Results of Proficiency Test
MTBE
February 2019

CONTENTS

Appendices:

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 2018/2019, it was decided to continue the round robin for the analyzes of MTBE.

In this interlaboratory study for MTBE 18 laboratories in 15 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2019 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 analyzes 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 liter bottle, labelled #19003). 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 June 2018 (iis-protocol, version 3.5). 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 20 liters of MTBE bulk material was obtained from a local supplier. After homogenization, the bulk material was transferred into 39 amber glass bottles of 500 mL and labelled #19003. 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 #19003-1	0.74189	1770
sample #19003-2	0.74190	1760
sample #19003-3	0.74188	1760
sample #19003-4	0.74190	1760
sample #19003-5	0.74190	1760
sample #19003-6	0.74188	1770
sample #19003-7	0.74188	1770
sample #19003-8	0.74190	1780

Table 1: homogeneity test results of subsamples #19003

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility 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)	0.00003	20.8
reference test method	ASTM D4052:18	ASTM E1064:16
0.3 x R (reference test method)	0.00015	58.5

Table 2: evaluation of the repeatabilities of the subsamples #19003

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one bottle of 500 ml, labelled #19003 was sent on January 23, 2019. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYZES

The participants were requested to determine on sample #19003: Appearance, Carbonyls, Density at 15°C, Refractive index at 20°C, Water, Purity (on dry basis), Methanol, and some impurities (Sum of 5 Di-isobutylenes [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-Butanol, Hydrocarbons (C4 and C5) and other impurities.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, 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 evaluations.

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 appropriate 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 reanalyses). 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 June 2018 (iis-protocol, version 3.5).
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. In this PT, the criterion of ISO13528, paragraph 9.2.1 was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2   GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as 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:

$$| z | < 1 \quad \text{good}$$
$$1 < \quad | z | < 2 \quad \text{satisfactory}$$
$$2 < \quad | z | < 3 \quad \text{questionable}$$
$$3 < \quad | z | \quad \text{unsatisfactory}$$

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 due to problems at customs. All participants reported results, but not all were able to report all analyzes requested.
In total 18 reporting laboratories submitted 158 numerical test results. Observed were 11 outlying test results, which is 7.0%. 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", "suspect" or "unknown". 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 original data. 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 these tests 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 #19003, which is pass or clear and bright.

Carbonyls: Two laboratories reported a test result, therefore, no z-scores were calculated.

Density at 15°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D4052:18.

Refractive Index: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not 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 E1064:16.

Purity: 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(2017).

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(2017).

Diisobutylene (=sum 5 DIB): This determination is problematic. No statistical outliers were
                  observed. However, the calculated reproducibility is not in agreement with
                  the requirements of ASTM D5441:98(2017).

2,4,4-Trimethyl-1-pentene: This determination was problematic for some participants. Three
                  statistical outliers were observed. However, the calculated reproducibility
                  after rejection of the statistical outliers is in agreement with the requirements
                  of ASTM D5441:98(2017).

2,4,4-Trimethyl-2-pentene: This determination may be problematic for some participants. Two
                  statistical outliers were observed. However, the calculated reproducibility
                  after rejection of the statistical outliers is in agreement with the estimated
                  reproducibility using the Horwitz equation.

Other DIB: It should be noticed that for the other DIB, test method D5441:98(2017) is
                  applicable for concentrations >0.02 %M/M. Only few numerical test results
                  were reported. Therefore, no z-scores were calculated.

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(2017).

Hydrocarbons C4: This determination was problematic. One statistical outlier was observed.
                  The calculated reproducibility after rejection of the statistical outlier is not in
                  agreement with the requirements of ASTM D5441:98(2017).

Hydrocarbons C5: This determination was very problematic. No statistical outliers were
                  observed. However, the calculated reproducibility is not at all in agreement
                  with the requirements of ASTM D5441:98(2017).

Other Impurities: Other impurities may be all components listed in table 3 of D5441:98(2017) and not evaluated separately in this proficiency test.

ASTM D5441:98(2017) 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 may be 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 the reproducibility as found for the group of participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu the ASTM standards) are presented in the next table.

	unit	n	average	2.8 * sd	R (lit)
Appearance		14	Pass	n.a.	n.a.
Carbonyls	µg/g	2	220.8	n.a.	n.a.
Density at 15°C	kg/L	16	0.7471	0.0002	0.0005
Refractive Index at 20°C		5	1.3698	0.0012	0.0005
Water	mg/kg	17	1750	111	194
Purity (on dry basis)	%M/M	16	98.236	0.288	0.404
Methanol	%M/M	16	0.439	0.058	0.081
Diisobutylene (=sum of 5)	%M/M	8	0.232	0.079	0.069
2,4,4-Trimethyl-1-pentene	%M/M	8	0.170	0.033	0.053
2,4,4-Trimethyl-2-pentene	%M/M	6	0.045	0.008	0.008
tert-Butanol	%M/M	16	0.247	0.045	0.132
Hydrocarbons C4	%M/M	12	0.052	0.022	0.018
Hydrocarbons C5	%M/M	12	0.086	0.074	0.032
Other impurities	%M/M	6	0.483	0.243	0.181

Table 3: reproducibilities of tests on sample #19003

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 reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2019 WITH PREVIOUS PTS

	February 2019	February 2017	February 2015	February 2013	February 2012
Number of reporting labs	18	16	17	16	17
Number of results reported	158	132	159	154	178
Statistical outliers	11	10	13	16	16
Percentage outliers	7.0%	7.6%	8.2%	10.4%	9.0%

Table 4: comparison with previous proficiency tests

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

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

Determination	February 2019	February 2017	February 2015	February 2013	February 2012
Carbonyls	n.e.	n.e.	n.e.	--	n.e.
Density at 15°C	++	++	++	++	+
Refractive Index at 20°C	--	+	+	+	+/-
Water	++	+	++	--	+/-
Purity	+	+	--	+	--
Methanol	+	+	+/-	--	-
Diisobutylene (=sum of 5)	-	+/-	+/-	-	+
2,4,4-Trimethyl-1-pentene	+	+	+	+/-	++
2,4,4-Trimethyl-2-pentene	+/-	++	+	-	+
tert-Butanol	++	++	++	++	++
Hydrocarbons C4	-	++	--	-	+/-
Hydrocarbons C5	--	+/-	+	++	--

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
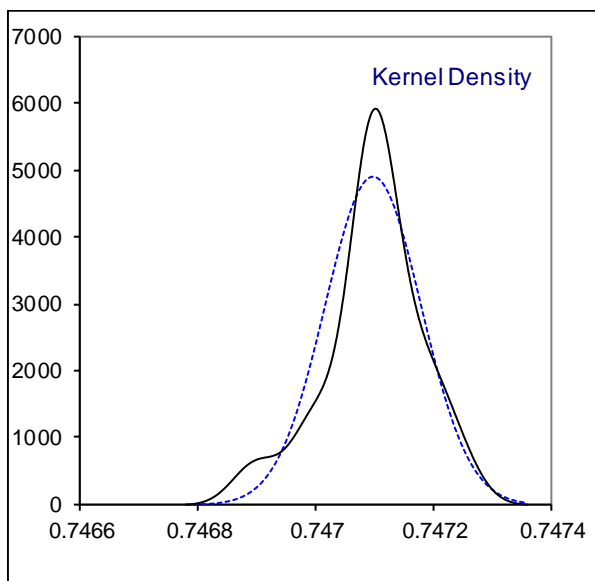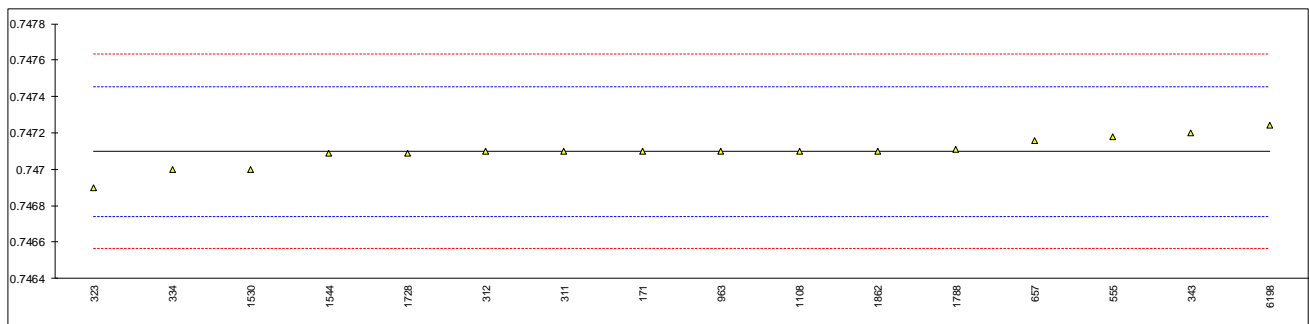Determination of Appearance on sample #19003;

lab	method	value	mark	z(targ)	remarks
171	E2680	Pass		-----	
311		-----		-----	
312	Visual	Br&Cl		-----	
323	E2680	clear & bright		-----	
334	Visual	Clear & Bright		-----	
343	E2680	Pass		-----	
555	Visual	Pass		-----	
657	E2680	Pass		-----	
963	E2680	Pass		-----	
1108		-----		-----	
1120	E2680	PASS		-----	
1530		-----		-----	
1544	Visual	Cl&Br		-----	
1728	Visual	Clear and bright		-----	
1788	Visual	Clear		-----	
1862	Visual	Cl & Br		-----	
1940		-----		-----	
6198	D4176	Pass		-----	
	n	14			
	mean (n)	Pass			

Determination of Carbonyls on sample #19003; results in µg/g

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343		-----		-----	
555		-----		-----	
657	E411	217.5084		-----	
963	E411	224.0		-----	
1108		-----		-----	
1120		-----		-----	
1530		-----		-----	
1544		-----		-----	
1728		-----		-----	
1788		-----		-----	
1862		-----		-----	
1940		-----		-----	
6198		-----		-----	
	n	2			
	mean (n)	220.754			

Determination of Density at 15°C on sample #19003; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.7471		0.01	
311	D4052	0.7471		0.01	
312	D4052	0.7471		0.01	
323	D4052	0.7469		-1.11	
334	D4052	0.7470		-0.55	
343	D4052	0.7472		0.57	
555	D4052	0.74718		0.46	
657	D4052	0.74716		0.35	
963	D4052	0.7471		0.01	
1108	D4052	0.74710		0.01	
1120		-----		-----	
1530	D4052	0.7470		-0.55	
1544	D4052	0.74709		-0.05	
1728	D4052	0.74709		-0.05	
1788	D4052	0.74711		0.07	
1862	D4052	0.74710		0.01	
1940		-----		-----	
6198	D4052	0.74724		0.79	

normality	suspect
n	16
outliers	0
mean (n)	0.74710
st.dev. (n)	0.000082
R(calc.)	0.00023
st.dev.(D4052:18)	0.000179
R(D4052:18)	0.00050

Determination of Refractive Index at 20°C on sample #19003;

lab	method	value	mark	z(targ)	remarks
171	D1218	1.3704		3.19	
311		-----		-----	
312		-----		-----	
323	D1218	1.36958		-1.40	
334		-----		-----	
343		-----		-----	
555		-----		-----	
657	D1218	1.3701		1.51	
963		-----		-----	
1108		-----		-----	
1120		-----		-----	
1530	D1218	1.3694		-2.41	
1544		-----		-----	
1728		1.36967		-0.90	
1788		-----		-----	
1862		-----		-----	
1940		-----		-----	
6198		-----		-----	

normality	unknown
n	5
outliers	0
mean (n)	1.36983
st.dev. (n)	0.000409
R(calc.)	0.00115
st.dev.(D1218:12)	0.000179
R(D1218:12)	0.0005
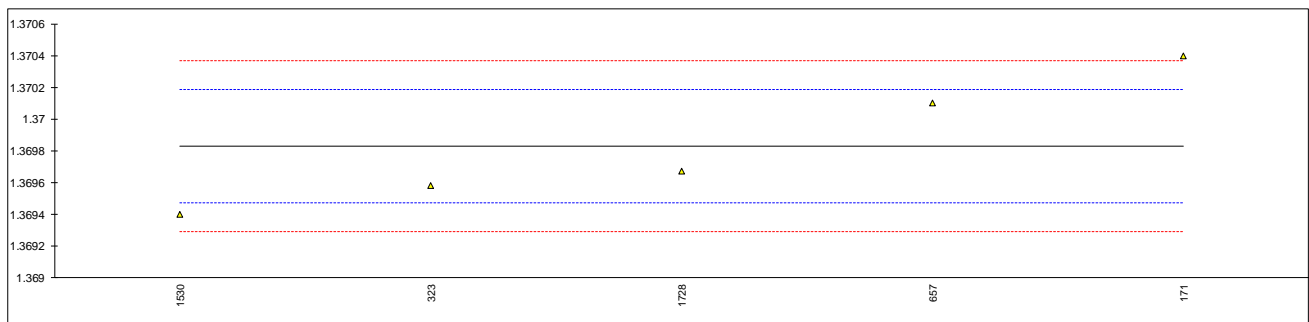
Determination of Water on sample #19003; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	1718		-0.46	
311	E1064	1775		0.36	
312	E1064	1700		-0.72	
323	D1364	1705		-0.65	
334	D1364	1651		-1.42	
343	E1064	1737		-0.18	
555	D4017	1775.27		0.37	
657	E1064	1739.85		-0.14	
963	D1364	1770		0.29	
1108	ISO12937	1805		0.80	
1120	D1364	1803		0.77	
1530	D1364	1746.5		-0.05	
1544	E1064	1758		0.12	
1728	E203	1753		0.05	
1788	D6304	1747.7		-0.03	
1862	D1364	1790		0.58	
1940		-----		-----	
6198	E1064	1772		0.32	

normality	OK
n	17
outliers	0
mean (n)	1749.78
st.dev. (n)	39.760
R(calc.)	111.33
st.dev.(E1064:16)	69.383
R(E1064:16)	194.27

Kernel Density
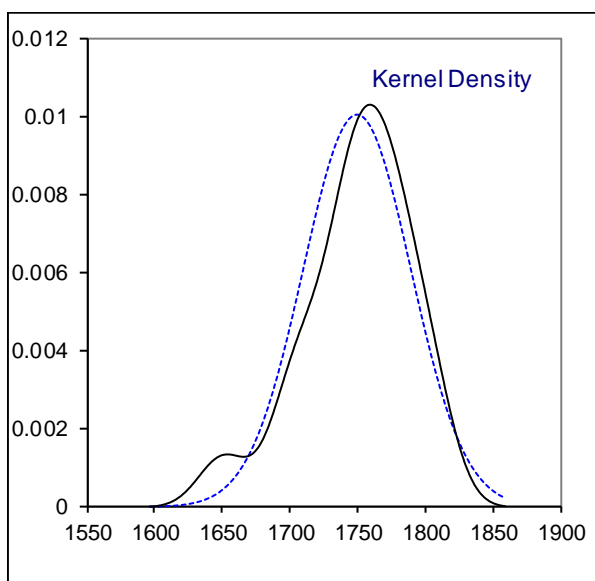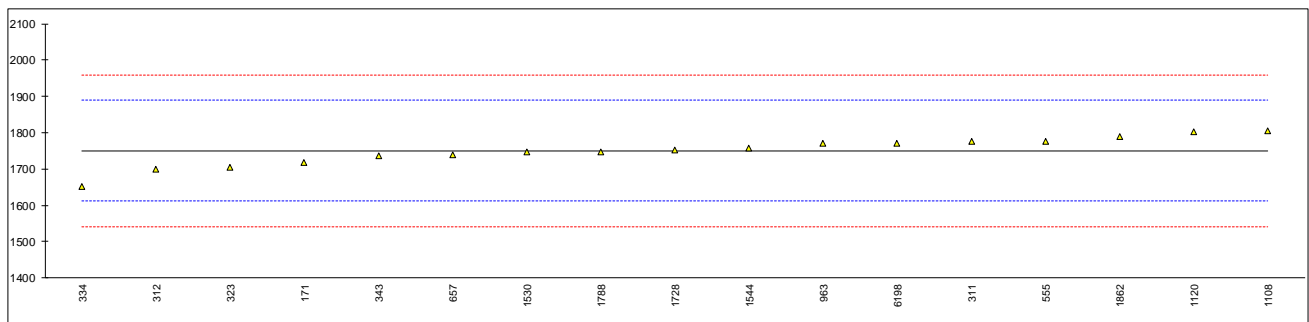
Determination of Purity by GC on dry basis on sample #19003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	98.164		-0.50	
311	D5441	98.30		0.45	
312	D5441	98.157		-0.55	
323	D5441	98.32		0.58	
334	D5441	98.36		0.86	
343	D5441	98.090		-1.01	
555	D5441	98.09		-1.01	
657	D5441	98.1994		-0.25	
963	D5441	98.20		-0.25	
1108	D5441	98.38		1.00	
1120	D5441	98.262		0.18	
1530	D5441	99.168	G(0.01)	6.46	
1544	D5441	98.141		-0.66	
1728		-----		-----	
1788	D5441	98.4432		1.44	
1862	D5441	98.21		-0.18	
1940	D5441	98.20		-0.25	
6198	SH/T1550	98.255		0.13	

normality	OK
n	16
outliers	1
mean (n)	98.2357
st.dev. (n)	0.10293
R(calc.)	0.2882
st.dev.(D5441:98)	0.14426
R(D5441:98)	0.4039
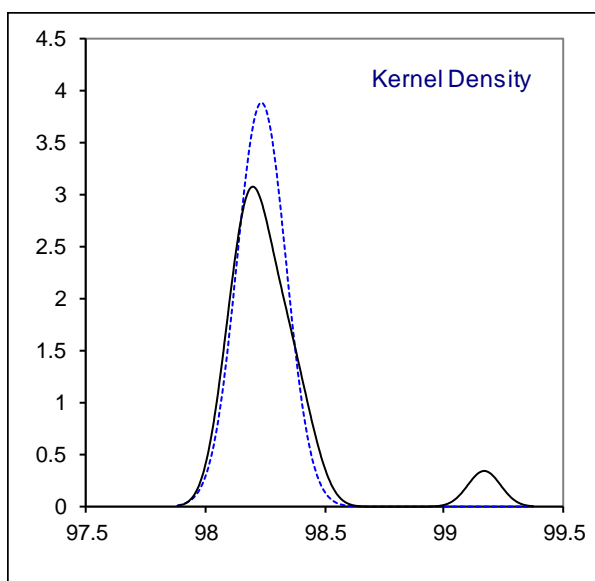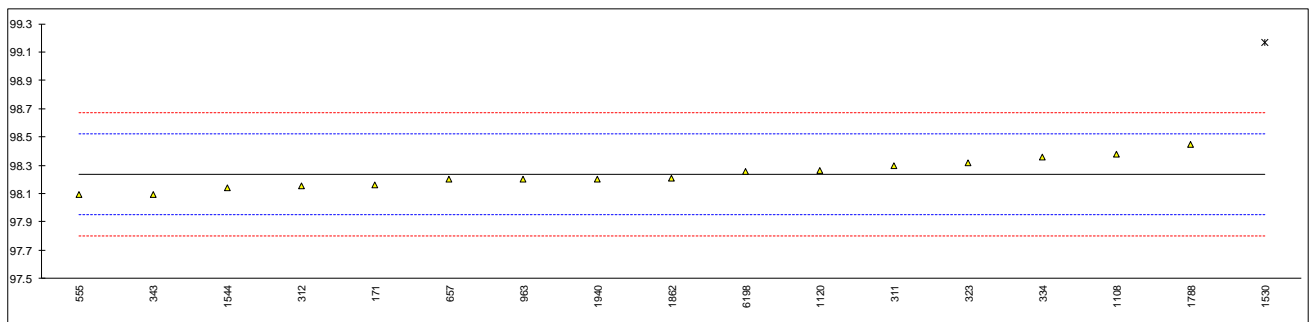
Determination of Methanol on sample #19003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.427		-0.40	
311	D5441	0.43		-0.30	
312	D5441	0.437		-0.06	
323	D5441	0.44		0.05	
334	D5441	0.46		0.74	
343	D5441	0.424		-0.51	
555	D5441	0.15	G(0.01)	-9.99	
657	D5441	0.4515		0.45	
963	D5441	0.457		0.64	
1108	D5441	0.386		-1.82	
1120	D5441	0.446		0.26	
1530	D5441	0.420		-0.64	
1544	D5441	0.476		1.29	
1728		-----		-----	
1788	D5441	0.4242		-0.50	
1862	D5441	0.454		0.53	
1940	D5441	0.449		0.36	
6198	SH/T1550	0.436		-0.09	

|	
normality	suspect
n	16
outliers	1
mean (n)	0.4386
st.dev. (n)	0.02084
R(calc.)	0.0584
st.dev.(D5441:98)	0.02890
R(D5441:98)	0.0809
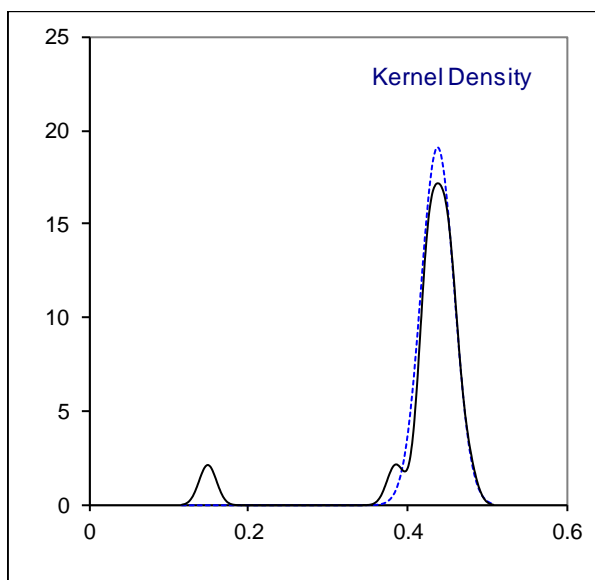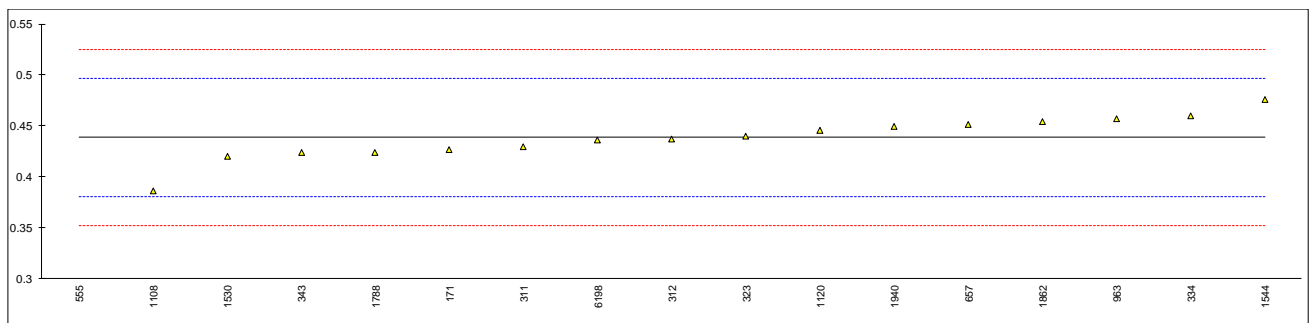
Determination of Diisobutylene (=sum 5 DIB*) on sample #19003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334	D5441	0.20		-1.30	
343		-----		-----	
555	D5441	0.23		-0.09	
657	D5441	0.2331		0.04	
963	D5441	0.21		-0.90	
1108	D5441	0.284		2.10	
1120	D5441	0.215		-0.69	
1530		-----		-----	
1544		-----		-----	
1728		-----		-----	
1788	D5441	0.2628		1.24	
1862		-----		-----	
1940	D5441	0.222		-0.41	
6198		-----		-----	

|		
normality	unknown	
n	8	
outliers	0	
mean (n)	0.2321	
st.dev. (n)	0.02817	
R(calc.)	0.0789	
st.dev.(D5441:98)	0.02465	
R(D5441:98)	0.0690	

*) 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.
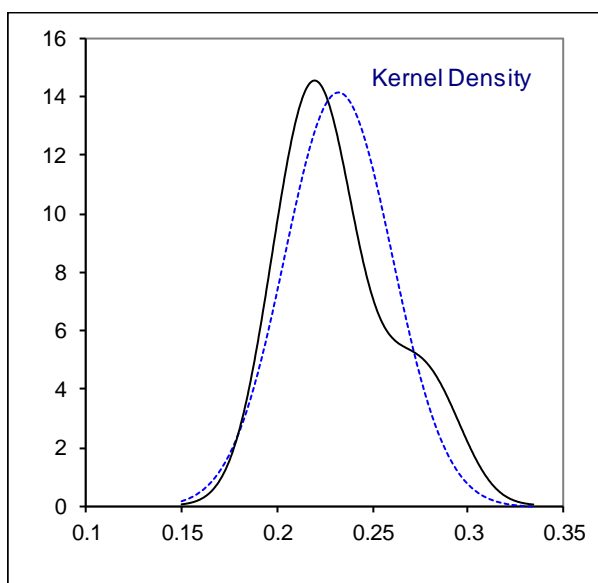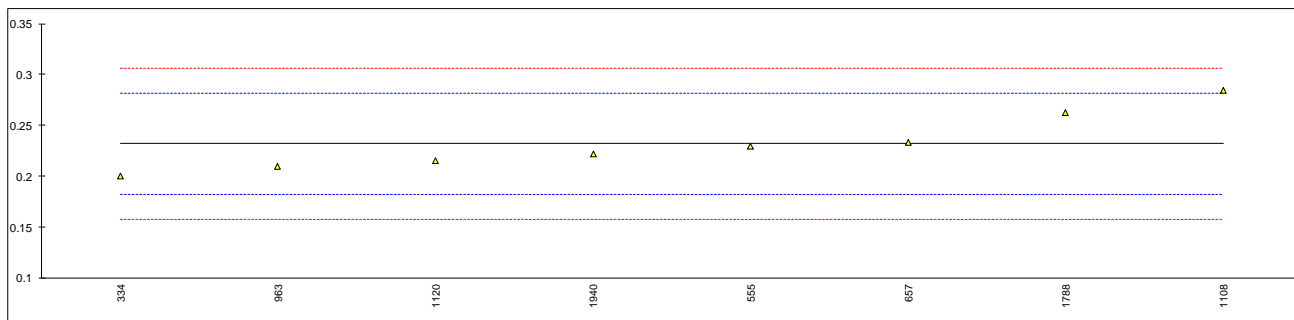
Determination of 2,4,4-Trimethyl-1-pentene on sample #19003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.187		0.91	
311	D5441	0.15		-1.03	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	0.161		-0.45	
555		-----		-----	
657	D5441	0.1732		0.19	
963	D5441	0.161		-0.45	
1108	D5441	0.171		0.07	
1120		-----		-----	
1530	D5441	0.041	G(0.01)	-6.76	
1544	D5441	0.180		0.54	
1728		-----		-----	
1788	D5441	0.0418	DG(0.01)	-6.72	
1862	D5441	0.174		0.23	
1940	D5441	0.044	DG(0.01)	-6.60	
6198		-----		-----	

normality	OK
n	8
outliers	3
mean (n)	0.1697
st.dev. (n)	0.01182
R(calc.)	0.0331
st.dev.(D5441:98)	0.01903
R(D5441:98)	0.0533
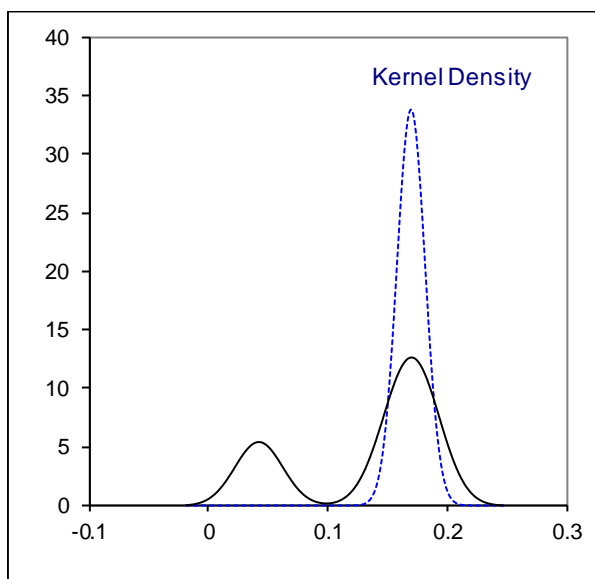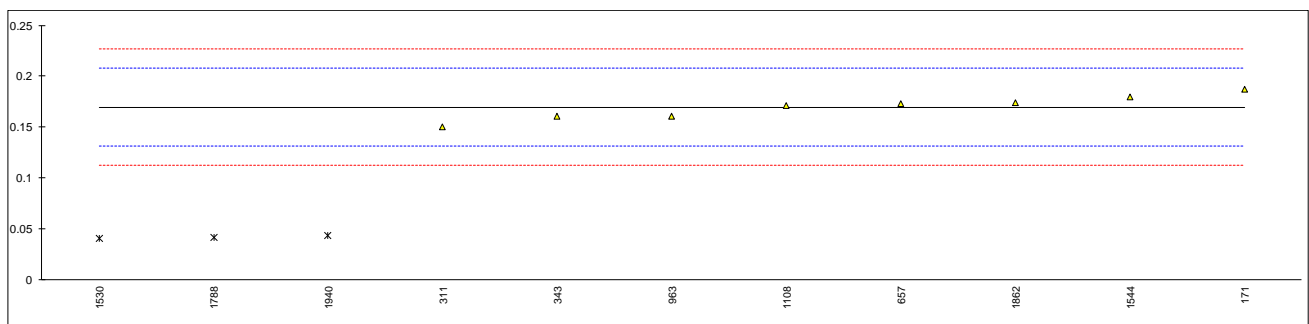
Determination of 2,4,4-Trimethyl-2-pentene on sample #19003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.044		-0.31	
311	D5441	0.04		-1.71	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	0.045		0.03	
555		-----		-----	
657	D5441	0.0464		0.52	
963	D5441	0.046		0.38	
1108	D5441	0.048		1.08	
1120		-----		-----	
1530		-----		-----	
1544		-----		-----	
1728		-----		-----	
1788	D5441	0.0044	DG(0.01)	-14.13	
1862		-----		-----	
1940	D5441	0.004	DG(0.01)	-14.27	
6198		-----		-----	

|	
normality	unknown
n	6
outliers	2
mean (n)	0.0449
st.dev. (n)	0.00275
R(calc.)	0.0077
st.dev.(Horwitz)	0.00287
R(Horwitz)	0.0080
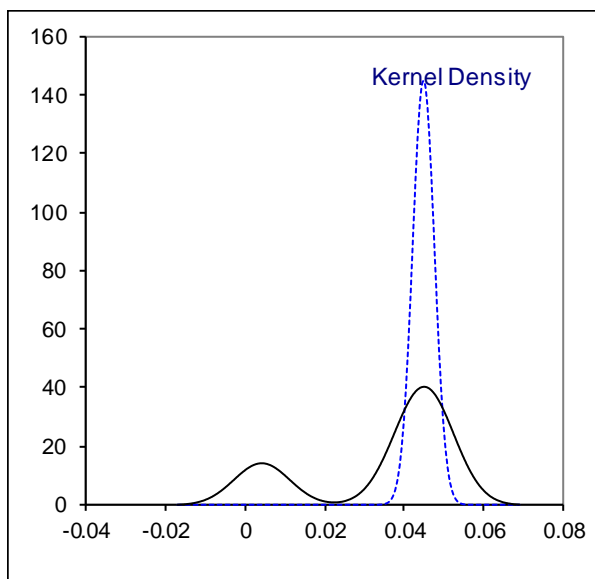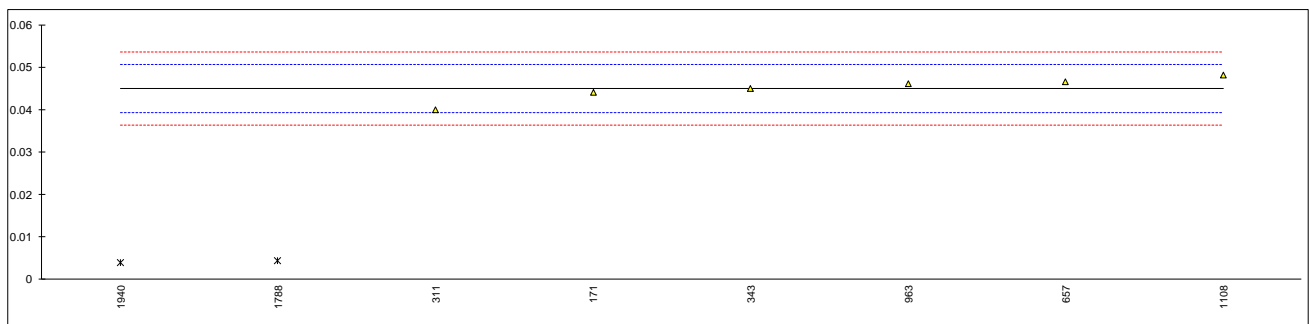
Determination of other individual diisobutylenes on sample #19003; results in %M/M

lab	method	2,3,4-Trimethyl-2-pentene	3,4,4-Trimethyl-1-pentene	3,5-Dimethyl-1-hexene	Remarks
171		-----	-----	-----	
311		-----	-----	-----	
312		-----	-----	-----	
323		-----	-----	-----	
334		-----	-----	-----	
343		-----	-----	-----	
555		-----	-----	-----	
657	D5441	< 0.02	0.0136	< 0.02	
963	D5441	0.014	-----	-----	
1108	D5441	-----	0.065	-----	
1120		-----	-----	-----	
1530		-----	-----	-----	
1544		-----	-----	-----	
1728		-----	-----	-----	
1788	D5441	0.0047	0.0639	0.1481*	*) Possibly a false positive result?
1862		-----	-----	-----	
1940	D5441	0.004	0.012	0.158*	*) Possibly a false positive result?
6198		-----	-----	-----	
	n	4	4	1	
	mean (n)	<0.02	<0.07	<0.02.	

Determination of tert-Butanol on sample #19003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.253		0.14	
311	D5441	0.25		0.07	
312	D5441	0.246		-0.01	
323	D5441	0.25		0.07	
334	D5441	0.21		-0.78	
343	D5441	0.252		0.12	
555	D5441	0.27		0.50	
657	D5441	0.2441		-0.05	
963	D5441	0.249		0.05	
1108	D5441	0.238		-0.18	
1120	D5441	0.284		0.79	
1530	D5441	0.238		-0.18	
1544	D5441	0.242		-0.10	
1728		-----		-----	
1788	D5441	0.2291		-0.37	
1862		-----		-----	
1940	D5441	0.249		0.05	
6198	SH/T1550	0.241		-0.12	

normality	not OK
n	16
outliers	0
mean (n)	0.2466
st.dev. (n)	0.01613
R(calc.)	0.0452
st.dev.(D5441:98)	0.04714
R(D5441:98)	0.132

Kernel Density
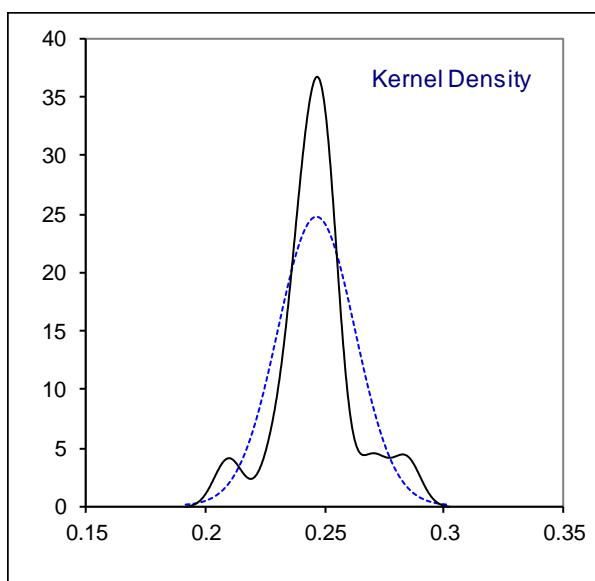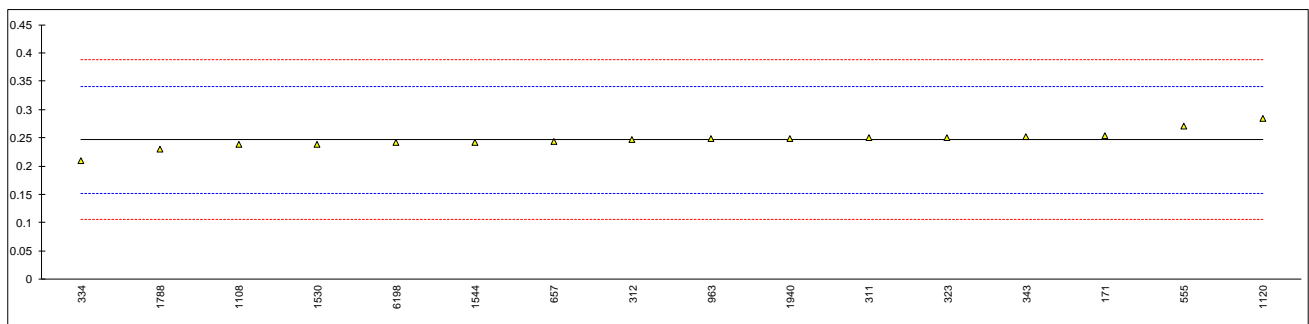
Determination of Hydrocarbons C4 on sample #19003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311	D5441	0.05		-0.29	
312		-----		-----	
323	D5441	0.04		-1.83	
334		-----		-----	
343	D5441	0.097	C,G(0.01)	6.96	first reported: 0.091
555	D5441	0.07		2.80	
657	D5441	0.0487		-0.49	
963	D5441	0.05		-0.29	
1108	D5441	0.051		-0.13	
1120	D5441	0.0419		-1.54	
1530		-----		-----	
1544	D5441	0.055		0.48	
1728		-----		-----	
1788	D5441	0.0578		0.91	
1862	D5441	0.047		-0.75	
1940	D5441	0.054		0.33	
6198	SH/T1550	0.057		0.79	

normality	suspect
n	12
outliers	1
mean (n)	0.0519
st.dev. (n)	0.00790
R(calc.)	0.0221
st.dev.(D5441:98)	0.00649
R(D5441:98)	0.0182

Kernel Density
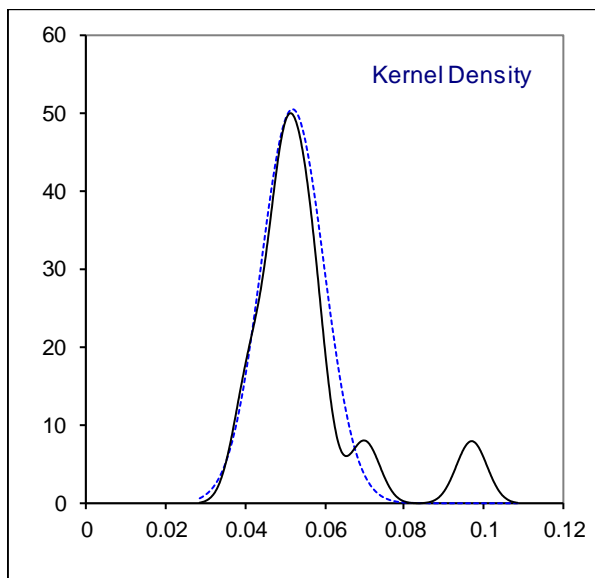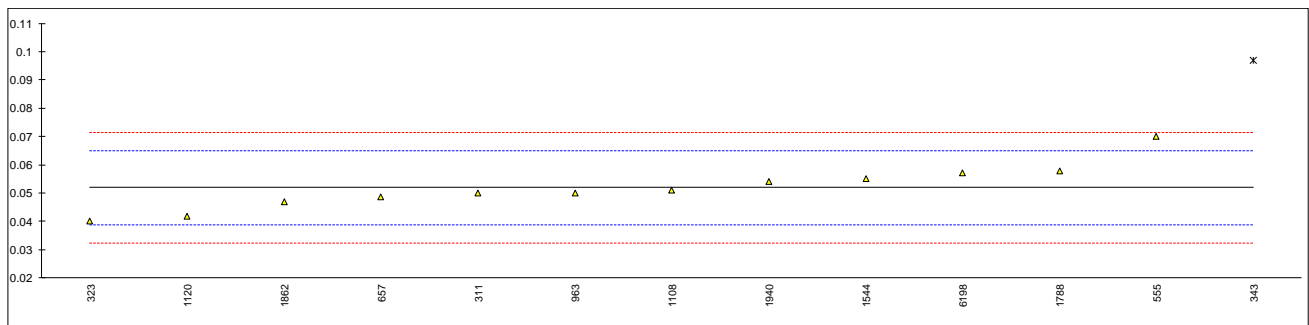
Determination of Hydrocarbons C5 on sample #19003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311	D5441	0.12		3.00	
312		-----		-----	
323	D5441	0.08		-0.50	
334		-----		-----	
343	D5441	0.062	C	-2.07	first reported: 0.021
555	D5441	0.05		-3.12	
657	D5441	0.0695		-1.42	
963	D5441	0.08		-0.50	
1108	D5441	0.109		2.04	
1120	D5441	0.051		-3.04	
1530		-----		-----	
1544	D5441	0.100		1.25	
1728		-----		-----	
1788	D5441	0.1167		2.71	
1862	D5441	0.120		3.00	
1940	D5441	0.070		-1.37	
6198		-----		-----	

normality	OK
n	12
outliers	0
mean (n)	0.0857
st.dev. (n)	0.02642
R(calc.)	0.0740
st.dev.(D5441:98)	0.01142
R(D5441:98)	0.0320
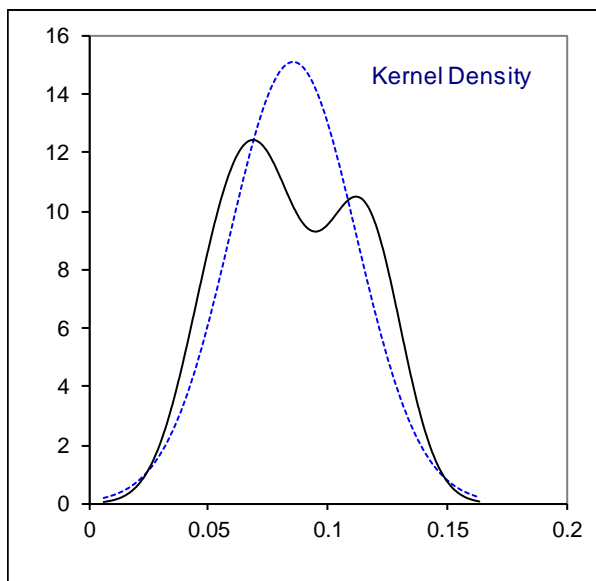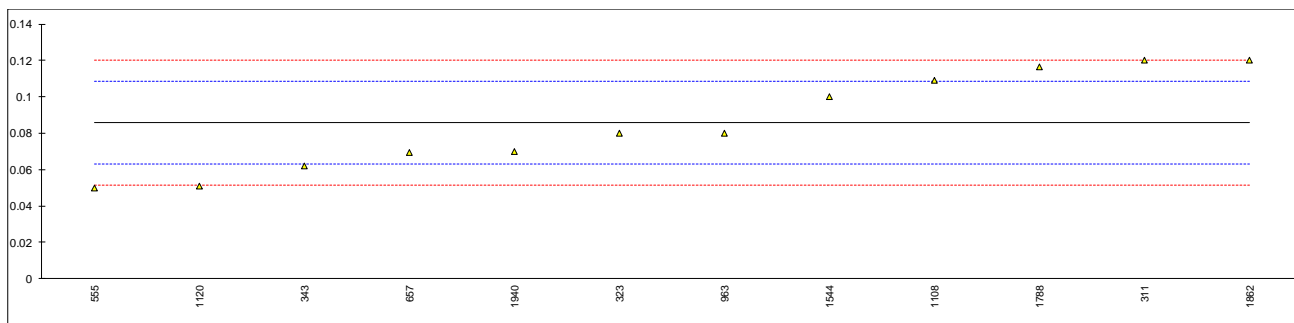
Determination of Other Impurities on sample #19003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343		-----		-----	
555	D5441	0.44		-0.66	
657	D5441	0.5793		1.49	
963	D5441	0.34		-2.21	
1108	D5441	0.551		1.06	
1120	D5441	0.518		0.55	
1530		-----		-----	
1544		-----		-----	
1728		-----		-----	
1788	D5441	0.4678		-0.23	
1862		-----		-----	
1940	D5441	0.93	G(0.05)	6.92	
6198		-----		-----	

normality	unknown
n	6
outliers	1
mean (n)	0.4827
st.dev. (n)	0.08678
R(calc.)	0.2430
st.dev.(Horwitz (n=9))	0.06463
R(Horwitz (n=9))	0.1810
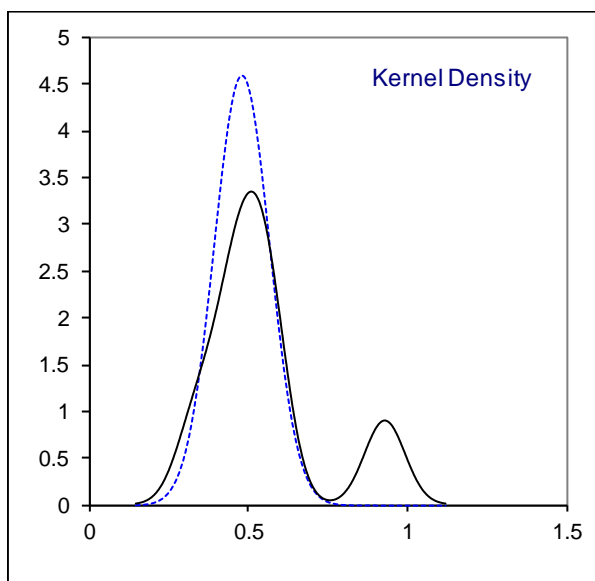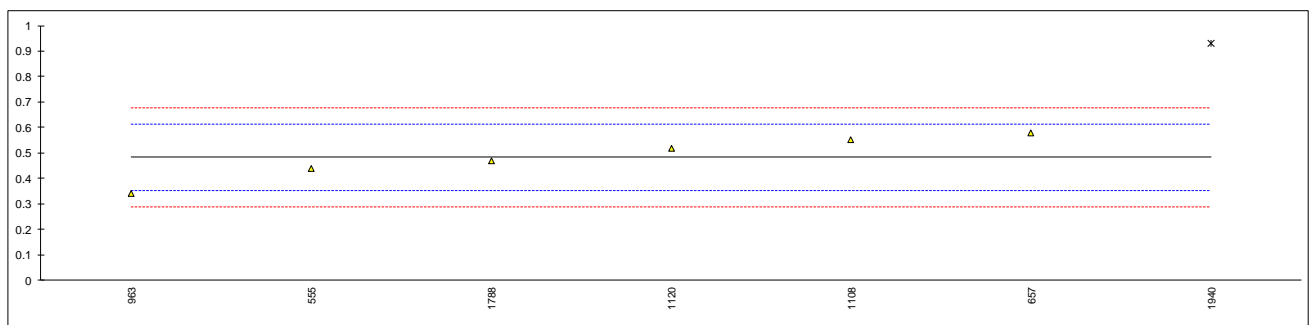
APPENDIX 2

Number of participants per country

1 lab in BELGIUM

1 lab in BRAZIL

1 lab in BULGARIA

1 lab in CHINA, People's Republic

1 lab in FRANCE

1 lab in GERMANY

1 lab in GREECE

2 labs in NETHERLANDS

1 lab in PORTUGAL

1 lab in ROMANIA

1 lab in RUSSIAN FEDERATION

3 labs in SAUDI ARABIA

1 lab in SINGAPORE

1 lab in SPAIN

1 lab in UNITED STATES OF AMERICA

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	= possibly an error in calculations
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, June 2018

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)