Institute for
Interlaboratory Studies

Results of Proficiency Test
Methyl-tert-Butylether (MTBE)
February 2023

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-tert-Butylether (MTBE) once every two years. During the annual proficiency testing program 2022/2023 it was decided to continue the round robin for the analysis of Methyl-tert-Butylether (MTBE).

In this interlaboratory study 14 laboratories in 13 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the MTBE proficiency test 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 sample MTBE in a 0.5-liter bottle labelled #23003.
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 15 liters of Methyl-tert-Butylether (MTBE) was obtained from a local supplier. After homogenization 20 amber glass bottles of 0.5 liter were filled and labelled #23003.
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 #23003-1	0.74134
sample #23003-2	0.74134
sample #23003-3	0.74133
sample #23003-4	0.74135
sample #23003-5	0.74133
sample #23003-6	0.74133
sample #23003-7	0.74135
sample #23003-8	0.74134

Table 1: homogeneity test results of subsamples #23003

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 D4052:22
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of the subsamples #23003

The calculated repeatability is 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 0.5 L MTBE labelled #23003 was sent on January 11, 2023. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Methyl-tert-Butylether (MTBE) 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 Diisobutylenes and the individual 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-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 (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in 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. Therefore, the reporting time on the data entry portal was extended with one week. One participant reported the test results after the extended reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.
In total 11 reporting laboratories submitted 85 numerical test results. No outlying test results were observed. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all 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 in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

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. D1218:21).

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.

Appearance: This determination was not problematic. All reporting participants agreed about the appearance as Clear and Bright (Pass).

Carbonyls: Only two laboratories reported a test result. Therefore, no z-scores are 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 D4052:22

Refractive Index at 20 °C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1218:21.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full 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 D5441:21.

Methanol: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D5441:21.

Diisobutylene (=sum 5 DIBs): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5441:21.

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 D5441:21.

2,4,4-Trimethyl-2-pentene: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

For other DIBs test method D5441:21 is applicable for concentrations above 0.02 %M/M, however a low number of test results is reported. Therefore, no z-scores were calculated for these components. 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 D5441:21.

Hydrocarbons C4: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5441:21.

Hydrocarbons C5: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D5441:21.

Other Impurities: Four participants reported a test result. Therefore, no z-scores are calculated. The test results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method 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 reference methods are presented in the next table.

	unit	n	average	2.8 * sd	R(lit)
Appearance		10	C&B (Pass)	n.a.	n.a.
Carbonyls	µg/g	2	n.e.	n.e.	n.e.
Density at 15 °C	kg/L	10	0.7466	0.0002	0.0005
Refractive Index at 20 °C		3	1.3694	0.0001	0.0005
Water	mg/kg	11	279	46	44
Purity by GC on dry basis	%M/M	10	97.594	0.332	0.455
Methanol	%M/M	10	0.644	0.123	0.089
Diisobutylene (=sum of 5 DIBs)	%M/M	5	0.188	0.029	0.058
2,4,4-Trimethyl-1-pentene	%M/M	7	0.141	0.013	0.046
2,4,4-Trimethyl-2-pentene	%M/M	6	0.040	0.002	0.007
tert-Butanol	%M/M	9	0.180	0.044	0.132
Hydrocarbons C4	%M/M	7	0.121	0.030	0.040
Hydrocarbons C5	%M/M	7	0.045	0.030	0.021

Table 3: reproducibilities of tests on sample #23003

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 2023 WITH PREVIOUS PTS

	February 2023	February 2021	February 2019	February 2017	February 2015
Number of reporting laboratories	11	16	18	16	17
Number of test results	85	142	158	132	159
Number of statistical outliers	0	10	11	10	13
Percentage of statistical outliers	0%	7.0%	7.0%	7.6%	8.2%

Table 4: comparison with previous proficiency tests

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

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

Determination	February 2023	February 2021	February 2019	February 2017	February 2015
Carbonyls	n.e.	--	n.e.	n.e.	n.e.
Density at 15 °C	++	++	++	++	++
Refractive Index at 20 °C	++	++	--	+	+
Water	+/-	-	++	+	++
Purity by GC on dry basis	+	+/-	+	+	--
Methanol	-	+/-	+	+	+/-
Diisobutylene (=sum of 5 DIBs)	++	+	-	+/-	+/-
2,4,4-Trimethyl-1-pentene	++	++	+	+	+
2,4,4-Trimethyl-2-pentene	++	+/-	+/-	++	+
tert-Butanol	++	+	++	++	++
Hydrocarbons C4	+	-	-	++	--
Hydrocarbons C5	-	-	--	+/-	+

Table 5: comparison determinations to 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 #23003;

lab	method	value	mark	z(targ)	remarks
171	E2680	Clear without any suspended matter		-----	
311	D4176	clear		-----	
312	Visual	br&cl		-----	
323		-----		-----	
334	Visual	clear & bright		-----	
343	E2680	PASS		-----	
395	E2680	PASS		-----	
657	E2680	Pass		-----	
963	E2680	Pass		-----	
1544		-----		-----	
1728	Visual	Clear and bright		-----	
1788	Visual	Clear		-----	
6198		-----		-----	
6506		-----		-----	
	n	10			
	mean (n)	clear & bright (Pass)			

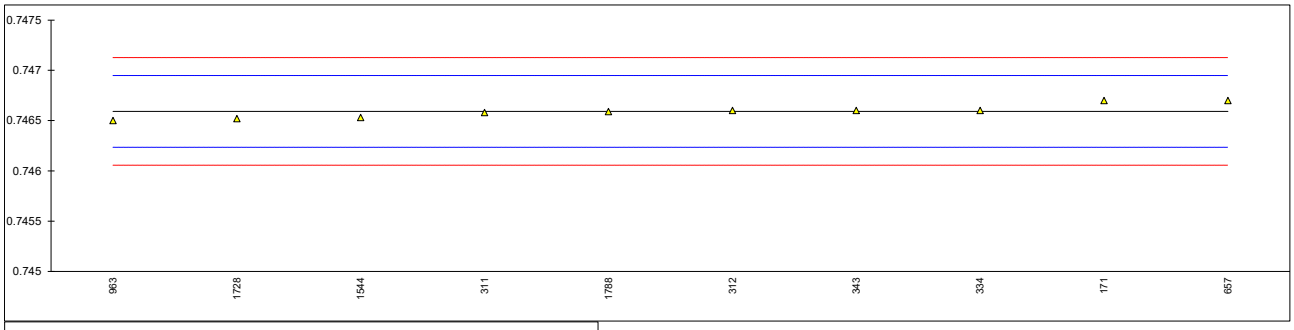
Determination of Carbonyls on sample #23003; results in µg/g

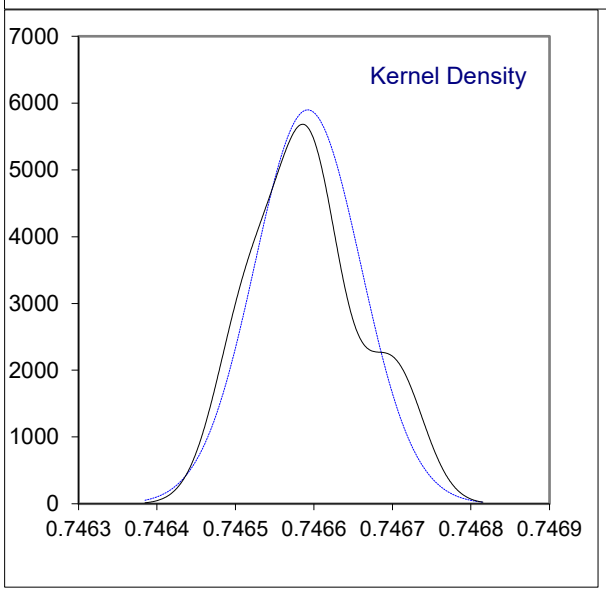
lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343		-----		-----	
395		-----		-----	
657	E411	292.26		-----	
963	E411	81.6		-----	
1544		-----		-----	
1728		-----		-----	
1788		-----		-----	
6198		-----		-----	
6506		-----		-----	

Determination of Density at 15 °C on sample #23003; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.7467		0.60	
311	D4052	0.74658		-0.07	
312	D4052	0.7466		0.04	
323		-----		-----	
334	D4052	0.7466		0.04	
343	D4052	0.7466		0.04	
395		-----		-----	
657	D4052	0.7467		0.60	
963	D4052	0.7465		-0.52	
1544	D4052	0.74653		-0.35	
1728	D4052	0.74652	C	-0.40	first reported 0.74620
1788	D4052	0.74659		-0.01	
6198		-----		-----	
6506		-----		-----	

normality	OK
n	10
outliers	0
mean (n)	0.74659
st.dev. (n)	0.000068
R(calc.)	0.00019
st.dev.(D4052:22)	0.000179
R(D4052:22)	0.0005

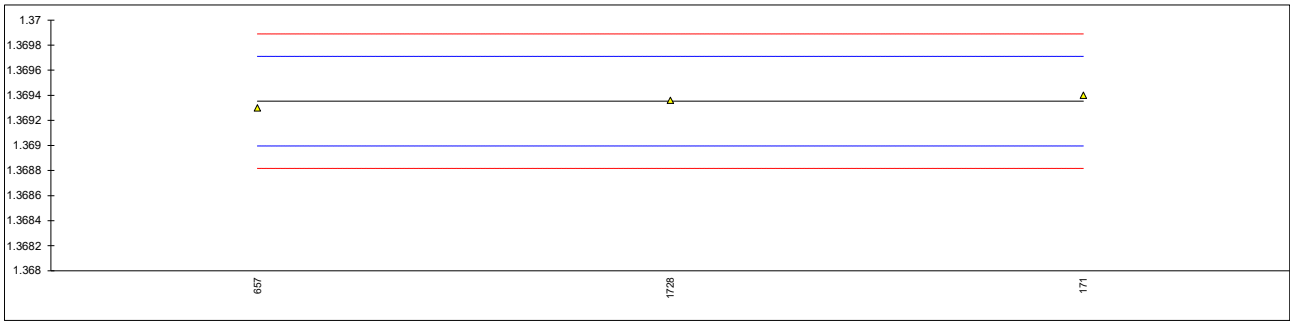




Determination of Refractive Index at 20 °C on sample #23003;

lab	method	value	mark	z(targ)	remarks
171	D1218	1.3694		0.26	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343		-----		-----	
395		-----		-----	
657	D1218	1.3693		-0.30	
963		-----		-----	
1544		-----		-----	
1728		1.36936		0.04	
1788		-----		-----	
6198		-----		-----	
6506		-----		-----	

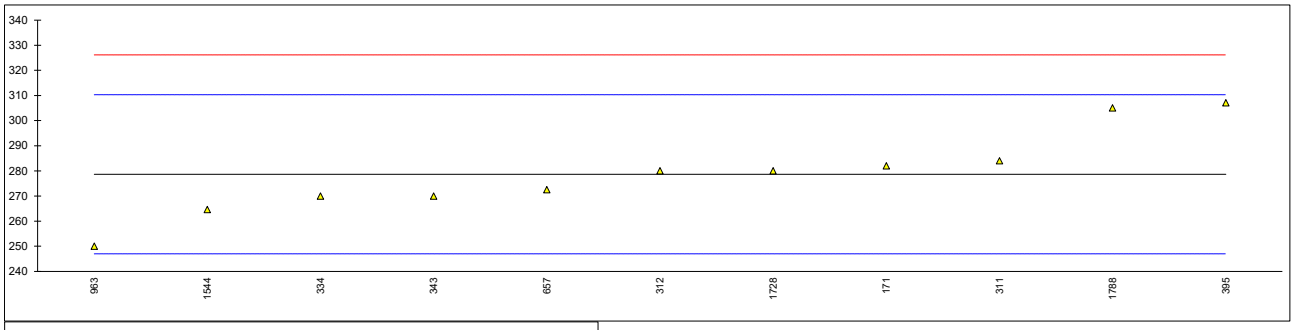
normality	unknown
n	3
outliers	0
mean (n)	1.36935
st.dev. (n)	0.000050
R(calc.)	0.00014
st.dev.(D1218:21)	0.000179
R(D1218:21)	0.0005

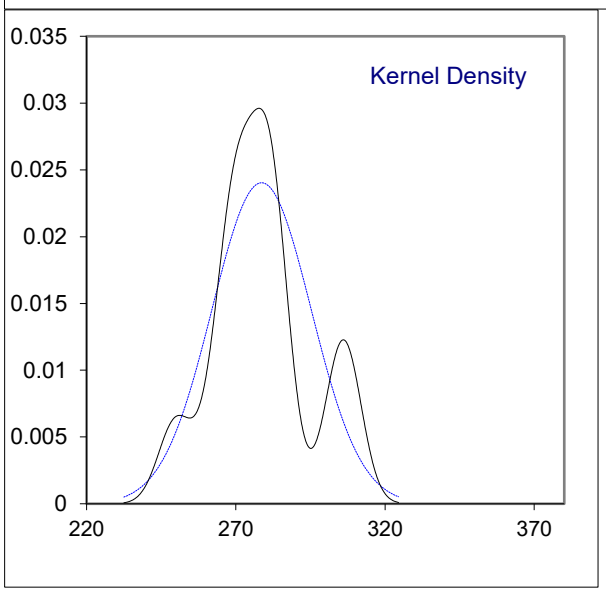


Determination of Water on sample #23003; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	282		0.21	
311	E203	284		0.34	
312	E1064	280		0.08	
323		-----		-----	
334	D1364	270		-0.55	
343	E1064	270		-0.55	
395	D1364	307.1		1.80	
657	E1064	272.5		-0.39	
963	D1364	250		-1.81	
1544	E1064	264.6		-0.89	
1728	E1064	280		0.08	
1788	E1064	305.03		1.67	
6198		-----		-----	
6506		-----		-----	

normality	OK
n	11
outliers	0
mean (n)	278.66
st.dev. (n)	16.597
R(calc.)	46.47
st.dev.(E1064:16)	15.824
R(E1064:16)	44.31

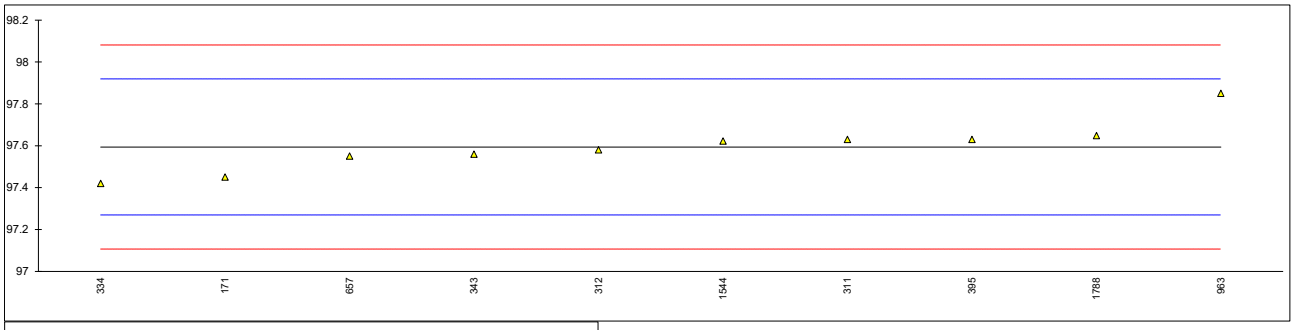


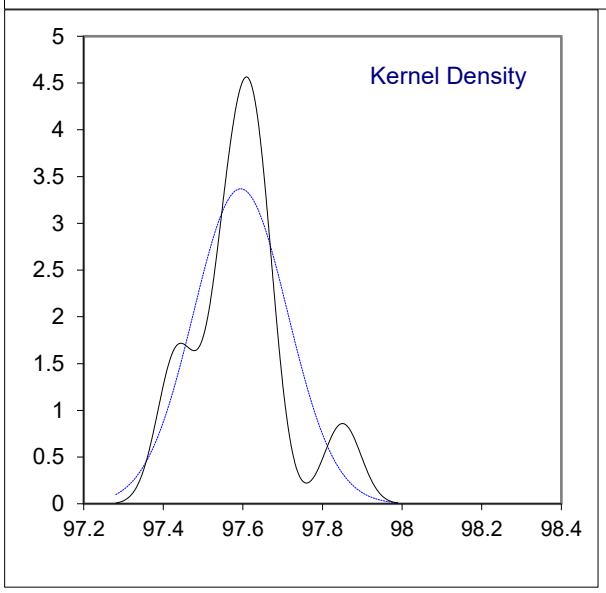


Determination of Purity by GC on dry basis on sample #23003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	97.45		-0.89	
311	D5441	97.63		0.22	
312	D5441	97.58	C	-0.09	first reported 97.96
323		-----		-----	
334	D5441	97.42		-1.07	
343	D5441	97.56		-0.21	
395	D5441	97.63		0.22	
657	D5441	97.5502		-0.27	
963	D5441	97.85		1.58	
1544	D5441	97.622		0.17	
1728		-----		-----	
1788	D5441	97.6485		0.34	
6198		-----		-----	
6506		-----		-----	

normality	suspect
n	10
outliers	0
mean (n)	97.5941
st.dev. (n)	0.11840
R(calc.)	0.3315
st.dev.(D5441:21)	0.16232
R(D5441:21)	0.4545

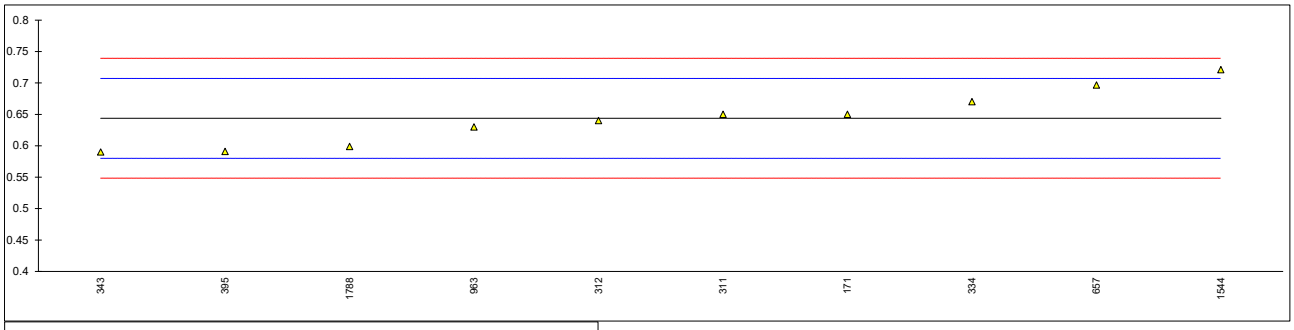


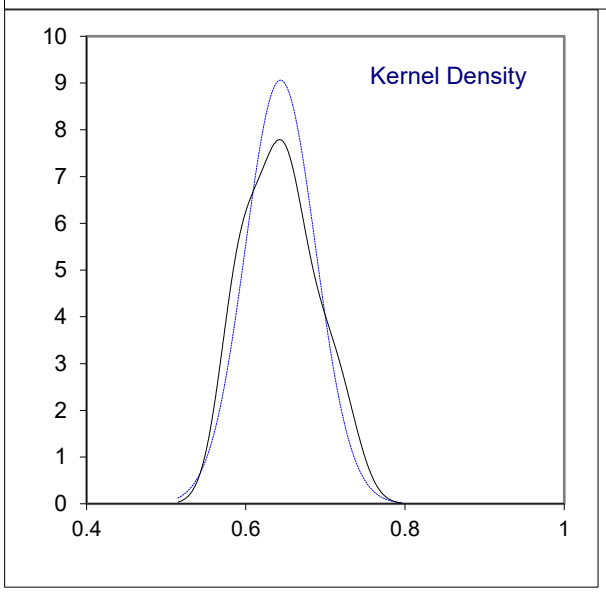


Determination of Methanol on sample #23003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.65		0.20	
311	D5441	0.65		0.20	
312	D5441	0.64		-0.12	
323		-----		-----	
334	D5441	0.67		0.83	
343	D5441	0.59		-1.69	
395	D5441	0.591		-1.66	
657	D5441	0.6966		1.66	
963	D5441	0.63		-0.43	
1544	D5441	0.7210		2.43	
1728		-----		-----	
1788	D5441	0.5986		-1.42	
6198		-----		-----	
6506		-----		-----	

normality	OK
n	10
outliers	0
mean (n)	0.6437
st.dev. (n)	0.04402
R(calc.)	0.1233
st.dev.(D5441:21)	0.03180
R(D5441:21)	0.0890



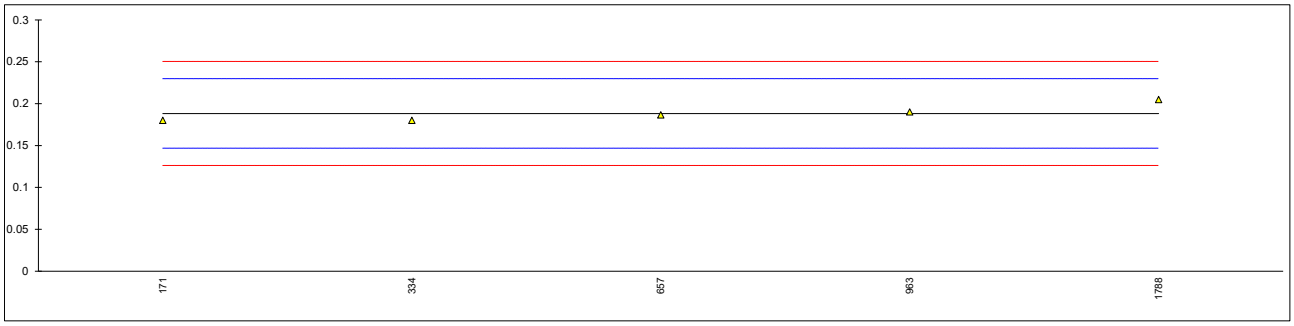


Determination of Diisobutylene (=sum 5 DIBs*) on sample #23003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.18	E	-0.40	iis calculated 0.25
311		-----		-----	
312		-----		-----	
323		-----		-----	
334	D5441	0.18		-0.40	
343		-----		-----	
395		-----		-----	
657	D5441	0.1865		-0.09	
963	D5441	0.19		0.08	
1544		-----		-----	
1728		-----		-----	
1788	D5441	0.2049		0.80	
6198		-----		-----	
6506		-----		-----	

normality	unknown
n	5
outliers	0
mean (n)	0.1883
st.dev. (n)	0.01024
R(calc.)	0.0287
st.dev.(D5441:21)	0.02070
R(D5441:21)	0.0580

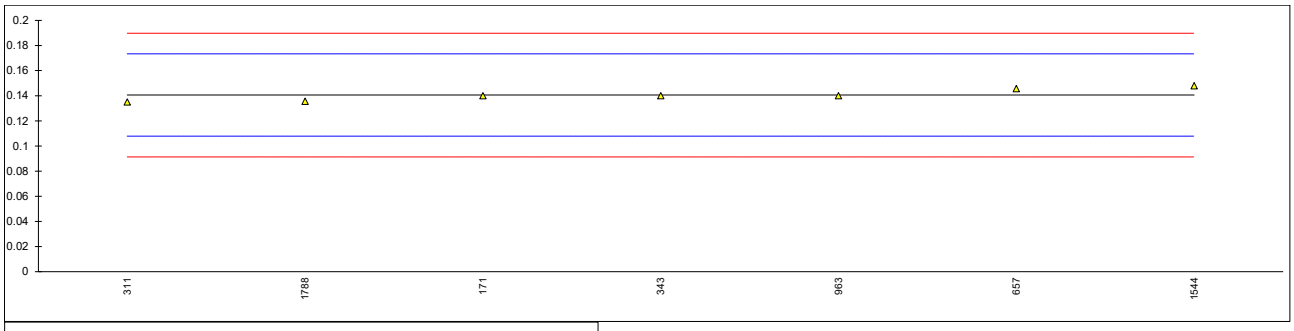
*) Sum of 5 DIBs: 2,4,4-Trimethyl-1-pentene; 2,4,4-Trimethyl-2-pentene; 2,3,4-Trimethyl-2-pentene; 3,4,4-Trimethyl-1-pentene and 3,5-Dimethyl-1-hexene.

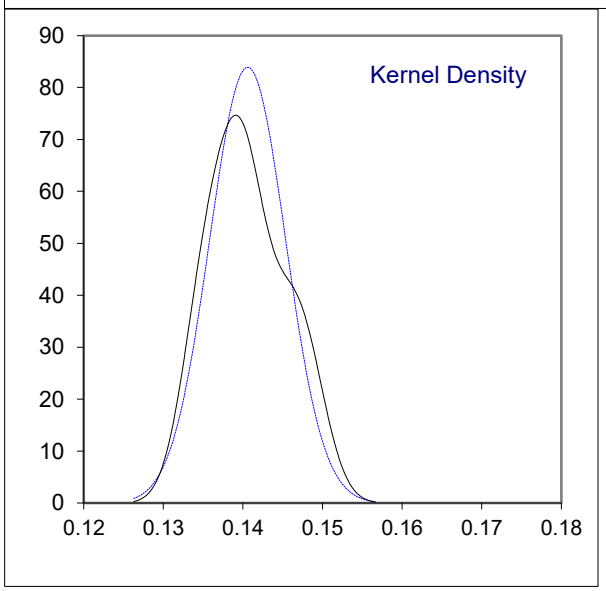


Determination of 2,4,4-Trimethyl-1-pentene on sample #23003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.14		-0.04	
311	D5441	0.135		-0.34	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	0.14		-0.04	
395		-----		-----	
657	D5441	0.1456		0.31	
963	D5441	0.14		-0.04	
1544	D5441	0.1479		0.45	
1728		-----		-----	
1788	D5441	0.1356		-0.30	
6198		-----		-----	
6506		-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	0.1406
st.dev. (n)	0.00477
R(calc.)	0.0133
st.dev.(D5441:21)	0.01641
R(D5441:21)	0.0459

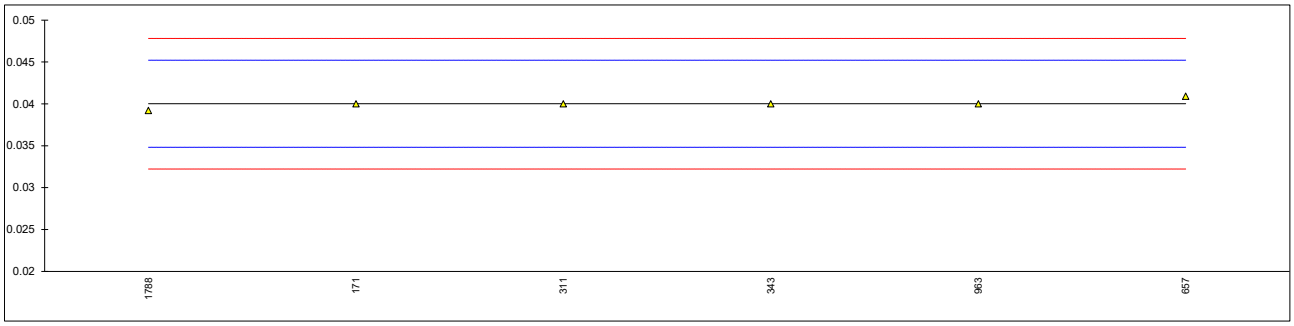




Determination of 2,4,4-Trimethyl-2-pentene on sample #23003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.04		-0.01	
311	D5441	0.04		-0.01	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	0.04		-0.01	
395		-----		-----	
657	D5441	0.0409		0.34	
963	D5441	0.04		-0.01	
1544		-----		-----	
1728		-----		-----	
1788	D5441	0.0392		-0.31	
6198		-----		-----	
6506		-----		-----	

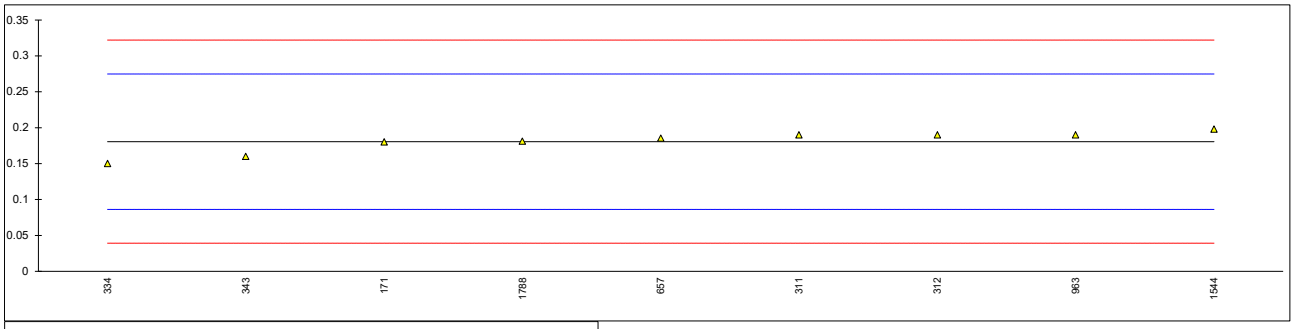
normality	unknown
n	6
outliers	0
mean (n)	0.0400
st.dev. (n)	0.00054
R(calc.)	0.0015
st.dev.(Horwitz)	0.00260
R(Horwitz)	0.0073

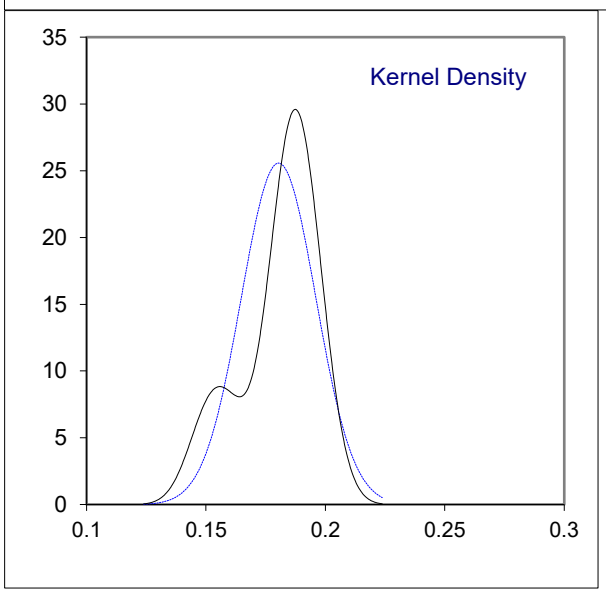


Determination of tert-Butanol on sample #23003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.18		-0.01	
311	D5441	0.19		0.20	
312	D5441	0.19		0.20	
323		-----		-----	
334	D5441	0.15		-0.65	
343	D5441	0.16		-0.43	
395		-----		-----	
657	D5441	0.1853		0.10	
963	D5441	0.19		0.20	
1544	D5441	0.1979		0.37	
1728		-----		-----	
1788	D5441	0.1810		0.01	
6198		-----		-----	
6506		-----		-----	

normality	OK
n	9
outliers	0
mean (n)	0.1805
st.dev. (n)	0.01560
R(calc.)	0.0437
st.dev.(D5441:21)	0.04714
R(D5441:21)	0.132

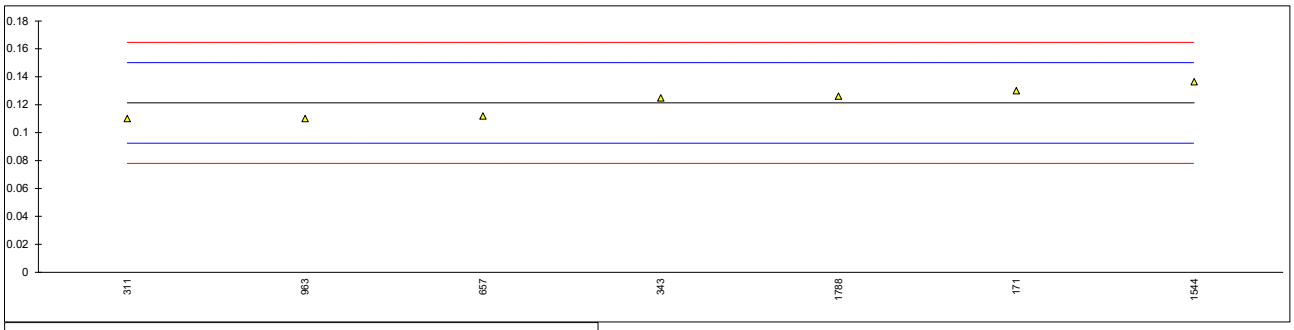


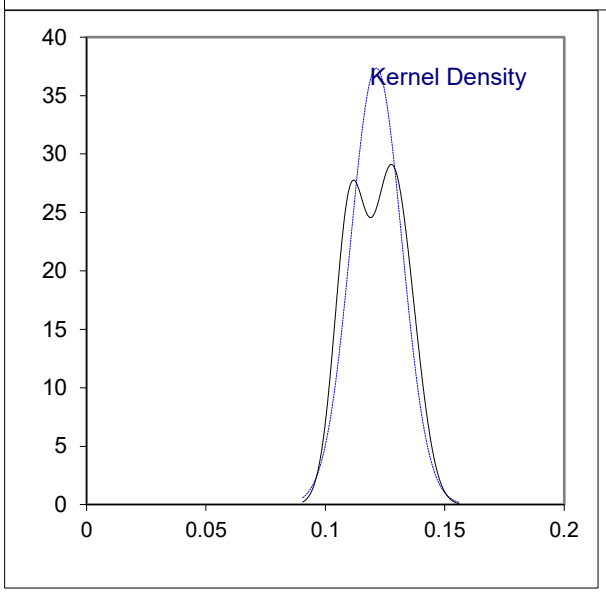


Determination of Hydrocarbons C4 on sample #23003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.13		0.60	
311	D5441	0.11		-0.78	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	0.1248		0.24	
395		-----		-----	
657	D5441	0.1118		-0.66	
963	D5441	0.11		-0.78	
1544	D5441	0.1364		1.05	
1728		-----		-----	
1788	D5441	0.1261		0.33	
6198		-----		-----	
6506		-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	0.1213
st.dev. (n)	0.01068
R(calc.)	0.0299
st.dev.(D5441:21)	0.01442
R(D5441:21)	0.0404

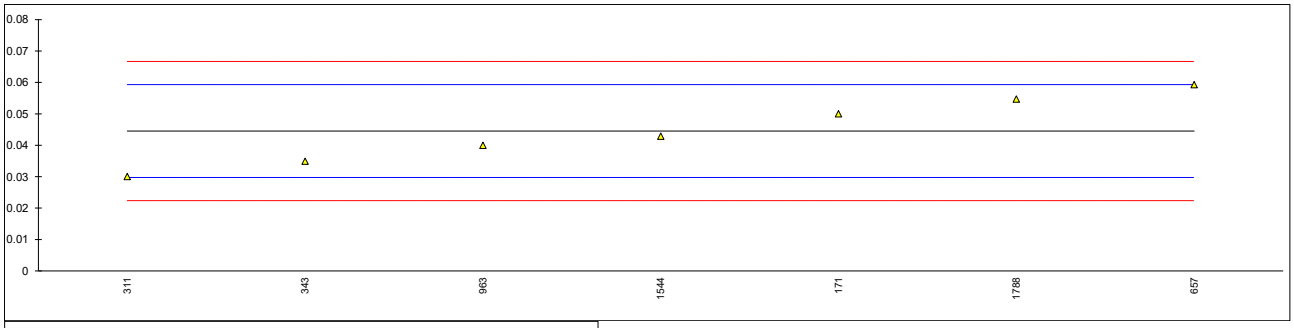


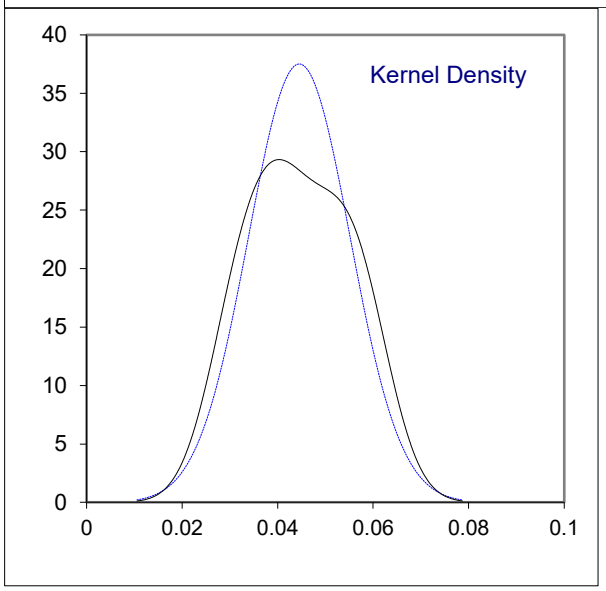


Determination of Hydrocarbons C5 on sample #23003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.05	C	0.74	first reported 0.24
311	D5441	0.03		-1.97	
312		-----		-----	
323		-----		-----	
334		-----		-----	
343	D5441	0.0349		-1.31	
395		-----		-----	
657	D5441	0.0593		2.00	
963	D5441	0.04		-0.62	
1544	D5441	0.0429		-0.22	
1728		-----		-----	
1788	D5441	0.0547		1.38	
6198		-----		-----	
6506		-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	0.0445
st.dev. (n)	0.01063
R(calc.)	0.0298
st.dev.(D5441:21)	0.00739
R(D5441:21)	0.0207





APPENDIX 2

Determination of other reported impurities on sample #23003; results in %M/M

lab	method	2,3,4-Trimethyl-2-pentene	3,4,4-Trimethyl-1-pentene	3,5-Dimethyl-1-hexene	Other Impurities
171	D5441	0.03	0.04	-----	0.23
311		-----	-----	-----	-----
312		-----	-----	-----	-----
323		-----	-----	-----	-----
334		-----	-----	-----	-----
343		-----	-----	-----	-----
395		-----	-----	-----	-----
657	D5441	<0.02	<0.02	<0.02	1.1834
963	D5441	-----	-----	-----	0.87
1544		-----	-----	-----	-----
1728		-----	-----	-----	-----
1788	D5441	0.000	0.0301	0.000	1.1864
6198		-----	-----	-----	-----
6506		-----	-----	-----	-----

APPENDIX 3

Number of participants per country

1 lab in BELGIUM

1 lab in BULGARIA

1 lab in CHINA, People's Republic

1 lab in FRANCE

1 lab in ITALY

1 lab in MALAYSIA

2 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
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
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3 ISO5725 parts 1-6:94
4 ISO13528:05
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11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
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