Results of Proficiency Test
Methyl-tert-Butylether (MTBE)
February 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 1995 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Methyl Tertiary Butyl Ether (MTBE) every two year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis of Methyl Tertiary Butyl Ether (MTBE).

In this interlaboratory study 16 laboratories in 12 different countries registered for participation. See appendix 3 for the number of participants per country. In this report the results of the MTBE 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 (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one bottle of 0.5L MTBE, labelled #21003.
The 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/IEC17043:2010. 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 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). The protocol is electronically available through the 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

A batch of approximately 27 liters of Methyl Tertiary Butyl Ether (MTBE) was obtained from a supplier. After homogenization 54 amber glass bottles of 0.5L were filled and labelled #21003.
The homogeneity of the subsamples was checked by determination of Density at 20˚C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

	Density at 20°C in kg/L
sample #21003-1	0.74073
sample #21003-2	0.74074
sample #21003-3	0.74073
sample #21003-4	0.74072
sample #21003-5	0.74073
sample #21003-6	0.74073
sample #21003-7	0.74074
sample #21003-8	0.74073

Table 1: homogeneity test results of subsamples #21003

From the above test results the repeatability is calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 20°C in kg/L
r (observed)	0.00002
reference test method	ASTM D4052:18a
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of the subsamples #21003

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

To each of the participating laboratories one sample of 0.5L MTBE labelled #21003 was sent on January 13, 2021. 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 sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine: Appearance, Carbonyls, Density at 15°C, Refractive index at 20°C, Water, Purity by GC 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 reference test methods (when applicable) 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 appendices 1 and 2 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 reanalyzes). 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

4 EVALUATION

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with one week. All participants reported test results, but one participant reported test results after the extended final reporting date. Not all participants were able to report all tests requested.
In total 16 reporting laboratories submitted 142 numerical test results. Observed were 10 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 were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data. The abbreviations, used in these tables, are explained in appendix 4.

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 estimated reproducibility calculated with 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: This determination was not problematic. All labs agreed about the appearance of sample #21003, which is pass or clear and bright, except one.

Carbonyls: Four laboratories reported a test result with a large variation. 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 agreement with the requirements of ASTM D4052:18a

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 problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM E1064:16.

Purity by GC on dry basis: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5441:98(2017).

Methanol: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5441:98(2017).

Diisobutylene (=sum 5 DIB): This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5441:98(2017).

2,4,4-Trimethyl-1-pentene: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5441:98(2017).

2,4,4-Trimethyl-2-pentene: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with 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. The test results are given in appendix 2.

Tert-Butanol: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5441:98(2017).

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

Hydrocarbons C5: 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).

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. The estimated reproducibility calculated with the Horwitz equation based on 9 components is used.
This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation (9 components).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method or as declared by the estimated target reproducibility calculated with Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu the ASTM standards) or estimated with the Horwitz equation are presented in the next table.

	unit	n	average	2.8 * sd	R(target)
Appearance		14	Pass	n.a.	n.a.
Carbonyls	µg/g	4	311	467	(53)
Density at 15°C	kg/L	16	0.7460	0.0002	0.0005
Refractive Index at 20°C		7	1.3693	0.0002	0.0005
Water	mg/kg	15	101	30	16
Purity by GC on dry basis	%M/M	15	98.389	0.363	0.393
Methanol	%M/M	15	0.421	0.082	0.080
Diisobutylene (=sum of 5)	%M/M	6	0.121	0.035	0.041
2,4,4-Trimethyl-1-pentene	%M/M	8	0.099	0.016	0.036
2,4,4-Trimethyl-2-pentene	%M/M	6	0.027	0.005	0.005
tert-Butanol	%M/M	15	0.307	0.101	0.132
Hydrocarbons C4	%M/M	10	0.317	0.143	0.103
Hydrocarbons C5	%M/M	9	0.075	0.044	0.029
Other impurities	%M/M	6	0.323	0.122	0.129

Table 3: reproducibilities of tests on sample #21003

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2021 WITH PREVIOUS PTS

	February 2021	February 2019	February 2017	February 2015	February 2013
Number of reporting laboratories	16	18	16	17	16
Number of test results	142	158	132	159	154
Number of statistical outliers	10	11	10	13	16
Percentage of statistical outliers	7.0%	7.0%	7.6%	8.2%	10.4%

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 to the requirements of the reference test methods. The conclusions are given the following table.

Determination	February 2021	February 2019	February 2017	February 2015	February 2013
Carbonyls	--	n.e.	n.e.	n.e.	--
Density at 15°C	++	++	++	++	++
Refractive Index at 20°C	++	--	+	+	+
Water	-	++	+	++	--
Purity by GC on dry basis	+/-	+	+	--	+

Determination	February 2021	February 2019	February 2017	February 2015	February 2013
Methanol	+/-	+	+	+/-	--
Diisobutylene (=sum of 5 DIB's)	+	-	+/-	+/-	-
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 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

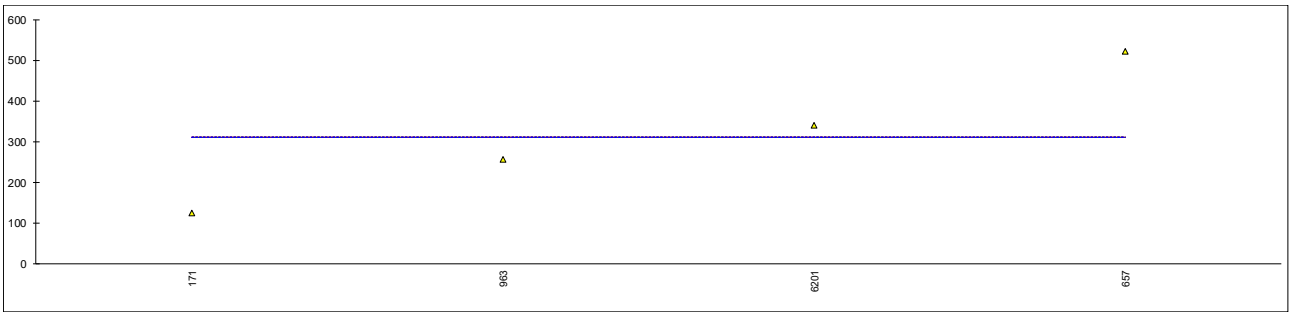
Determination of Appearance on sample #21003;

lab	method	value	mark	z(targ)	remarks
171	E2680	Pass		-----	
311		-----		-----	
312	Visual	Br&Cl		-----	
323	In house	Clear and bright		-----	
334	Visual	clear and bright FFSM		-----	
343	E2680	pass		-----	
555	Visual	Limpid liquid and isent of impurities		-----	
657	E2680	Pass		-----	
963	E2680	Pass		-----	
1457	Visual	pass		-----	
1530	Visual	C & B		-----	
1728	Visual	Clear and bright		-----	
1788	Visual	CLEAR		-----	
6198	D4176	Pass		-----	
6201	Visual	Pass		-----	
6262	Visual	Bright & clear		-----	
	n	14			
	mean (n)	Pass			

Determination of Carbonyls on sample #21003; results in µg/g

lab	method	value	mark	z(targ)	remarks
171	E411	124.63		-----	
311		-----		-----	
312		-----		-----	
323	E411	>50		-----	
334		-----		-----	
343		-----		-----	
555		-----		-----	
657	E411	522.678		-----	
963	E411	257.0		-----	
1457		-----		-----	
1530		-----		-----	
1728		-----		-----	
1788		-----		-----	
6198		-----		-----	
6201	E411	340		-----	
6262		-----		-----	

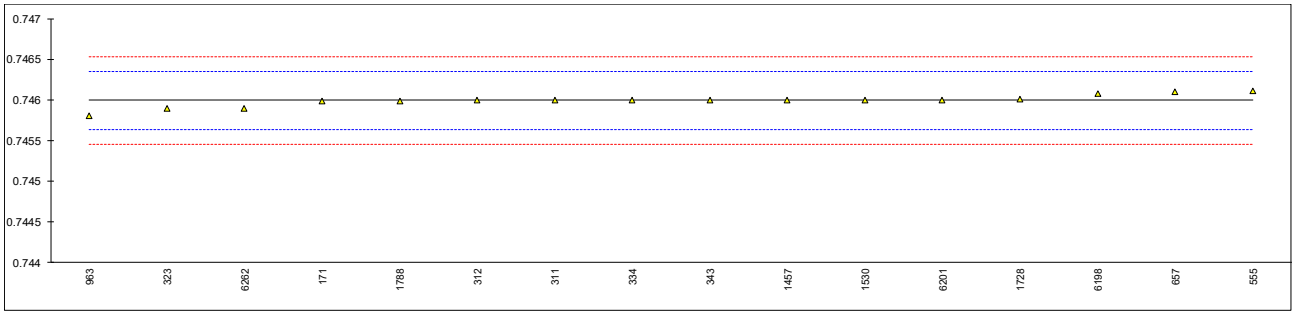
normality	unknown
n	4
outliers	0
mean (n)	311.08
st.dev. (n)	166.632
R(calc.)	466.578
st.dev.(E411:17)	(18.887)
R(E411:17)	(52.88)

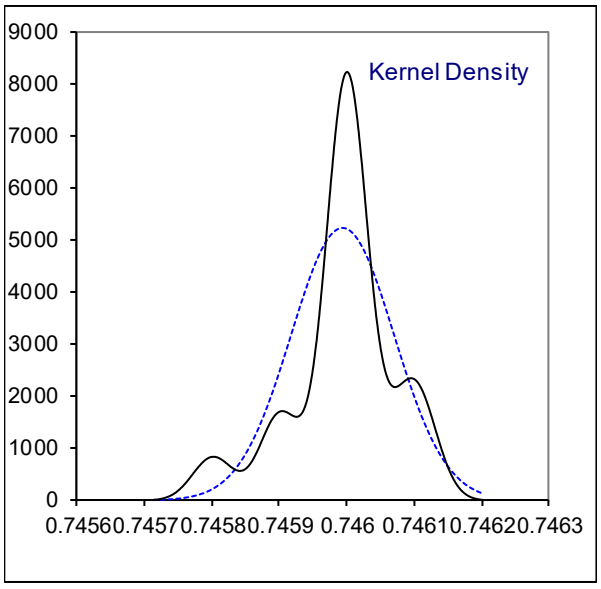


Determination of Density at 15°C on sample #21003; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.74599		-0.01	
311	D4052	0.7460		0.04	
312	D4052	0.7460		0.04	
323	D4052	0.7459		-0.52	
334	D4052	0.7460		0.04	
343	D4052	0.7460		0.04	
555	D4052	0.74611		0.66	
657	D4052	0.7461		0.60	
963	D4052	0.7458		-1.08	
1457	D4052	0.7460		0.04	
1530	ISO12185	0.74600		0.04	
1728	D4052	0.74601		0.10	
1788	D4052	0.74599		-0.01	
6198	D4052	0.74608		0.49	
6201	D4052	0.7460		0.04	
6262	D4052	0.7459		-0.52	

normality	suspect
n	16
outliers	0
mean (n)	0.74599
st.dev. (n)	0.000077
R(calc.)	0.00021
st.dev.(D4052:18a)	0.000179
R(D4052:18a)	0.0005

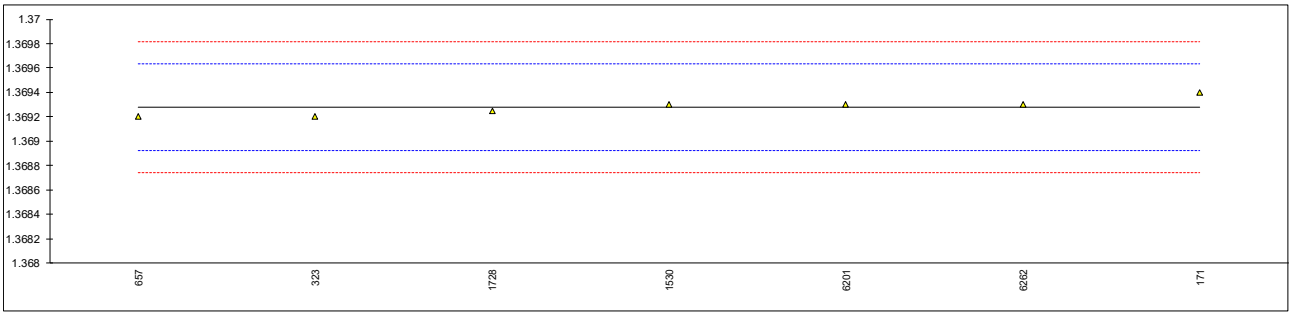


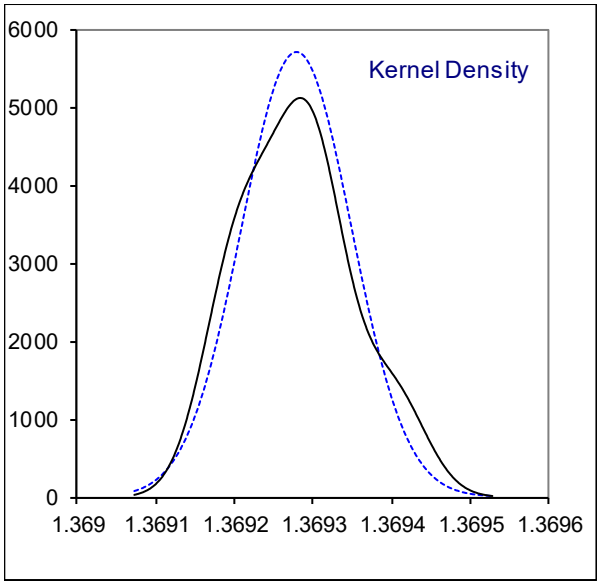


Determination of Refractive Index at 20°C on sample #21003;

lab	method	value	mark	z(targ)	remarks
171	D1218	1.3694		0.68	
311		-----		-----	
312		-----		-----	
323	D1218	1.3692		-0.44	
334		-----		-----	
343		-----		-----	
555		-----		-----	
657	D1218	1.36920		-0.44	
963		-----		-----	
1457		-----		-----	
1530	D1218	1.3693		0.12	
1728		1.36925		-0.16	
1788		-----		-----	
6198		-----		-----	
6201	D1218	1.3693		0.12	
6262	D1218	1.3693		0.12	

normality	unknown
n	7
outliers	0
mean (n)	1.36928
st.dev. (n)	0.00007
R(calc.)	0.00020
st.dev.(D1218:12)	0.000179
R(D1218:12)	0.0005

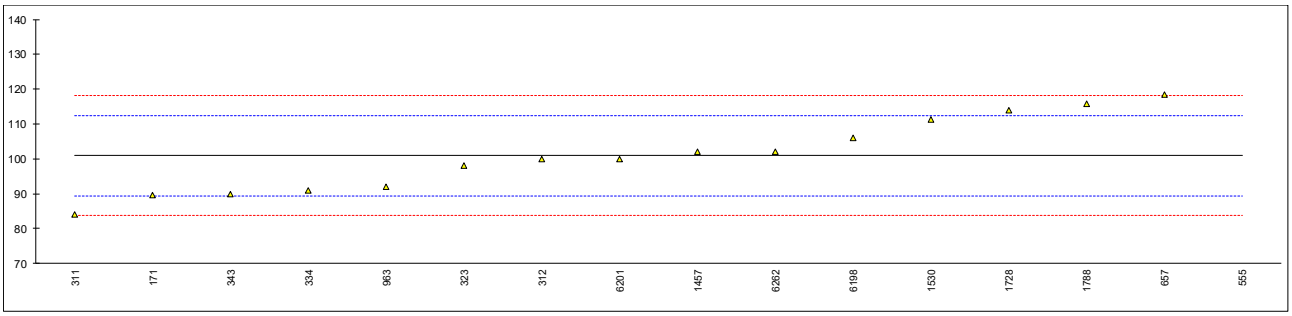


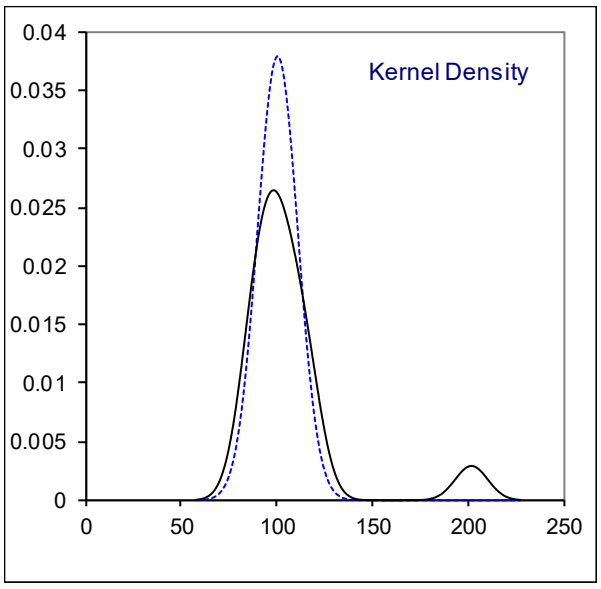


Determination of Water on sample #21003; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	E1064	89.6024		-1.98	
311	E1064	84		-2.95	
312	E1064	100		-0.16	
323	E1064	98		-0.51	
334	D1364	91		-1.73	
343	E1064	90		-1.91	
555	D4017	202.15	G(0.01)	17.66	
657	E1064	118.5		3.06	
963	D1364	92.00		-1.56	
1457	E1064	102		0.19	
1530	ISO12937	111.2		1.79	
1728	E1064	114		2.28	
1788	D6304	115.70		2.58	
6198	E1064	106	C	0.88	First reported 135
6201	E1064	100		-0.16	
6262	D1364	102		0.19	

normality	OK
n	15
outliers	1
mean (n)	100.93
st.dev. (n)	10.536
R(calc.)	29.50
st.dev.(E1064:16)	5.732
R(E1064:16)	16.05

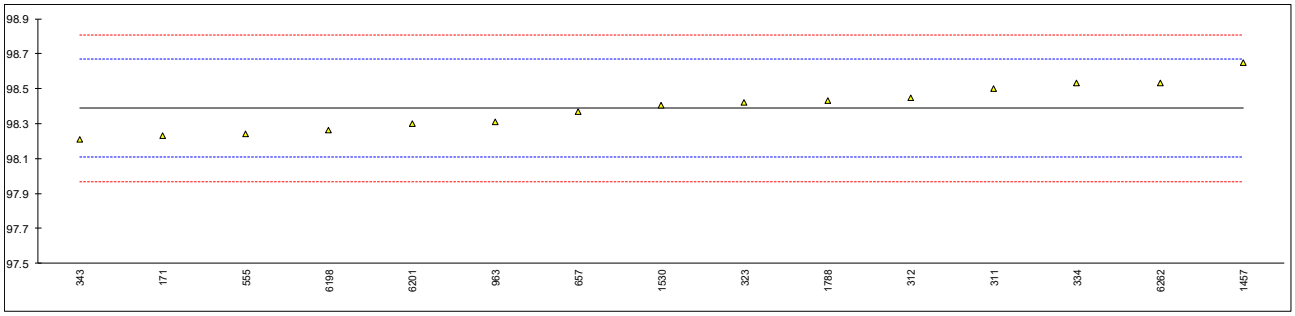


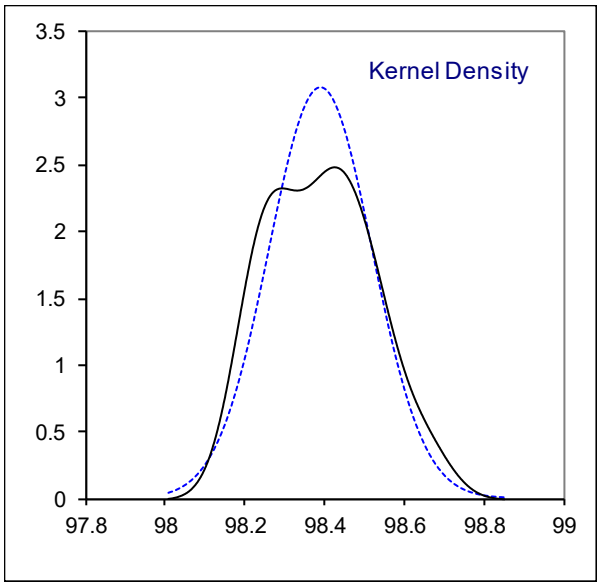


Determination of Purity by GC on dry basis on sample #21003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	98.23		-1.13	
311	D5441	98.50		0.79	
312	D5441	98.446		0.41	
323	D5441	98.42		0.22	
334	D5441	98.53		1.01	
343	D5441	98.21		-1.28	
555	D5441	98.242		-1.05	
657	D5441	98.3693		-0.14	
963	D5441	98.31		-0.56	
1457	D5441	98.648		1.85	
1530	D5441	98.408		0.14	
1728		-----		-----	
1788	D5441	98.4294		0.29	
6198	SH/T1550	98.262		-0.91	
6201	D5441	98.3		-0.63	
6262	D5441	98.53		1.01	

normality	OK
n	15
outliers	0
mean (n)	98.3890
st.dev. (n)	0.12955
R(calc.)	0.3627
st.dev.(D5441:98)	0.14027
R(D5441:98)	0.3928

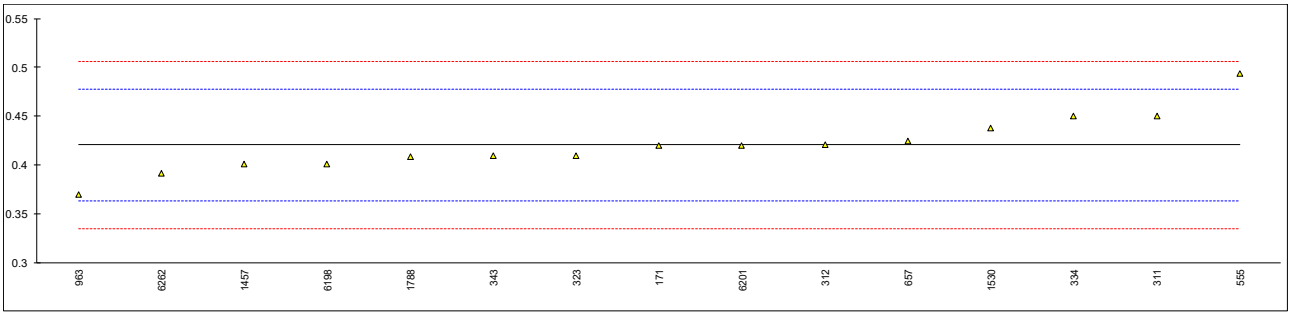


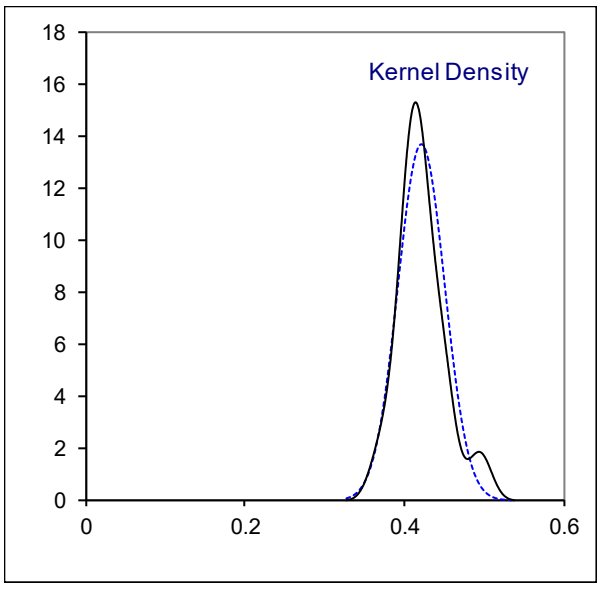


Determination of Methanol on sample #21003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.42		-0.03	
311	D5441	0.45		1.02	
312	D5441	0.421		0.01	
323	D5441	0.41		-0.38	
334	D5441	0.45		1.02	
343	D5441	0.41		-0.38	
555	D5441	0.494		2.56	
657	D5441	0.4253		0.16	
963	D5441	0.37		-1.77	
1457	D5441	0.401		-0.69	
1530	D5441	0.438		0.60	
1728		-----		-----	
1788	D5441	0.40885		-0.42	
6198	SH/T1550	0.401		-0.69	
6201	D5441	0.42		-0.03	
6262	D5441	0.3918		-1.01	

normality	suspect
n	15
outliers	0
mean (n)	0.4207
st.dev. (n)	0.02923
R(calc.)	0.0818
st.dev.(D5441:98)	0.02859
R(D5441:98)	0.0801



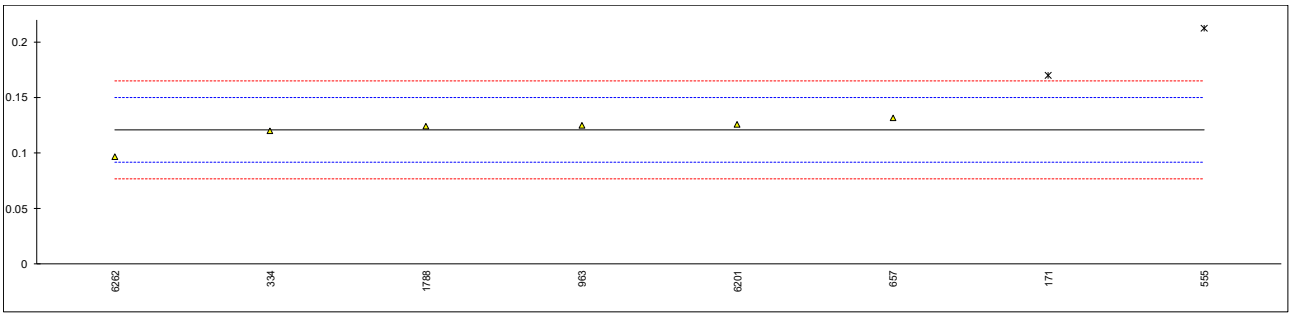


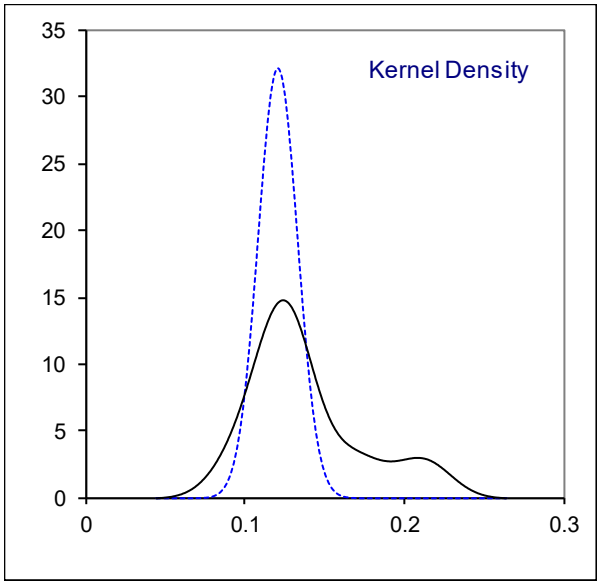
Determination of Diisobutylene (=sum 5 DIB's*) on sample #21003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.17	DG(0.05)	3.38	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334	D5441	0.12		-0.04	
343		-----		-----	
555	D5441	0.212	DG(0.05)	6.26	
657	D5441	0.1318		0.77	
963	D5441	0.125		0.30	
1457		-----		-----	
1530		-----		-----	
1728		-----		-----	
1788	D5441	0.1242		0.25	
6198		-----		-----	
6201	D5441	0.126	E	0.37	calculation difference, iis calculated 0.173
6262	D5441	0.0965		-1.65	

normality	unknown
n	6
outliers	2
mean (n)	0.1206
st.dev. (n)	0.01239
R(calc.)	0.0347
st.dev.(D5441:98)	0.01460
R(D5441:98)	0.0409

*) Sum of 5 DIB's: 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.

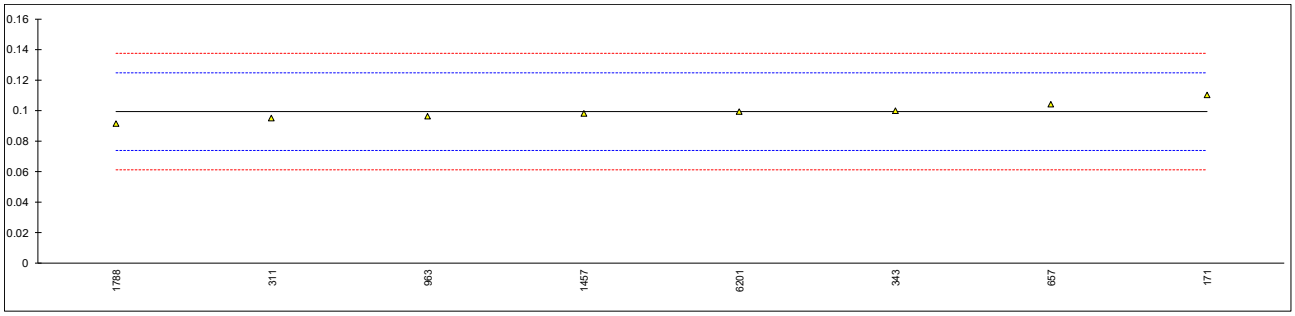


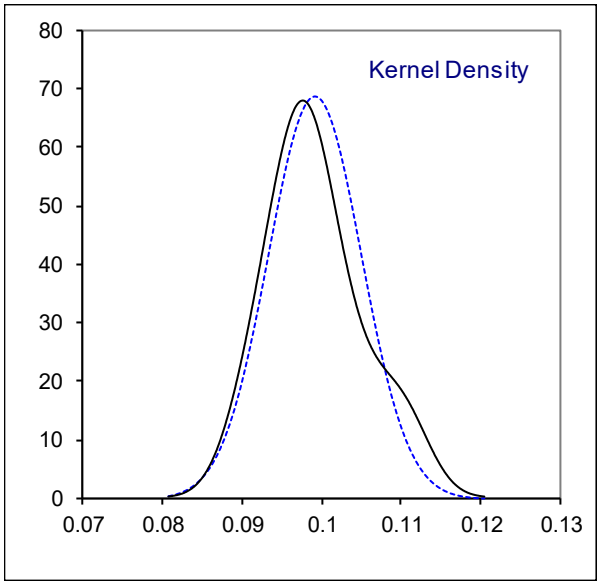


Determination of 2,4,4-Trimethyl-1-pentene on sample #21003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.11		0.85	
311	D5441	0.095	C	-0.33	First reported 0.09
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	0.10		0.07	
555		-----		-----	
657	D5441	0.1042		0.40	
963	D5441	0.096		-0.25	
1457	D5441	0.098		-0.09	
1530		-----		-----	
1728		-----		-----	
1788	D5441	0.0912		-0.63	
6198		-----		-----	
6201	D5441	0.099		-0.01	
6262		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	0.0992
st.dev. (n)	0.00580
R(calc.)	0.0162
st.dev.(D5441:98)	0.01268
R(D5441:98)	0.0355

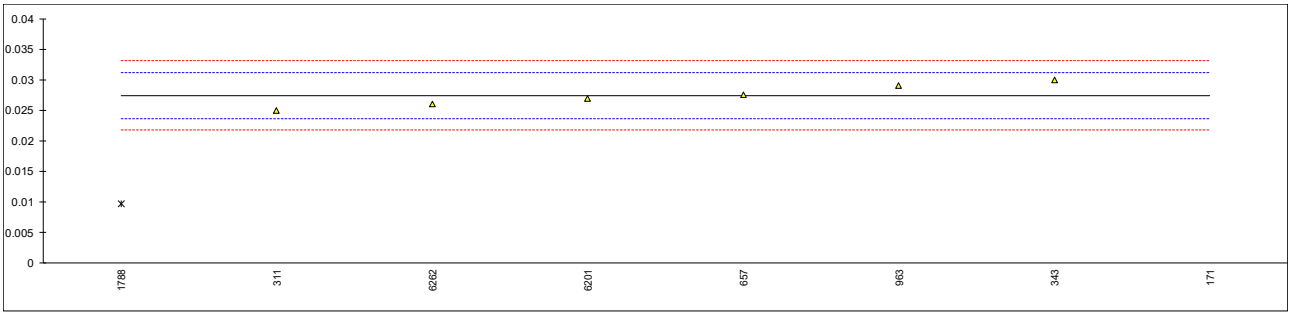


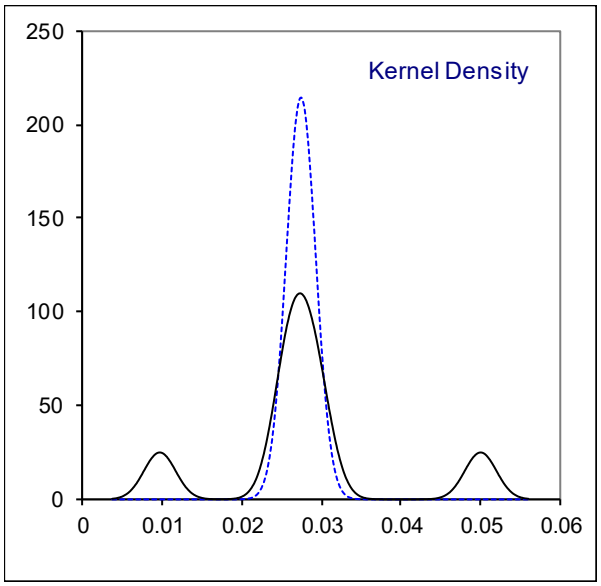


Determination of 2,4,4-Trimethyl-2-pentene on sample #21003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.05	D(0.01)	11.97	
311	D5441	0.025	C	-1.29	First reported 0.02
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	0.03		1.36	
555		-----		-----	
657	D5441	0.0276		0.09	
963	D5441	0.029		0.83	
1457		-----	W	-----	Test result withdrawn, reported 0
1530		-----		-----	
1728		-----		-----	
1788	D5441	0.0097	D(0.01)	-9.41	
6198		-----		-----	
6201	D5441	0.027		-0.23	
6262	D5441	0.0260		-0.76	

normality	unknown
n	6
outliers	2
mean (n)	0.0274
st.dev. (n)	0.00186
R(calc.)	0.0052
st.dev.(Horwitz)	0.00189
R(Horwitz)	0.0053

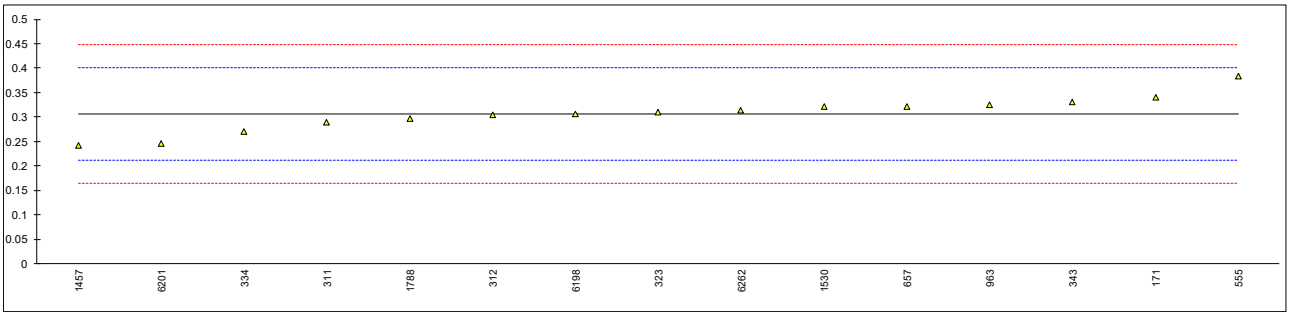


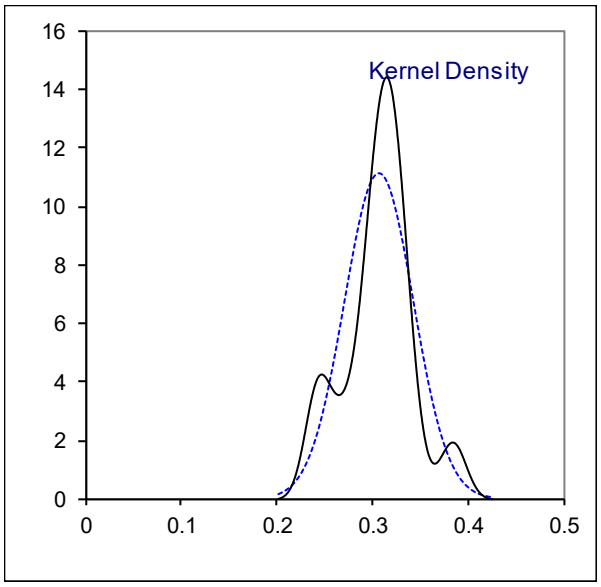


Determination of tert-Butanol on sample #21003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.34		0.70	
311	D5441	0.29		-0.36	
312	D5441	0.304		-0.06	
323	D5441	0.31		0.07	
334	D5441	0.27		-0.78	
343	D5441	0.33		0.49	
555	D5441	0.384		1.64	
657	D5441	0.3216		0.31	
963	D5441	0.325		0.39	
1457	D5441	0.243		-1.35	
1530	D5441	0.321		0.30	
1728		-----		-----	
1788	D5441	0.2970		-0.21	
6198	SH/T1550	0.307		0.00	
6201	D5441	0.245		-1.31	
6262	D5441	0.3143		0.16	

normality	OK
n	15
outliers	0
mean (n)	0.3068
st.dev. (n)	0.03591
R(calc.)	0.1005
st.dev.(D5441:98)	0.04714
R(D5441:98)	0.132

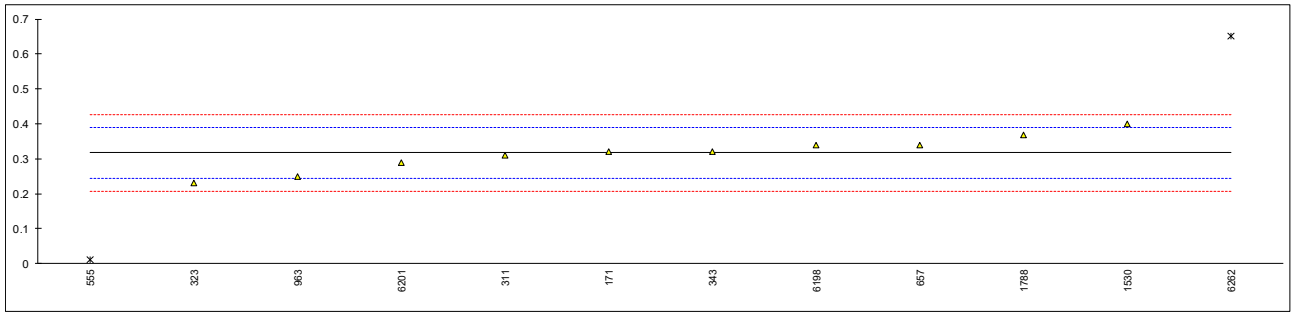


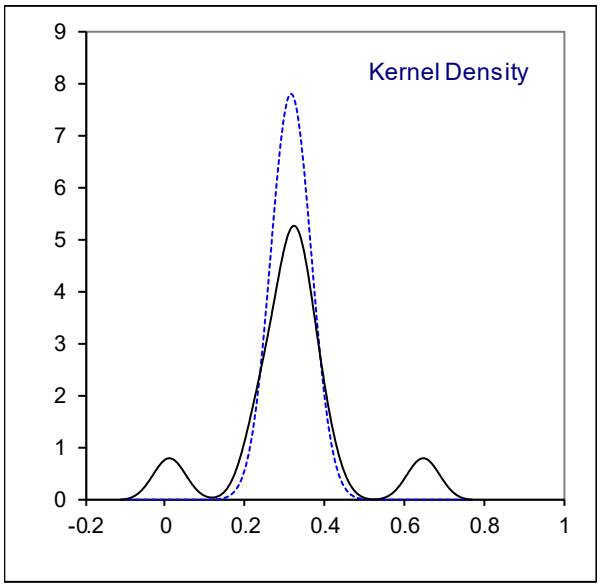


Determination of Hydrocarbons C4 on sample #21003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.32		0.09	
311	D5441	0.31		-0.18	
312		-----		-----	
323	D5441	0.23		-2.36	
334		-----		-----	
343	D5441	0.32		0.09	
555	D5441	0.01	D(0.01)	-8.35	
657	D5441	0.3402		0.64	
963	D5441	0.249		-1.84	
1457		-----		-----	
1530	D5441	0.401		2.30	
1728		-----		-----	
1788	D5441	0.3669		1.37	
6198	SH/T1550	0.339		0.61	
6201	D5441	0.29		-0.72	
6262	D5441	0.6502	D(0.01)	9.08	

normality	OK
n	10
outliers	2
mean (n)	0.3166
st.dev. (n)	0.05116
R(calc.)	0.1432
st.dev.(D5441:98)	0.03673
R(D5441:98)	0.1029

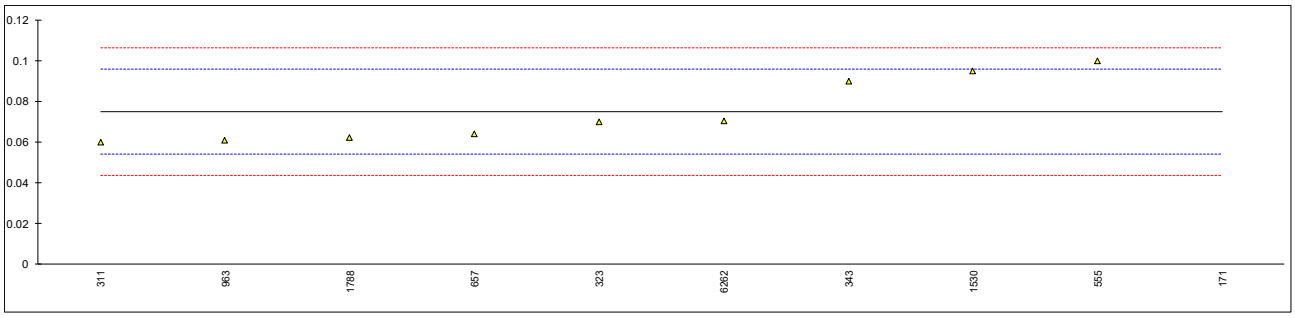


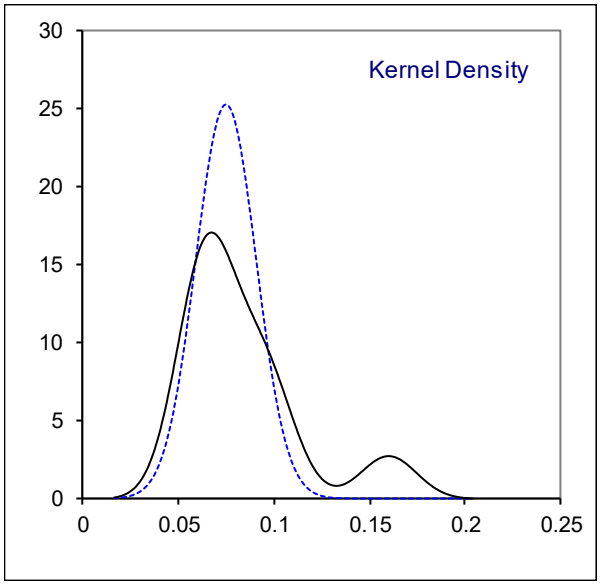


Determination of Hydrocarbons C5 on sample #21003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.16	G(0.01)	8.18	
311	D5441	0.06		-1.41	
312		-----		-----	
323	D5441	0.07		-0.45	
334		-----		-----	
343	D5441	0.09		1.47	
555	D5441	0.1		2.43	
657	D5441	0.0640		-1.03	
963	D5441	0.061		-1.31	
1457		-----		-----	
1530	D5441	0.095		1.95	
1728		-----		-----	
1788	D5441	0.0620		-1.22	
6198		-----		-----	
6201		-----		-----	
6262	D5441	0.0702		-0.43	

normality	OK
n	9
outliers	1
mean (n)	0.0747
st.dev. (n)	0.01584
R(calc.)	0.0444
st.dev.(D5441:98)	0.01042
R(D5441:98)	0.0292

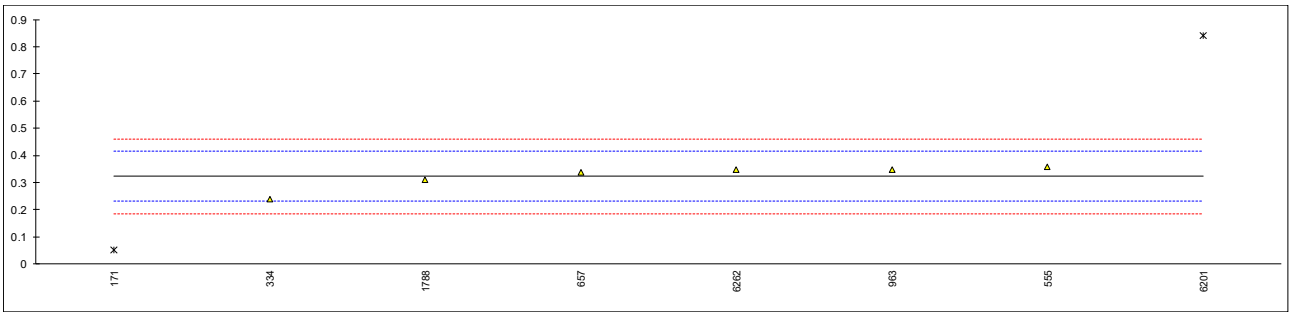


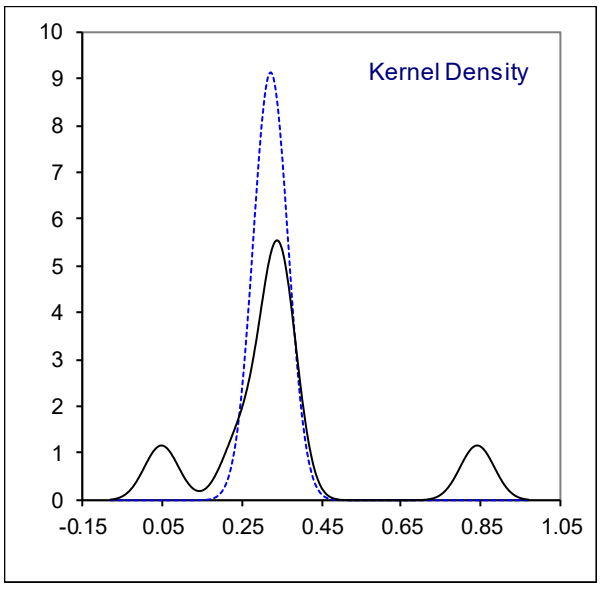


Determination of Other Impurities on sample #21003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.05	G(0.05)	-5.94	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334	D5441	0.24		-1.81	
343		-----		-----	
555	D5441	0.356		0.72	
657	D5441	0.3358		0.28	
963	D5441	0.348		0.54	
1457		-----		-----	
1530		-----		-----	
1728		-----		-----	
1788	D5441	0.3108		-0.27	
6198		-----		-----	
6201	D5441	0.84	G(0.05)	11.25	
6262	D5441	0.3478		0.54	

normality	unknown
n	6
outliers	2
mean (n)	0.3231
st.dev. (n)	0.04366
R(calc.)	0.1222
st.dev.(Horwitz (n=9))	0.04595
R(Horwitz (n=9))	0.1287





APPENDIX 2

Determination of other individual diisobutylenes on sample #21003; results in %M/M

lab	method	2,3,4-Trimethyl-2-pentene	3,4,4-Trimethyl-1-pentene	3,5-Dimethyl-1-hexene
171	D5441	<0.02	-----	-----
311		-----	-----	-----
312		-----	-----	-----
323		-----	-----	-----
334		-----	-----	-----
343		-----	-----	-----
555		-----	-----	-----
657	D5441	< 0.02	< 0.02	< 0.02
963		-----	-----	-----
1457		-----	-----	-----
1530		-----	-----	-----
1728		-----	-----	-----
1788	D5441	0.0000	0.0000	0.0233
6198		-----	-----	-----
6201	D5441	0.017	0.030	<0,02
6262	D5441	0.0043	-----	0.0005

APPENDIX 3

Number of participants per country

2 labs in BELGIUM
1 lab in BRAZIL
1 lab in CHINA, People's Republic
1 lab in FRANCE
1 lab in GERMANY
4 labs in NETHERLANDS
1 lab in PORTUGAL
1 lab in ROMANIA
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
1 lab in SPAIN
1 lab in UNITED STATES OF AMERICA

APPENDIX 4

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	= calculation difference between reported test result and result calculated by iis
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 Horwitz, R. Albert, J. AOAC Int, 79, 3, 589, (1996)
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4 ASTM E1301:03
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6 ISO5725, parts 1-6:94
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8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
9 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
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11 DIN38402 T41/42
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13 J.N. Miller, Analyst, 118, 455, (1993)
14 Analytical Methods Committee, Technical Brief, No 4, January 2001
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