Results of Proficiency Test
MTBE - ETBE
March 2010

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 1995, a proficiency test for Methyl Tertiary Butyl Ether (MTBE) and since 2007 a proficiency test for Ethyl Tertiary Butyl Ether (ETBE) was organized every year by The Institute for Interlaboratory Studies. During the annual proficiency testing program 2009/2010, it was decided to continue the round robin for the analyses of MTBE and ETBE. In these international interlaboratory studies for MTBE, 21 laboratories in 13 different countries and for ETBE, 18 laboratories in 11 countries have participated. See appendix 2 for a list of participants in alphabetical country order. In this report, the results are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (i.i.s.) in Spijkenisse, The Netherlands, was the organizer of this proficiency test. It was decided, depending on the registration, to send one sample of MTBE (0.5 litre, labelled #1032) and/or one sample of ETBE (0.5 litre, labelled #1033) to the participants. Analyses for fit-four-use and homogeneity were subcontracted to an accredited laboratory. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO guide 43 and ILAC-G13:2007, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures 100% confidentiality of participant's data. Also customer's satisfaction is measured on a regular basis by sending out questionnaires (see www.rva.nl).

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 January 2010 (i.i.s.-protocol, version 3.2).

2.3 CONFIDENTIALITY STATEMENT

All data, present 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

MTBE:
The necessary 25 litres of MTBE bulk material were obtained from a local producer. After homogenisation, the bulk material was transferred into 53 brown glass bottles of 500 mL and labelled #1032. The homogeneity of the subsamples was checked by determination of Density @15˚C in accordance with ASTM D4052:02e1, Water in accordance with ASTM E1064:05 and Total Impurities on 8 stratified random selected samples:

MTBE	Density at 15˚C in kg/L	Water in mg/kg	Total impurities in %M/M
sample #1032-1	0.74660	1590	1.25
sample #1032-2	0.74659	1580	1.28
sample #1032-3	0.74661	1580	1.29
sample #1032-4	0.74661	1580	1.31
sample #1032-5	0.74661	1570	1.25
sample #1032-6	0.74661	1570	1.30
sample #1032-7	0.74661	1580	1.28
sample #1032-8	0.74662	1580	1.28

table 1: homogeneity test of subsamples #1032

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the target methods or with the reproducibility calculate using the Horwitz equation in agreement with the procedure of ISO 13528, Annex B2 in the next table;

	Density at 15˚C in kg/L	Water in mg/kg	Total impurities in %M/M
r (sample #1032)	0.00002	20	0.06
reference test method	ASTM D4052:02e1	ASTM E1064:05	Horwitz
0.3xR (reference test)	0.00015	80	0.12

table 2: evaluation of the subsamples #1032

The calculated repeatabilities were in agreement with 0.3 times the corresponding target reproducibility. Therefore, homogeneity of the samples was assumed.

ETBE:
The necessary 25 litres of ETBE bulk material were obtained from a local producer. After homogenisation, the bulk material was transferred into 50 brown glass bottles of 500 mL and labelled #1033. The homogeneity of the subsamples was checked by determination of Density @15˚C in accordance with ASTM D4052:02e1 and total impurities on 8 stratified random selected samples:

ETBE	Density at 15˚C in kg/L	Total impurities in %M/M
sample #1033-1	0.74632	2.60
sample #1033-2	0.74633	2.59
sample #1033-3	0.74633	2.59
sample #1033-4	0.74633	2.60
sample #1033-5	0.74633	2.58
sample #1033-6	0.74633	2.59
sample #1033-7	0.74634	2.58
sample #1033-8	0.74633	2.63

table 3: homogeneity test of subsamples #1033

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the target methods or with the reproducibility calculate using the Horwitz equation in agreement with the procedure of ISO 13528, Annex B2 in the next table;

	Density at 15˚C in kg/L	Total impurities in %M/M
r (sample #1033)	0.00001	0.05
reference test method	ASTM D4052:02e1	Horwitz
0.3xR (reference test)	0.00015	0.23

table 4: evaluation of the subsamples #1033

The calculated repeatabilities were in agreement with 0.3 times the corresponding target reproducibility. Therefore, homogeneity of the samples was assumed.

Depending on the registration to each participant was dispatched on March 24, 2010: one 500 mL bottle of MTBE, labelled #1032 and/or one 500 mL bottle of ETBE, labelled #1033

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were asked to determine on sample #1032 containing MTBE: Appearance, Density @ 15°C, Refractive index @ 20°C, Water, Purity (both on “as-is” and on dry basis) and some GC-impurities (Sum of 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], Methanol, Tert-butanol, C4-, C5-, Other and Unknown Hydrocarbons). The participants were asked to determine on sample #1033 containing ETBE: Appearance,

Density @ 15°C, Sulphur, Nitrogen, Water, Purity (both on “as-is” and on dry basis) and some GC-impurities (Sum of 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], Ethanol, Tert-butanol, C4-, C5-, Other and Unknown Hydrocarbons).
To get comparable results a detailed report form, on which the units were prescribed, was sent together with each set of samples. Also, a letter of instructions and two SDS’s were added to the package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code number.

Directly after the deadline the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. Laboratories that produced these suspect data were asked to check the results. Additional or corrected data are put under 'Remarks' in the result tables in appendix 1. Results that came in after deadline were not taken into account in the screening for suspect data and thus these participants were not requested to check the raw data for obvious errors.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test is described in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (i.i.s.-protocol, version 3.2).

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

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. After removal of outliers this check was repeated. In case a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

Finally, the reproduciblities were calculated from the standard deviations by multiplying these 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 analysis results are plotted. The corresponding laboratory numbers are under 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 standard. 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 method is for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nr.13 and 14).

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 spread of this interlaboratory study.
The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated in accordance with:

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

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

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

4 EVALUATION

In this proficiency test, some problems were encountered during the execution. Participants in Brazil, Saudi Arabia, Turkey and U.A.E received the samples late or not at all due to problems at customs. From the 39 participants, 3 participants reported results after the deadline for reporting and 6 participants did not report any results at all. The 33 reporting laboratories submitted 305 numerical results. Observed were 22 outlying results, which is 7.2 %. In proficiency studies, outlier percentages of 3 % - 7.5 % are quite normal.

Not for all determinations, a normal distribution was found. Anormal distributions were found for: Density (#1032), Water (#1032), Purity “as received” (#1032) and 2,4,4-Trimethyl-1-pentene (#1033). The statistical evaluations for these determinations with anormal distributions should be used with care.

4.1 EVALUATION PER TEST

In this section, the results are discussed per test. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are listed in appendix 3. Regretfully, there is no standard method available for the analysis of ETBE, therefore ASTM D5441, which is valid for MTBE, was also used as reference method for evaluation of the analyses of ETBE. Therefore the evaluation should be used with due care.

Sample #1032 MTBE

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #1032, which is “pass”(acc. ASTM D2680) or bright, clear and free of suspended matter. The uniformity of reporting can be improved. A new standardized method is available for Appearance since 2009, being ASTM E2680. According this method the appearance should be reported as ‘pass’ (or ‘fail’).

Density @ 15°C: This determination was problematic for one laboratory. Only one statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outliers, is in agreement with the requirements of ASTM D4052:02e1.

Refractive Index: This determination was problematic for three laboratories. Three statistical outliers were observed. However, the calculated reproducibility, after rejection of the statistical outliers, is in full agreement with the requirements of ASTM D1218:07.

Water: This determination was not problematic at this level (assigned value of the group = 1571 mg/kg). However, the calculated reproducibility is, after rejection of the statistical outliers, in good agreement with the requirements of ASTM E1064:05.

Purity “as received”: This determination was problematic for several laboratories. Three laboratories reported a result for purity “as received” that was smaller then purity result “on dry basis”. These results were excluded from statistical evaluation. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5441:08e1.

Purity on dry basis: This determination was problematic for several laboratories. Three laboratories reported a result for purity “on dry basis” that was larger then purity result “as received”. These results were excluded from statistical evaluation. One statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is in full agreement with the requirements of ASTM D5441:08e1.

Methanol: This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility, after rejection of the statistical outlier, is in full agreement with the requirements of ASTM D5441:08e1.

Diisobutylene (=sum): This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the estimated reproducibility limits, calculated using the Horwitz equation. When the results were evaluated after manual summation of the reported DIB’s, it was observed that after rejection of one statistical outlier, the consensus value was somewhat smaller, but the spread was the same.

2,4,4-Trime-1-pent.: This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility, after rejection of the statistical outlier, is in full agreement with the requirements of ASTM D5441:08e1.

2,4,4-Trime-2-pent.: This determination was problematic for some laboratories. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the estimated reproducibility limits, calculated using the Horwitz equation.

Other DIB.'s: It should be noticed that for the other DIB's, method D5441:08e1 is applicable for concentrations >0.02 %M/M. Only a few numerical results were reported. Therefore, no significant conclusions were drawn.

Tert-butanol: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D5441:08e1.

C4-hydrocarbons: This determination was problematic. One statistical outlier was observed and the calculated reproducibility, after rejection of the statistical outlier, is not in agreement with the requirements of ASTM D5441:08e1.

C5-hydrocarbons: This determination may be problematic for several laboratories. Two statistical outliers were observed. However, the calculated reproducibility, after rejection of the statistical outliers, is in agreement with the requirements of ASTM D5441:08e1.

Other hydrocarb.+ Unknown Impurities:

The observed large spreads may be explained by the uncertainty of component identification, differences in response factors and co-elution. Other hydrocarbons are all components listed in table 2 of D5441:08e1 and not evaluated separately in this proficiency test.
For unknown impurities, it should be noticed that ASTM D5441:08e1 does not give a definition which "unknown impurities" might be present in MTBE. ASTM states however that for unknown contaminants a response factor of 1.00 should be used.
Finally, it is hard to judge the overall group performance, because ASTM D5441:08e1 does not specify requirements for the calculated reproducibility for these components.

Sample #1033 ETBE

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #1033, which is “pass”(acc. ASTM D2680) or bright, clear and free of suspended matter. The uniformity of reporting can be improved. A new standardized method is available for Appearance since 2009, being ASTM E2680. According this method the appearance should be reported as ‘pass’ (or ‘fail’).

Density @ 15°C: This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility, after rejection of the statistical outlier, is in good agreement with the requirements of ASTM D4052:02e1.

Nitrogen: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D4629:09.

Sulphur: The application range of D5453 is 1.0 – 8000 mg/kg and most participants reported a numerical result near and below this application range. Therefore no significant conclusions were drawn. Only one statistical outlier was observed.

Water: This determination is problematic for several laboratories. Two statistical outliers were observed and the calculated reproducibility, after rejection of the statistical outliers, is not at all in agreement with the requirements of ASTM E1064:05.

Purity “as received”: This determination may be problematic. Two laboratories reported a result for purity “as received” that was smaller then purity result “on dry basis”. These results were excluded from statistical evaluation. One statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is not at all in agreement with the requirements of ASTM D5441:08e1 for MTBE.

Purity “on dry basis”: This determination may be problematic. Two laboratories reported a result for the purity “on dry basis” that was larger then purity result “as received”. These results were excluded from statistical evaluation. One statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is not at all in agreement with the requirements of ASTM D5441:08e1 for MTBE.

Ethanol: This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D5441:08e1 for MTBE.

Diisobutylene (=sum): This determination may be not problematic. One statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier, is in agreement with the estimated reproducibility limits, calculated using the Horwitz equation. When the results were evaluated after manual summation of the reported DIB’s, it was observed that after rejection of one statistical outlier, the consensus value was the same. The spread was somewhat smaller.

2,4,4-Trime-1-pent.: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility, after rejection of the statistical outlier, is in full agreement with the requirements of ASTM D5441:08e1 for MTBE.

2,4,4-Trime-2-pent.: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with the estimated reproducibility limits, calculated using the Horwitz equation.

Other DIB.'s: It should be noticed that for the other DIB's, method D5441:08e1 for MTBE is applicable for concentrations >0.02 %M/M. Only a few numerical results were reported. Therefore, no significant conclusions were drawn.

Tert-butanol: This determination may be problematic for one or more laboratories. Two statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers, is in good agreement with the requirements of ASTM D5441:08e1 for MTBE.

C4-hydrocarbons: This determination may be problematic for one laboratory. One statistical outlier was observed. However, the calculated reproducibility, after rejection of the statistical outlier, is in full agreement with the requirements of ASTM D5441:08e1 for MTBE.

C5-hydrocarbons: This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D5441:08e1 for MTBE.

Other hydrocarb.+ Unknown impurities:

Only a few numerical results were reported for Other Hydrocarbons and Unknown impurities. Therefore, no significant conclusions were drawn.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	R (Calc.)	R (lit)
Density @ 15°C	kg/L	18	0.74651	0.00045	0.00050
Refractive Index @ 20ºC		9	1.36966	0.00047	0.00050
Water	mg/kg	18	1571	144	250
Purity	%M/M	11	98.3123	0.2979	0.3023
Purity on dry basis	%M/M	12	98.4722	0.2905	0.3014
Methanol	%M/M	14	0.2184	0.0637	0.0680
Diisobutylene (=sum of 5)	% M/M	7	0.2139	0.0896	0.0676
2,4,4-Trimethyl-1-pentene	%M/M	9	0.1333	0.0225	0.0441
2,4,4-Trimethyl-2-pentene	%M/M	11	0.0404	0.0137	0.0073
Tert-butanol	%M/M	14	0.2083	0.0287	0.1320
C4 – hydrocarbons	%M/M	10	0.1163	0.0564	0.0388
C5 – hydrocarbons	%M/M	9	0.2360	0.0408	0.0629
Other hydrocarbons	%M/M	5	0.3907	0.6375	unknown
Unknown impurities	%M/M	6	0.5847	1.0192	unknown

table 5: reproducibilities of results of sample #1032 (MTBE)

Parameter	unit	n	average	2.8*sd	R(lit)
Density @ 15°C	kg/L	13	0.74631	0.00021	0.00050
Nitrogen	mg/kg	8	4.14	3.17	1.68
Sulphur	mg/kg	7	(0.32)	(0.49)	(0.25)
Water	mg/kg	12	64.07	27.99	10.19
Purity	%M/M	7	97.3584	0.5170	0.3077
Purity on dry basis	%M/M	8	97.3667	0.5017	0.3076
Ethanol	%M/M	10	0.8521	0.1472	0.0955
Diisobutylene (=sum of 5)	%M/M	5	0.3848	0.0897	0.1113
2,4,4-Trimethyl-1-pentene	%M/M	6	0.3029	0.0491	0.0869
2,4,4-Trimethyl-2-pentene	%M/M	6	0.0841	0.0138	0.0137
Tert-butanol	%M/M	7	0.3904	0.0180	0.1320
C4 – hydrocarbons	%M/M	8	0.3383	0.0945	0.1098
C5 – hydrocarbons	%M/M	8	0.0422	0.0387	0.0199
Other hydrocarbons	%M/M	4	0.5904	1.6294	unknown
Unknown impurities	%M/M	3	0.6017	unknown	unknown

table 6: reproducibilities of results of sample #1033 (ETBE)

Note: Results between brackets are estimates, as the assigned value is below or the detection limit.

Without further statistical calculations, it can be concluded that for many tests for MTBE there is a good compliance of the group of participating laboratories with the relevant standards. However, several tests for ETBE are much more problematic. However, it can be concluded that ASTM D5441 is suitable for the analysis of ETBE.
The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2010 WITH PREVIOUS PT'S

	March 2010	March 2008	April 2007
Number of reporting labs	33	23	19
Number of Results reported	305	400	364
Statistical outliers	22	38	19
Percentage outliers	7.2 %	9.5 %	8.2 %

table 7: comparison with previous proficiency tests.

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

The performance of the determinations for the MTBE and ETBE samples of the proficiency test were compared against the requirements of the respective standards. The conclusions are given the following table:

Determination	March 2010	March 2008	April 2007
MTBE			
Density @ 15°C	+	+	++
Refractive Index @ 20ºC	+	--	--
Water	++	++	--
Purity as received	+	-	--
Purity on dry basis	+	-	--
Methanol	+	-	+/-
2,4,4-Trimethyl-1-pentene	++	+	+/-
2,4,4-Trimethyl-2-pentene	-- *)	+ *)	-- *)
Tert-butanol	++	++	+
C4 – hydrocarbons	--	+	+
C5 – hydrocarbons	++	+	--

table 8: comparison determinations against the standard for MTBE only

*) compared against the strict Horwitz equation

result between brackets is an estimate, as the assigned value is below or the detection limit.

Determination	March 2010	March 2008	April 2007
ETBE			
Density @ 15°C	++	++	++
Nitrogen	--	--	--
Sulphur	(--)	(+)	(--)
Water	--	++	--
Purity	--	--	--
Purity on dry basis	--	--	--
Ethanol	--	+	--
2,4,4-Trimethyl-1-pentene	++	+/-	-
2,4,4-Trimethyl-2-pentene	+/- *)	++ *)	++ *)
Tert-butanol	++	-	-
C4 – hydrocarbons	+	--	+
C5 – hydrocarbons	--	--	--

table 9: comparison determinations against the standard for ETBE only

*) compared against the strict Horwitz equation

result between brackets is an estimate, as the assigned value is below or near the detection limit.

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

- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- - : group performed worse than the standard
- -- : group performed much worse than the standard

APPENDIX 1

Determination of Appearance on MTBE sample #1032;

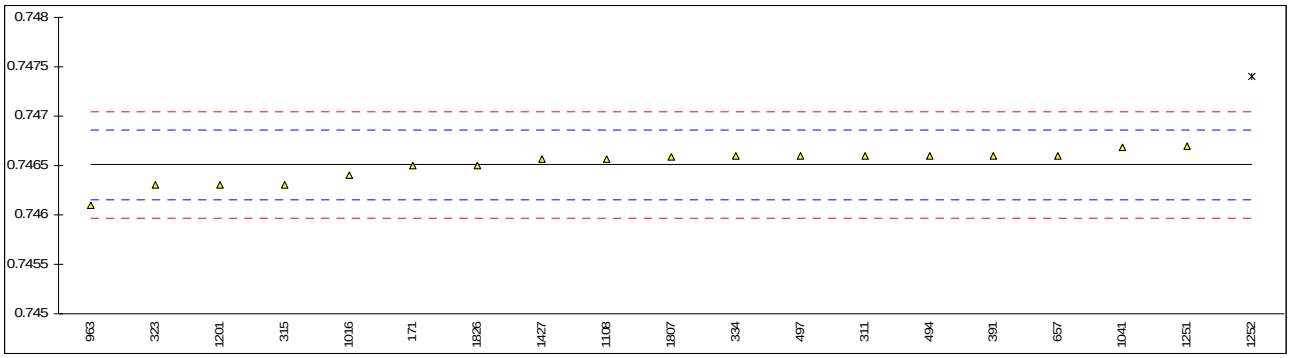
lab	method	value	mark	z(targ)	Remarks
171	E2680	C&F		-----	
311	INH-402	C&F		-----	
315	INH-402	B&C		-----	
323	E2680	PASS		-----	
334		-----		-----	
391		-----		-----	
494	E2680	PASS		-----	
497	E2680	B&C		-----	
657	E2680	PASS		-----	
963	E2680	PASS		-----	
974		-----		-----	
1016	In house	PASS		-----	
1041		-----		-----	
1108		-----		-----	
1201	E2680	B&C		-----	
1251	E2680	B&C		-----	
1252	E2680	Clear		-----	
1427		-----		-----	
1807	D4176	B&C		-----	
1826	E2680	PASS		-----	
1833		-----		-----	

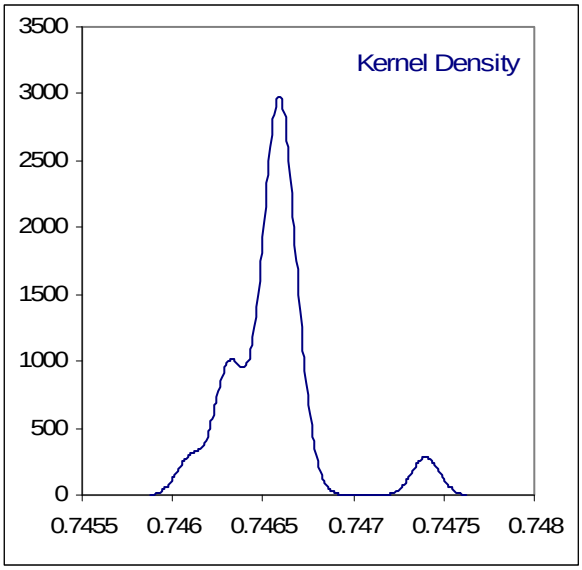
C&F = Clear and Free of particles
B&C = Bright and clear

Determination of Density @ 15°C on MTBE sample #0822; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.7465		-0.03	
311	D4052	0.7466		0.53	
315	D4052	0.7463		-1.15	
323	D4052	0.7463		-1.15	
334	D4052	0.7466		0.53	
391	D4052	0.7466		0.53	
494	D4052	0.7466		0.53	
497	D4052	0.7466		0.53	
657	D4052	0.7466		0.53	
963	D4052	0.7461		-2.27	
974		-----		-----	
1016	D4052	0.7464		-0.59	
1041	D4052	0.74669	C	1.03	First reported 746.69
1108	D4052	0.74657		0.36	
1201	D4052	0.7463		-1.15	
1251	D4052	0.7467		1.09	
1252	D4052	0.7474	G(0.01)	5.01	
1427	D4052	0.74656		0.30	
1807	D4052	0.74659		0.47	
1826	D4052	0.7465		-0.03	
1833		-----		-----	

normality	not OK
n	18
outliers	1
mean (n)	0.74651
st.dev. (n)	0.000161
R(calc.)	0.00045
R(D4052:02e1)	0.00050

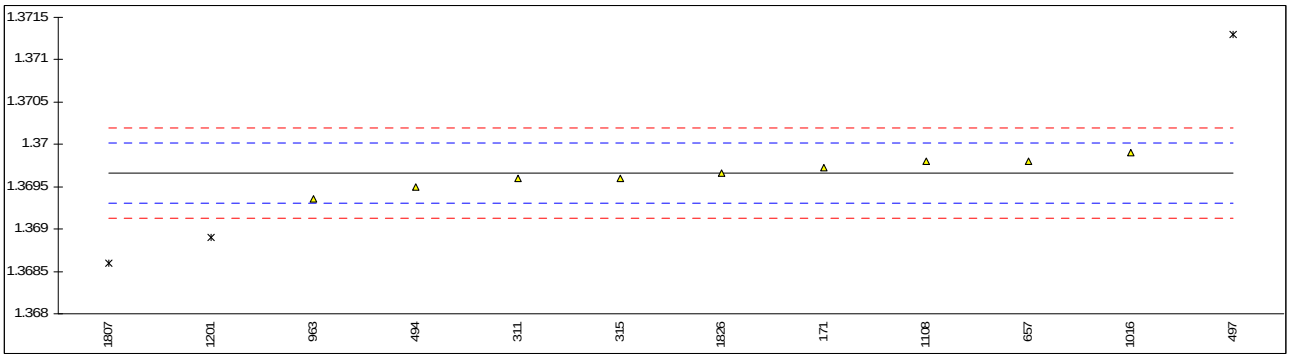


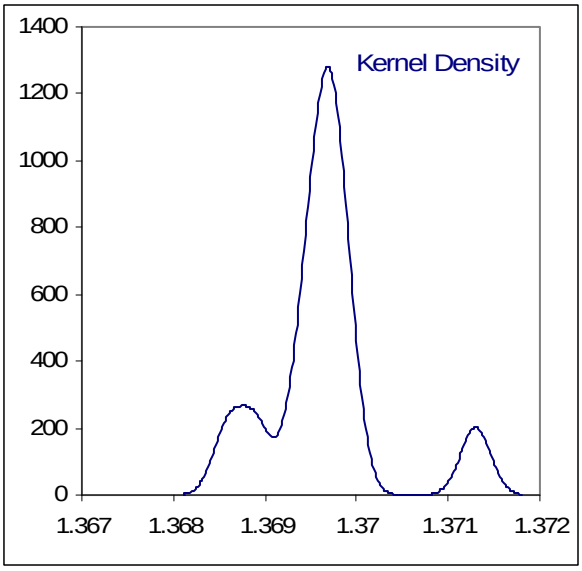


Determination of Refractive Index @ 20°C on MTBE sample #1032;

Lab	method	value	mark	z(targ)	remarks
171	D1218	1.36973		0.39	
311	D1218	1.3696	C	-0.34	First reported 1.3656
315	D1218	1.3696		-0.34	
323		-----		-----	
334		-----		-----	
391		-----		-----	
494	D1218	1.3695		-0.90	
497	D1218	1.3713	G(0.05)	9.18	
657	D1218	1.36980		0.78	
963	D1218	1.36936		-1.69	
974		-----		-----	
1016	D1218	1.3699		1.34	
1041		-----		-----	
1108	D1218	1.3698		0.78	
1201	D1218	1.3689	DG(0.01)	-4.26	
1251		-----		-----	
1252		-----		-----	
1427		-----		-----	
1807	D1218	1.3686	DG(0.01)	-5.94	
1826	D1218	1.36966		-0.01	
1833		-----		-----	

normality	OK
n	9
outliers	3
mean (n)	1.36966
st.dev. (n)	0.000168
R(calc.)	0.00047
R(D1218:07)	0.00050

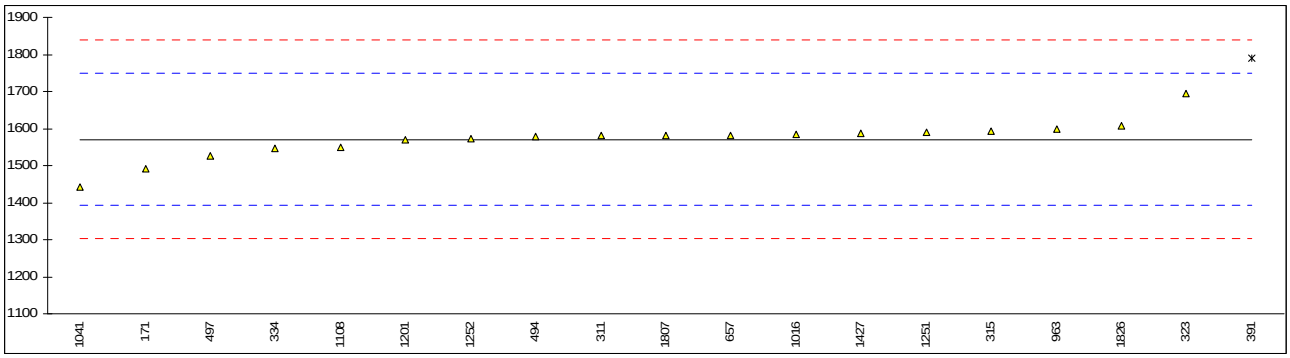


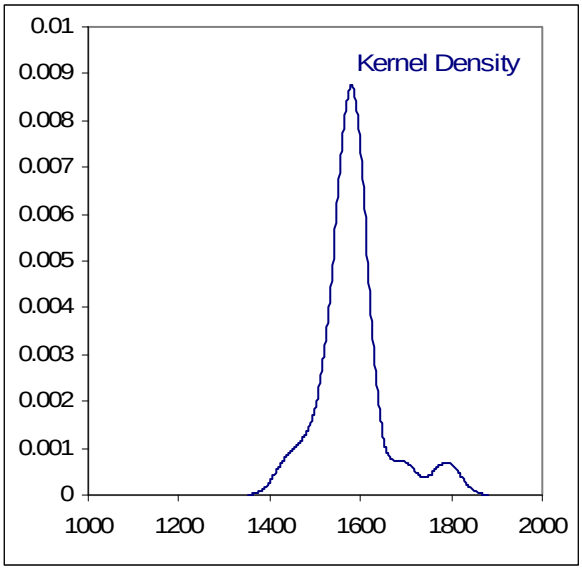


Determination of Water on MTBE sample #1032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	E1064	1492	C	-0.88	First reported 0.1492
311	E1064	1580		0.11	
315	E1064	1593		0.25	
323	E1064	1693		1.37	
334	E1064	1545		-0.29	
391	E1064	1790	G(0.05)	2.46	
494	E1064	1579		0.09	
497	E1064	1526		-0.50	
657	E1064	1580		0.11	
963	E1064	1600		0.33	
974		-----		-----	
1016	D1364	1583	C	0.14	First reported 0.1583
1041	E1064	1441		-1.45	
1108	E1064	1550		-0.23	
1201	E1064	1570		-0.01	
1251	E1064	1590		0.22	
1252	E1064	1573		0.03	
1427	D1364	1587		0.18	
1807	D1744	1580		0.11	
1826	E1064	1608	C	0.42	First reported 0.1608
1833		-----		-----	

normality	not OK
n	18
outliers	1
mean (n)	1570.6
st.dev. (n)	51.41
R(calc.)	143.9
R(E1064:05)	249.7

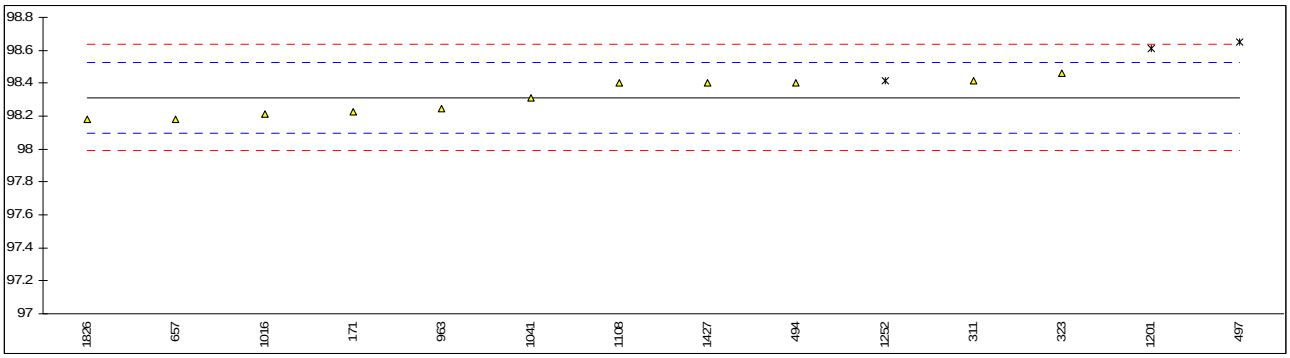


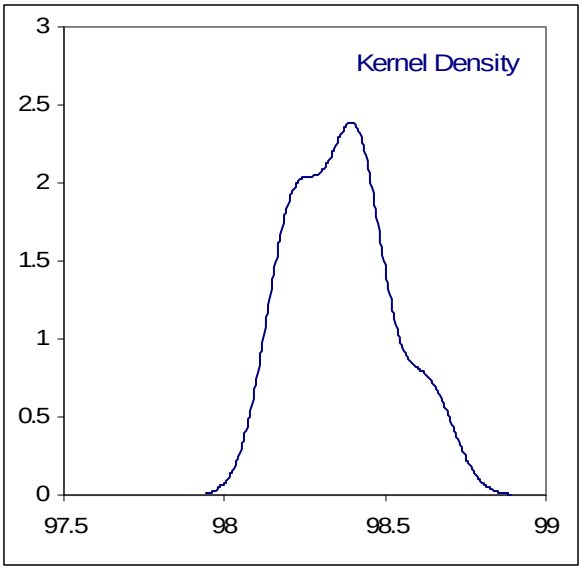


Determination of Purity “as received” of MTBE sample #1032; results in %M/M

Lab	method	value	mark	z(targ)	remarks
171	D5441	98.227	C	-0.79	First reported 98.248
311	D5441	98.418		0.98	
315		-----		-----	
323	D5441	98.46		1.37	
334		-----		-----	
391		-----		-----	
494	D5441	98.40		0.81	
497	D5441	98.653	ex	3.16	Result exclude as purity “as received” > purity on dry basis
657	D5441	98.179		-1.23	
963	D5441	98.246		-0.61	
974		-----		-----	
1016	D5441	98.215		-0.90	
1041	D5441	98.3126		0.00	
1108	D5441	98.40		0.81	
1201	D5441	98.61	ex	2.76	Result exclude as purity “as received” > purity on dry basis
1251		-----		-----	
1252	D5441	98.4163	ex	0.96	Result exclude as purity “as received” > purity on dry basis
1427	D5441	98.400		0.81	
1807		-----		-----	
1826	INHOUSE	98.1772		-1.25	
1833		-----		-----	

normality	not OK
n	11
outliers	0
mean (n)	98.3123
st.dev. (n)	0.10641
R(calc.)	0.2979
R(D5441:08e1)	0.3023

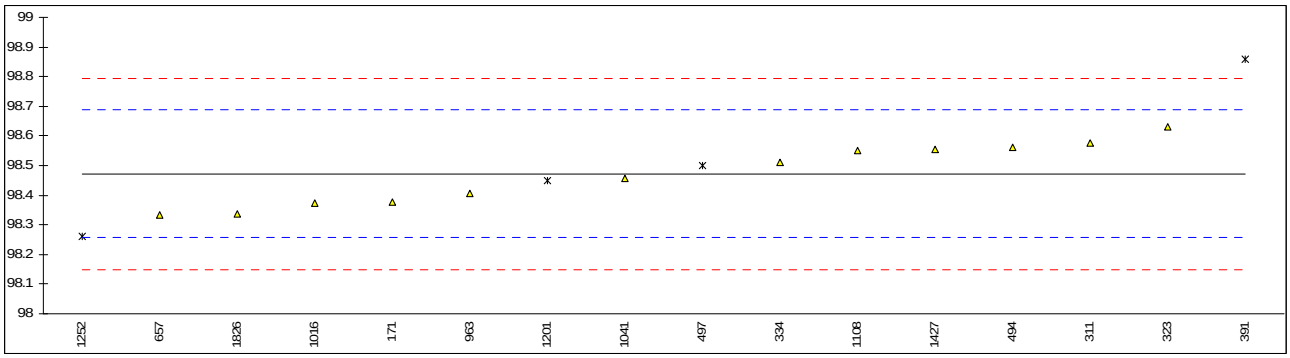


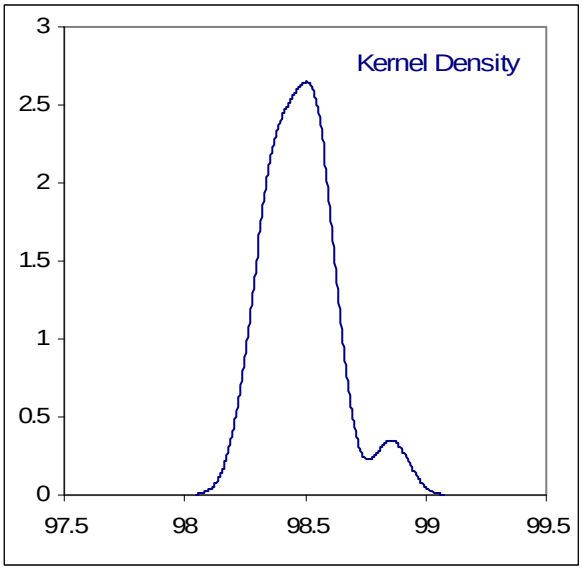


Determination of Purity on dry basis on MTBE sample #1032; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	98.376	C	-0.89	First reported 98.397
311	D5441	98.576		0.96	
315		-----		-----	
323	D5441	98.63		1.47	
334	D5441	98.51		0.35	
391	D5441	98.86	G(0.01)	3.60	
494	D5441	98.56		0.82	
497	D5441	98.500	ex	0.26	Result exclude as purity on dry basis < purity “as received”
657	D5441	98.335		-1.27	
963	D5441	98.406		-0.62	
974		-----		-----	
1016	D5441	98.373		-0.92	
1041	D5441	98.4567		-0.14	
1108	D5441	98.55		0.72	
1201	D5441	98.45	ex	-0.21	Result exclude as purity on dry basis < purity “as received”
1251		-----		-----	
1252	D5441	98.2615	ex	-1.96	Result exclude as purity on dry basis < purity “as received”
1427	D5441	98.556		0.78	
1807		-----		-----	
1826	INHOUSE	98.338		-1.25	
1833		-----		-----	

normality	OK
n	12
outliers	1
mean (n)	98.4722
st.dev. (n)	0.10377
R(calc.)	0.2905
R(D5441:08e1)	0.3014

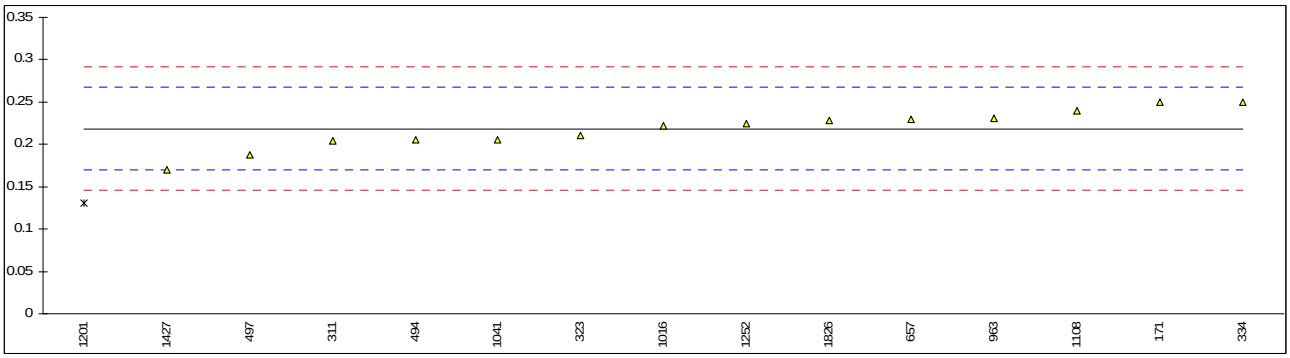


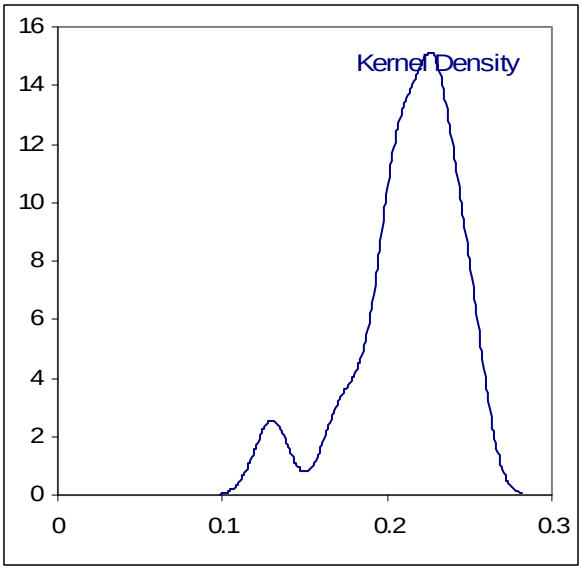


Determination of Methanol on MTBE sample #1032; results in %M/M

Lab	method	value	mark	z(targ)	remarks
171	D5441	0.250		1.30	
311	D5441	0.204		-0.59	
315		-----		-----	
323	D5441	0.21		-0.35	
334	D5441	0.25		1.30	
391		-----		-----	
494	D5441	0.205		-0.55	
497	D5441	0.188		-1.25	
657	D5441	0.230		0.48	
963	D5441	0.231		0.52	
974		-----		-----	
1016	D5441	0.222		0.15	
1041	D5441	0.2055		-0.53	
1108	D5441	0.24		0.89	
1201	D5441	0.13	G(0.05)	-3.64	
1251		-----		-----	
1252	D5441	0.2239		0.23	
1427	D5441	0.1705		-1.97	
1807		-----		-----	
1826	INHOUSE	0.228		0.39	
1833		-----		-----	

normality	OK
n	14
outliers	1
mean (n)	0.2184
st.dev. (n)	0.02276
R(calc.)	0.0637
R(D5441:08e1)	0.0680

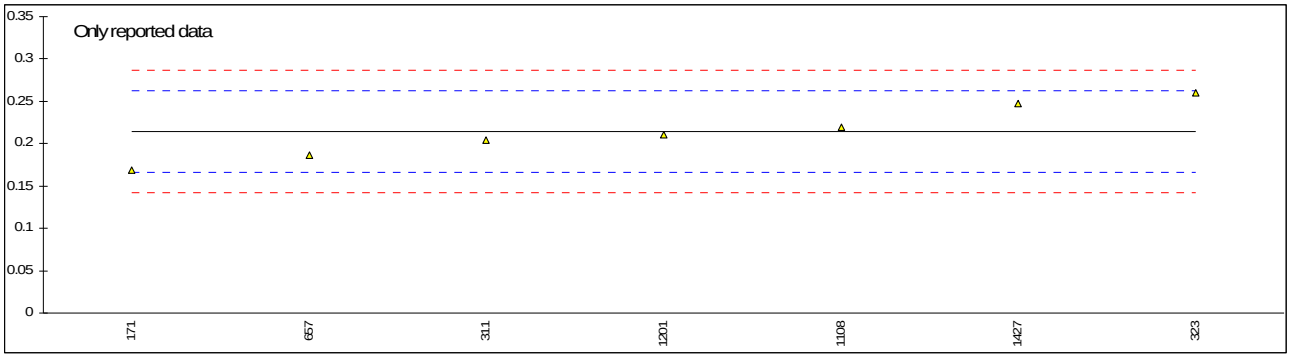


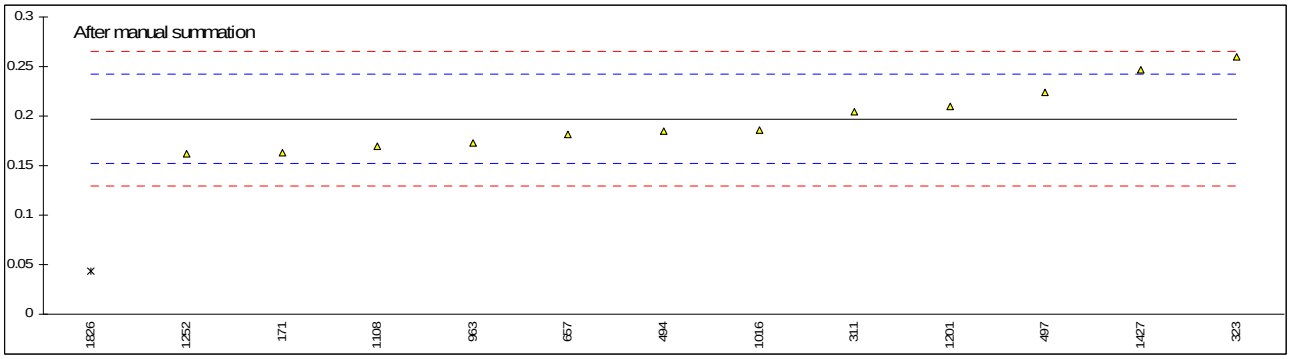


Determination of Diisobutylene (=sum 5 DIB's*) on MTBE sample #1032; results in %M/M

lab	method	value	mark	z(targ)	value after manual summation	mark	z(targ)	remarks
171	D5441	0.169	C	-1.86	0.163		-1.52	First reported 0.158
311	D5441	0.204		-0.41	0.204		0.30	
315		-----		-----	-----		-----	
323	D5441	0.26		1.91	0.26		2.79	
334		-----		-----	-----		-----	
391		-----		-----	-----		-----	
494		-----		-----	0.185		-0.54	
497		-----		-----	0.224		1.19	
657	D5441	0.187		-1.12	0.182		-0.67	
963		-----		-----	0.173		-1.07	
974		-----		-----	-----		-----	
1016		-----		-----	0.186		-0.50	
1041		-----		-----			-----	
1108	D5441	0.22		0.25	0.170		-1.21	
1201	D5441	0.21		-0.16	0.21		0.57	
1251		-----		-----	-----		-----	
1252		-----		-----	0.162		-1.56	
1427	D5441	0.2475		1.39	0.247		2.21	
1807		-----		-----	-----		-----	
1826		-----		-----	0.044	G(0.05)	-6.80	
1833		-----		-----	-----		-----	
	normality	OK			OK			
	n	7			12			
	outliers	0			1			
	mean (n)	0.2139			0.1972			
	st.dev. (n)	0.03200			0.03246			
	R(calc.)	0.0896			0.0909			
	R(Horwitz.)	0.0676			0.0630			

* 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.

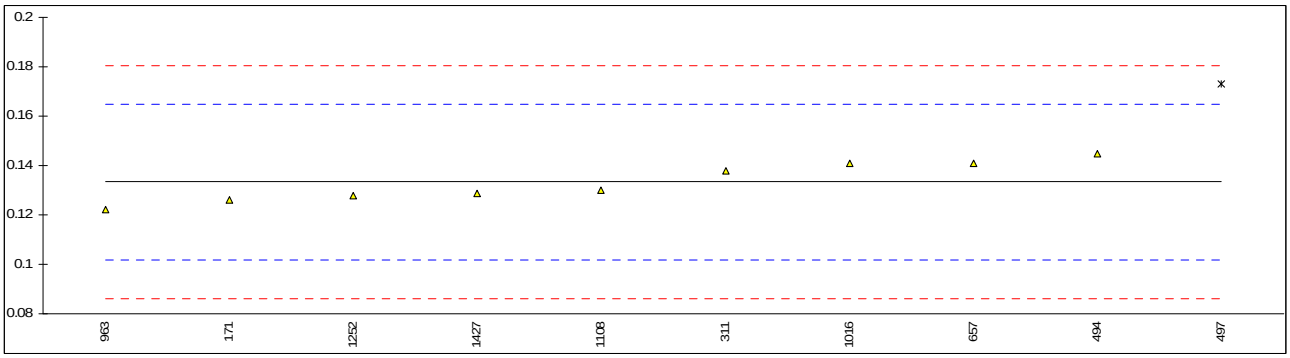


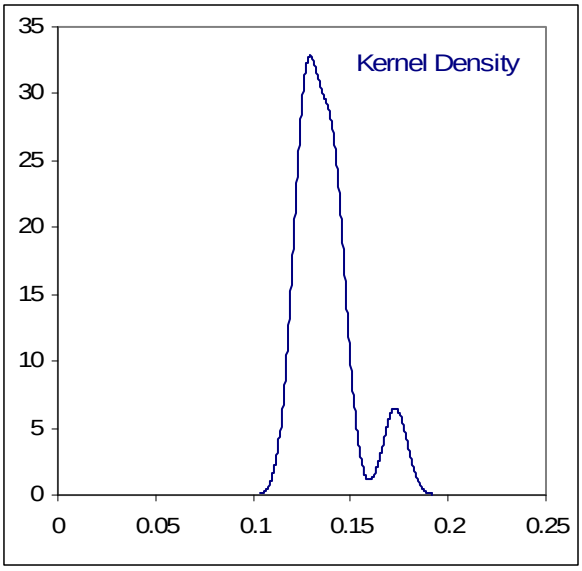


Determination of 2,4,4-Trimethyl-1-pentene on MTBE sample #1032; results in %M/M

Lab	method	value	mark	z(targ)	remarks
171	D5441	0.126		-0.46	
311	D5441	0.138		0.30	
315		-----		-----	
323		-----		-----	
334		-----		-----	
391		-----		-----	
494	D5441	0.145		0.74	
497	D5441	0.173	G(0.01)	2.52	
657	D5441	0.141		0.49	
963	D5441	0.122		-0.72	
974		-----		-----	
1016	D5441	0.141		0.49	
1041		-----		-----	
1108	D5441	0.13		-0.21	
1201		-----		-----	
1251		-----		-----	
1252	D5441	0.1280		-0.34	
1427	D5441	0.1287		-0.29	
1807		-----		-----	
1826		-----		-----	
1833		-----		-----	

normality	OK
n	9
outliers	1
mean (n)	0.1333
st.dev. (n)	0.00805
R(calc.)	0.0225
R(D5441:08e1)	0.0441

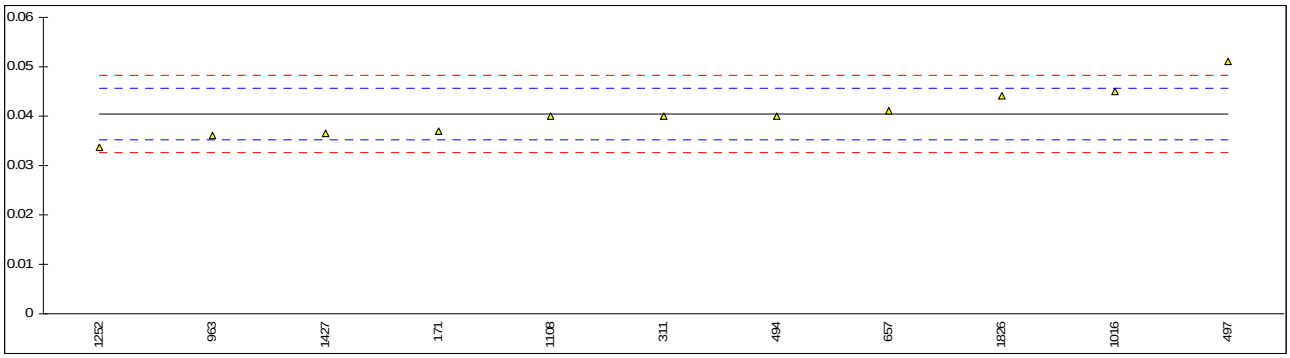


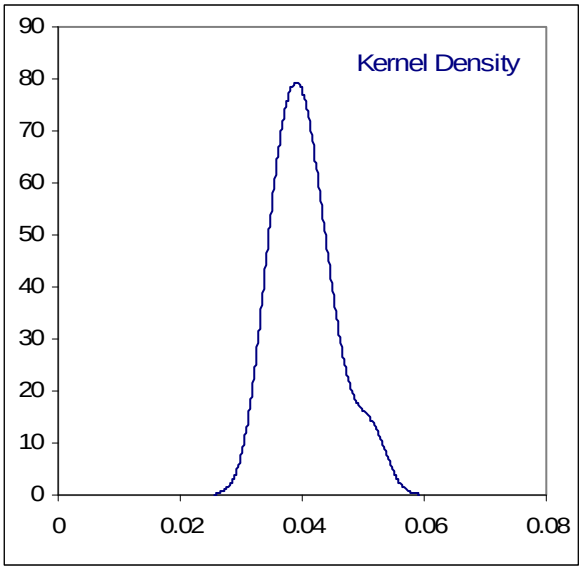


Determination of 2,4,4-Trimethyl-2-pentene on MTBE sample #1032; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.037	C	-1.30	First reported 0.030
311	D5441	0.040		-0.15	
315		-----		-----	
323		-----		-----	
334		-----		-----	
391		-----		-----	
494	D5441	0.04		-0.15	
497	D5441	0.051		4.05	
657	D5441	0.041		0.23	
963	D5441	0.036		-1.68	
974		-----		-----	
1016	D5441	0.045		1.76	
1041		-----		-----	
1108	D5441	0.04		-0.15	
1201		-----		-----	
1251		-----		-----	
1252	D5441	0.0338		-2.52	
1427	D5441	0.0365		-1.49	
1807		-----		-----	
1826	INHOUSE	0.0441		1.41	
1833		-----		-----	

normality	OK
n	11
outliers	0
mean (n)	0.0404
st.dev. (n)	0.00488
R(calc.)	0.0137
R(Horwitz)	0.0073





Determination of other individual diisobutylenes on MTBE sample #1032; results in %M/M

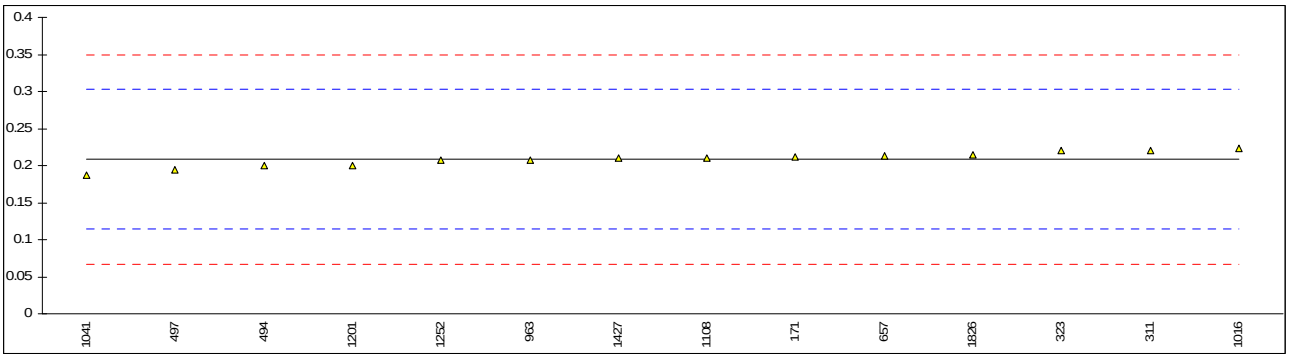
Lab	method	243T2P *)	mark	z(targ)	344T1P *)	mark	z(targ)	35D1H *)	mark	z(targ)	remarks
171	D5441	<0.01		-----	<0.01		-----	<0.01		-----	
311	D5441	0.026		-----	<0.02		-----	<0.02		-----	
315		-----		-----	n		-----	n		-----	
323		-----		-----	n		-----	n		-----	
334		-----		-----	n		-----	n		-----	
391		-----		-----	n		-----	n		-----	
494		-----		-----	n		-----	n		-----	
497		-----		-----	n		-----	n		-----	
657	D5441	<0.02		-----	<0.02		-----	<0.02		-----	
963	D5441	0.015		-----	n		-----	n		-----	
974		-----		-----	n		-----	n		-----	
1016		-----		-----	n		-----	n		-----	
1041		-----		-----	n		-----	n		-----	
1108		-----		-----	n		-----	n		-----	
1201		-----		-----	n		-----	n		-----	
1251		-----		-----	n		-----	n		-----	
1252		-----		-----	n		-----	n		-----	
1427	D5441	0.0277		-----	0.0545		-----	n		-----	
1807		-----		-----	n		-----	n		-----	
1826		-----		-----	n		-----	n		-----	
1833		-----		-----	n		-----	n		-----	
	normality	n.a.			n.a.			n.a.			
	n	3			1			0			
	outliers	0			0			0			
	mean (n)	0.0229			n.a.			n.a.			
	st.dev. (n)	n.a.			n.a.			n.a.			
	R(calc.)	n.a.			n.a.			n.a.			
	R(lit.)	n.a.			n.a.			n.a.			

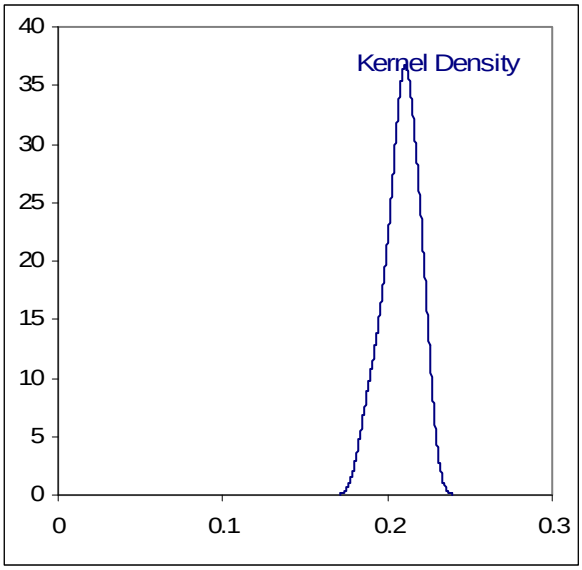
* 2,4,3-Trimethyl-2-pentene
* 3,4,4-Trimethyl-1-pentene
* 3,5-Dimethyl-1-hexene
** reported as sum of 2,3,4-Trimethyl-2-pentene, 3,4,4-Trimethyl-1-pentene and 3,5-Dimethyl-1-hexene

Determination of Tert-butanol on MTBE sample #1032; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.211		0.06	
311	D5441	0.220		0.25	
315		-----		-----	
323	D5441	0.22		0.25	
334		-----		-----	
391		-----		-----	
494	D5441	0.20		-0.18	
497	D5441	0.194		-0.30	
657	D5441	0.213		0.10	
963	D5441	0.207		-0.03	
974		-----		-----	
1016	D5441	0.223		0.31	
1041	D5441	0.1870		-0.45	
1108	D5441	0.21		0.04	
1201	D5441	0.20		-0.18	
1251		-----		-----	
1252	D5441	0.2066		-0.04	
1427	D5441	0.2099		0.03	
1807		-----		-----	
1826	INHOUSE	0.215		0.14	
1833		-----		-----	

normality	OK
n	14
outliers	0
mean (n)	0.2083
st.dev. (n)	0.01027
R(calc.)	0.0287
R(D5441:08e1)	0.1320

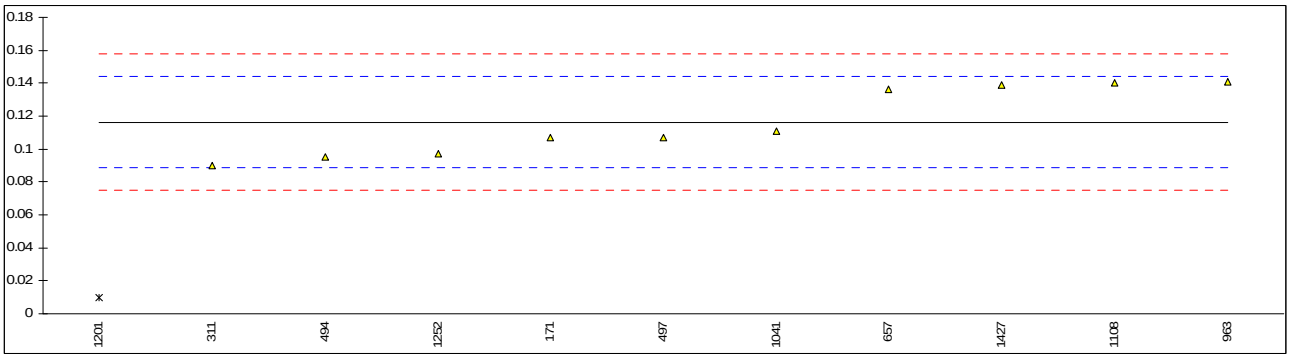


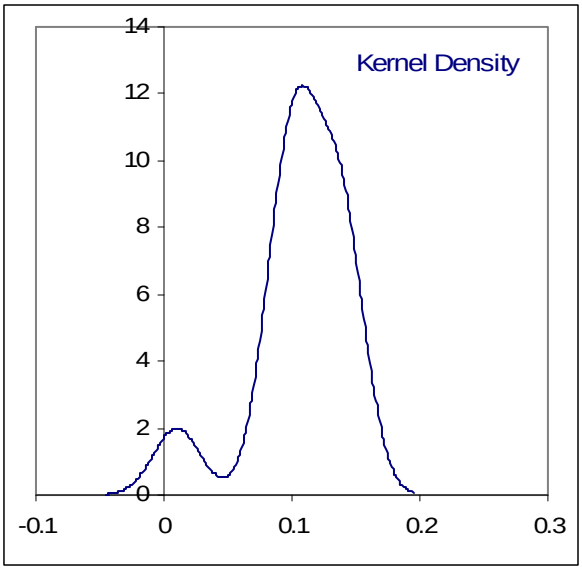


Determination of C4-hydrocarbons on MTBE sample #1032; results in %M/M

Lab	method	value	mark	z(targ)	remarks
171	D5441	0.107		-0.67	
311	D5441	0.09		-1.90	
315		-----		-----	
323		-----		-----	
334		-----		-----	
391		-----		-----	
494	D5441	0.095		-1.54	
497	D5441	0.107		-0.67	
657	D5441	0.136		1.42	
963	D5441	0.141		1.78	
974		-----		-----	
1016		-----		-----	
1041	D5441	0.1110		-0.39	
1108	D5441	0.14		1.71	
1201	D5441	0.01	G(0.01)	-7.68	
1251		-----		-----	
1252	D5441	0.0973		-1.37	
1427	D5441	0.1391		1.64	
1807		-----		-----	
1826		-----		-----	
1833		-----		-----	

normality	OK
n	10
outliers	1
mean (n)	0.1163
st.dev. (n)	0.02051
R(calc.)	0.0574
R(D5441:08e1)	0.0388

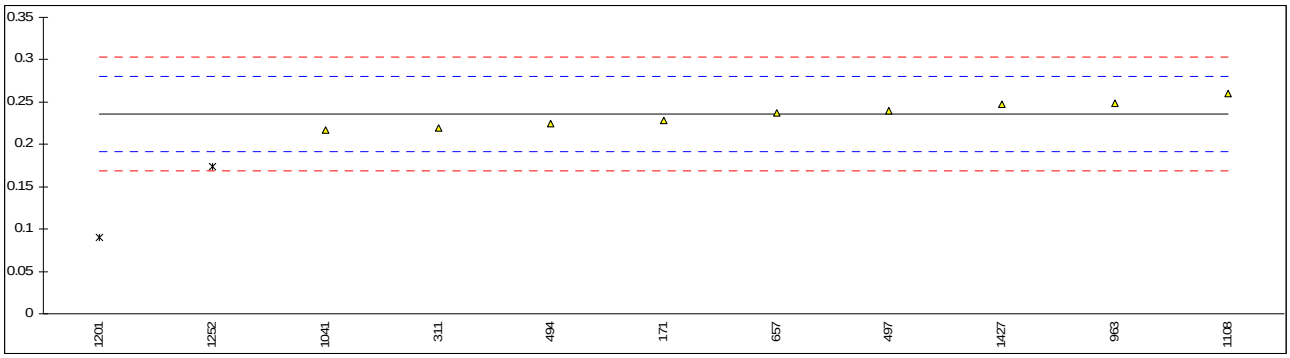


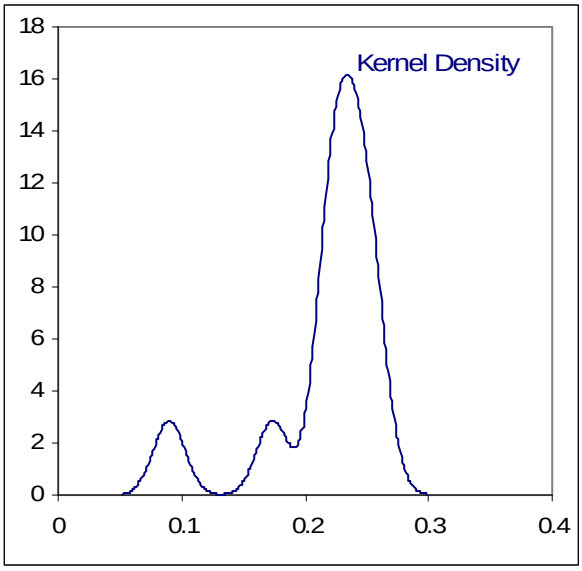


Determination of C5-hydrocarbons on MTBE sample #1032; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.228	C	-0.35	First reported 0.244
311	D5441	0.22		-0.71	
315		-----		-----	
323		-----		-----	
334		-----		-----	
391		-----		-----	
494	D5441	0.225		-0.49	
497	D5441	0.240		0.18	
657	D5441	0.237		0.05	
963	D5441	0.249		0.58	
974		-----		-----	
1016		-----		-----	
1041	D5441	0.2170		-0.84	
1108	D5441	0.26		1.07	
1201	D5441	0.09	G(0.01)	-6.50	
1251		-----		-----	
1252	D5441	0.1736	G(0.05)	-2.78	
1427	D5441	0.2477		0.52	
1807		-----		-----	
1826		-----		-----	
1833		-----		-----	

normality	OK
n	9
outliers	2
mean (n)	0.2360
st.dev. (n)	0.01458
R(calc.)	0.0408
R(D5441:08e1)	0.0629

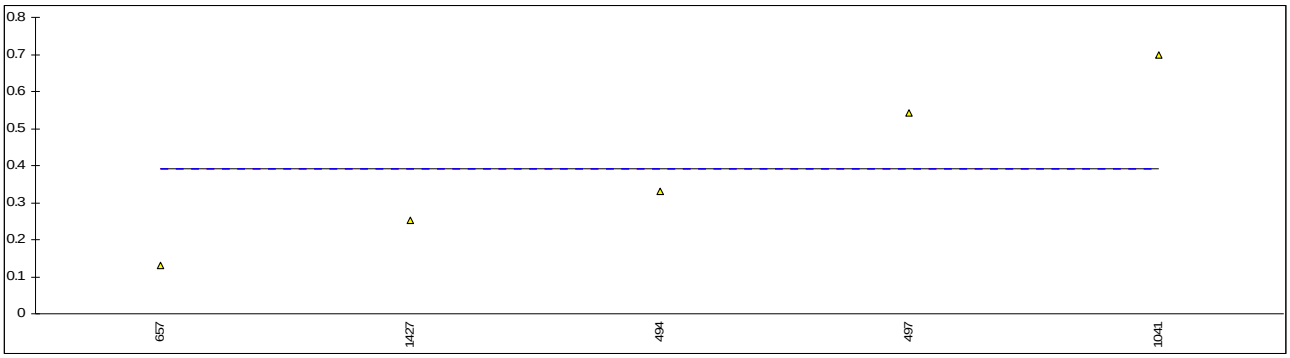




Determination of Other Hydrocarbons on MTBE sample #1032; results in %M/M

Lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311		-----		-----	
315		-----		-----	
323		-----		-----	
334		-----		-----	
391		-----		-----	
494	D5441	0.33		-----	
497	D5441	0.541		-----	
657	D5441	0.131		-----	
963		-----		-----	
974		-----		-----	
1016		-----		-----	
1041	D5441	0.6985		-----	
1108		-----		-----	
1201		-----		-----	
1251		-----		-----	
1252		-----		-----	
1427	D5441	0.2532		-----	
1807		-----		-----	
1826		-----		-----	
1833		-----		-----	

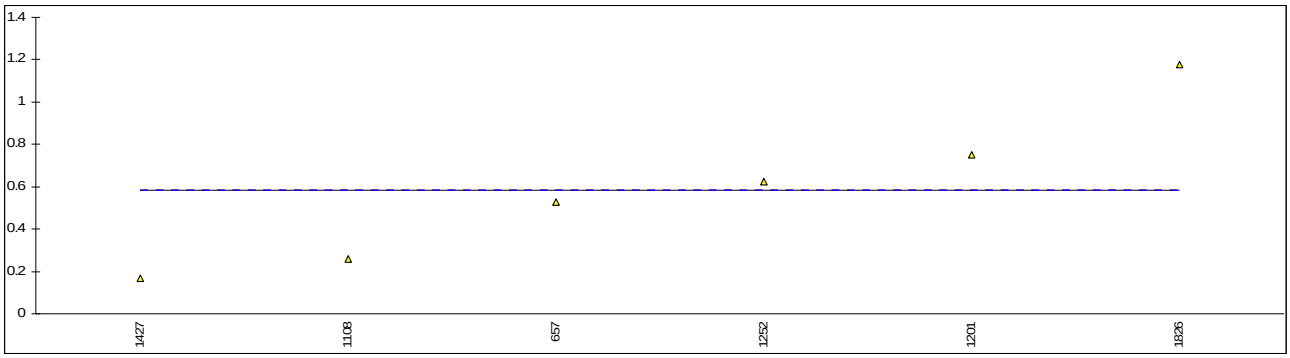
normality	OK
n	5
outliers	0
mean (n)	0.3907
st.dev. (n)	0.22769
R(calc.)	0.6375
R(lit.)	unknown



Determination of Unknown Impurities on MTBE sample #1032; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311		-----		-----	
315		-----		-----	
323		-----		-----	
334		-----		-----	
391		-----		-----	
494		-----		-----	
497		-----		-----	
657	D5441	0.529		-----	
963		-----		-----	
974		-----		-----	
1016		-----		-----	
1041		-----		-----	
1108	D5441	0.26		-----	
1201	D5441	0.75		-----	
1251		-----		-----	
1252	D5441	0.6226		-----	
1427	D5441	0.1686		-----	
1807		-----		-----	
1826	INHOUSE	1.178		-----	
1833		-----		-----	

normality	OK
n	6
outliers	0
mean (n)	0.5847
st.dev. (n)	0.36399
R(calc.)	1.0192
R(lit.)	unknown



Determination of Appearance on ETBE sample #1033;

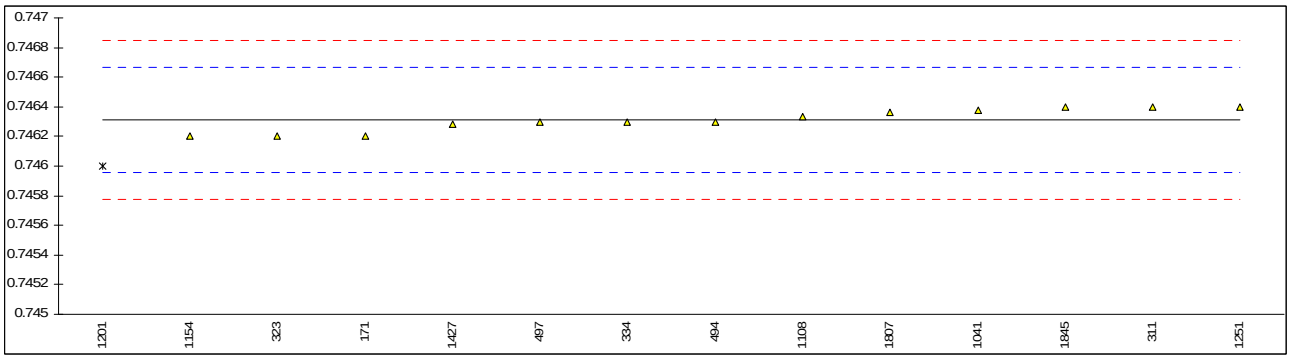
lab	method	value	mark	z(targ)	remarks
171	E2680	C&F		-----	
311	INH-402	C&F		-----	
323	E2680	PASS		-----	
334		-----		-----	
494	E2680	PASS		-----	
497	E2680	B&C		-----	
555		-----		-----	
974		-----		-----	
1041		-----		-----	
1108		-----		-----	
1154		-----		-----	
1201	E2680	B&C		-----	
1251	E2680	B&C		-----	
1291		-----		-----	
1427		-----		-----	
1653		-----		-----	
1807	D4176	B&C		-----	
1845		-----		-----	

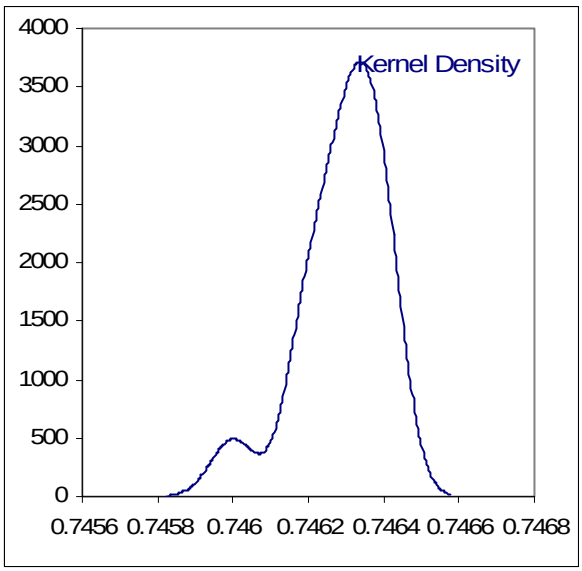
C&F = Clear and Free of particles
B&C = Bright and clear

Determination of Density @ 15°C on ETBE sample #1033; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.7462		-0.62	
311	D4052	0.7464		0.50	
323	D4052	0.7462		-0.62	
334	D4052	0.7463		-0.06	
494	D4052	0.7463		-0.06	
497	D4052	0.7463		-0.06	
555		-----		-----	
974		-----		-----	
1041	D4052	0.74638	C	0.38	
1108	D4052	0.74633		0.10	
1154	ISO12185	0.7462		-0.62	
1201	D4052	0.7460	G(0.05)	-1.74	
1251	D4052	0.7464		0.50	
1291		-----		-----	
1427	D4052	0.74628		-0.18	
1653		-----		-----	
1807	D4052	0.74636		0.27	
1845	D4052	0.7464		0.50	

normality	OK
n	13
outliers	1
mean (n)	0.74631
st.dev. (n)	0.000076
R(calc.)	0.00021
R(D4052:02e1)	0.00050

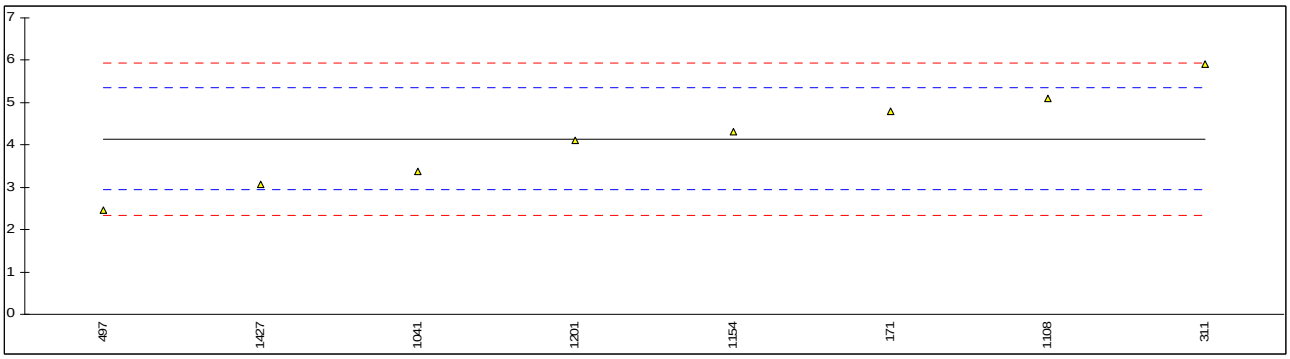


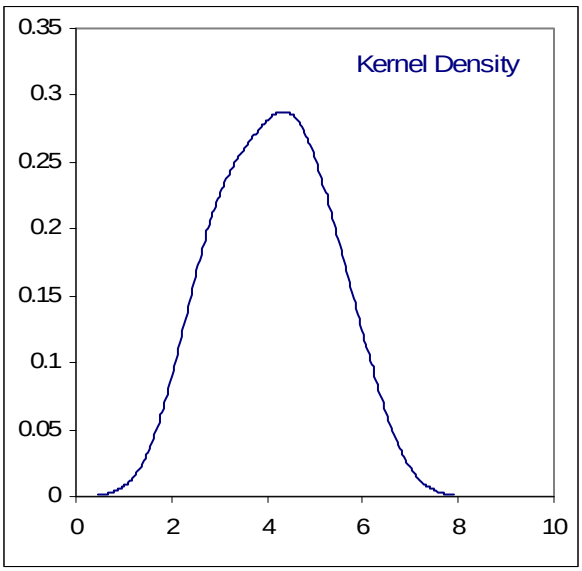


Determination of Nitrogen on ETBE sample #1033; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D4629	4.79		1.08	
311	D4629	5.9	C	2.93	First reported 7.1
323		-----		-----	
334		-----		-----	
494		-----		-----	
497	D4629	2.47		-2.78	
555		-----		-----	
974		-----		-----	
1041	D4629	3.38		-1.26	
1108	D4629	5.1		1.60	
1154	D4629	4.3		0.27	
1201	D4629	4.1		-0.06	
1251		-----		-----	
1291		-----		-----	
1427	D4629	3.07		-1.78	
1653		-----		-----	
1807		-----		-----	
1845		-----		-----	

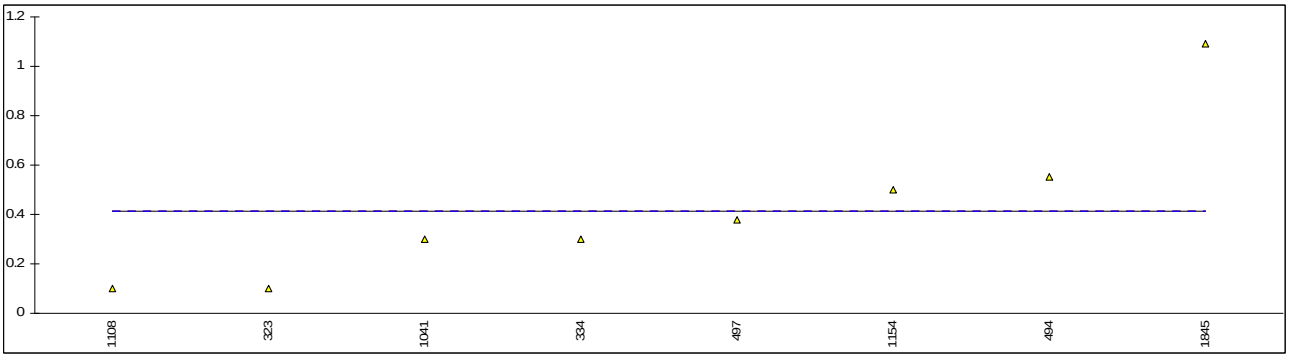
normality	OK
n	8
outliers	0
mean (n)	4.14
st.dev. (n)	1.133
R(calc.)	3.17
R(D4629:09)	1.68

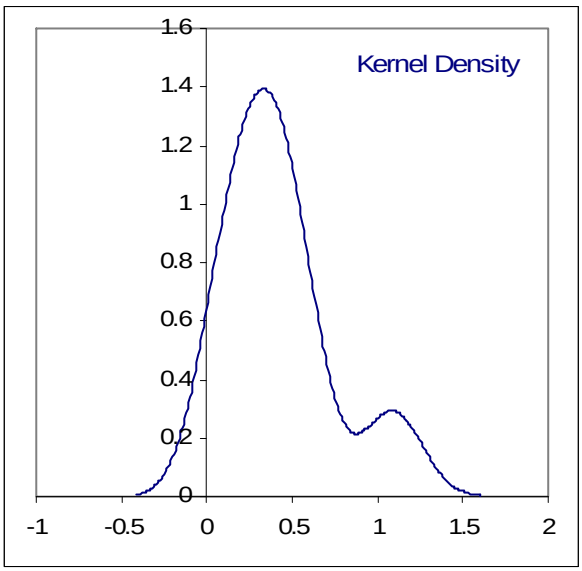




Determination of Sulphur on ETBE sample #1033; results in mg/kg

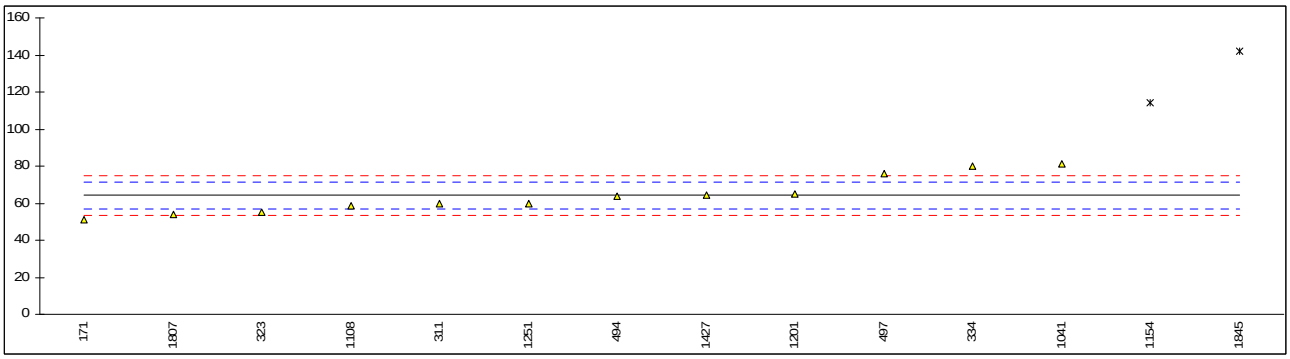
lab	method	value	mark	z(targ)	remarks
171	D5453	<1		-----	
311	D5453	<1		-----	
323	D5453	0.1		-----	
334	D5453	0.3		-----	
494	D5453	0.55		-----	
497	D5453	0.38		-----	
555		-----		-----	
974		-----		-----	
1041	D5453	0.30		-----	
1108	D5453	0.1		-----	
1154	ISO20846	0.5		-----	
1201	D5453	<1		-----	
1251	D5453	<1		-----	
1291		-----		-----	
1427	D5453	<1		-----	
1653		-----		-----	
1807		-----		-----	
1845	D5453	1.09	G(0.05)	-----	
	normality	OK			
	n	7			
	outliers	1			
	mean (n)	0.32			
	st.dev. (n)	0.176			
	R(calc.)	0.49			
	R(D5453:09)	(0.25)			

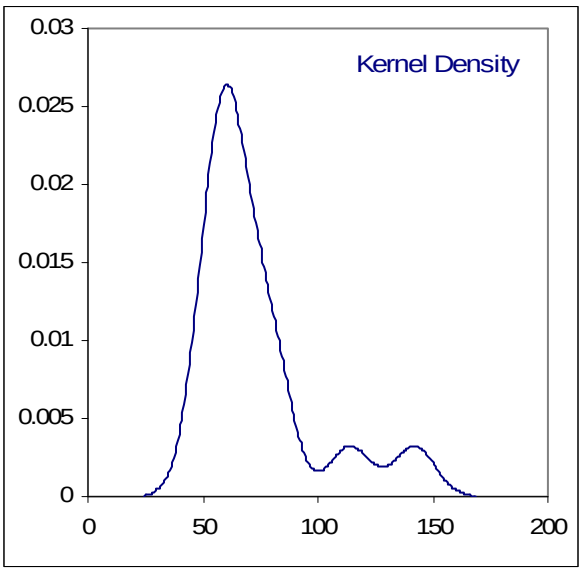




Determination of Water on ETBE sample #1033; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	E1064	51	C	-3.59	
311	E1064	60		-1.12	
323	E1064	55		-2.49	
334	E1064	80		4.38	
494	E1064	64		-0.02	
497	E1064	76		3.28	
555		-----		-----	
974		-----		-----	
1041	E1064	81		4.65	
1108	E1064	58.7		-1.48	
1154	ISO12937	114	G(0.01)	13.72	
1201	E1064	65		0.25	
1251	E1064	60		-1.12	
1291		-----		-----	
1427	D1364	64.13		0.02	
1653		-----		-----	
1807	D1744	54.06		-2.75	
1845	D4928	142	G(0.05)	21.42	
	normality	OK			
	n	12			
	outliers	2			
	mean (n)	64.07			
	st.dev. (n)	9.995			
	R(calc.)	27.99			
	R(E1064:05)	10.19			

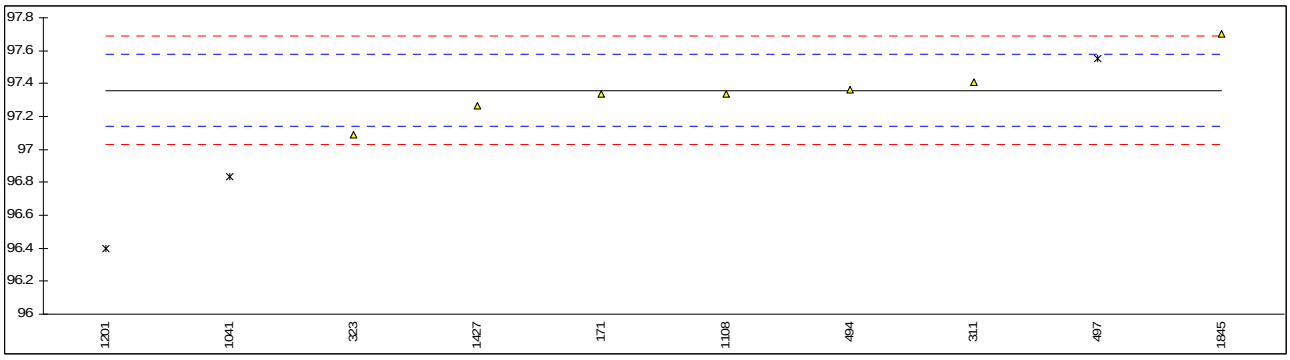


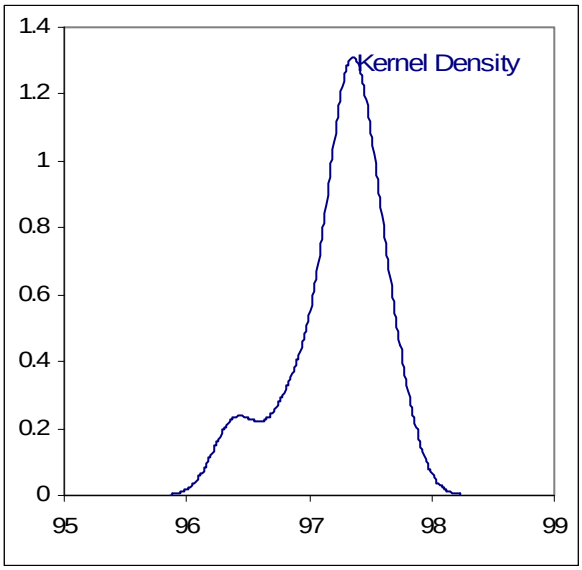


Determination of Purity “as received” on ETBE sample #1033; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	97.340		-0.17	
311	INH-284	97.411		0.48	
323	D5441	97.09		-2.44	
334		-----		-----	
494	D5441	97.36		0.01	
497	D5441	97.554	ex	1.78	Result excluded, “as received” < “dry basis”
555		-----		-----	
974		-----		-----	
1041	D5441	96.8341	G(0.05)	-4.77	
1108	D5441	97.34		-0.17	
1154		-----		-----	
1201	D5441	96.40	ex	-8.72	Result excluded, “as received” < “dry basis”
1251		-----		-----	
1291		-----		-----	
1427	D5441	97.2655		-0.85	
1653		-----		-----	
1807		-----		-----	
1845	D5441	97.702		3.13	
	normality	OK			
	n	7			
	outliers	1			
	mean (n)	97.3584			
	st.dev. (n)	0.18357			
	R(calc.)	0.5140			
	R(D5441:08e1) *	0.3077			

* ASTM D5441 is applicable for MTBE, but as no suitable test method for ETBE is available ASTM D5441 is used as reference test method, see also §4.1

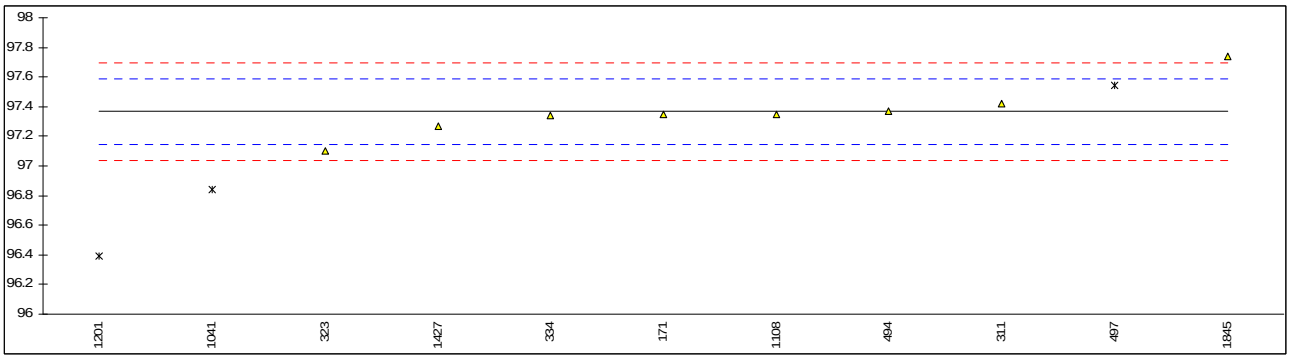


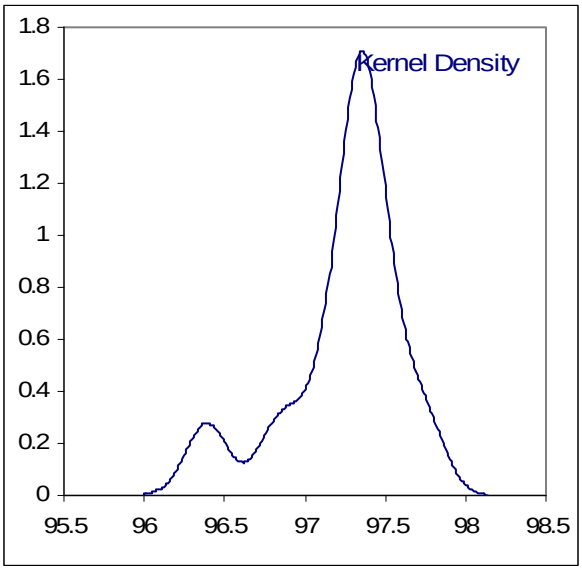


Determination of Purity on dry basis on ETBE sample #1033; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	97.345		-0.20	
311	INH-284	97.417		0.46	
323	D5441	97.10		-2.43	
334	D5441	97.34		-0.24	
494	D5441	97.37		0.03	
497	D5441	97.546	ex	1.63	Result excluded, “as received” < “dry basis”
555		-----		-----	
974		-----		-----	
1041	D5441	96.8422	G(0.05)	-4.77	
1108	D5441	97.35		-0.15	
1154		-----		-----	
1201	D5441	96.39	ex	-8.89	Result excluded, “as received” < “dry basis”
1251		-----		-----	
1291		-----		-----	
1427	D5441	97.2707		-0.87	
1653		-----		-----	
1807		-----		-----	
1845	D5441	97.741		3.41	
	normality	OK			
	n	8			
	outliers	1			
	mean (n)	97.3667			
	st.dev. (n)	0.17906			
	R(calc.)	0.5014			
	R(D5441:08e1)	0.3076			

* ASTM D5441 is applicable for MTBE, but as no suitable test method for ETBE is available ASTM D5441 is used as reference test method, see also §4.1

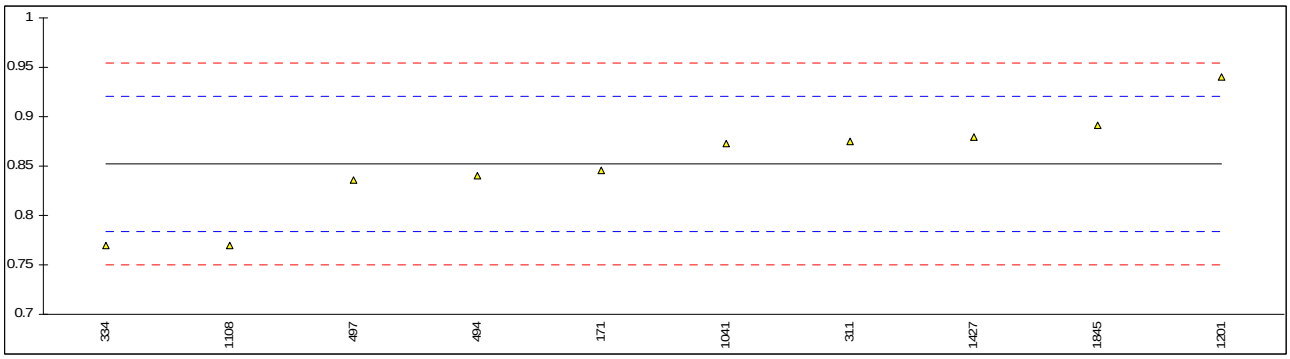


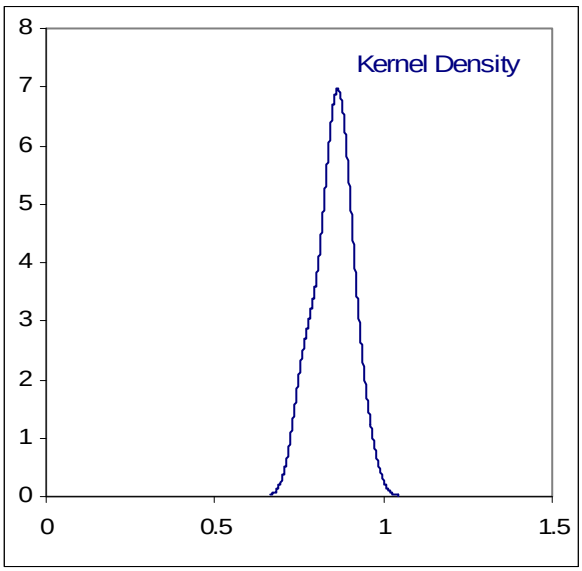


Determination of Ethanol on ETBE sample #1033; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.846		-0.18	
311	INH-284	0.875		0.67	
323		-----		-----	
334	D5441	0.77		-2.41	
494	D5441	0.84		-0.35	
497	D5441	0.836		-0.47	
555		-----		-----	
974		-----		-----	
1041	D5441	0.8730		0.61	
1108	D5441	0.77		-2.41	
1154		-----		-----	
1201	D5441	0.94		2.58	
1251		-----		-----	
1291		-----		-----	
1427	D5441	0.8797		0.81	
1653		-----		-----	
1807		-----		-----	
1845	D5441	0.891		1.14	

normality	OK
n	10
outliers	0
mean (n)	0.8521
st.dev. (n)	0.05259
R(calc.)	0.1472
R(D5441:08e1)	0.0955

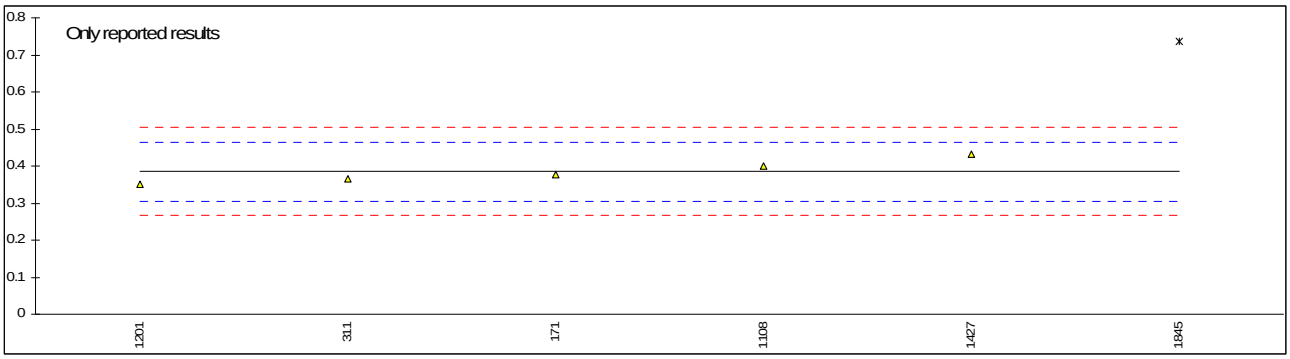


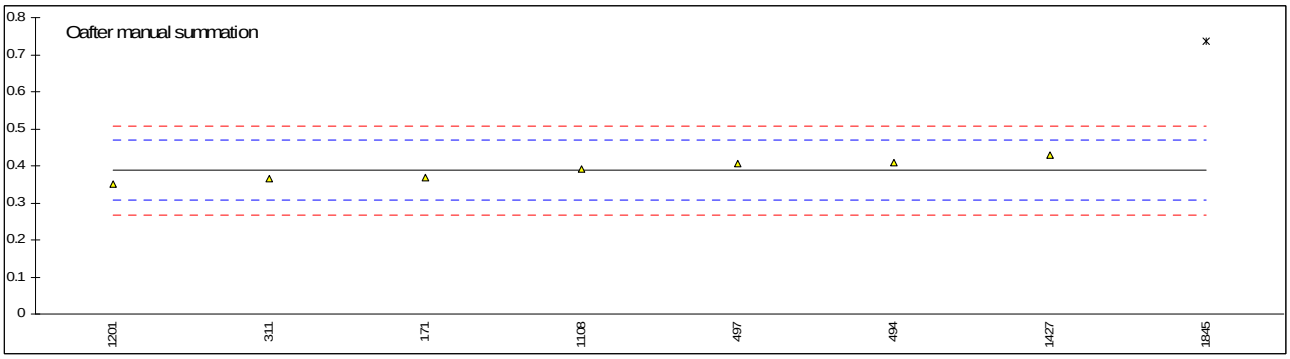


Determination of Diisobutylene (=sum 5 DIB's*) on ETBE sample #1033; results in %M/M

lab	method	value	mark	z(targ)	value after manual summation	mark	z(targ)	remarks
171	D5441	0.376		-0.22	0.368		-0.50	
311	INH-284	0.366		-0.47	0.366		-0.55	
323		-----		-----	-----		-----	
334		-----		-----	-----		-----	
494		-----		-----	0.410		0.55	
497		-----		-----	0.405		0.42	
555		-----		-----	-----		-----	
974		-----		-----	-----		-----	
1041		-----		-----	-----		-----	
1108	D5441	0.40		0.38	0.390		0.05	
1154		-----		-----	-----		-----	
1201	D5441	0.35		-0.88	0.35		-0.95	
1251		-----		-----	-----		-----	
1291		-----		-----	-----		-----	
1427	D5441	0.432		1.19	0.428		1.00	
1653		-----		-----	-----		-----	
1807		-----		-----	-----		-----	
1845	D5441	0.735	G(0.01)	8.81	0.735	G(0.01)	8.66	
	normality	OK			OK			
	n	5			7			
	outliers	1			1			
	mean (n)	0.3848			0.3881			
	st.dev. (n)	0.03202			0.02800			
	R(calc.)	0.0897			0.0784			
	R(Horwitz.)	0.1113			0.1121			

* 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.



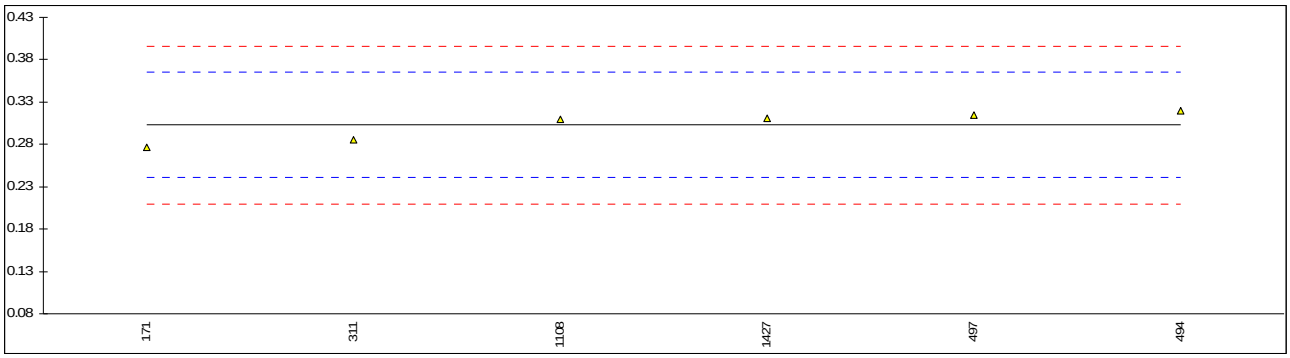


Determination of 2,4,4-Trimethyl-1-pentene on ETBE sample #1033; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.277		-0.83	
311	INH-284	0.285		-0.58	
323		-----		-----	
334		-----		-----	
494	D5441	0.32		0.55	
497	D5441	0.315		0.39	
555		-----		-----	
974		-----		-----	
1041		-----		-----	
1108	D5441	0.31		0.23	
1154		-----		-----	
1201		-----		-----	
1251		-----		-----	
1291		-----		-----	
1427	D5441	0.3103		0.24	
1653		-----		-----	
1807		-----		-----	
1845		-----		-----	

normality	not OK
n	6
outliers	0
mean (n)	0.3029
st.dev. (n)	0.01752
R(calc.)	0.0491
R(D5441:08e1)	0.0869

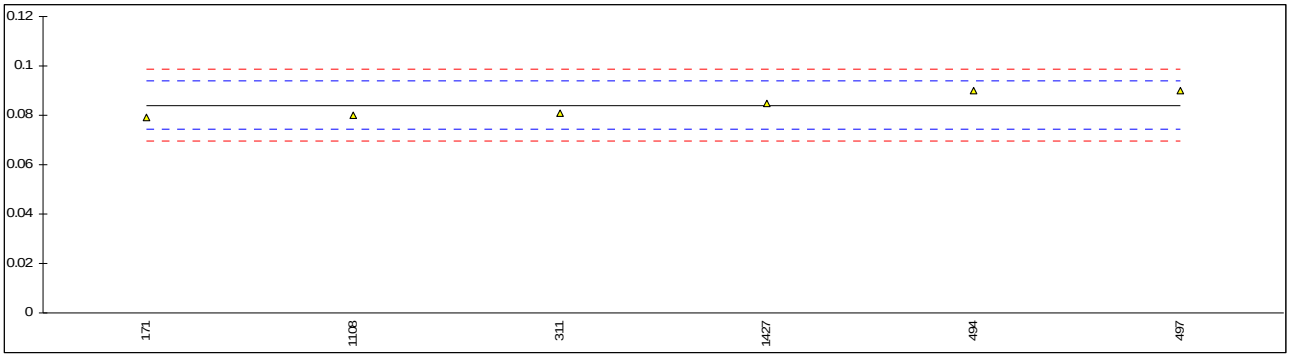
* ASTM D5441 is applicable for MTBE, but as no suitable test method for ETBE is available ASTM D5441 is used as reference test method, see also §4.1



Determination of 2,4,4-Trimethyl-2-pentene on ETBE sample #1033; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.079		-1.04	
311	INH-284	0.081		-0.63	
323		-----		-----	
334		-----		-----	
494	D5441	0.09		1.21	
497	D5441	0.090		1.21	
555		-----		-----	
974		-----		-----	
1041		-----		-----	
1108	D5441	0.08		-0.84	
1154		-----		-----	
1201		-----		-----	
1251		-----		-----	
1291		-----		-----	
1427	D5441	0.0846		0.10	
1653		-----		-----	
1807		-----		-----	
1845		-----		-----	

normality	OK
n	6
outliers	0
mean (n)	0.0841
st.dev. (n)	0.00495
R(calc.)	0.0138
R(Horwitz)	0.0137



Determination of other individual diisobutylenes on ETBE sample #1033; results in %M/M

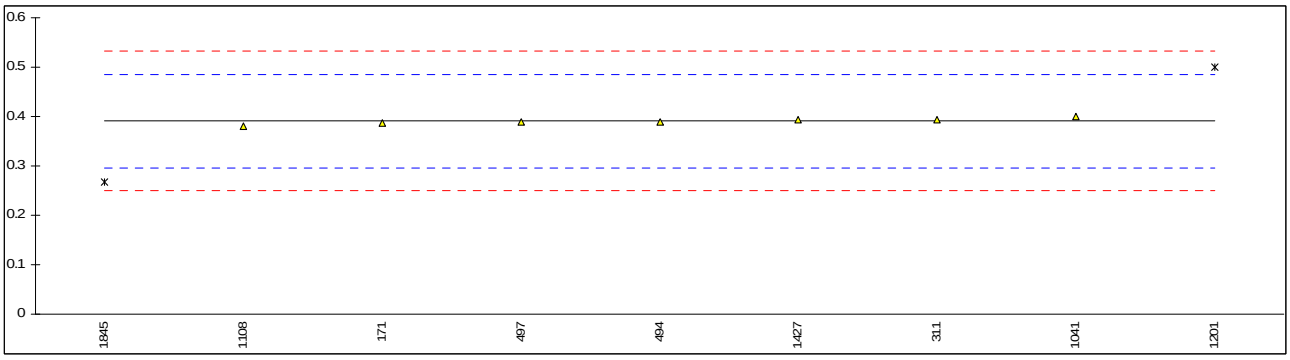
lab	method	243T2P *)	mark	z(targ)	344T1P *)	mark	z(targ)	35D1H *)	mark	z(targ)	remarks
171	D5441	<0.01		-----	<0.01		-----	0.012	C	-----	Fr 0.148
311	INH-284	<0.02		-----	<0.02		-----	<0.02		-----	
323		-----		-----	-----		-----	-----		-----	
334		-----		-----	-----		-----	-----		-----	
494		-----		-----	-----		-----	-----		-----	
497		-----		-----	-----		-----	-----		-----	
555		-----		-----	-----		-----	-----		-----	
974		-----		-----	-----		-----	-----		-----	
1041		-----		-----	-----		-----	-----		-----	
1108		-----		-----	-----		-----	-----		-----	
1154		-----		-----	-----		-----	-----		-----	
1201		-----		-----	-----		-----	-----		-----	
1251		-----		-----	-----		-----	-----		-----	
1291		-----		-----	-----		-----	-----		-----	
1427	D5441	0.012		-----	0.021		-----	-----		-----	
1653		-----		-----	-----		-----	-----		-----	
1807		-----		-----	-----		-----	-----		-----	
1845		-----		-----	-----		-----	-----		-----	
	normality	n.a.			n.a.			n.a.			
	n	1			1			0			
	outliers	0			0			0			
	mean (n)	n.a.			n.a.			n.a.			
	st.dev. (n)	n.a.			n.a.			n.a.			
	R(calc.)	n.a.			n.a.			n.a.			
	R(lit.)	n.a.			n.a.			n.a.			

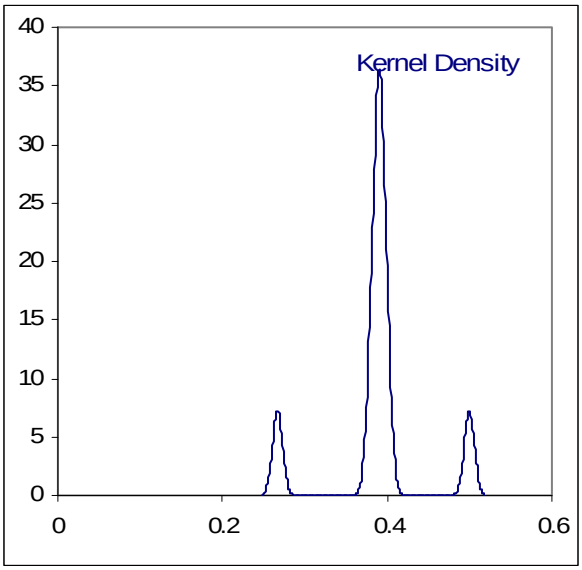
* 2,4,3-Trimethyl-2-pentene
* 3,4,4-Trimethyl-1-pentene
* 3,5-Dimethyl-1-hexene

Determination of Tert-butanol on ETBE sample #1033; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.386	C	-0.09	First reported 0.625
311	INH-284	0.394		0.08	
323		-----		-----	
334		-----		-----	
494	D5441	0.39		-0.01	
497	D5441	0.390		-0.01	
555		-----		-----	
974		-----		-----	
1041	D5441	0.4005		0.21	
1108	D5441	0.38		-0.22	
1154		-----		-----	
1201	D5441	0.50	D(0.01)	2.32	
1251		-----		-----	
1291		-----		-----	
1427	D5441	0.3924		0.04	
1653		-----		-----	
1807		-----		-----	
1845	D5441	0.267	D(0.01)	-2.62	
	normality	OK			
	n	7			
	outliers	2			
	mean (n)	0.3904			
	st.dev. (n)	0.00641			
	R(calc.)	0.0180			
	R(D5441:08e1)	0.1320			

* ASTM D5441 is applicable for MTBE, but as no suitable test method for ETBE is available ASTM D5441 is used as reference test method, see also §4.1

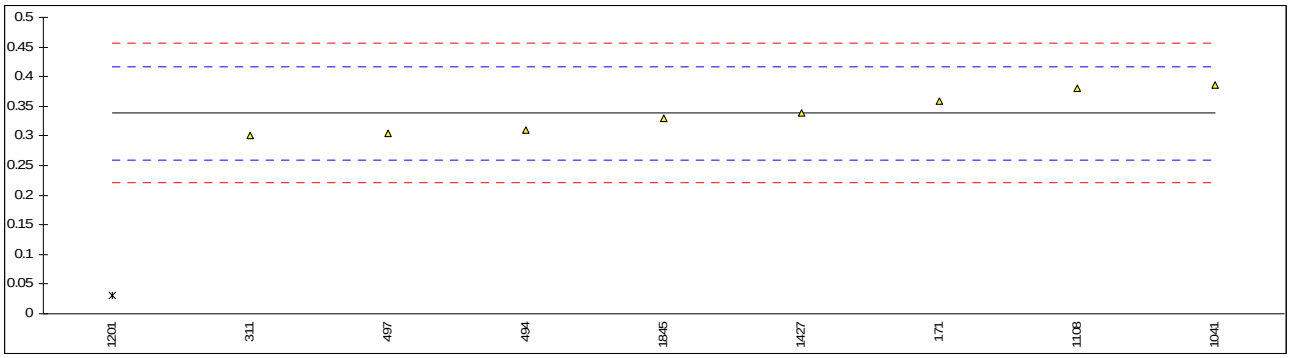


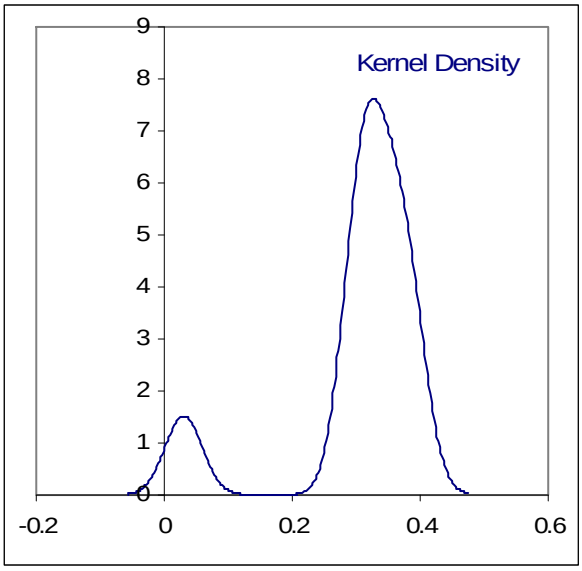


Determination of C4-hydrocarbons on ETBE sample #1033; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.359	C	0.53	First reported 0.006
311	INH-284	0.30		-0.98	
323		-----		-----	
334		-----		-----	
494	D5441	0.31		-0.72	
497	D5441	0.304		-0.87	
555		-----		-----	
974		-----		-----	
1041	D5441	0.3860		1.22	
1108	D5441	0.38		1.06	
1154		-----		-----	
1201	D5441	0.03	G(0.01)	-7.86	
1251		-----		-----	
1291		-----		-----	
1427	D5441	0.3382		0.00	
1653		-----		-----	
1807		-----		-----	
1845	D5441	0.329		-0.24	
	normality	OK			
	n	8			
	outliers	1			
	mean (n)	0.3383			
	st.dev. (n)	0.03375			
	R(calc.)	0.0945			
	R(D5441:08e1)	0.1098			

* ASTM D5441 is applicable for MTBE, but as no suitable test method for ETBE is available ASTM D5441 is used as reference test method, see also §4.1

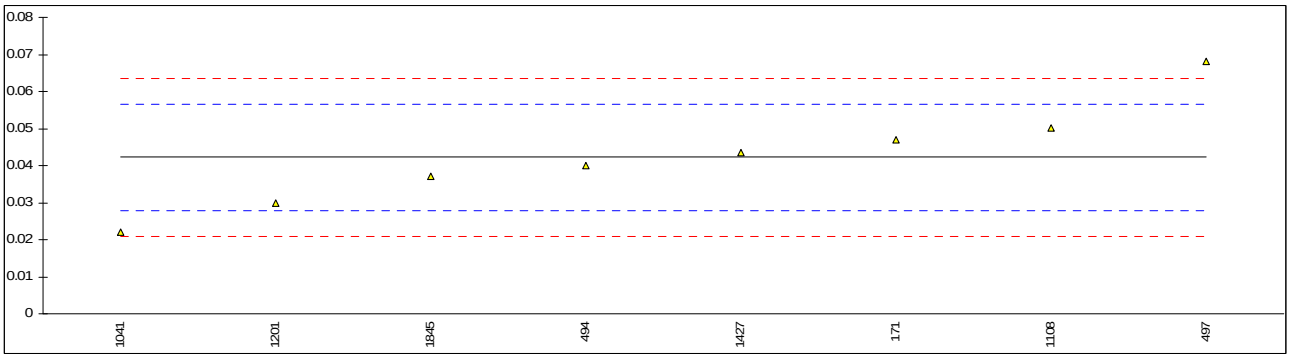


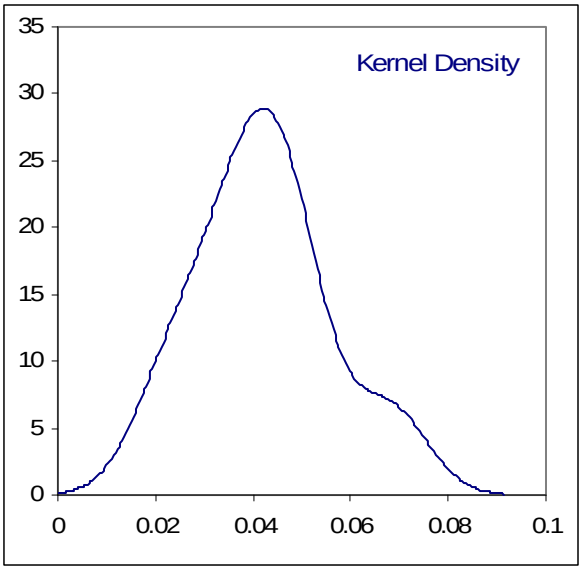


Determination of C5-hydrocarbons on ETBE sample #1033; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.047	C	0.67	First reported 0.035
311	INH-284	<0.10		-----	
323		-----		-----	
334		-----		-----	
494	D5441	0.04		-0.31	
497	D5441	0.068		3.62	
555		-----		-----	
974		-----		-----	
1041	D5441	0.0220		-2.84	
1108	D5441	0.05		1.09	
1154		-----		-----	
1201	D5441	0.03		-1.71	
1251		-----		-----	
1291		-----		-----	
1427	D5441	0.0436		0.20	
1653		-----		-----	
1807		-----		-----	
1845	D5441	0.037		-0.73	
	normality	OK			
	n	8			
	outliers	0			
	mean (n)	0.0422			
	st.dev. (n)	0.01384			
	R(calc.)	0.0387			
	R(D5441:08e1)	0.0199			

* ASTM D5441 is applicable for MTBE, but as no suitable test method for ETBE is available ASTM D5441 is used as reference test method, see also §4.1





Determination of Other Hydrocarbons on sample #1033; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311		-----		-----	
323		-----		-----	
334		-----		-----	
494	D5441	0.56		-----	
497	D5441	0.394		-----	
555		-----		-----	
974		-----		-----	
1041	D5441	1.3932		-----	
1108		-----		-----	
1154		-----		-----	
1201		-----		-----	
1251		-----		-----	
1291		-----		-----	
1427	D5441	0.0143		-----	
1653		-----		-----	
1807		-----		-----	
1845		-----		-----	

normality	OK
n	4
outliers	0
mean (n)	0.5904
st.dev. (n)	0.58192
R(calc.)	1.6294
R(lit)	unknown

Determination of Unknown Impurities on ETBE sample #1033; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311		-----		-----	
323		-----		-----	
334		-----		-----	
494		-----		-----	
497		-----		-----	
555		-----		-----	
974		-----		-----	
1041		-----		-----	
1108	D5441	0.52		-----	
1154		-----		-----	
1201	D5441	0.63		-----	
1251		-----		-----	
1291		-----		-----	
1427	D5441	0.6551		-----	
1653		-----		-----	
1807		-----		-----	
1845		-----		-----	
	normality	n.a.			
	n	3			
	outliers	0			
	mean (n)	0.6017			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit.)	unknown			

APPENDIX 2

Number of participating laboratories per country

For MTBE:

1 laboratory in	BELGIUM
1 laboratory in	FRANCE
3 laboratories in	GERMANY
1 laboratory in	GREECE
1 laboratory in	ITALY
1 laboratory in	PORTUGAL
3 laboratories in	SAUDI ARABIA
1 laboratory in	SINGAPORE
1 laboratory in	SPAIN
6 laboratories in	THE NETHERLANDS
1 laboratory in	TURKEY
1 laboratory in	U.A.E.
1 laboratory in	U.S.A.

For ETBE:

1 laboratory in	BELGIUM
3 laboratories in	BRAZIL
2 laboratories in	FRANCE
3 laboratories in	GERMANY
1 laboratory in	GREECE
1 laboratory in	PORTUGAL
1 laboratory in	SPAIN
1 laboratory in	SWEDEN
3 laboratories in	THE NETHERLANDS
1 laboratory in	U.A.E.
1 laboratory in	U.S.A.

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
ex	= excluded from calculations
S	= scope of the reported method is not applicable
n.a.	= not applicable
MSDS	= Material Safety Data Sheet
Fr.	= first reported
U	= reported in different unit
E	= possibly an error in the calculation

Literature:

1 i.i.s. Interlaboratory Studies. Protocol for the Organisation, Statistics and Evaluation, January 2010
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3 ASTM E1301-89
4 ISO 5725-86
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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)
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12 J.N. Miller. Analyst. 118. 455. (1993)
13 Analytical Methods Committee Technical brief, No4 January 2001
14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page 1359-1364, P.J. Lowthian and M. Thompson (see http://www.rsc.org/suppdata/an/b2/b205600n)