Results of Proficiency Test
MTBE
February 2013

CONTENTS

Appendices:

1   INTRODUCTION

Since 1995, the Institute for Interlaboratory Studies organizes a proficiency test for the analysis of Methyl Tertiary Butyl Ether (MTBE). During the annual proficiency testing program 2012/2013, it was decided to continue the round robin for the analyses of MTBE.

In this interlaboratory study for MTBE 18 laboratories in 14 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2013 MTBE proficiency test are presented and discussed. This report is also electronically available through the iis internet site ww.iisnl.com.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test. It was decided to send one sample of MTBE (0.5 litre bottle, labelled #13003) to the participants. Analyses for fit-for-use and homogeneity were subcontracted. Participants were requested to report rounded and unrounded results. The unrounded 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 guide 43, ILAC-G13:2007 and IEC/ISO17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2   PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).

2.3   CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The necessary 25 litres of MTBE bulk material were obtained from a local producer. After homogenisation, the bulk material was transferred into 50 brown glass bottles of 500 mL and labelled #13003. The homogeneity of the subsamples was checked by determination of Density @20˚C in accordance with ASTM D4052:12 and Water in accordance with ASTM E1064:12 on respectively 8 and 5 stratified randomly selected samples:

	Density at 20˚C in kg/L	Water in mg/kg
sample #13003-1	0.74144	110
sample #13003-2	0.74146	110
sample #13003-3	0.74144	113
sample #13003-4	0.74144	111
sample #13003-5	0.74146	110
sample #13003-6	0.74146	--
sample #13003-7	0.74146	--
sample #13003-8	0.74147	--

Table 1: homogeneity test results of subsamples #13003

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 20˚C in kg/L	Water in mg/kg
r (sample #13003)	0.00003	4
reference test method	ASTM D4052:02e1	ASTM E1064:12
0.3xR (reference test)	0.00015	6

Table 2: repeatabilities of the subsamples #13003

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

To each of the participating laboratories one bottle of 500 ml, labelled #13003, was sent on January 30, 2013.

2.5 STABILITY OF THE SAMPLES

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

2.6   ANALYSES

The participants were asked to determine on sample #13003 containing MTBE: Appearance, Density @15°C, Refractive index @ 20°C, Carbonyls, Water, Purity (both on as received and on dry basis), Methanol, 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], tert-Butylalcohol, Hydrocarbons (C4- and C5), Other hydrocarbons and Unknown impurities.

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website (www.iisnl.com).

A SDS and a form to confirm receipt of the samples were added to the sample package.

3   RESULTS

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

Directly after deadline, a reminder fax was sent to those laboratories that had not yet reported any results at that moment.

Shortly 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. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1   STATISTICS

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

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying 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; nos.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.

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 the fit-for-useness of the reported test result.

In case no literature reproducibility was available, other target values were used. In some cases literature repeatability is available; in other cases a reproducibility of a former iis proficiency test could be used and also the Horwitz equation can be used to estimate target reproducibility.

The z-scores were calculated according to:

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

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:

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

4   EVALUATION

In this proficiency test, some problems were encountered during the execution. Participants in Brazil, Saudi Arabia and Venezuela received the samples late or not at all due to problems at customs. From the 18 participants, 6 participants reported results after the deadline for reporting and 2 participants did not report any results at all. The 16 reporting laboratories submitted 154 numerical results. Observed were 16 outlying results, which is 10.4%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.
For all determinations a normal distribution was found.

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.

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #13003, which is pass or bright and clear.

Density @ 15°C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D4052:02e1. The current version of this method ASTM D4052:11 only give reproducibilities for gasoline, distillates, base stocks and lubricating oils. Therefore this 2011 version may not be applicable for MTBE.

Refractive Index: This determination was problematic for two laboratories. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D1218:12.

Carbonyls: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM E411:12. This may be explained by the small number of reported test results.

Water: This determination was very problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM E1064:12.

Purity: This determination was problematic for two laboratories. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5441:08e1.

Methanol: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D5441:08e1.

Diisobutylene (=sum): This determination may be very problematic. 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.

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

2,4,4-Trime-2-pent.: This determination may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers 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 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, although the concentration (0.117 %M/M) is outside the application range of the test method (0.474 - 0.8763 %M/M).

C4-hydrocarbons: It is hard to draw conclusions, because the C4-hydrocarbon content is below or near the detection limit of the test method.

C5-hydrocarbons: This determination was problematic for a number of laboratories. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5441:08e1.

Other hydrocarb.+ Unknown Impurities:

Other hydrocarbons may be 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.

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

In general, it is hard to judge an overall group performance, because ASTM D5441:08e1 does not specify requirements for the reproducibility for these components.

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 standards, see tables in appendix 1).

	unit	n	average	R (Calc.)	R (lit)
Appearance		13	pass	n.a	n.a
Density @ 15°C	kg/L	16	0.74664	0.00030	0.00050
Refractive Index @ 20ºC		9	1.36955	0.00045	0.00050
Carbonyls	µg/g	4	14.8	5.0	2.5
Water	mg/kg	13	115.8	33.3	18.4
Purity	%M/M	11	98.251	0.271	0.303
Methanol	%M/M	12	0.637	0.147	0.089
Diisobutylene (=sum of 5)	% M/M	8	0.220	0.119	0.066
2,4,4-Trimethyl-1-pentene	%M/M	9	0.142	0.043	0.046
2,4,4-Trimethyl-2-pentene	%M/M	7	0.041	0.010	0.007
Tert-butanol	%M/M	14	0.117	0.028	0.132
C4 – hydrocarbons	%M/M	7	0.013	0.009	(0.006)
C5 – hydrocarbons	%M/M	7	0.088	0.020	0.033
Other hydrocarbons	%M/M	4	0.483	n.a	unknown
Unknown impurities	%M/M	6	0.443	n.a	unknown

Table 3: performance evaluation of sample #13003

Result between brackets is near or below the detection limit of the test method

Without further statistical calculations, it can be concluded that for number of tests for MTBE there is a good compliance of the group of participating laboratories with the relevant standards. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2013 WITH PREVIOUS PTS

A good comparison with the previous PT'S can not be made. The PT's from April 2008 and March 2010 also include the Ethyl Tertiary Butyl Ether (ETBE) data.

	February 2013	February 2012	March 2010	March 2008
Number of reporting labs	16	17	33	23
Number of Results reported	154	178	305	400
Statistical outliers	16	16	22	38
Percentage outliers	10.4%	9.0%	7.2%	9.5%

Table 4: comparison with previous proficiency tests.

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

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

Determination	February 2013	February 2012	March 2010	March 2008
Density @ 15°C	++	+	+	+
Refractive Index @ 20ºC	+	+/-	+	--
Carbonyls	--	n.e	n.e	n.e
Water	--	+/-	++	++
Purity	+	--	+	-
Methanol	--	-	+	-
2,4,4-Trimethyl-1-pentene	+/-	++	++	+
2,4,4-Trimethyl-2-pentene	- *)	+ *)	-- *)	+ *)
Tert-butanol	++	++	++	++
C4 – hydrocarbons	(-)	+/-	--	+
C5 – hydrocarbons	++	--	++	+

Table 5: comparison determinations against the standard for MTBE

*) compared against the strict Horwitz equation

Result between brackets is an estimate, as the assigned value is below or 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
 n.e.:   not evaluated

APPENDIX 1

Determination of Appearance on MTBE sample #13003;

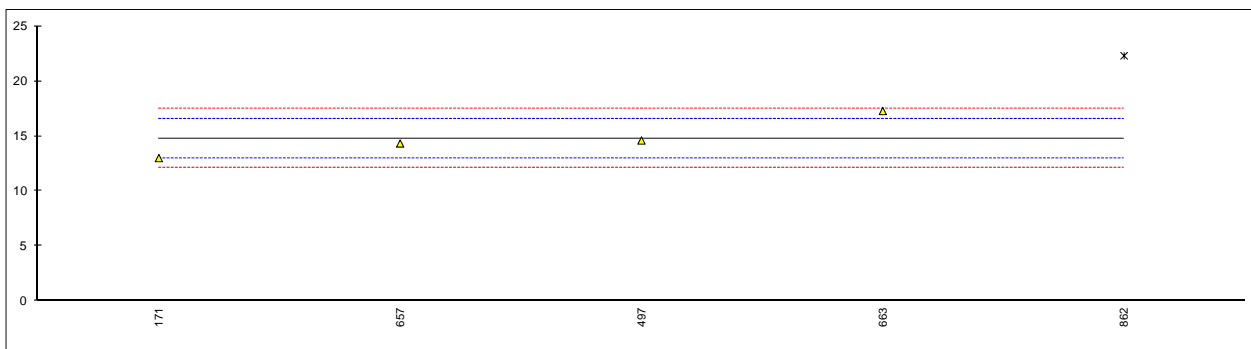| lab	method	value	mark	z(targ)	remarks
171	E2680	Pass		-----	
311	E2680	Pass		-----	
312	Visual	Pass		-----	
323	E2680	Pass		-----	
334		-----		-----	
347	E2680	Pass		-----	
395	E2680	Pass		-----	
497	E2680	B&C		-----	
555		-----		-----	
657	E2680	Pass		-----	
663	E2680	Pass		-----	
862	E2680	Pass		-----	
963	E2680	Pass		-----	
1108		-----		-----	
1200		-----		-----	
1221		-----		-----	
1386	E2680	Pass		-----	
1866	E2680	Pass		-----	
	normality	n.a.			
	n	13			
	outliers	n.a.			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

B&C   = Bright and clear

Determination of Density @ 15°C on MTBE sample #13003; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.7465		-0.80	
311	D4052	0.7467		0.32	
312	D4052	0.7466		-0.24	
323	D4052	0.7465		-0.80	
334	D4052	0.7467		0.32	
347	D4052	0.7466		-0.24	
395	D4052	0.7466		-0.24	
497	D4052	0.74665		0.04	
555		-----		-----	
657	D4052	0.74658		-0.35	
663	D4052	0.74670		0.32	
862	D4052	0.74692		1.55	
963	D4052	0.7465		-0.80	
1108	D4052	0.74662		-0.13	
1200		-----		-----	
1221	D4052	0.74672		0.43	
1386	D4052	0.74671		0.38	
1866	D4052	0.74668		0.21	

normality	OK
n	16
outliers	0
mean (n)	0.74664
st.dev. (n)	0.000106
R(calc.)	0.00030
R(D4052:02e1)	0.00050
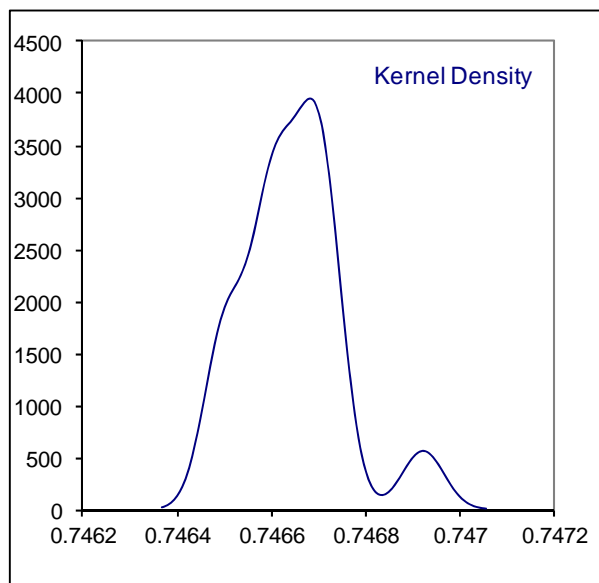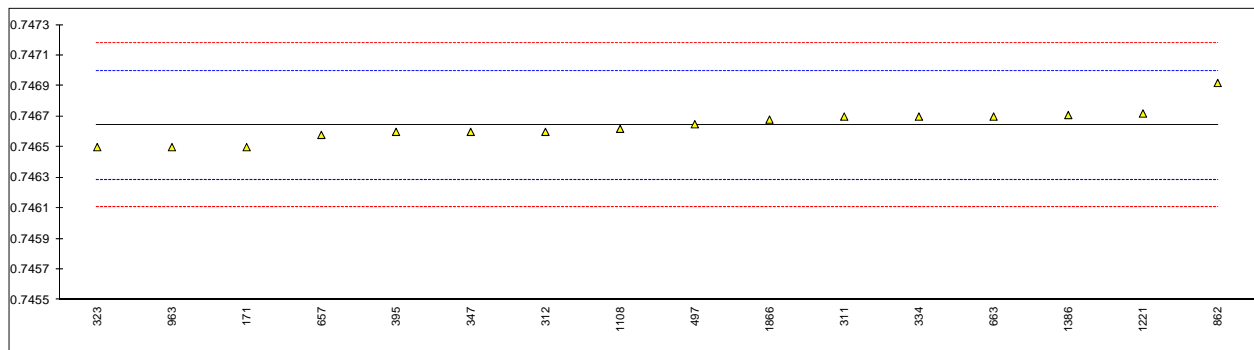
Determination of Refractive Index @ 20°C on MTBE sample #13003;

lab	method	value	mark	z(targ)	remarks
171	D1218	1.3699		1.98	
311	D1218	1.3695		-0.26	
312		-----		-----	
323	D1218	1.36952		-0.15	
334		-----		-----	
347		-----		-----	
395		-----		-----	
497	D1218	1.3709	DG(0.05)	7.58	
555		-----		-----	
657	D1218	1.3696		0.30	
663	D1218	1.3695		-0.26	
862	D1218	1.36954		-0.04	
963	D1218	1.3693		-1.38	
1108	D1218	1.3696		0.30	
1200		-----		-----	
1221		-----		-----	
1386	D1218	1.36946		-0.49	
1866	D1218	1.3683	DG(0.05)	-6.98	

normality	OK
n	9
outliers	2
mean (n)	1.36955
st.dev. (n)	0.000160
R(calc.)	0.00045
R(D1218:12)	0.00050
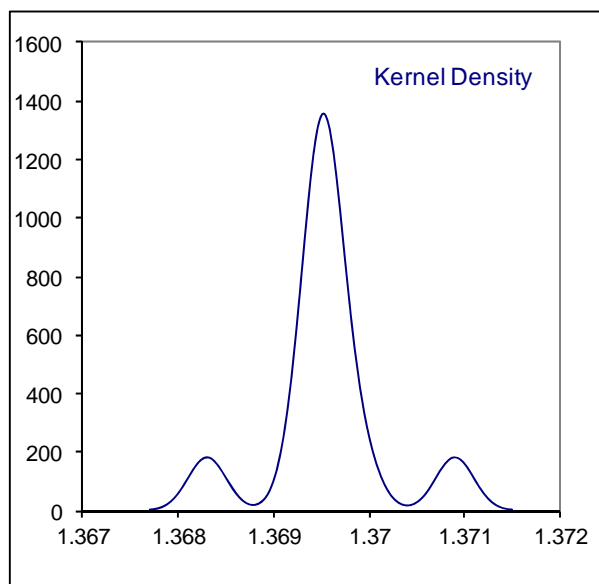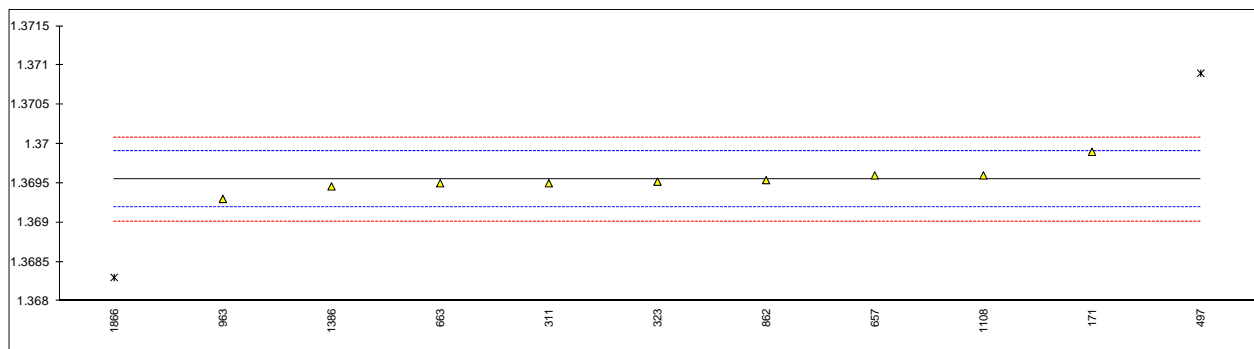
Determination of Carbonyls as CO on MTBE sample #13003; results in µg/g

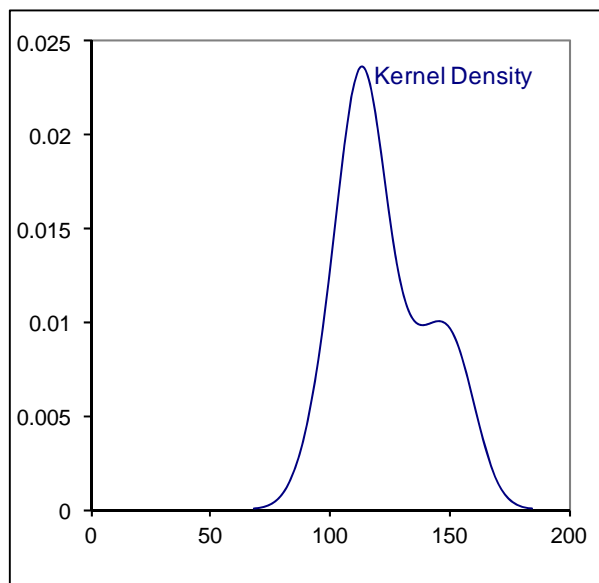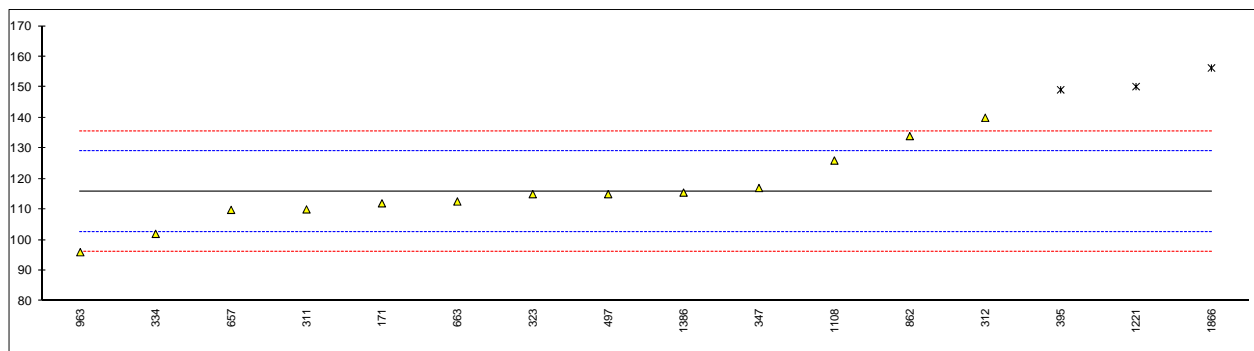| lab	method	value	mark	z(targ)	remarks
171	E411	13.0		-2.01	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
347		-----		-----	
395		-----		-----	
497	E411	14.6		-0.23	
555		-----		-----	
657	E411	14.33		-0.53	
663	E411	17.28		2.76	
862	E411	22.3	G(0.05)	8.34	
963		-----		-----	
1108		-----		-----	
1200		-----		-----	
1221		-----		-----	
1386		-----		-----	
1866		-----		-----	

normality	OK	
n	4	
outliers	1	
mean (n)	14.80	
st.dev. (n)	1.794	
R(calc.)	5.02	
R(E411:12)	2.52	Application range: 0.5 – 50 µg/g calculated as CO

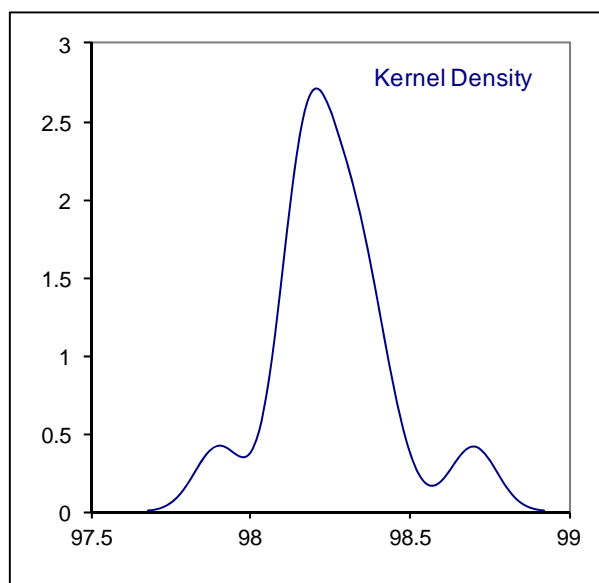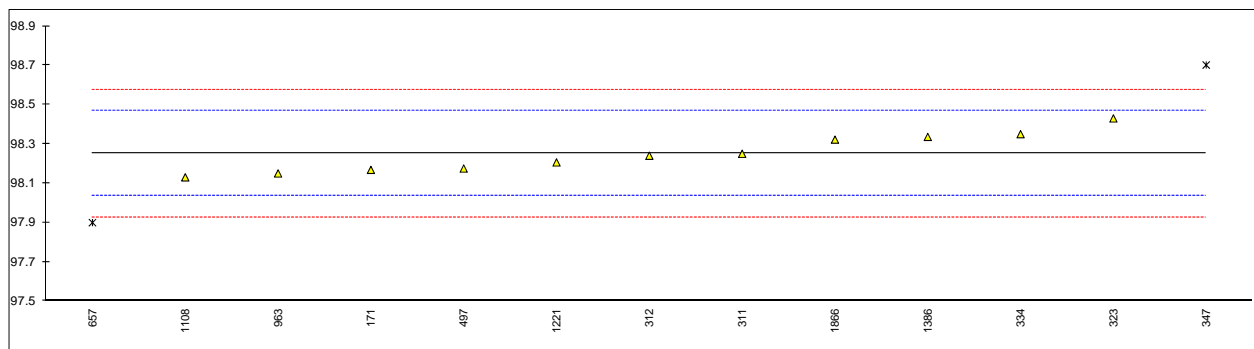## Determination of Water on MTBE sample #13003; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	E1064	112		-0.57	
311	E1064	110		-0.88	
312	ISO12937	140		3.69	
323	E1064	115		-0.12	
334	E1064	102		-2.09	
347	E1064	117		0.19	
395	E1364	149.15	G(0.05)	5.08	
497	E1064	115		-0.12	
555		-----		-----	
657	E1064	109.85		-0.90	
663	E1064	112.6		-0.48	
862	E1064	134		2.77	
963	E1064	96		-3.01	
1108	E1064	126		1.56	
1200		-----		-----	
1221	E1064	150.20	G(0.05)	5.24	
1386	E1064	115.5		-0.04	
1866	E1064	156.3	G(0.05)	6.17	

normality	OK
n	13
outliers	3
mean (n)	115.77
st.dev. (n)	11.902
R(calc.)	33.33
R(E1064:12)	18.41

## Determination of Purity by GLC of MTBE sample #13003; results in %M/M

lab	method	value	mark	z(targ)	Remarks
171	D5441	98.168		-0.76	
311	D5441	98.25		-0.01	
312	D5441	98.24		-0.10	
323	D5441	98.43		1.66	
334	D5441	98.35		0.92	
347	D5441	98.702	DG(0.05)	4.18	
395		-----		-----	
497	D5441	98.175		-0.70	
555		-----		-----	
657	D5441	97.8994	DG(0.05)	-3.25	
663		-----		-----	
862		-----		-----	
963	D5441	98.15		-0.93	
1108	D5441	98.13		-1.12	
1200		-----		-----	
1221	D5441	98.206		-0.41	
1386	D5441	98.336		0.79	
1866	D5441	98.3220		0.66	

|	
normality	OK
n	11
outliers	2
mean (n)	98.2506
st.dev. (n)	0.09683
R(calc.)	0.2711
R(D5441:08e1)	0.3027

Determination of Methanol on MTBE sample #13003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.657		0.62	
311	D5441	0.59		-1.50	
312	D5441	0.61		-0.87	
323	D5441	0.57		-2.13	
334	D5441	0.74		3.23	
347	D5441	0.489	DG(0.05)	-4.68	
395		-----		-----	
497	D5441	0.840	DG(0.05)	6.39	
555		-----		-----	
657	D5441	0.668		0.96	
663		-----		-----	
862	D5441	0.553		-2.66	
963	D5441	0.66		0.71	
1108	D5441	0.63		-0.23	
1200		-----		-----	
1221	D5441	0.656		0.59	
1386	D5441	0.691		1.69	
1866	D5441	0.6243		-0.41	

normality	OK
n	12
outliers	2
mean (n)	0.6374
st.dev. (n)	0.05263
R(calc.)	0.1474
R(D5441:08e1)	0.0888
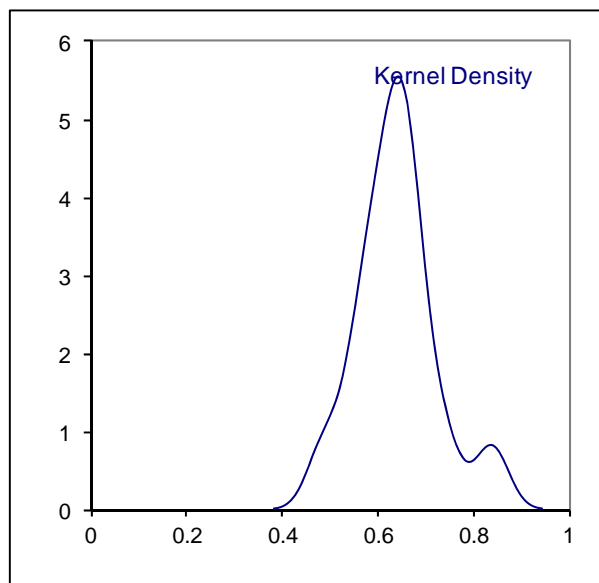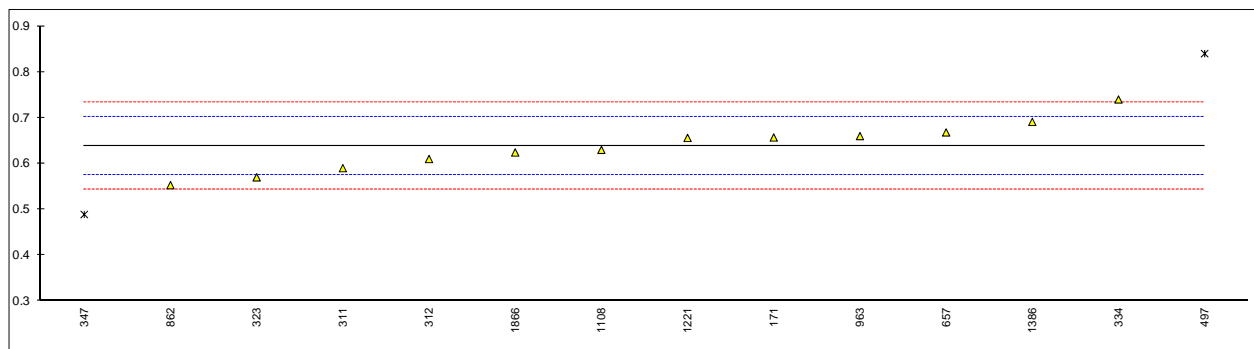
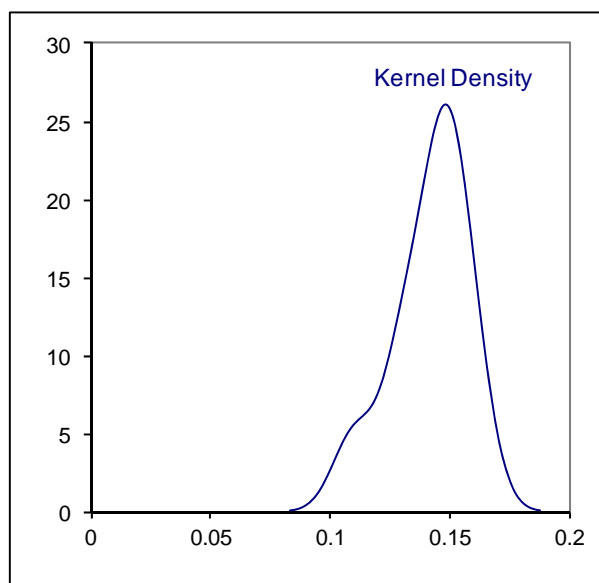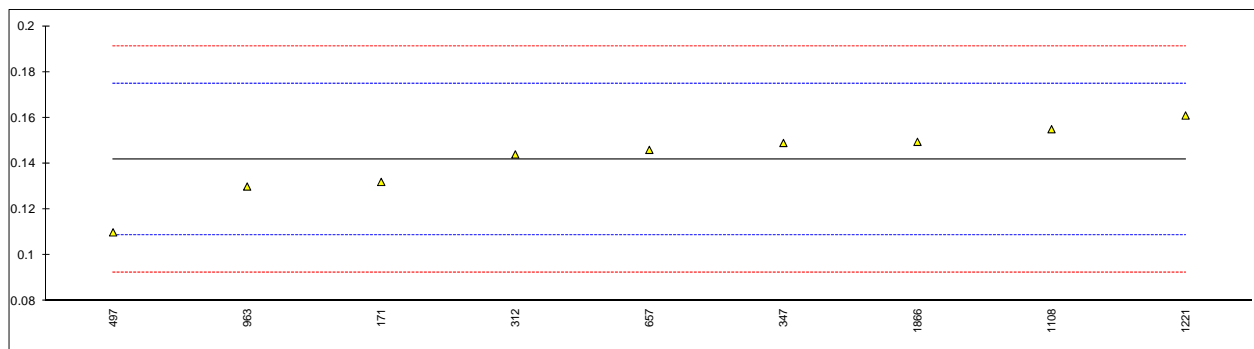## Determination of Diisobutylene (=sum 5 DIB's*) on MTBE sample #13003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.279		2.49	
311	D5441	0.17		-2.13	
312		-----		-----	
323		-----		-----	
334		-----		-----	
347	D5441	0.194		-1.11	
395		-----		-----	
497		-----		-----	
555		-----		-----	
657	D5441	0.224		0.16	
663		-----		-----	
862	D5441	0.253		1.39	
963	D5441	0.17		-2.13	
1108	D5441	0.332	G(0.05)	4.74	
1200		-----		-----	
1221	D5441	0.205		-0.64	
1386		-----		-----	
1866	D5441	0.2666		1.97	

normality	OK
n	8
outliers	1
mean (n)	0.2202
st.dev. (n)	0.04252
R(calc.)	0.1191
R(D5441:08e1)	0.0660

* 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.
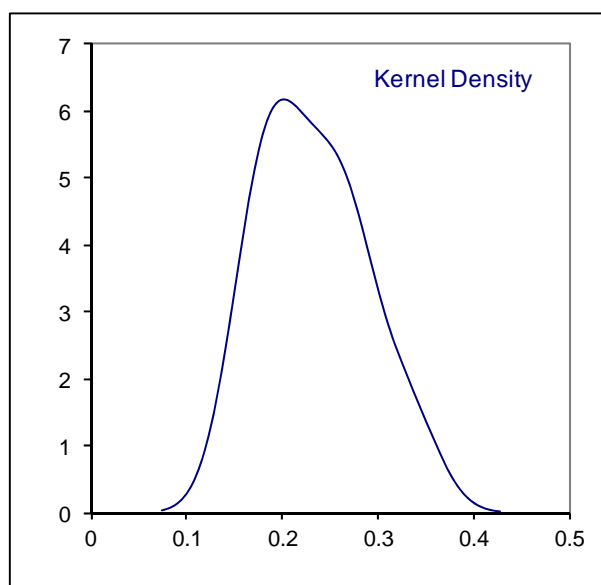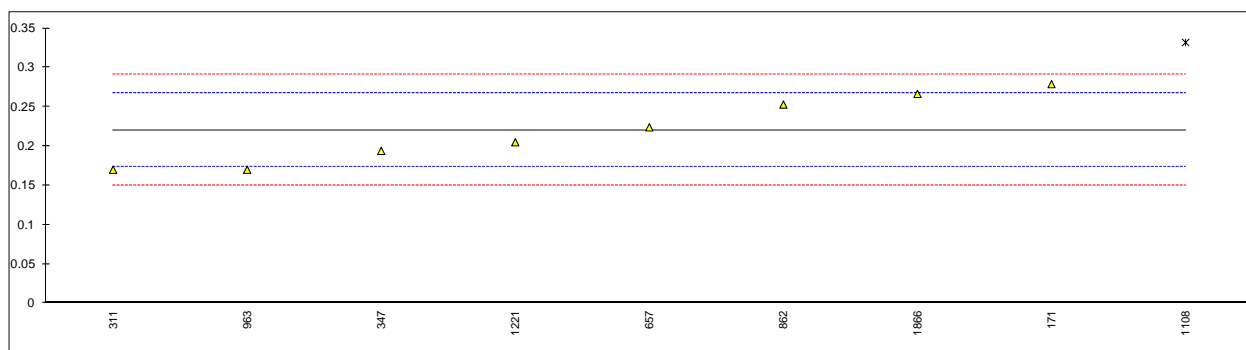
Determination of 2,4,4-Trimethyl-1-pentene on MTBE sample #13003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.132		-0.60	
311		-----		-----	
312	D5441	0.144		0.13	
323		-----		-----	
334		-----		-----	
347	D5441	0.149		0.43	
395		-----		-----	
497	D5441	0.110		-1.93	
555		-----		-----	
657	D5441	0.146		0.25	
663		-----		-----	
862		-----		-----	
963	D5441	0.13		-0.72	
1108	D5441	0.155		0.80	
1200		-----		-----	
1221	D5441	0.161		1.16	
1386		-----		-----	
1866	D5441	0.1495		0.46	

normality	OK
n	9
outliers	0
mean (n)	0.1418
st.dev. (n)	0.01550
R(calc.)	0.0434
R(D5441:08e1)	0.0463

Kernel Density

Determination of 2,4,4-Trimethyl-2-pentene on MTBE sample #13003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.038		-1.18	
311		-----		-----	
312	D5441	0.036		-1.93	
323		-----		-----	
334		-----		-----	
347	D5441	0.045		1.45	
395		-----		-----	
497	D5441	0.026	DG(0.05)	-5.69	
555		-----		-----	
657	D5441	0.041		-0.05	
663		-----		-----	
862		-----		-----	
963	D5441	0.04		-0.43	
1108	D5441	0.044		1.07	
1200		-----		-----	
1221	D5441	0.044		1.07	
1386		-----		-----	
1866	D5441	0.0173	DG(0.05)	-8.96	

normality	OK
n	7
outliers	2
mean (n)	0.0411
st.dev. (n)	0.00339
R(calc.)	0.0095
R(Horwitz)	0.0074
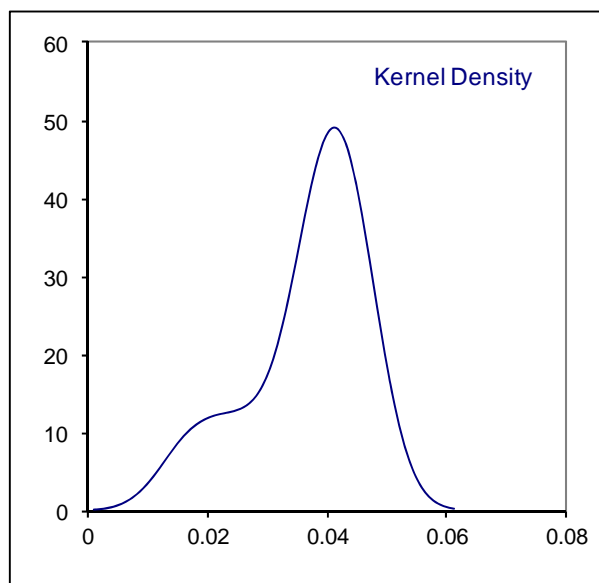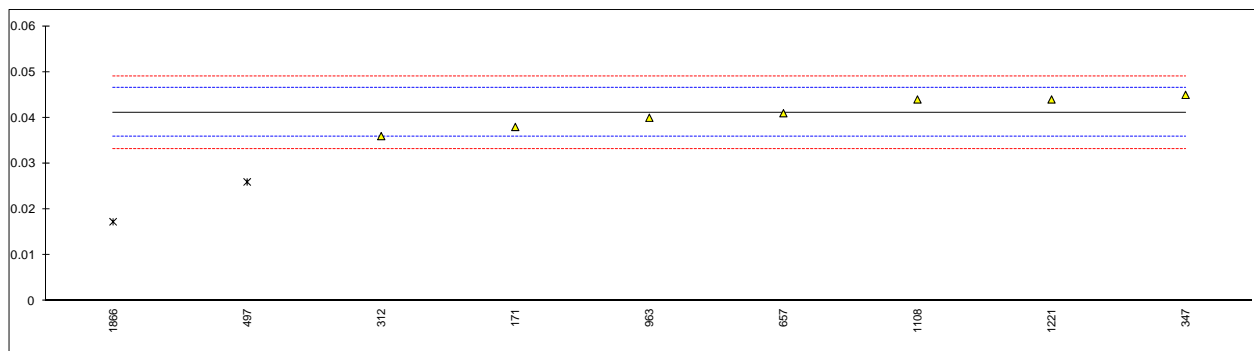
Determination of other individual diisobutylenes on MTBE sample #13003; results in %M/M

| Lab	method	243T2P *)	mark	z(targ)	344T1P *)	mark	z(targ)	35D1H *)	mark	z(targ)	remarks
171	D5441	0.001	-----	-----	0.105	-----	-----	0.003	-----	-----	
311	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
312	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
323	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
334	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
347	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
395	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
497	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
555	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
657	D5441	0.008	-----	-----	0.023	-----	-----	0.007	-----	-----	
663	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
862	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
963	D5441	<0.02	-----	-----	<0.02	-----	-----	<0.02	-----	-----	
1108	D5441	-----	-----	-----	0.131	-----	-----	0.002	-----	-----	
1200	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
1221	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
1386	D5441	-----	-----	-----	-----	-----	-----	-----	-----	-----	
1866	D5441	0.0425	-----	-----	0.0386	-----	-----	0.0187	-----	-----	
	normality	n.a.			n.a.			n.a.			
	n	3			4			4			
	outliers	0			0			0			
	mean (n)	0.0172			0.0744			0.0077			
	st.dev. (n)	n.a.			n.a.			n.a.			
	R(calc.)	n.a.			n.a.			n.a.			
	R(Horwitz)	n.a.			n.a.			n.a.			

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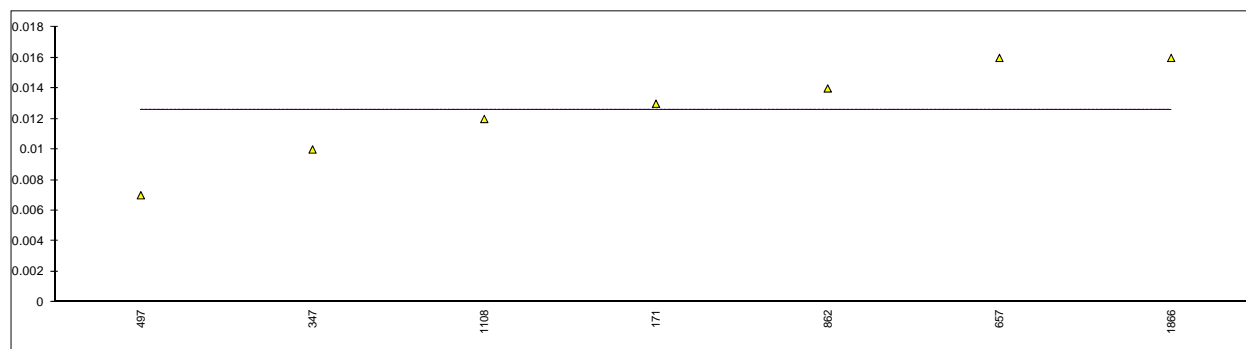*) 3,4,4-Trimethyl-1-pentene
*) 3,5-Dimethyl-1-hexene

Determination of Tert-Butanol on MTBE sample #13003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.116		-0.01	
311	D5441	0.12		0.07	
312	D5441	0.118		0.03	
323	D5441	0.12		0.07	
334	D5441	0.10		-0.35	
347	D5441	0.114		-0.06	
395		-----		-----	
497	D5441	0.093		-0.50	
555		-----		-----	
657	D5441	0.123		0.13	
663		-----		-----	
862	D5441	0.122		0.11	
963	D5441	0.12		0.07	
1108	D5441	0.126		0.20	
1200		-----		-----	
1221	D5441	0.129		0.26	
1386	D5441	0.109		-0.16	
1866	D5441	0.1236		0.15	

|	
normality	OK
n	14
outliers	0
mean (n)	0.1167
st.dev. (n)	0.00998
R(calc.)	0.0279
R(D5441:08e1)	0.1320
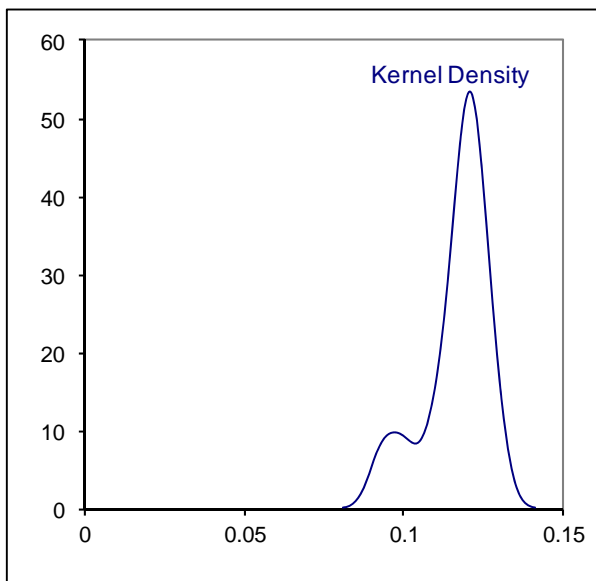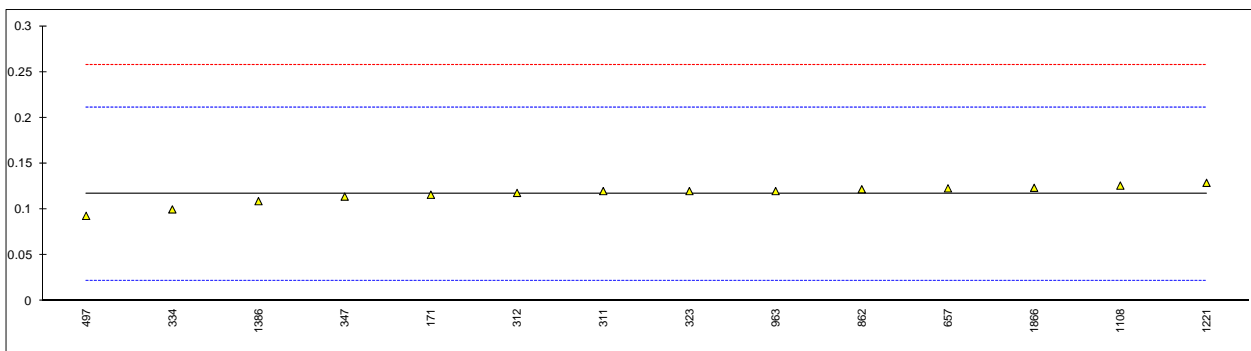
Determination of C4-hydrocarbons on MTBE sample #13003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.013		-----	
311	D5441	<0.02		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
347	D5441	0.01		-----	
395		-----		-----	
497	D5441	0.007		-----	
555		-----		-----	
657	D5441	0.016		-----	
663		-----		-----	
862	D5441	0.014		-----	
963	D5441	<0.02		-----	
1108	D5441	0.012		-----	
1200		-----		-----	
1221		-----		-----	
1386		-----		-----	
1866	D5441	0.0160		-----	

normality	OK
n	7
outliers	0
mean (n)	0.0126
st.dev. (n)	0.00326
R(calc.)	0.0091
R(D5441:08e1)	(0.0056)

Determination of C5-hydrocarbons on MTBE sample #13003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D5441	0.097		0.75	
311	D5441	0.08	C	-0.71	First reported 0.17
312		-----		-----	
323		-----		-----	
334		-----		-----	
347	D5441	0.079		-0.79	
395		-----		-----	
497	D5441	0.026	G(0.01)	-5.34	
555		-----		-----	
657	D5441	0.096		0.66	
663		-----		-----	
862	D5441	0.243	DG(0.01)	13.28	
963	D5441	0.09		0.15	
1108	D5441	0.18	DG(0.01)	7.87	
1200		-----		-----	
1221	D5441	0.089		0.06	
1386		-----		-----	
1866	D5441	0.0868		-0.13	

|	
normality	OK
n	7
outliers	3
mean (n)	0.0883
st.dev. (n)	0.00702
R(calc.)	0.0197
R(D5441:08e1)	0.0326
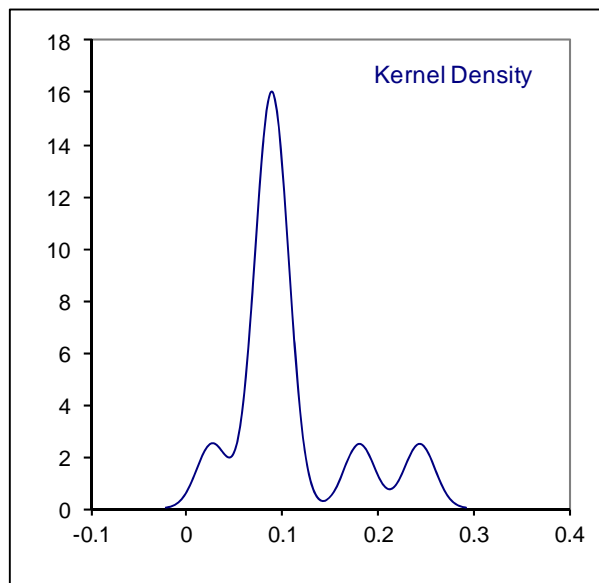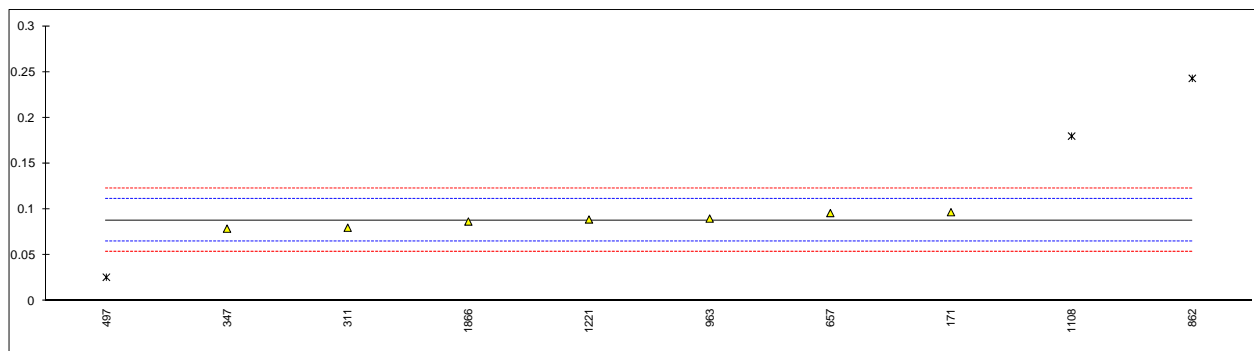
Determination of Other Hydrocarbons on MTBE sample #13003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		0.006		-----	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
347		-----		-----	
395		-----		-----	
497		1.281		-----	
555		-----		-----	
657	D5441	0.334		-----	
663		-----		-----	
862		-----		-----	
963		-----		-----	
1108		-----		-----	
1200		-----		-----	
1221		-----		-----	
1386		-----		-----	
1866	D5441	0.3099		-----	

normality	n.a.
n	4
outliers	0
mean (n)	0.4827
st.dev. (n)	n.a.
R(calc.)	n.a.
R(lit.)	n.a.

Determination of Unknown Impurities on MTBE sample #13003; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		0.653		-----	
311		-----		-----	
312		-----		-----	
323		-----		-----	
334		-----		-----	
347		-----		-----	
395		-----		-----	
497		0.282		-----	
555		-----		-----	
657	D5441	0.630		-----	
663		-----		-----	
862		-----		-----	
963	D5441	0.42		-----	
1108	D5441	0.42		-----	
1200		-----		-----	
1221		-----		-----	
1386		-----		-----	
1866	D5441	0.2499		-----	

normality	OK
n	6
outliers	0
mean (n)	0.4425
st.dev. (n)	n.a.
R(calc.)	n.a.
R(lit)	n.a.
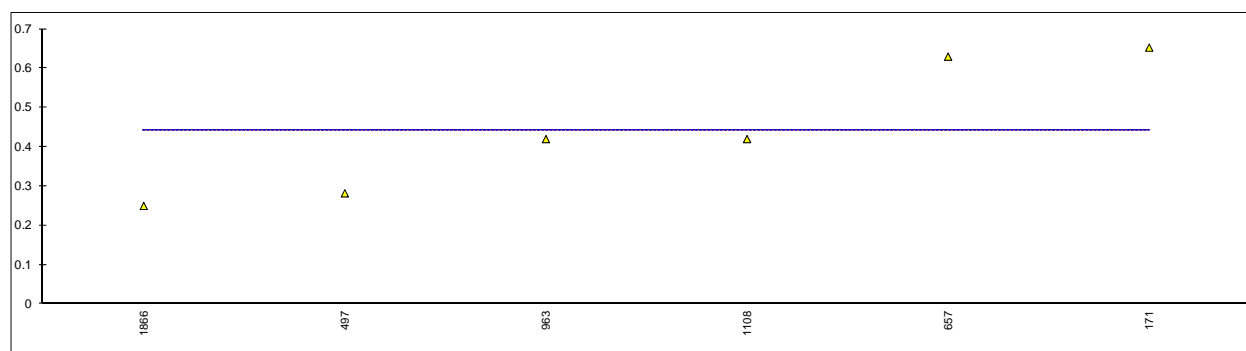
APPENDIX 2

Number of participating laboratories per country

1 lab in BELGIUM
1 lab in BRAZIL
1 lab in FRANCE
1 lab in GERMANY
1 lab in GREECE
1 lab in ITALY
2 labs in P.R. of CHINA
3 labs in SAUDI ARABIA
1 lab in SINGAPORE
1 lab in SPAIN
1 lab in THAILAND
2 labs in THE NETHERLANDS
1 lab in U.S.A.
1 lab in VENEZUELA

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
n.e.   = not evaluated
MSDS   = Material Safety Data Sheet
Fr.   = first reported
U   = reported in different unit
E   = possibly an error in the calculation

Literature:

1   iis Interlaboratory Studies. Protocol for the Organisation, Statistics and Evaluation, January 2010
2   ASTM E178-89
3   ASTM E1301-89
4   ISO 5725-86
5   ISO 5725. parts 1-6. 1994
6   ISO13528-05
7   M. Thompson and R. Wood. J. AOAC Int. 76. 926. (1993)
8   W.J. Youden and E.H. Steiner. Statistical Manual of the AOAC. (1975)
9   IP 367/84
10   DIN 38402 T41/42
11   P.L. Davies. First reported Z. Anal. Chem. 331. 513. (1988)
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)