of Other Impurities on sample #17003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	1.19	G(0.05)	14.11	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	0.386		-0.34	
347		-----		-----	
555		-----		-----	
657	D5441	0.481		1.37	
963		-----		-----	
1201		-----		-----	
1221		-----		-----	
1251	D5441	0.599		3.49	
1530		-----		-----	
1728		-----		-----	
1788	D5441	0.29		-2.06	
1866	D5441	0.106		-5.37	
1940	D5441	0.566		2.90	
3166		-----		-----	

normality	unknown
n	6
outliers	1
mean (n)	0.4047
st.dev. (n)	0.18564
R(calc.)	0.5198
R(Horwitz (n=9))	0.1558



APPENDIX 2

Number of participating laboratories per country

1 lab in BELGIUM
1 lab in BRAZIL
1 lab in FRANCE
1 lab in GERMANY
4 labs in NETHERLANDS
1 lab in PORTUGAL
1 lab in ROMANIA
3 labs in SAUDI ARABIA
1 lab in SINGAPORE
2 labs in SPAIN
2 labs in UNITED STATES OF AMERICA
1 lab in VENEZUELA

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test 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's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
2 ASTM E178:89
3 ASTM E1301:89
4 ISO 5725:86
5 ISO 5725, parts 1-6, 1994
6 ISO13528:05
7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9 IP 367:84
10 DIN 38402 T41/42
11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12 J.N. Miller, Analyst, 118, 455, (1993)
13 Analytical Methods Committee Technical brief, No 4 January 2001
14 P.J. Lowthian and M.Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2),165-172, (1983)