

Results of Proficiency Test
Styrene
September 2012

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 1995, the Institute for Interlaboratory Studies organizes a proficiency test for the analysis of Styrene. During the annual proficiency testing program 2012/2013, it was decided to organize again a round robin for the analysis of Styrene. In this interlaboratory study, 44 laboratories from 21 different countries have participated. See appendix 2 for the number of participants per country.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted.

It was decided to send one 0.5 L bottle with Styrene (labelled #12100).

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 agreement with ISO/IEC 17043:2010, since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on 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' (iis-protocol, version 3.2) of January 2010.

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 bulk material was obtained from a local Styrene producer. The approximately 50 litre was spiked with 145.3 mg Chloroform, 759.2 mg Benzene, 87.3 mg Dibenzothiophene and 709.5 mg Polystyrene.

The bulk sample was, after homogenisation, divided over 90 amber glass bottles of 0.5 L (labelled #12100). The homogeneity of the subsamples #12100 was checked by determination of Density @ 15°C in accordance with ASTM D4052:02e1, Organic Chloride in accordance with ASTM D5808:09a and Polymers in accordance with ASTM 2121:07 on 8 stratified random selected samples.

	<i>Density @15°C in kg/L</i>	<i>Organic Chloride in mg/kg</i>	<i>Polymers in mg/kg</i>
sample #12100-1	0.91070	2.9	10.1
sample #12100-2	0.91070	3.0	9.3
sample #12100-3	0.91070	2.9	9.4
sample #12100-4	0.91070	3.0	9.2
sample #12100-5	0.91070	2.8	9.9
sample #12100-6	0.91070	2.8	9.7
sample #12100-7	0.91070	2.7	9.7
sample #12100-8	0.91070	2.9	9.7

Table 1: homogeneity test results of subsamples #12100

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	<i>Density @15°C in kg/L</i>	<i>Organic Chloride in mg/kg</i>	<i>Polymers in mg/kg</i>
r (sample #12100)	0.00000	0.3	0.9
Ref. method	ASTM D4052:02e1	ASTM5808:09a	Horwitz
0.3 x R (ref. method)	0.00015	0.4	0.9

Table 2: repeatabilities of subsamples #12100

Each calculated repeatability was less than 0.3 times the corresponding reproducibility of the reference method.

To each of the participating laboratories, 1 bottle of 0.5 L (labelled #12100) was sent on September 5, 2012.

2.5 STABILITY OF THE SAMPLES

The stability of Styrene, packed in a brown glass bottle, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine on sample #12100: Aldehydes as benzaldehyde, Appearance, Colour Pt/Co, Inhibitor, Density @ 20°C, Peroxide as H₂O₂, Polymer, Organic Chloride, Sulphur, Water, Purity and the Impurities: Benzene, Ethylbenzene, m- & p-Xylenes, Cumene, o-Xylene, n-Propylbenzene, m- & p-Ethyltoluenes, alpha-Methylstyrene, 1,2-diethylbenzene, Phenylacetylene, 3,4-dimethylstyrenes, Benzaldehyde and Nonaromatics.

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 were 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 results.

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 were used for data analysis and original results were placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as 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 did 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, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. 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 visualise 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 is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nrs.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 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.

The z-scores were calculated according to:

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

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare. Therefore, 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 with the despatch of the samples to the laboratories. Probably due to problems with custom clearance laboratories in Brazil, India and Saudi Arabia did not receive the samples in time or did not receive any sample at all. Eight participants reported results after the final reporting date and two laboratories did not report results at all. Finally, 38 laboratories did report 552 numerical results. Observed were 30 outlying results, which is 5.4%. In proficiency studies outlier percentages of 3 - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section the results were discussed per test. The standard reproducibilities used for the evaluation of the GC impurities were extrapolated from the reproducibilities, as mentioned in ASTM D 5135:07 (table 3).

Not all original data sets proved to have a normal distribution. Not normal distributions were found for the following determinations: Colour Pt/Co, Density, Purity, Water, Ethylbenzene, o-Xylene, Benzaldehyde, Nonaromatics. In these cases the statistical evaluation should be used with due care.

Aldehydes as benzaldehyde: This determination was very problematic as in previous years. No statistical outliers were observed. However, the observed reproducibility, is not at all in agreement with the requirements of ASTM D2119:09. Two test results were excluded as the results of total Aldehydes were lower than the results of Benzaldehyde by GC.

Appearance: A new standardized method is available for Appearance since 2009, being ASTM E2680. However, not all participants did report according this method. All participants agreed about the appearance of sample #12100, except one. This laboratory reported that the sample failed to be CFMS. Participants who used the ASTM E2680 should report the Appearance as

'pass' (or 'fail'). Twenty-two participants reported the appearance correctly as pass. The other laboratories used different kind of terms or abbreviations like: Clear, C&B, CFFSM and CFSM. The explanations for the used abbreviations are given on page 15.

- Colour Pt/Co: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with the requirements of ASTM D1209:11.
- Density: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is in good agreement with the requirements of ASTM D4052:02e1. The current version of this method ASTM D4052:11 only gives reproducibilities for the density range 0.71 g/ml to 0.88 g/ml, being valid for gasolines, distillates, basestocks and lubricating oils. Therefore this 2011 version is not applicable for Styrene.
- Inhibitor: The determination of pTBC was problematic for a number of laboratories. Four statistical outliers were observed. However, the observed reproducibility, after rejection of the statistical outliers, is in full agreement with the requirements of ASTM D4590:09.
- Peroxides: This determination was very problematic. Two statistical outliers were observed. The observed reproducibility is not at all in agreement with the requirements of ASTM D2340:09.
- Polymers: This determination was very problematic. Three statistical outliers were observed. The calculated reproducibility is not at all in agreement with the requirements estimated from the Horwitz equation. Remarkable is the significant difference between the Horwitz equation and reproducibility of the method. Therefore the conclusion can be drawn that the reproducibility of the method probably can not be met in practice.
- The average recovery of Polymers (theoretical increment of 14.2 mg/kg) may be unsatisfactory: "less then 75%" (the actual blank Polymers is unknown).
The reported test results vary over a large range: 1.2 – 15.2 mg/kg. The application range of the determination is 1 – 15 mg/kg.
- Org. chloride: This determination was not problematic. Three statistical outliers were observed. However, the observed reproducibility, after rejection of the statistical outliers, is in good agreement with the requirements of ASTM D5808:09a.
The average recovery of Organic Chloride (theoretical increment of 2.59 mg/kg) may be good: "less then 113%" (the actual blank Organic Chloride is unknown).

- Sulphur: This determination was not problematic. No statistical outliers were observed and the observed reproducibility is in agreement with the requirements of ASTM D5453:09. One false negative result was observed.
The average recovery of Sulphur (theoretical increment of 1.75 mg/kg) may be good: "less than 115%" (the actual blank Sulphur is unknown).
- Water: This determination was very problematic. One statistical outlier was observed. However, the observed reproducibility, after rejection of the statistical outlier is not at all in agreement with the requirement of ASTM E1064:05.
- Purity: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with the requirements of ASTM D5135:07. Due to false positive reported test results for Benzene, 1,2-Diethylbenzene or Phenylacetylene three test results were excluded for determination of the purity.
- Benzene: This determination may be very problematic. Two statistical outliers were observed. However, the observed reproducibility is not at all in agreement with the requirement estimated from the Horwitz equation.
The average recovery of Benzene (theoretical increment of 15.2 mg/kg) may be good: "less than 117%" (the actual blank Benzene content is unknown).
- Ethylbenzene: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility, after rejection of the statistical outliers is in agreement with the requirements of ASTM D5135:07.
- m- & p-Xylenes: This determination was very problematic. Two statistical outliers were observed. However, the observed reproducibility, after rejection of the statistical outliers, is not at all in agreement with the requirements of ASTM D5135:07. It must be noted that the observed reproducibility is in agreement with the requirements estimated from the Horwitz equation. Remarkable is the significant difference between the Horwitz equation and reproducibility of the method. Therefore the conclusion can be drawn that the reproducibility of the method probably can not be met in practice.
- Cumene: This determination was problematic for a number of laboratories. Seven (!) statistical outliers were observed. However, the observed reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D5135:07.

- o-Xylene: This determination was not problematic. No statistical outliers were observed and the observed reproducibility is in good agreement with the requirements of ASTM D5135:07. Remarkable is the significant difference between reproducibility of the method and the Horwitz equation. Therefore the conclusion can be drawn that the reproducibility of the method is probably not realistic.
- n-Prop.benzene: No significant conclusions were drawn as the concentration n-Propylbenzene was below the application range (10 – 10000 mg/kg) of the test method ASTM D5135:07.
- m- & p-Ethyltol.: No significant conclusions were drawn as the concentration m- & p-Ethyltoluenes was below the application range (10 – 10000 mg/kg) of the test method ASTM D5135:07. One laboratory reported a false positive test result for m- & p-Ethyltoluenes.
- α -Methylstyrene: The determination of this component was problematic. One statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is not in agreement with the requirements of ASTM D5135:07. The problem may be caused by the fact that the peak of this component is present on the long tail of the styrene peak, which may lead to integration differences between the various laboratories.
- 1,2-Diethylbenz.: Six laboratories reported a 'less than' result for this component, while one result was possibly false positive. Therefore no significant conclusions were drawn.
- Phenylacetylene: Seventeen reported a 'less than' result for this component and two laboratories reported a false positive test result. Therefore no significant conclusions were drawn.
- 3- & 4 -Me-Styrenes: Eight of the eleven laboratories reported a 'less than' result for this component. Therefore no significant conclusions were drawn.
- Benzaldehyde: This determination was very problematic. No statistical outliers were observed. However, the observed reproducibility is not at all in agreement with the requirement estimated from the Horwitz equation. Two test results were excluded as both results for Benzaldehyde were higher than the results of total Aldehydes. One laboratory reported a false negative test result.
- Nonaromatics: This determination may be very problematic. As the group seems to be divided bimodally, no significant conclusions were drawn. However, the large spread may be caused by problems with the identification of the various peaks.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The results and the calculated reproducibilities and the reproducibilities, derived from literature standards (in casu ASTM standards) are compared in the next table.

Parameter	unit	n	average	2.8 *sd _R	R (lit)
Aldehydes as benzaldehyde	mg/kg	31	105.8	122.3	42.3
Appearance		36	pass	n.a.	n.a.
Colour		35	7.5	5.7	7.0
Density @20°C	kg/L	33	0.9063	0.0002	0.0005
Inhibitor (p-TBC)	mg/kg	29	5.3	1.7	1.9
Peroxides as H ₂ O ₂	mg/kg	30	40.6	21.6	13.0
Polymers	mg/kg	24	10.7	5.9	3.4
Organic Chlorides	mg/kg	15	2.93	0.49	1.30
Sulphur	mg/kg	23	2.02	0.88	0.98
Water	mg/kg	34	189.3	53.8	30.1
Purity	%M/M	32	99.925	0.036	0.033
Benzene	mg/kg	29	17.8	9.6	5.2
Ethylbenzene	mg/kg	33	90.9	25.4	26.0
m- & p-Xylenes	mg/kg	28	39.3	10.2	2.2
Cumene	mg/kg	25	47.1	5.3	5.9
o-Xylene	mg/kg	30	18.3	5.1	25.6
n-Propylbenzene	mg/kg	27	<10	n.a.	n.a.
m- & p-Ethyltoluenes	mg/kg	24	<10	n.a.	n.a.
α-Methylstyrene	mg/kg	30	249.5	50.5	35.6
1,2-diethylbenzene	mg/kg	6	<10	n.a.	n.a.
Phenylacetylene	mg/kg	23	<10	n.a.	n.a.
3- & 4-Methylstyrenes	mg/kg	11	<10	n.a.	n.a.
Benzaldehyde	mg/kg	14	98.1	48.8	22.0
Nonaromatics	mg/kg	17	60.7	100.3	(46.3)

Table 3: reproducibilities for sample #12100
between brackets: below LOD

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2012 WITH PREVIOUS PTS

	September 2012	October 2011	October 2010	October 2009
Number of reporting labs	38	31	35	33
Number of Results reported	552	403	496	613
Statistical outliers	30	15	30	44
Percentage outliers	5.4%	3.7%	6.1%	7.2%

Table 4: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal. The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given in the following table:

Determination	September 2012	October 2011	October 2010	October 2009	
Aldehydes	--	--	--	--	--
Colour	+	++	++	++	
Density	++	++	++	+	
Inhibitor	+	-	-	+/-	
Peroxides as H ₂ O ₂	--	--	-	-	
Polymer	--	+/-	n.e.	--	
Organic chloride	++	n.e	n.e.	n.e.	
Sulphur	+	n.e	n.e.	n.e.	
Water	--	--	--	-	
Purity	+/-	+	++	++	++
Benzene	--	n.e	n.e.	n.e.	+
Ethylbenzene	+/-	++	++	++	+
m+p-Xylenes	--	--	n.e.	n.e.	--
Cumene	+	--	-	n.e.	--
o-Xylene	++	++	n.e.	n.e.	n.e.
n-Propylbenzene	n.e	-	++	n.e.	n.e.
m+p-Ethyltoluenes	n.e	n.e	--	n.e.	n.e.
α-Methylstyrene	-	--	-	-	--
1,2-diethylbenzene	n.e	n.e	--	n.e.	n.e.
Phenylacetylene	(--)	--	--	n.e.	n.e.
3-&4-methylstyrenes	n.e	n.e	--	n.e.	n.e.
Benzaldehyde	--	--	--	--	--
Nonaromatics	n.e.	--	n.e	n.e.	n.e.

Table 5: comparison of overall performance per parameter against the standard requirements

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

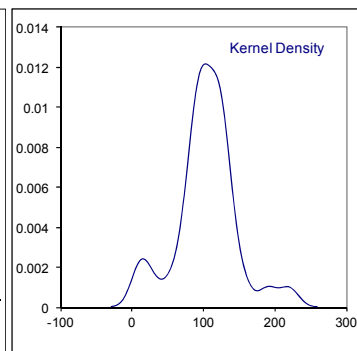
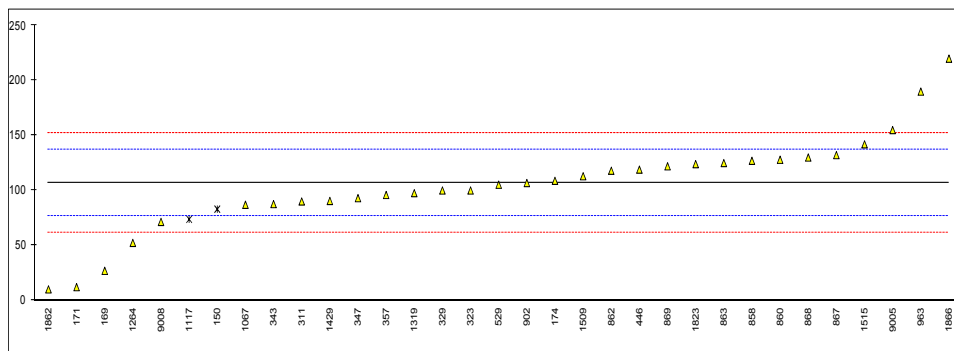
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.e.: not evaluated

APPENDIX 1

Determination of Aldehydes as benzaldehyde on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D2119	83	ex	-1.51	see § 4.1
169	D2119	26.9		-5.22	
171	D2119	12		-6.21	
174	D1209	108.8		0.20	
311	D2119	90		-1.05	
323	D2119	100		-0.38	
329	D2119	100		-0.38	
333		----		----	
343	D2119	87.60		-1.20	
347	D2119	93		-0.85	
357	D2119	95.9		-0.66	
396		----		----	
446	D2119	119		0.87	
529	D2119	105.25		-0.04	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858	D2119	127		1.40	
860	D2119	128		1.47	
862	D2119	118		0.81	
863	D2119	125	C	1.27	first reported: 159
867	D2119	132.2		1.75	
868	D2119	130		1.60	
869	D2119	122	C	1.07	first reported:162
902	D2119	106.8		0.07	
913		----		----	
963	D2119	190		5.57	
1067	D2119	87		-1.24	
1085		----		----	
1107		----		----	
1117	D2119	73.8	ex	-2.12	see § 4.1
1169		----		----	
1252		----		----	
1264	D2119	52.3	C	-3.54	first reported:51.8
1319	D2119	97.6		-0.54	
1429	D2119	90.5		-1.01	
1509	D2119	113		0.48	
1515	D2119	142		2.39	
1823	D2119	124		1.20	
1862	D2119	10.0		-6.34	
1866	D2119	220		7.55	
9005	D2119	155	C	3.25	first reported: 0.0155
9008	D2119	71.4		-2.28	

normality not OK
n 31
outliers 0
mean (n) 105.81
st.dev. (n) 43.683
R(calc.) 122.31
R(D2119:09) 42.33



Determination of Appearance on sample #12100;

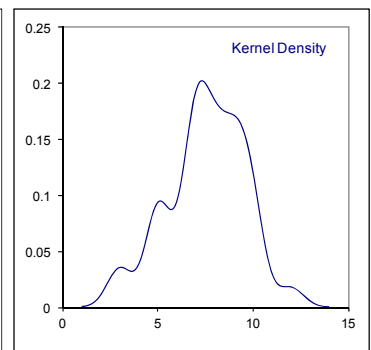
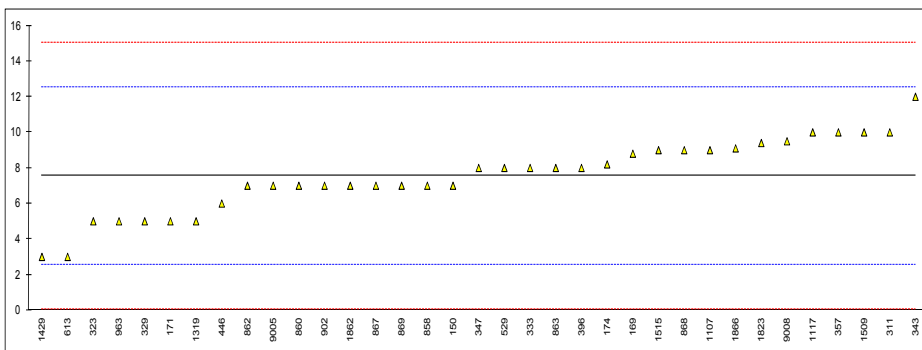
lab	method	value	mark	z(targ)	Remarks
150	E2680	pass		----	
169	D4176	CB&FSM		----	
171	E2680	pass		----	
174	E2680	pass		----	
311	E2680	pass		----	
323	E2680	pass		----	
329	E2680	pass		----	
333	E2680	C&B		----	
343	E2680	pass		----	
347	E2680	pass		----	
357	E2680	pass		----	
396	E2680	pass		----	
446	E2680	pass		----	
529	E2680	pass		----	
551		----		----	
557		----		----	
613	E2680	C&B		----	
663		----		----	
858	E2680	pass		----	
860	E2680	pass		----	
862	VISUAL	C&B		----	
863	E2680	pass		----	
867	E2680	pass		----	
868	E2680	pass		----	
869	E2680	pass		----	
902	E2680	pass		----	
913		----		----	
963	E2680	pass		----	
1067	E2680	pass		----	
1085		----		----	
1107	E2680	C&B		----	
1117	D4176	CFFSM		----	
1169		----		----	
1252		----		----	
1264	E2680	Clear		----	
1319	E2680	C&B		----	
1429	E2680	C&B		----	
1509	E2680	CFSM		----	
1515	E2680	C&B		----	
1823	E2680	----		----	fail to be CFSM
1862	visual	CLFSH		----	
1866	E2680	Clear		----	
9005	E2680	pass		----	
9008	E2680	Clear		----	
	n	36			
	average	pass			

Abbreviations:

C&B:	Clear and Bright
C&F:	Clear and Free
CFFSM:	Clear and Free from Matter in Suspension
CFSM:	Clear and free from Suspended Matter
CLFSH:	Clear Liquid Free of Sediment and Haze

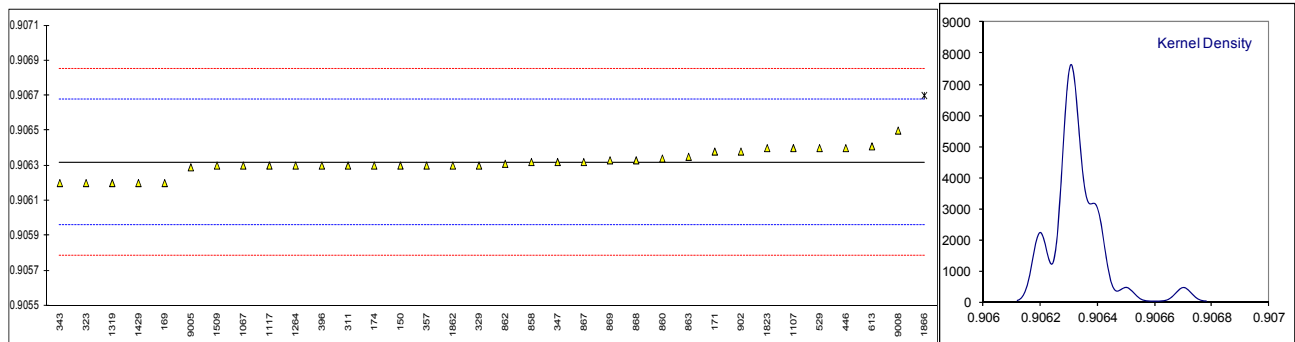
Determination of Colour Pt/Co on sample #12100;

lab	method	value	mark	z(targ)	Remarks
150	D1209	7		-0.22	
169	D5386	8.8		0.50	
171	D1209	5		-1.02	
174	D1209	8.2		0.26	
311	D1209	10		0.98	
323	D1209	5		-1.02	
329	D1209	5		-1.02	
333	D1209	8		0.18	
343	D1209	12		1.78	
347	D5386	8		0.18	
357	D1209	10		0.98	
396	D1209	8		0.18	
446	D1209	6		-0.62	
529	D1209	8		0.18	
551		----		----	
557		----		----	
613	D1209	3		-1.82	
663		----		----	
858	D1209	7		-0.22	
860	D1209	7		-0.22	
862	D1209	7		-0.22	
863	D1209	8		0.18	
867	D1209	7		-0.22	
868	D1209	9		0.58	
869	D1209	7		-0.22	
902	D5386	7		-0.22	
913		----		----	
963	D1209	5		-1.02	
1067		----		----	
1085		----		----	
1107	D1209	9		0.58	
1117	D1209	10		0.98	
1169		----		----	
1252		----		----	
1264	D1209	<10		----	
1319	D1209	5		-1.02	
1429	D5386	3		-1.82	
1509	D1209	10		0.98	
1515	D1209	9		0.58	
1823	D5386	9.4		0.74	
1862	D1209	7		-0.22	
1866	D1209	9.1		0.62	
9005	D5386	7.0		-0.22	
9008	D1209	9.5		0.78	
normality		not OK			
n		35			
outliers		0			
mean (n)		7.54			
st.dev. (n)		2.042			
R(calc.)		5.72			
R(D1209:11)		7.00			



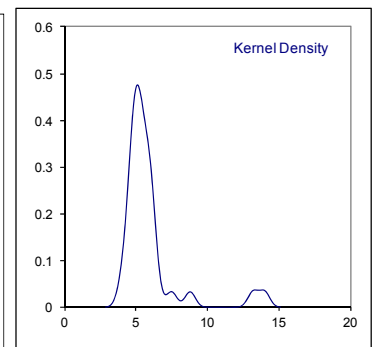
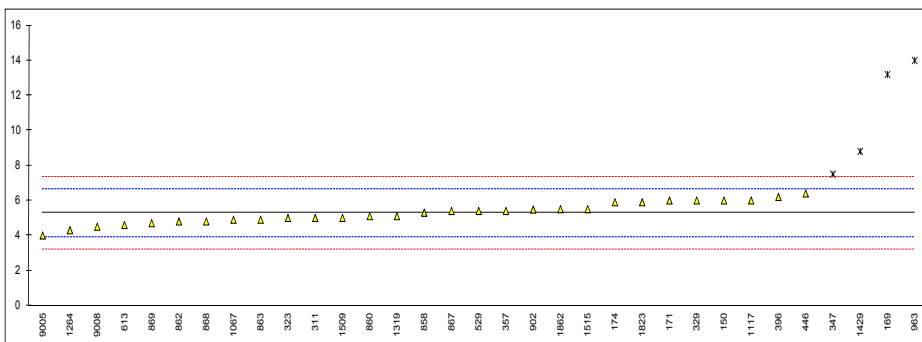
Determination of Density at 20°C on sample #12100; results in kg/L

lab	method	value	mark	z(targ)	Remarks
150	D4052	0.9063		-0.10	
169	D4052	0.9062		-0.66	
171	D4052	0.90638		0.35	
174	D4052	0.9063		-0.10	
311	D4052	0.9063		-0.10	
323	D4052	0.9062		-0.66	
329	D4052	0.9063		-0.10	
333		----		----	
343	D4052	0.9062		-0.66	
347	D4052	0.90632		0.01	
357	D4052	0.9063		-0.10	
396	D4052	0.9063		-0.10	
446	D4052	0.9064		0.46	
529	D4052	0.9064		0.46	
551		----		----	
557		----		----	
613	D4052	0.90641		0.52	
663		----		----	
858	D4052	0.90632		0.01	
860	D4052	0.90634		0.13	
862	D4052	0.90631		-0.04	
863	D4052	0.90635		0.18	
867	D4052	0.90632		0.01	
868	D4052	0.90633		0.07	
869	D4052	0.90633		0.07	
902	D4052	0.90638		0.35	
913		----		----	
963		----		----	
1067	D4052	0.9063		-0.10	
1085		----		----	
1107	D4052	0.9064		0.46	
1117	D4052	0.9063		-0.10	
1169		----		----	
1252		----		----	
1264	D4052	0.9063		-0.10	
1319	D4052	0.9062		-0.66	
1429	D4052	0.9062		-0.66	
1509	D4052	0.9063		-0.10	
1515		----		----	
1823	D4052	0.9064		0.46	
1862	D4052	0.9063		-0.10	
1866	D4052	0.9067	C, G(0.01)	2.14	reported 906.700
9005	D4052	0.90629		-0.15	
9008	D4052	0.9065		1.02	
normality		not OK			
n		33			
outliers		1			
mean (n)		0.90632			
st.dev. (n)		0.000007			
R(calc.)		0.00019			
R(D4052:02e1)		0.00050			



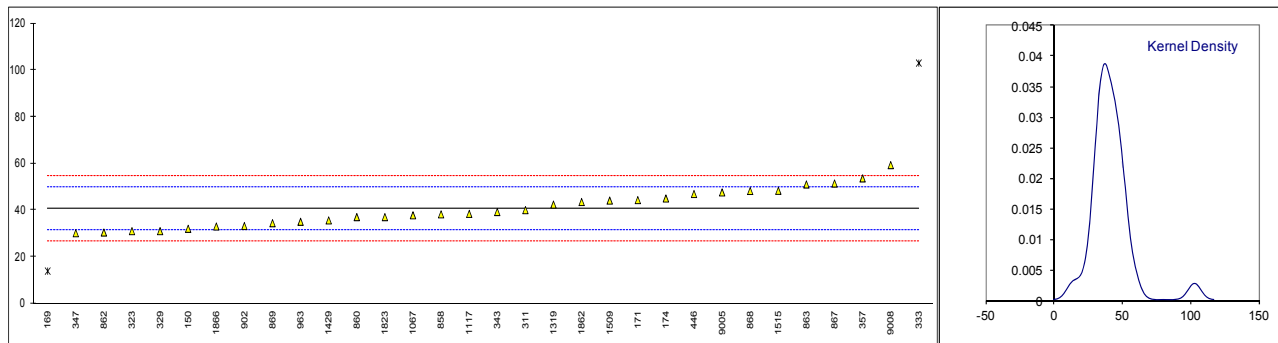
Determination of Inhibitor (pTBC) on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D4590	6		1.05	
169	D4590	13.2	G(0.01)	11.50	
171	D4590	6		1.05	
174	D4590	5.9		0.90	
311	D4590	5		-0.40	
323	D4590	5		-0.40	
329	D4590	6		1.05	
333		----		----	
343		----		----	
347	D4590	7.5	G(0.05)	3.22	
357	D4590	5.4		0.18	
396	D4590	6.2		1.34	
446	D4590	6.4		1.63	
529	D4590	5.4		0.18	
551		----		----	
557		----		----	
613	D4590	4.6		-0.98	
663		----		----	
858	D4590	5.3		0.03	
860	D4590	5.1		-0.26	
862	D4590	4.8		-0.69	
863	D4590	4.9		-0.55	
867	D4590	5.4		0.18	
868	D4590	4.8		-0.69	
869	D4590	4.7		-0.84	
902	D4590	5.48		0.29	
913		----		----	
963	D4590	14	G(0.01)	12.66	
1067	D4590	4.9		-0.55	
1085		----		----	
1107		----		----	
1117	D4590	6.0		1.05	
1169		----		----	
1252		----		----	
1264	D4590	4.3		-1.42	
1319	D4590	5.1		-0.26	
1429	D4590	8.8	G(0.01)	5.11	
1509	D4590	5.0		-0.40	
1515	D4590	5.5		0.32	
1823	D4590	5.9		0.90	
1862	D4590	5.5		0.32	
1866		----		----	
9005	D4590	4.00		-1.86	
9008	D4590	4.5		-1.13	
normality		OK			
n		29			
outliers		4			
mean (n)		5.28			
st.dev. (n)		0.603			
R(calc.)		1.69			
R(D4590:09)		1.93			



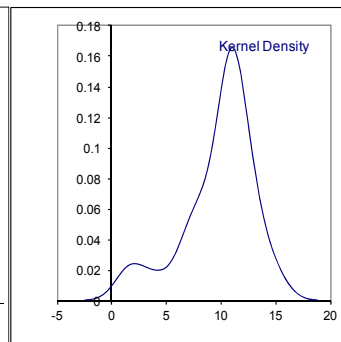
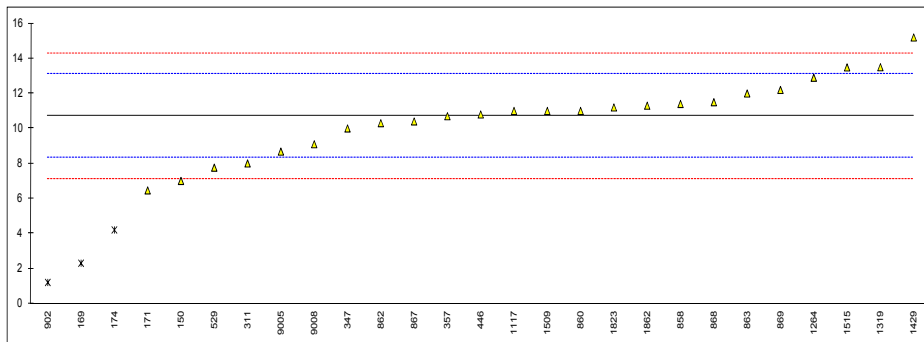
Determination of Peroxides as H₂O₂ on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D2340	32		-1.86	
169	D2340	13.858	D(0.05)	-5.77	
171	D2340	44.3		0.79	
174	D2340	45.0		0.94	
311	D2340	40		-0.14	
323	D2340	31	C	-2.07	first reported: 23
329	D2340	31		-2.07	
333	D2340	103	D(0.01)	13.43	
343	D2340	39.2		-0.31	
347	D2340	30.1		-2.27	
357	D2340	53.6		2.79	
396		----		----	
446	D2340	46.9		1.35	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858	D2340	38.2		-0.52	
860	D2340	37		-0.78	
862	D2340	30.4		-2.20	
863	D2340	51.0		2.23	
867	D2340	51.4		2.32	
868	D2340	48.2		1.63	
869	D2340	34.4		-1.34	
902	D2340	33.18		-1.60	
913		----		----	
963	D2340	35		-1.21	
1067	D2340	37.8		-0.61	
1085		----		----	
1107		----		----	
1117	D2340	38.4		-0.48	
1169		----		----	
1252		----		----	
1264		----		----	
1319	D2340	42.4		0.38	
1429	D2340	35.6		-1.08	
1509	D2340	44.1		0.75	
1515	D2340	48.3		1.65	
1823	D2340	37		-0.78	
1862	D2340	43.47		0.61	
1866	D2340	33		-1.64	
9005	D2340	47.6		1.50	
9008	D2340	59.29		4.02	
normality		OK			
n		30			
outliers		2			
mean (n)		40.63			
st.dev. (n)		7.729			
R(calc.)		21.64			
R(D2340:09)		13.00			



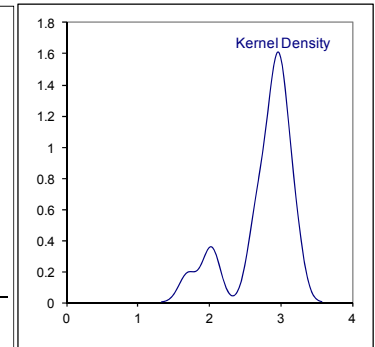
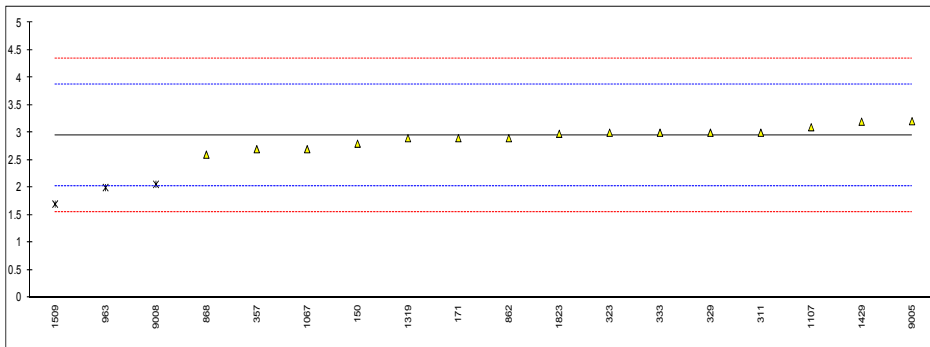
Determination of Polymers on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D2121	7		-3.09	
169	D2121	2.3	DG(0.05)	-7.01	
171	D2121	6.459		-3.54	
174	D2121	4.2	C, DG(0.05)	-5.43	first reported: <1
311	D2121	8		-2.26	
323		----		----	
329		----		----	
333		----		----	
343		----		----	
347	D2121	10		-0.59	
357	D2121	10.7		0.00	
396		----		----	
446	D2121	10.8		0.08	
529	D2121	7.76		-2.46	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858	D2121	11.4		0.58	
860	D2121	11.0		0.25	
862	D2121	10.3		-0.34	
863	D2121	12.0		1.08	
867	D2121	10.4		-0.25	
868	D2121	11.5		0.66	
869	D2121	12.2		1.25	
902	D2121	1.2	C, DG(0.05)	-7.93	first reported: 3.2
913		----		----	
963		----		----	
1067		----		----	
1085		----		----	
1107		----		----	
1117	D2121	11.0		0.25	
1169		----		----	
1252		----		----	
1264	D2121	12.9		1.83	
1319	D2121	13.5		2.33	
1429	D2121	15.2		3.75	
1509	D2121	11.0		0.25	
1515	D2121	13.49		2.32	
1823	D2121	11.2		0.41	
1862	D2121	11.3		0.50	
1866		----		----	
9005	D2121	8.68		-1.69	
9008	D2121	9.1		-1.34	
	normality	OK		<u>spike</u>	
	n	24			
	outliers	3			
	mean (n)	10.70		14.2	Recovery: <75%
	st.dev. (n)	2.102			
	R(calc.)	5.89			
	R(Horwitz)	3.36			Compare R(D2121:07): 1.00, application range: 1 – 15 mg/kg,



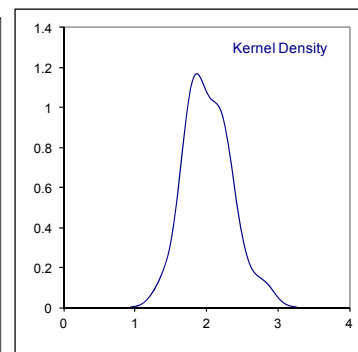
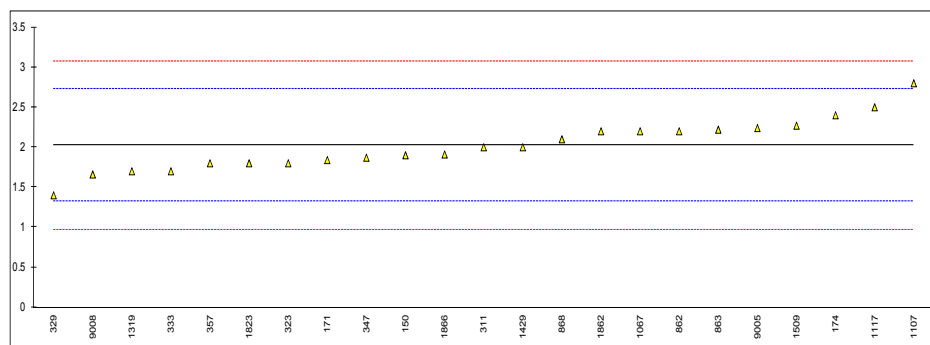
Determination of Organic Chloride on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D7359	2.8		-0.29	
169		----		----	
171	D5808	2.90		-0.07	
174		----		----	
311	D5808	3		0.15	
323	UOP779	3		0.15	
329	UOP779	3		0.15	
333	UOP779	3		0.15	
343		----		----	
347		----		----	
357	D5808	2.7	C	-0.50	first reported: 2
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858		----		----	
860		----		----	
862	D5808	2.9		-0.07	
863		----		----	
867		----		----	
868	D5808	2.6		-0.72	
869		----		----	
902		----		----	
913		----		----	
963	UOP779Mod.	2	DG(0.01)	-2.01	
1067		2.7		-0.50	
1085		----		----	
1107	UOP779	3.1		0.36	
1117		----		----	
1169		----		----	
1252		----		----	
1264		----		----	
1319	D5808	2.9		-0.07	
1429	D5808	3.2		0.58	
1509	D5808	1.70	C, G(0.05)	-2.65	first reported: 1.36
1515		----		----	
1823	D5808	2.98		0.10	
1862		----		----	
1866		----		----	
9005	D5808	3.21		0.60	
9008		2.06	DG(0.01)	-1.88	
	normality	OK		<u>spike</u>	
	n	15			
	outliers	3			
	mean (n)	2.933		2.59	recovery: <113%
	st.dev. (n)	0.1766			
	R(calc.)	0.494			
	R(D5808:09a)	1.300			



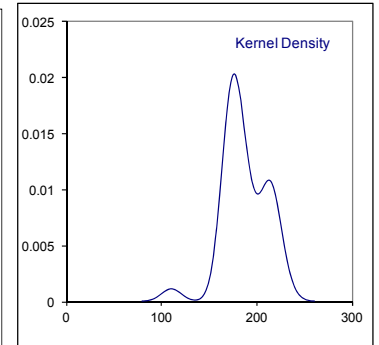
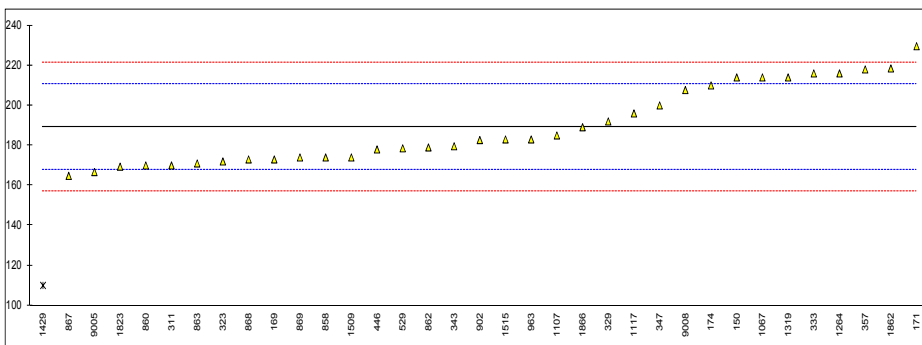
Determination of Sulphur on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D5453	1.9		-0.35	
169		----		----	
171	D5453	1.84		-0.52	
174	D5453	2.4		1.08	
311	D5453	2		-0.06	
323	D5453	1.8		-0.63	
329	D5453	1.4		-1.77	
333	D5453	1.7		-0.92	
343		----		----	
347	D5453	1.87		-0.43	
357	D5453	1.8		-0.63	
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858		----		----	
860		----		----	
862	D5453	2.2		0.51	
863	D5453	2.22		0.56	
867		----		----	
868	D3120	2.1		0.22	
869		----		----	
902		----		----	
913		----		----	
963	D4045	<0.02		<-5.47	false negative result?
1067	D5453	2.2		0.51	
1085		----		----	
1107	D5453	2.8		2.22	
1117	D5453	2.5		1.36	
1169		----		----	
1252		----		----	
1264		----		----	
1319	D5453	1.7		-0.92	
1429	D5453	2.0		-0.06	
1509	D5453	2.27		0.71	
1515		----		----	
1823	D5453	1.8		-0.63	
1862	ISO20846	2.20		0.51	
1866	D5453	1.91		-0.32	
9005	D5453	2.24		0.62	
9008	D5453	1.66	C	-1.03	first reported: 0.66
	normality	OK		<u>spike</u>	
	n	23			
	outliers	0			
	mean (n)	2.022		1.75	recovery: <115%
	st.dev. (n)	0.3151			
	R(calc.)	0.882			
	R(D5453:09)	0.983			



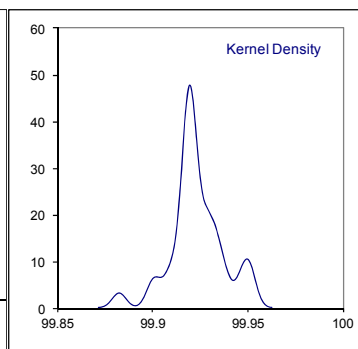
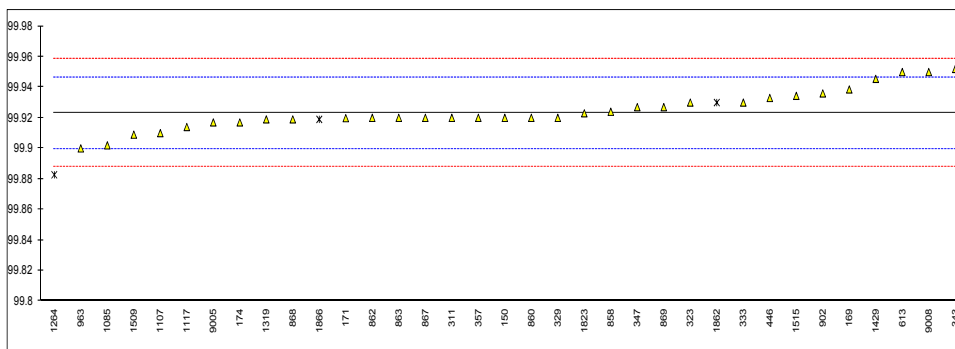
Determination of Water on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	E1064	214		2.30	
169	E1064	173		-1.52	
171	E1064	229.608		3.75	
174	E1064	210		1.93	
311	E1064	170		-1.79	
323	E1064	172		-1.61	
329	E1064	192		0.25	
333	E1064	216		2.49	
343	E1064	179.6		-0.90	
347	E1064	200		1.00	
357	E1064	218		2.67	
396		----		----	
446	E203	178		-1.05	
529	E1064	178.59		-1.00	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858	E1064	174		-1.42	
860	E1064	170		-1.79	
862	E1064	179		-0.96	
863	E1064	171		-1.70	
867	E1064	164.8		-2.28	
868	E1064	173		-1.52	
869	E1064	174		-1.42	
902	E1064	182.7		-0.61	
913		----		----	
963	E1064	183		-0.58	
1067	E1064	214		2.30	
1085		----		----	
1107	E1064	185		-0.40	
1117	E1064	196.0		0.62	
1169		----		----	
1252		----		----	
1264	E1064	216		2.49	
1319	E1064	214		2.30	
1429	D1364	110	G(0.01)	-7.38	
1509	E1064	174		-1.42	
1515	E1064	183		-0.58	
1823	E1064	169.4		-1.85	
1862	E1064	218.5		2.72	
1866	E1064	189.1		-0.02	
9005	E1064	166.7		-2.10	
9008	E1064	207.7		1.71	
normality		not OK			
n		34			
outliers		1			
mean (n)		189.29			
st.dev. (n)		19.223			
R(calc.)		53.83			
R(E1064:05)		30.10			



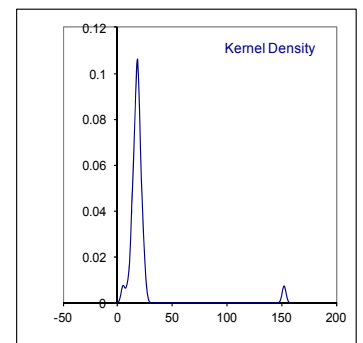
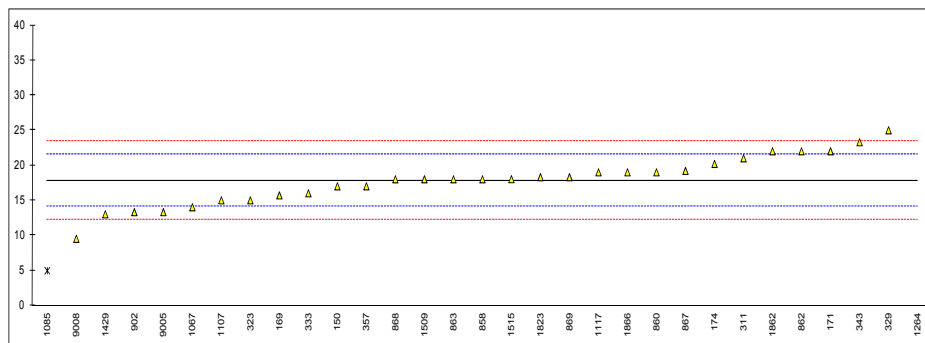
Determination of Purity on sample #12100; results in %M/M

lab	method	value	mark	z(targ)	Remarks
150	D5135	99.92		-0.39	
169	D5135	99.9386		1.19	
171	D5135	99.9198		-0.41	
174	D7504	99.917		-0.65	
311	D5135	99.92		-0.39	
323	D5135	99.93		0.46	
329	D5135	99.92		-0.39	
333	D5135	99.93		0.46	
343	D5135	99.952		2.33	
347	D5135	99.927		0.20	
357	D5135	99.92		-0.39	
396		----		----	
446	D5135	99.933		0.71	
529		----		----	
551		----		----	
557		----		----	
613	D5135	99.95		2.16	
663		----		----	
858	D5135	99.924		-0.05	
860	D5135	99.920		-0.39	
862	D5135	99.92		-0.39	
863	D5135	99.920		-0.39	
867	D5135	99.920		-0.39	
868	D5135	99.919		-0.48	
869	D5135	99.927		0.20	
902	D5135	99.936		0.97	
913		----		----	
963	D5135	99.90		-2.09	
1067		----		----	
1085	in house	99.9020		-1.92	
1107	in house	99.91		-1.24	
1117	D5135	99.914		-0.90	
1169		----		----	
1252		----		----	
1264	D5135	99.8827	C, ex	-3.56	first reported: 99.879, see § 4.1
1319	D5135	99.919		-0.48	
1429	D5135	99.9455		1.77	
1509	D5135	99.909		-1.32	
1515	in house	99.9344		0.83	
1823	D5135	99.923		-0.14	
1862	D5135	99.930	ex	0.46	see § 4.1
1866	D5135	99.919	ex	-0.48	see § 4.1
9005	D5135	99.917		-0.65	
9008	D5135	99.95		2.16	
normality		not OK			
n		32			
outliers		0			
mean (n)		99.9246			
st.dev. (n)		0.01282			
R(calc.)		0.0359			
R(D5135:07)		0.0330			



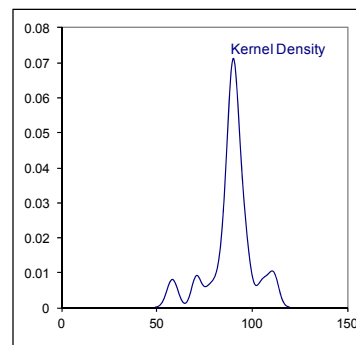
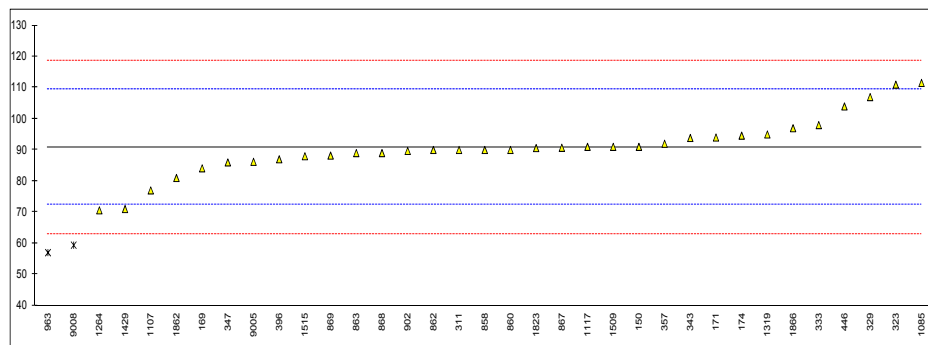
Determination of Benzene on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D5135	17		-0.45	
169	D5135Mod.	15.7		-1.15	
171	D5135	22		2.25	
174	D7504	20.2		1.28	
311	INH-222	21		1.71	
323	INH-189	15		-1.53	
329	INH-189	25		3.88	
333	D5135	16		-0.99	
343	INH-1456	23.3		2.96	
347		----		----	
357	D5135	17		-0.45	
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858	D5135	18		0.09	
860	D5135	19		0.63	
862	D5135	22		2.25	
863	D5135	18		0.09	
867	D5135	19.2		0.74	
868	D5135	18		0.09	
869	D5135	18.3		0.25	
902	INH-83	13.3		-2.45	
913		----		----	
963		----		----	
1067	D5135	14.0		-2.07	
1085	in house	4.94	G(0.05)	-6.97	
1107	in house	15		-1.53	
1117	D5135	19		0.63	
1169		----		----	
1252		----		----	
1264		152.69	C, G(0.01)	72.93	first reported: 187.4
1319		----		----	
1429	D5135Mod.	13		-2.61	
1509	D5135	18		0.09	
1515	in house	18.02		0.10	
1823	in house	18.3		0.25	
1862	in house	22.0		2.25	
1866	D5135	19		0.63	
9005	D4534	13.3		-2.45	
9008		9.5		-4.51	
	normality	OK		<u>spike</u>	
	n	29			
	outliers	2			
	mean (n)	17.83		15.2	recovery: <117%
	st.dev. (n)	3.430			
	R(calc.)	9.60			
	R(Horwitz)	5.18			



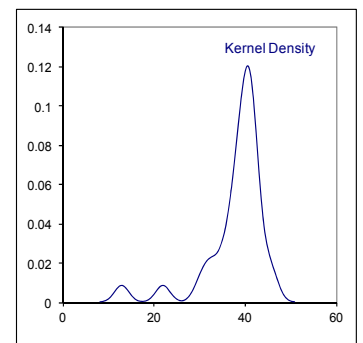
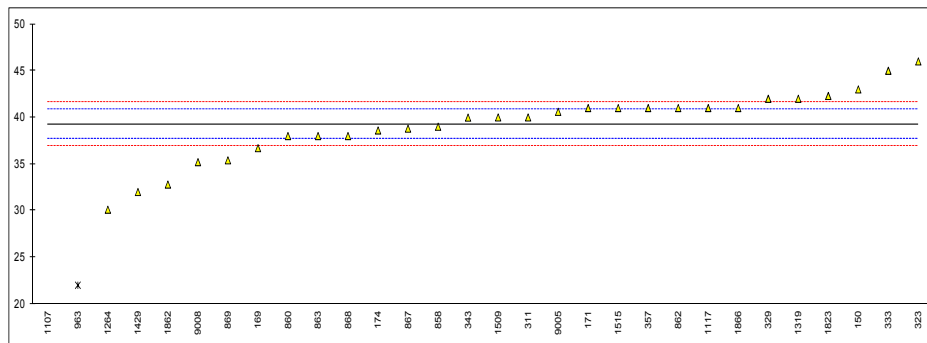
Determination of Ethylbenzene on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D5135	91		0.01	
169	D5135	84.1		-0.73	
171	D5135	94		0.34	
174	D7504	94.6		0.40	
311	D5135	90		-0.10	
323	D5135	111	C	2.17	first reported: 111
329	D5135	107		1.74	
333	D5135	98		0.77	
343	D5135	93.84		0.32	
347	D5135	86		-0.53	
357	D5135	92		0.12	
396	D5135	87		-0.42	
446	D5135	104	C	1.41	first reported: 136
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858	D5135	90		-0.10	
860	D5135	90		-0.10	
862	D5135	90		-0.10	
863	D5135	89		-0.20	
867	D5135	90.7		-0.02	
868	D5135	89		-0.20	
869	D5135	88.2		-0.29	
902	D5135	89.7	C	-0.13	first reported: 69.7
913		----		----	
963	D5135	57	DG(0.05)	-3.65	
1067		----		----	
1085	in house	111.54		2.23	
1107	in house	77		-1.50	
1117	D5135	91		0.01	
1169		----		----	
1252		----		----	
1264	D5135	70.61	C	-2.19	first reported: 64.3
1319	D5135	95		0.44	
1429	D5135	71		-2.14	
1509	D5135	91		0.01	
1515	in house	88		-0.31	
1823	D5135	90.6		-0.03	
1862	D5135	81.0		-1.07	
1866	D5135	97		0.66	
9005	D5135	86.2		-0.50	
9008	D5135	59.4	DG(0.05)	-3.39	
normality		not OK			
n		33			
outliers		2			
mean (n)		90.88			
st.dev. (n)		9.0703			
R(calc.)		25.40			
R(D5135:07)		25.97			



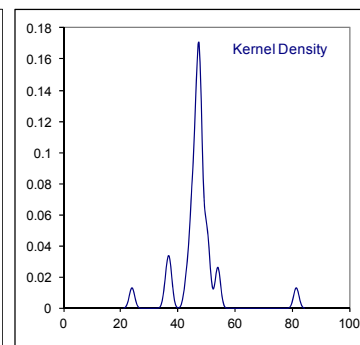
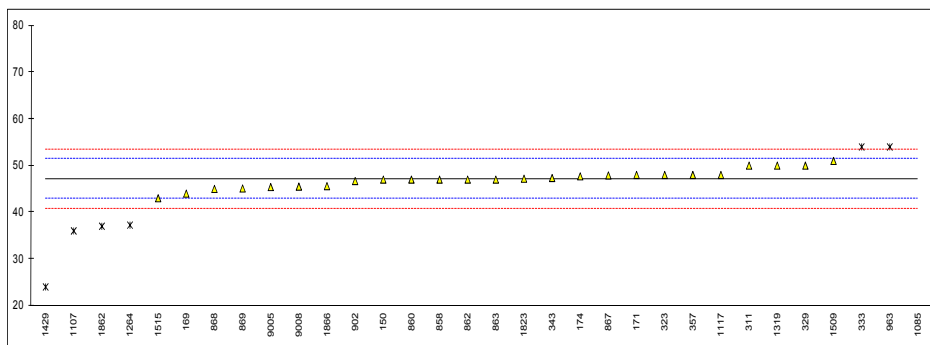
Determination of m- & p-Xylenes on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D5135	43		4.75	
169	D5135	36.7		-3.27	
171	D5135	41		2.21	
174	D7504	38.6		-0.85	
311	D5135	40		0.93	
323	D5135	46		8.57	
329	D5135	42		3.48	
333	D5135	45		7.30	
343	D5135	39.98		0.91	
347		----		----	
357	D5135	41		2.21	
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858	D5135	39		-0.34	
860	D5135	38		-1.61	
862	D5135	41		2.21	
863	D5135	38		-1.61	
867	D5135	38.8		-0.59	
868	D5135	38		-1.61	
869	D5135	35.4		-4.92	
902		----		----	
913		----		----	
963	D5135	22	G(0.01)	-21.99	
1067		----		----	
1085		----		----	
1107	in house	13	G(0.01)	-33.45	
1117	D5135	41		2.21	
1169		----		----	
1252		----		----	
1264	D5135	30.1		-11.67	
1319	D5135	42	C	3.48	first reported: 23
1429	D5135	32		-9.25	
1509	D5135	40		0.93	
1515	in house	41		2.21	
1823	D5135	42.3		3.86	
1862	D5135	32.8		-8.23	
1866	D5135	41		2.21	
9005	D5135	40.6	C	1.70	first reported: 22.0
9008	D5135	35.2		-5.18	
normality		OK			
n		28			
outliers		2			
mean (n)		39.27			
st.dev. (n)		3.651			
R(calc.)		10.22			
R(D5135:07)		2.20			
				Compare R(Horwitz) = 14.79	



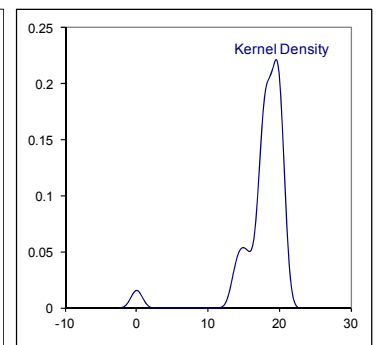
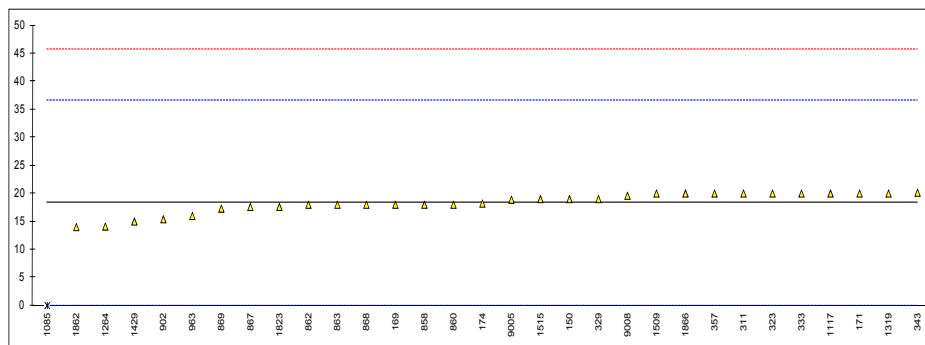
Determination of Cumene on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D5135	47		-0.07	
169	D5135	44.0		-1.49	
171	D5135	48		0.41	
174	D7504	47.7		0.27	
311	D5135	50		1.36	
323	D5135	48		0.41	
329	D5135	50		1.36	
333	D5135	54	DG(0.05)	3.26	
343	D5135	47.34		0.10	
347		----		----	
357	D5135	48		0.41	
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858	D5135	47		-0.07	
860	D5135	47		-0.07	
862	D5135	47		-0.07	
863	D5135	47		-0.07	
867	D5135	47.9		0.36	
868	D5135	45		-1.02	
869	D5135	45.1		-0.97	
902	D5135	46.7		-0.21	
913		----		----	
963	D5135	54	DG(0.05)	3.26	
1067		----		----	
1085	in house	81.42	G(0.01)	16.29	
1107	in house	36	DG(0.05)	-5.29	
1117	D5135	48		0.41	
1169		----		----	
1252		----		----	
1264	D5135	37.24	C,G(0.05)	-4.70	first reported: 33.7
1319	D5135	50		1.36	
1429	D5135	24	G(0.01)	-11.00	
1509	D5135	51		1.84	
1515	in house	43		-1.97	
1823	D5135	47.2		0.03	
1862	D5135	37.0	DG(0.05)	-4.82	
1866	D5135	45.6		-0.73	
9005	D5135	45.4		-0.83	
9008	D5135	45.5		-0.78	
normality		OK			
n		25			
outliers		7			
mean (n)		47.14			
st.dev. (n)		1.909			
R(calc.)		5.34			
R(D5135:07)		5.89			



Determination of o-Xylene on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D5135	19		0.08	
169	D5135	18.0		-0.03	
171	D5135	20		0.19	
174	D7504	18.2		-0.01	
311	D5135	20		0.19	
323	D5135	20		0.19	
329	D5135	19		0.08	
333	D5135	20		0.19	
343	D5135	20.14		0.20	
347		----		----	
357	D5135	20		0.19	
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858	D5135	18		-0.03	
860	D5135	18		-0.03	
862	D5135	18		-0.03	
863	D5135	18		-0.03	
867	D5135	17.6		-0.08	
868	D5135	18		-0.03	
869	D5135	17.3		-0.11	
902	D5135	15.4		-0.32	
913		----		----	
963	D5135	16		-0.25	
1067		----		----	
1085	in house	0.00	ex	-2.00	result excluded, zero is not a real value
1107		----		----	
1117	D5135	20		0.19	
1169		----		----	
1252		----		----	
1264	D5135	14.1		-0.46	
1319	D5135	20		0.19	
1429	D5135	15		-0.36	
1509	D5135	20		0.19	
1515	in house	19		0.08	
1823	D5135	17.6		-0.08	
1862	D5135	14.0		-0.47	
1866	D5135	20		0.19	
9005	D5135	18.9		0.07	
9008	D5135	19.6		0.14	
normality		not OK			
n		30			
outliers		0			
mean (n)		18.29			
st.dev. (n)		1.821			
R(calc.)		5.10			
R(D5135:07)		25.61			
				Compare R(Horwitz) = 5.29	



Determination of n-Propylbenzene on sample #12100; results in mg/kg

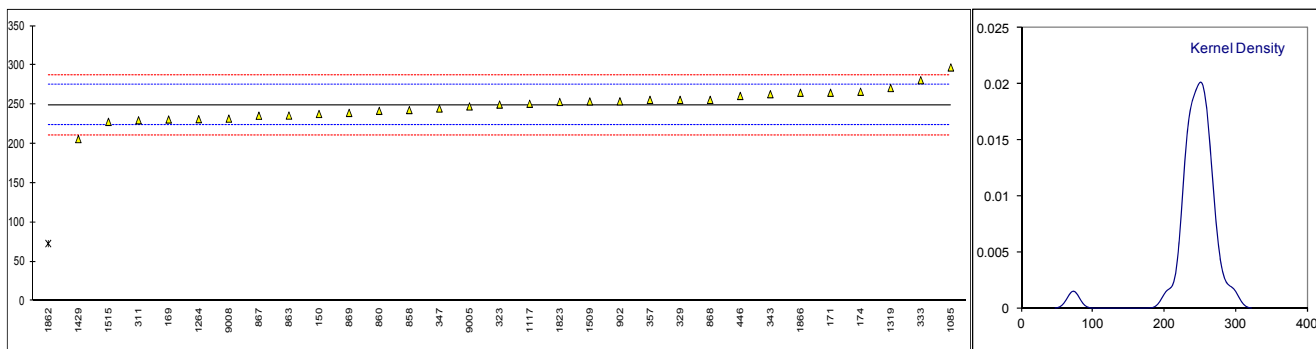
lab	method	value	mark	z(targ)	Remarks
150	D5135	<10		----	
169	D5135	<0.1		----	
171	D5135	<1		----	
174	D7504	<10		----	
311	D5135	<10		----	
323	D5135	<10		----	
329	D5135	<10		----	
333	D5135	<10		----	
343	D5135	0.99		----	
347		----		----	
357	D5135	<10		----	
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858	D5135	<10		----	
860	D5135	<10		----	
862	D5135	1.0		----	
863	D5135	<10		----	
867	D5135	<10		----	
868	D5135	<10		----	
869	D5135	<10		----	
902		----		----	
913		----		----	
963		----		----	
1067		----		----	
1085	in house	7.68		----	
1107		----		----	
1117	D5135	<5		----	
1169		----		----	
1252		----		----	
1264		----		----	
1319	D5135	0	ex	----	result excluded, zero is not a real value
1429	D5135	<5		----	
1509	D5135	<5		----	
1515	in house	<6		----	
1823	D5135	<10		----	
1862	D5135	<1		----	
1866	D5135	n.d.		----	
9005	D5135	<1		----	
9008	D5135	1.0		----	
	normality	n.a			
	n	27			
	outliers	0			
	mean (n)	<10			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(D5135:07)	n.a			Application range: 10 – 10000 mg/kg

Determination of m- & p-Ethyltoluenes on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D5135	<10		----	
169		----		----	
171	D5135	<1		----	
174		----		----	
311	D5135	<10		----	
323	D5135	<10		----	
329	D5135	<10		----	
333	D5135	<10		----	
343	D5135	2.32		----	
347		----		----	
357	D5135	<10		----	
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858		----		----	
860	D5135	<10		----	
862	D5135	1.3		----	
863	D5135	<10		----	
867	D5135	<10		----	
868	D5135	<10		----	
869	D5135	<10		----	
902		----		----	
913		----		----	
963		----		----	
1067		----		----	
1085	in house	5.25		----	
1107		----		----	
1117	D5135	<5		----	
1169		----		----	
1252		----		----	
1264		----		----	
1319	D5135	0	ex	----	result excluded, zero is not a real value
1429	D5135	<5		----	
1509	D5135	<5		----	
1515	in house	<6		----	
1823	D5135	<10		----	
1862	D5135	<1		----	
1866	D5135	36.5		----	false positive?
9005	D5135	<1		----	
9008	D5135	0.6		----	
	normality	n.a			
	n	24			
	outliers	0			
	mean (n)	<10			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(D5135:07)	n.a			Application range: 10 – 10000 mg/kg

Determination of alpha-Methylstyrene on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D5135	238		-0.90	
169	D5135	231.0		-1.45	
171	D5135	265		1.22	
174	D7504	266.3		1.32	
311	D5135	230		-1.53	
323	D5135	250		0.04	
329	D5135	256		0.51	
333	D5135	281		2.47	
343	D5135	263.2		1.08	
347	D5135	245		-0.35	
357	D5135	256		0.51	
396		----		----	
446	D5135	261		0.90	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858	D5135	243		-0.51	
860	D5135	242		-0.59	
862		----		----	
863	D5135	236		-1.06	
867	D5135	235.8		-1.08	
868	D5135	256		0.51	
869	D5135	239.4		-0.79	
902	D5135	254.1		0.36	
913		----		----	
963		----		----	
1067		----		----	
1085	in house	297.26		3.75	
1107		----		----	
1117	D5135	251		0.12	
1169		----		----	
1252		----		----	
1264	D5135	231.37	C	-1.43	first reported: 185.0
1319	D5135	271		1.69	
1429	D5135	206		-3.42	
1509	D5135	254		0.35	
1515	in house	228		-1.69	
1823	D5135	253.3		0.30	
1862	D5135	73.0	G(0.01)	-13.87	
1866	D5135	265		1.22	
9005	D5135	247.6		-0.15	
9008	D5135	232.1		-1.37	
normality		OK			
n		30			
outliers		1			
mean (n)		249.51			
st.dev. (n)		18.040			
R(calc.)		50.51			
R(D5135:07)		35.64			



Determination of 1,2-diethylbenzene on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150		----		----	
169		----		----	
171	D5135	<1		----	
174		----		----	
311	D5135	<10		----	
323		----		----	
329		----		----	
333		----		----	
343		----		----	
347		----		----	
357		----		----	
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858		----		----	
860		----		----	
862		----		----	
863		----		----	
867		----		----	
868		----		----	
869		----		----	
902		----		----	
913		----		----	
963		----		----	
1067		----		----	
1085		----		----	
1107		----		----	
1117	D5135	<5		----	
1169		----		----	
1252		----		----	
1264		----		----	
1319		----		----	
1429		----		----	
1509	in house	<5		----	
1515	in house	<7		----	
1823	in house	<10		----	
1862		----		----	
1866	D5135	138.9		----	false positive?
9005		----		----	
9008		----		----	
	normality	n.a			
	n	6			
	outliers	n.a			
	mean (n)	<10			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(Horwitz)	n.a			

Determination of Phenylacetylene on sample #12100; results in mg/kg

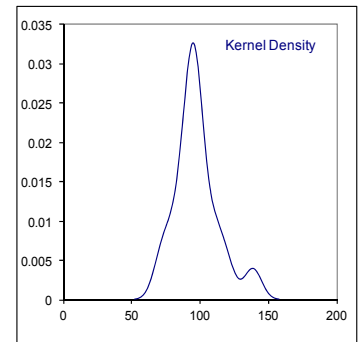
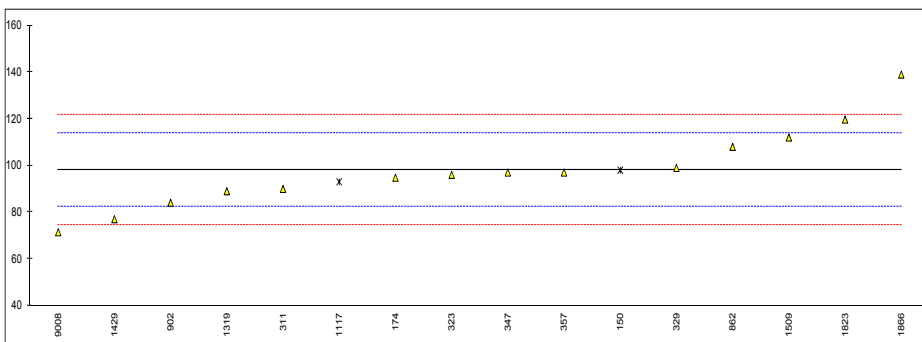
lab	method	value	mark	z(targ)	Remarks
150	D5135	<10		----	
169		----		----	
171	D5135	<1		----	
174	D7504	<10		----	
311	D5135	<10		----	
323	D5135	<10		----	
329	D5135	<10		----	
333	D5135	<10		----	
343	D5135	3.31		----	
347		----		----	
357	D5135	<10		----	
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858		----		----	
860	D5135	<10		----	
862	D5135	3.6		----	
863	D5135	<10		----	
867	D5135	<10		----	
868		----		----	
869	D5135	<10		----	
902		----		----	
913		----		----	
963		----		----	
1067		----		----	
1085		----		----	
1107	in house	20	G(0.01)	----	false positive?
1117	D5135	6		----	
1169		----		----	
1252		----		----	
1264	D5135	1.1		----	
1319	D5135	4		----	
1429	D5135	<5		----	
1509	in house	<5		----	
1515	in house	4		----	
1823	in house	<10		----	
1862	in house	229.5	G(0.01)	----	false positive?
1866	D5135	<10		----	
9005	D5135	<1		----	
9008		4.5		----	
	normality	OK			
	n	23			
	outliers	2			
	mean (n)	<10			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(Horwitz)	(1.39)			

Determination of 3- & 4-methylstyrenes on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150		----		----	
169		----		----	
171		----		----	
174		----		----	
311	D5135	<10		----	
323		----		----	
329		----		----	
333	D5135	<10		----	
343	D5135	3.17		----	
347		----		----	
357	D5135	<10		----	
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858		----		----	
860		----		----	
862		----		----	
863		----		----	
867		----		----	
868		----		----	
869		----		----	
902		----		----	
913		----		----	
963		----		----	
1067		----		----	
1085		----		----	
1107		----		----	
1117	D5135	<5		----	
1169		----		----	
1252		----		----	
1264		----		----	
1319		----		----	
1429	D5135	<5		----	
1509	in house	<5		----	
1515		----		----	
1823	in house	<10		----	
1862		----		----	
1866	D5135	<10		----	
9005	D5135	8.5		----	
9008		0.9		----	
	normality	n.a			
	n	11			
	outliers	n.a			
	mean (n)	<10			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(Horwitz)	n.a			

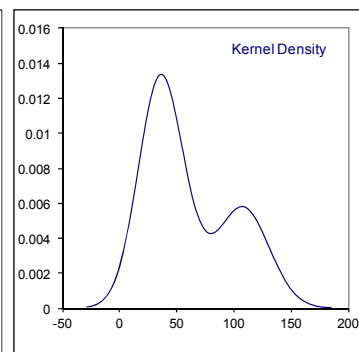
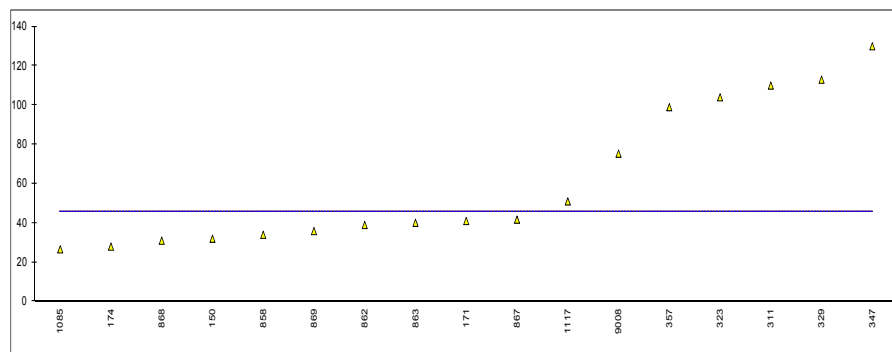
Determination of Benzaldehyde on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D5135	98	ex	-0.02	see § 4.1
169		----		----	
171	D5135	<10	C	<-10.45	first reported:<1, false negative?
174	D7504	94.8		-0.42	
311	D5135	90		-1.03	
323	D5135	96		-0.27	
329	D5135	99		0.11	
333		----		----	
343		----		----	
347	D5135	97		-0.15	
357	D5135	97		-0.15	
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858		----		----	
860		----		----	
862	D5135	108		1.25	
863		----		----	
867		----		----	
868		----		----	
869		----		----	
902	INH-83	84.1		-1.78	
913		----		----	
963		----		----	
1067		----		----	
1085		----		----	
1107		----		----	
1117	D5135	93	ex	-0.65	see § 4.1
1169		----		----	
1252		----		----	
1264		----		----	
1319	D5135	89		-1.16	
1429	D5135	77		-2.69	
1509	D5135	112		1.76	
1515		----		----	
1823	D5135	119.7		2.74	
1862		----		----	
1866	D5135	139		5.19	
9005		----		----	
9008		71.4		-3.40	
normality		OK			
n		14			
outliers		0			
mean (n)		98.14			
st.dev. (n)		17.435			
R(calc.)		48.82			
R(Horwitz)		22.05			



Determination of Nonaromatics on sample #12100; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
150	D5135	32		----	
169		----		----	
171	D5135	41	C	----	first reported:249
174	D7504	28		----	
311	D5135	110		----	
323	D5135	104		----	
329	D5135	113		----	
333		----		----	
343		----		----	
347	D5135	130		----	
357	D5135	99		----	
396		----		----	
446		----		----	
529		----		----	
551		----		----	
557		----		----	
613		----		----	
663		----		----	
858	D5135	34		----	
860		----		----	
862	D5135	39		----	
863	D5135	40.1		----	
867	D5135	41.7		----	
868	D5135	31		----	
869	D5135	35.9		----	
902		----		----	
913		----		----	
963		----		----	
1067		----		----	
1085	in house	26.60		----	
1107		----		----	
1117	D5135	51		----	
1169		----		----	
1252		----		----	
1264		----		----	
1319		----		----	
1429	D5135	<10		----	False negative?
1509		----		----	
1515	in house	<10		----	False negative?
1823		----		----	
1862		----		----	
1866	D5135	<10		----	False negative?
9005		----		----	
9008		75.3		----	
	normality	not OK			
	n	17			
	outliers	0			
	mean (n)	60.682			
	st.dev. (n)	35.8185			
	R(calc.)	100.292			
	R(Horwitz)	(46.341)			



APPENDIX 2

Number of participants per country

1 lab in AUSTRALIA
3 labs in BELGIUM
2 labs in BRAZIL
1 lab in CANADA
1 lab in FINLAND
1 lab in FRANCE
1 lab in INDIA
1 lab in ITALY
1 lab in JAPAN
2 labs in KUWAIT
1 lab in MEXICO
8 labs in P.R. of CHINA
1 lab in RUSSIA
4 labs in SAUDI ARABIA
1 lab in SINGAPORE
2 labs in SPAIN
1 lab in THAILAND
4 labs in THE NETHERLANDS
1 lab in TURKEY
5 labs in U.S.A.
2 labs in UNITED KINGDOM

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect result
C(0.01)	= outlier in Cochran's outlier test
C(0.05)	= straggler in Cochran's outlier test
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
E	= error in calculations
U	= reported wrong unit
W	= result withdrawn on request of participant
ex	= excluded from calculations
n.a.	= not applicable
n.d.	= not detected
n.e.	= not evaluated
Fr.	= first reported

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
- 2 ASTM E178-02
- 3 ASTM E1301-03
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- 5 ISO 5725-86
- 6 ISO 5725, parts 1-6, 1994
- 7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367/84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 Analytical Methods Committee Technical brief, No4 January 2001.
- 14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page 1359-1364, P.J. Lothian and M. Thompson (see <http://www.rsc.org/suppdata/an/b2/b205600n/>).