

Results of Proficiency Test
Styrene
September 2011

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 2010/2011, it was decided to organize again a round robin for the analysis of Styrene. In this interlaboratory study, 33 laboratories from 19 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 sample regular Styrene (1*500 mL, labelled #11070). 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 guide 43 and ILAC-G13:2007, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures 100% confidentiality of participant's data. 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 present in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

The necessary bulk material was obtained from a local Styrene producer. The approx. 50 litre bulk sample was homogenised in a precleaned drum and divided over 56 amber glass bottles of 500 mL (labelled #111070).

The homogeneity of the subsamples #111070 was checked by determination of Density @ 20°C in accordance with ASTM D4052:02e1 and inhibitor in accordance with ASTM D4590:00 on 8 stratified random selected samples.

	<i>Density @20°C in kg/L</i>	<i>Inhibitor in mg/kg</i>
sample #11070-1	0.90627	13
sample #11070-2	0.90626	12
sample #11070-3	0.90627	13
sample #11070-4	0.90627	12
sample #11070-5	0.90626	13
sample #11070-6	0.90628	13
sample #11070-7	0.90628	13
sample #11070-8	0.90626	13

Table 1: homogeneity tests of subsamples #11070

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 @20°C in kg/L</i>	<i>Inhibitor in mg/kg</i>
r (sample #11070)	0.00015	1.0
Ref. method	ASTM D4052:02	ASTM D4590:00
0.3 x R (ref. method)	0.00015	0.8

Table 2: repeatabilities of subsamples #11070

Each calculated repeatability was near or equal to 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories, 1 bottle of 500 mL (labelled #11070) was sent on September 7, 2011.

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 #11070: 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 and the standard methods were printed, was sent together with each set of samples. Also a letter of instructions and a SDS were added to the 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 to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers were marked by D(0.01)

for the Dixon test and by $G(0)$. 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.

Finally, the reproducibilities were calculated from the standard deviations by multiplying these with a factor of 2.8.

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.

01) or $DG(0.01)$ for the Grubbs test. Stragglers were 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.

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 4; nr.13 and 14).

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This result was an evaluation independent of the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated in accordance with:

The $z_{(target)}$ scores are listed in the result tables in appendix 1.

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.

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. The laboratories in Brazil, India and Saudi Arabia did not receive the samples in time due to problems with custom clearance. Three participants reported results after the final reporting date.

Finally, 31 laboratories did report 403 numerical results. Observed were 15 outlying results, which is 3.7%. 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: density, n-propylbenzene, organic chloride, o-xylene, sulphur, In these cases the statistical evaluation should be used with due care.

Aldehydes as benzaldehyde: This determination was 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.

Appearance: A new standardized method is available for Appearance since 2009, being ASTM E2680. According this method the appearance should be reported as 'pass' (or 'fail'). All participants, except two reported following the ASTM E2680. All participants agreed about the appearance of sample #11070. The uniformity of reporting can be improved. Fifteen participants reported the appearance correctly as pass. The other laboratories used different kind of abbreviations like C&B, CFFSM, CFMSM and C&F. 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 good agreement with the requirements of ASTM D1209:05e1.

Inhibitor: The determination of pTBC may be problematic. No statistical outliers were observed. However, the observed reproducibility, after rejection of the statistical outlier, is not in agreement with the requirements of ASTM D4590:09.

Density: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers, is in good agreement with the requirements of ASTM D4052:02e1.

- Peroxides: This determination was very problematic. The results vary over large range (3.4–41 mg/kg). No statistical outliers were observed. However, the observed reproducibility, is not at all in agreement with the requirements of ASTM D2340:09.
- Polymers: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D5135:07. The application range of the method is 1-15 mg/kg. Twelve participants reported a value below the limit of the method. Therefore the conclusion can be drawn that the actual lower limit may be below one.
- Org. chloride: Due to the low organic chloride concentration no significant conclusions were drawn. All participants agreed that less than 1 mg/kg of organic chlorides was present.
- Sulphur: No significant conclusions were drawn as the sulphur concentration was below the application range of the test method D5453:09. Not any laboratory reported a test result for sulphur above 1 mg/kg.
- Water: This determination was problematic. One statistical outlier was observed. However, the observed reproducibility, after rejection of the statistical outlier, is not in agreement with the requirement of ASTM E1064:05.
- Purity: No analytical problems were observed. Three statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers, is in good agreement with the requirements of ASTM D5135:07.
- Benzene: Due to the low benzene concentration no significant conclusions were drawn. All participants agreed that less than 10 mg/kg of benzene was present.
- Ethylbenzene: No analytical problems have been observed. Also, no statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D5135:07.
- m- & p-Xylenes: 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 requirements of ASTM D5135:07. It must be noted that the observed reproducibility is almost 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. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D5135:07. The large spread may partly be explained by unobserved impurities in the calibration standard used. Running a blank as mentioned in paragraph 12.3 of ASTM D5137:07.
- o-Xylene: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D5135:07. Five laboratories reported a "less than" result.
- n-Prop.benzene: This determination was problematic. Only one statistical outlier was detected. However the calculated reproducibility, after rejection of the statistical outlier, is not in good agreement with the requirements of ASTM D5135:07.
- m- & p-Ethyltol.: No significant conclusions were drawn as the m- & p- Ethyltoluenes concentration was below the application range of the test method D5135:07.
- α -Methylstyrene: The determination of this component was problematic. Three statistical outliers were observed and the calculated reproducibility, after rejection of the statistical outliers, 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.: Only six laboratories reported a test result for this component. Two results were 'less than' the detection limit, while the other four results were clearly positive. Therefore no significant conclusions were drawn.
- Phenylacetylene: This determination was very problematic. One statistical outlier was observed and two false negative test results were reported. The observed reproducibility is not at all in agreement with the requirements estimated from the Horwitz equation. 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.
- 3- & 4 -Me-Styrenes: Only six laboratories reported a test result for these components. Two results were less than the detection limit, while the other four results were clearly positive. Therefore no significant conclusions were drawn.
- Benzaldehyde: This determination was problematic. Two statistical outliers were observed and one false negative result was reported. The observed

reproducibility is not in agreement with the requirement estimated from the Horwitz equation.

Nonaromatics: This determination was very problematic. Three laboratories reported a false negative result. No statistical outliers were observed. However, the observed reproducibility is not at all in agreement with the requirement estimated from the Horwitz equation. 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	22	102.6	96.9	41.0
Appearance		28	pass	n.a.	n.a.
Colour		27	7.7	6.3	7.0
Inhibitor (p-TBC)	mg/kg	27	10.4	3.0	2.4
Density @20°C	kg/L	24	0.9063	0.0002	0.0005
Peroxides as H ₂ O ₂	mg/kg	24	26.3	26.0	13.0
Polymers	mg/kg	16	0.60	0.99	1.00
Organic Chlorides	mg/kg	6	0.20	0.47	(0.12)
Sulphur	mg/kg	8	0.13	0.23	(0.12)
Water	mg/kg	22	129.9	31.6	20.6
Purity	%M/M	25	99.946	0.028	0.033
Benzene	mg/kg	20	< 10	n.a	n.a
Ethylbenzene	mg/kg	26	71.0	12.8	20.3
m- & p-Xylenes	mg/kg	21	16.0	7.7	0.9
Cumene	mg/kg	21	12.8	3.5	1.6
o-Xylene	mg/kg	15	9.5	2.9	13.3
n-Propylbenzene	mg/kg	15	10.4	3.0	2.6
m- & p-Ethyltoluenes	mg/kg	18	<10	n.a	n.a
α-Methylstyrene	mg/kg	21	196.2	37.5	28.0
1,2-diethylbenzene	mg/kg	4	17.7	n.a	n.a
Phenylacetylene	mg/kg	16	24.2	22.9	6.7
3- & 4-Methylstyrenes	mg/kg	4	26.2	n.a	n.a
Benzaldehyde	mg/kg	14	88.7	28.1	20.2
Nonaromatics	mg/kg	12	34.9	54.5	13.0

Table 3: reproducibilities of sample #11070

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 OCTOBER 2011 WITH PREVIOUS PTS

	<i>October 2011</i>	<i>October 2010</i>	<i>October 2009</i>	<i>October 2008</i>
Number of reporting labs	31	35	33	33
Number of results reported	403	496	613	708
Statistical outliers	15	30	44	37
Percentage outliers	3.7%	6.1%	7.2%	5.2%

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	<i>October 2011</i>	<i>October 2010</i>	<i>October 2009</i>		<i>October 2008</i>	
Aldehydes	--	--	--	--	--	
Colour	++	++	++		++	
Inhibitor	-	-	+/-		+/-	
Density	++	++	+		++	
Peroxides as H ₂ O ₂	--	-	-		+	
Polymer	+/-	n.e.	--		--	
Organic chloride	n.e.	n.e.	n.e.		n.e.	
Sulphur	n.e.	n.e.	n.e.		n.e.	
Water	--	--	-		--	
Purity	+	++	++	++	++	++
Benzene	n.e.	n.e.	n.e.	+	n.e.	--
Ethylbenzene	++	++	++	+	n.e.	++
m+p-Xylenes	--	n.e.	n.e.	--	--	--
Cumene	--	-	n.e.	--	-	+
o-Xylene	++	n.e.	n.e.	n.e.	++	++
n-Propylbenzene	-	++	n.e.	n.e.	+	+
m+p-Ethyltoluenes	n.e.	--	n.e.	n.e.	--	--
α-Methylstyrene	--	-	-	--	-	+
1,2-diethylbenzene	n.e.	--	n.e.	n.e.	n.e.	n.e.
Phenylacetylene	--	--	n.e.	n.e.	+	+
3-&4-methylstyrenes	n.e.	--	n.e.	n.e.	n.e.	n.e.
Benzaldehyde	--	--	--	--	--	--
Nonaromatics	--	n.e.	n.e.	n.e.	n.e.	n.e.

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

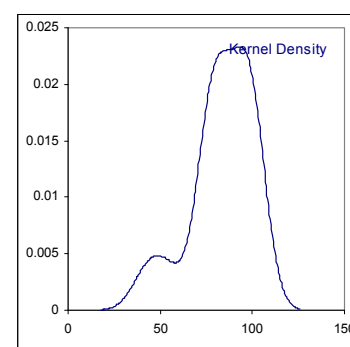
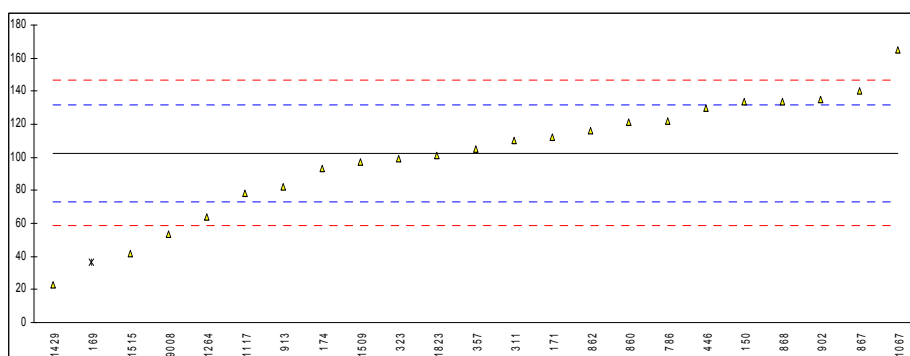
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: 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 #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D2119	134		2.14	
169	D2119	36.2	U	-4.53	different unit? reported:0.00362
171	D2119	112		0.64	
174	D2119	93	C	-0.65	first reported:30
311	D2119	110		0.51	
323	D2119	99		-0.24	
333		----		----	
342		----		----	
343		----		----	
347		----		----	
357	D2119	105		0.17	
396		----		----	
446	D2119	130	C	1.87	first reported:0.013
551		----		----	
557		----		----	
613		----		----	
786	D2119	122		1.33	
860	D2119	121		1.26	
862	D2119	116		0.92	
867	D2119	140		2.55	
868	D2119	134		2.14	
902	D2119	134.8		2.20	
913	D2119	81.9		-1.41	
1067	D2119	165		4.26	
1085		----		----	
1117	D2119	78.2		-1.66	
1264	D2119	64	C	-2.63	first reported:47
1429	D2119	23		-5.43	
1509	D2119	97		-0.38	
1515	D2119	42		-4.13	
1823	D2119	101		-0.11	
1866		----		----	
9008	D2119	53.8		-3.33	
normality		OK			
n		22			
outliers		0			
mean (n)		102.58			
st.dev. (n)		34.599			
R(calc.)		96.88			
R(D2119:09)		41.03			



Determination of Appearance on sample #11070;

lab	method	value	mark	z(targ)	remarks
150	E2680	pass		----	
169	E2680	pass		----	
171	E2680	C&F		----	
174	E2680	pass		----	
311	E2680	C&F		----	
323	E2680	pass		----	
333	E2680	C&B		----	
342	E2680	pass		----	
343		----		----	
347	E2680	pass		----	
357	E2680	pass		----	
396	E2680	pass		----	
446	D4176	pass		----	
551		----		----	
557		----		----	
613	E2680	C&B		----	
786	E2680	pass		----	
860	E2680	pass		----	
862	E2680	C&B		----	
867	E2680	C&B		----	
868	E2680	pass		----	
902	E2680	pass		----	
913	E2680	CFSM		----	
1067	E2680	pass		----	
1085		----		----	
1117	D4176	pass		----	
1264	E2680	clear		----	
1429	VISUAL	C&B		----	
1509	E2680	CFSM		----	
1515	E2680	C&B		----	
1823	E2680	CFFSM		----	
1866		----		----	
9008	E2680	clear		----	
	n	28			

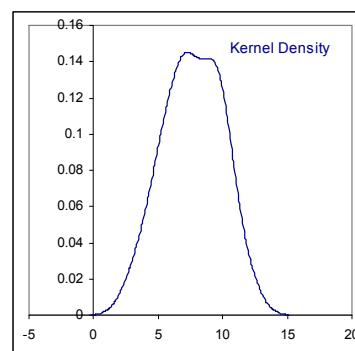
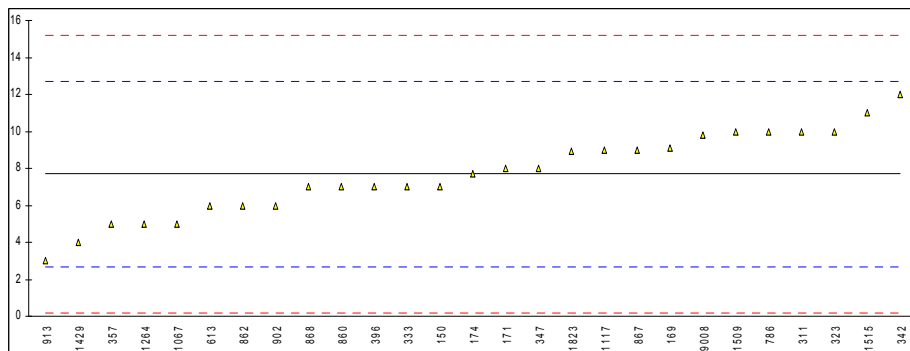
Abbreviations:

C&B: clear and bright
CFFSM: clear and free from matter in suspension
CFSM: clear and free from suspended matter
C&F: clear and free

Determination of Colour Pt/Co on sample #11070;

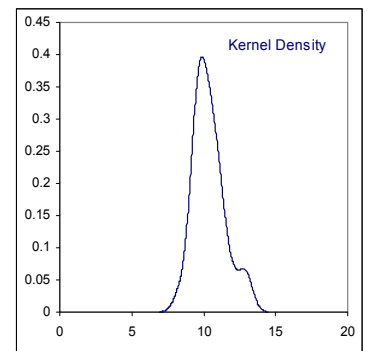
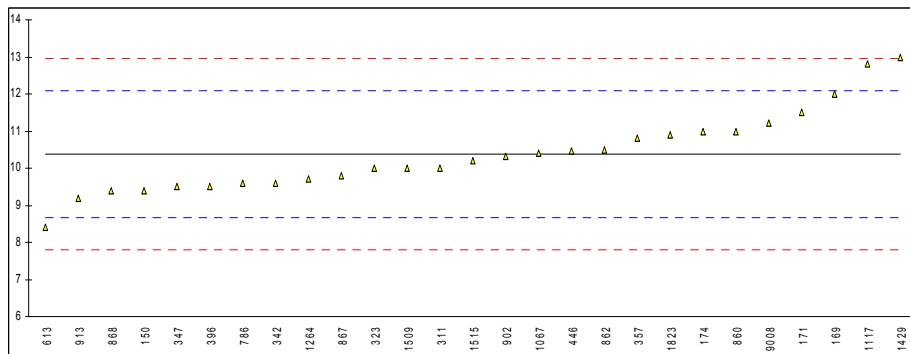
lab	method	value	mark	z(targ)	remarks
150	D1209	7		-0.27	
169	D5386	9.1		0.57	
171	D1209	8		0.13	
174	D1209	7.7		0.01	
311	D1209	10		0.93	
323	D1209	10		0.93	
333	D1209	7		-0.27	
342	D5386	12		1.73	
343		----		----	
347	D1209	8		0.13	
357	D1209	5		-1.07	
396	D1209	7		-0.27	
446	D1209	<10		----	
551		----		----	
557		----		----	
613	D1209	6		-0.67	
786	D1209	10		0.93	
860	D1209	7		-0.27	
862	D1209	6		-0.67	
867	D1209	9		0.53	
868	D1209	7		-0.27	
902	D5386	6		-0.67	
913	D5386	3.0		-1.87	
1067	D1209	5		-1.07	
1085		----		----	
1117	D1209	9		0.53	
1264	D1209	5		-1.07	
1429	D6045	4		-1.47	
1509	D1209	10		0.93	
1515	D1209	11		1.33	
1823	D5386	8.9		0.49	
1866		----		----	
9008	D1209	9.8		0.85	

normality OK
n 27
outliers 0
mean (n) 7.68
st.dev. (n) 2.242
R(calc.) 6.28
R(D1209:05e) 7.00



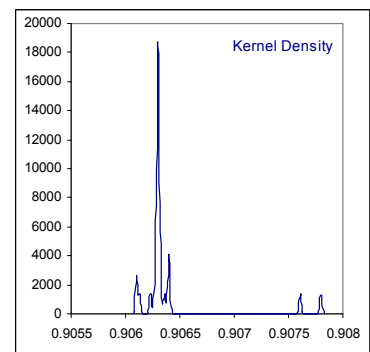
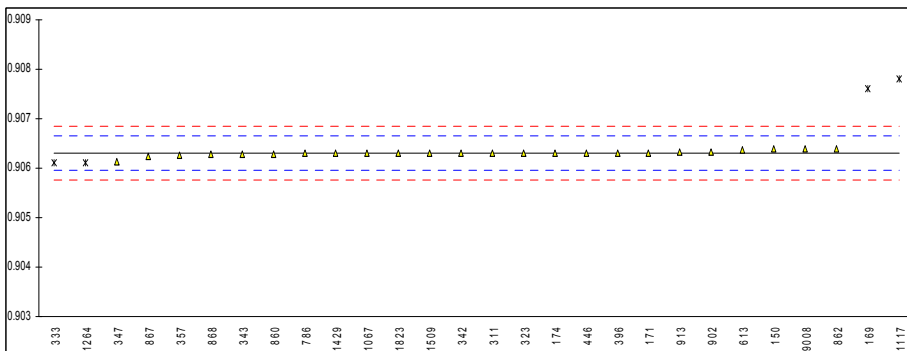
Determination of Inhibitor (pTBC) on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D4590	9.4		-1.14	
169	D4590	11.99		1.88	
171	D4590	11.5		1.31	
174	D4590	11		0.73	
311	D4590	10		-0.44	
323	D4590	10		-0.44	
333		----		----	
342	D4590	9.6		-0.91	
343		----		----	
347	D4590	9.5		-1.02	
357	D4590	10.8		0.49	
396	D4590	9.5		-1.02	
446	D4590	10.46		0.10	
551		----		----	
557		----		----	
613	D4590	8.4		-2.31	
786	D4590	9.6		-0.91	
860	D4590	11		0.73	
862	D4590	10.5		0.14	
867	D4590	9.8		-0.67	
868	D4590	9.4		-1.14	
902	D4590	10.32		-0.07	
913	D4590	9.2		-1.37	
1067	D4590	10.4		0.03	
1085		----		----	
1117	D4590	12.8		2.82	
1264	D4590	9.7		-0.79	
1429	D4590	13.0	C	3.06	First reported:13.6
1509	D4590	10.0		-0.44	
1515	D4590	10.2		-0.21	
1823	D4590	10.9		0.61	
1866		----		----	
9008	D4590	11.22		0.98	
normality		OK			
n		27			
outliers		0			
mean (n)		10.377			
st.dev. (n)		1.0702			
R(calc.)		3.000			
R(D4590:09)		2.401			



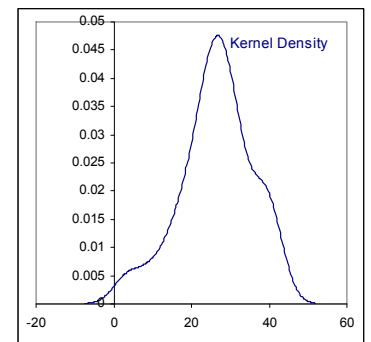
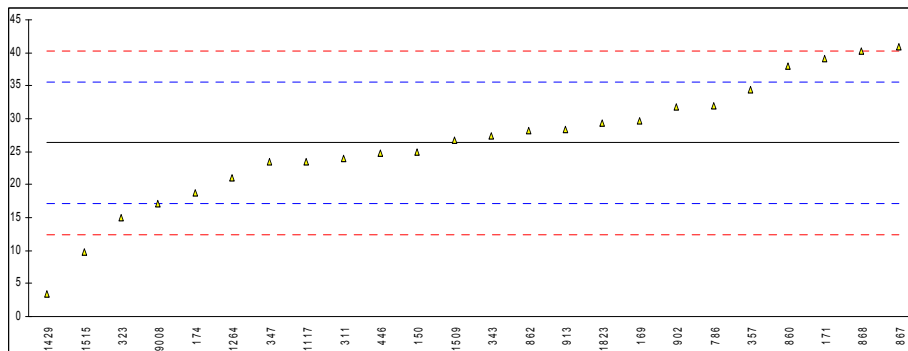
Determination of Density at 20°C on sample #11070; results in kg/L

lab	method	value	mark	z(targ)	remarks
150	D4052	0.9064		0.54	
169	D4052	0.90761	G(0.01)	7.31	
171	D4052	0.9063		-0.02	
174	D4052	0.9063		-0.02	
311	D4052	0.9063		-0.02	
323	D4052	0.9063		-0.02	
333	D4052	0.9061	U, C	-1.14	Different unit? reported:906.1
342	D4052	0.9063		-0.02	
343	D4052	0.90629		-0.08	
347	D4052	0.90613		-0.97	
357	D4052	0.90627		-0.19	
396	D4052	0.9063		-0.02	
446	D4052	0.9063		-0.02	
551		----		----	
557		----		----	
613	D4052	0.90636		0.31	
786	D4052	0.9063		-0.02	
860	D4052	0.90629		-0.08	
862	D4052	0.90640		0.54	
867	D4052	0.90623		-0.41	
868	D4052	0.90628		-0.13	
902	D4052	0.90632		0.09	
913	D4052	0.90632		0.09	
1067	D4052	0.9063		-0.02	
1085		----		----	
1117	D4052	0.9078	G(0.01)	8.38	
1264	D4052	0.9061	G(0.05)	-1.14	
1429	D4052	0.9063		-0.02	
1509	D4052	0.9063		-0.02	
1515		----		----	
1823	D4052	0.9063		-0.02	
1866		----		----	
9008	D4052	0.9064	C	0.54	first reported:0.0964
normality		not OK			
n		24			
outliers		3			
mean (n)		0.9063			
st.dev. (n)		0.00055			
R(calc.)		0.0002			
R(D4052:02e1)		0.0005			



Determination of Peroxides as H₂O₂ on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D2340	25		-0.29	
169	D2340	29.73		0.73	
171	D2340	39.2		2.77	
174	D2340	18.8		-1.62	
311	D2340	24		-0.50	
323	D2340	15		-2.44	
333		----		----	
342		----		----	
343	D2340	27.4		0.23	
347	D2340	23.4		-0.63	
357	D2340	34.4		1.74	
396		----		----	
446	D2340	24.8		-0.33	
551		----		----	
557		----		----	
613		----		----	
786	D2340	31.9		1.20	
860	D2340	38		2.51	
862	D2340	28.2		0.40	
867	D2340	41	C	3.16	first reported:55.4
868	D2340	40.2	C	2.99	first reported:52.7
902	D2340	31.8		1.18	
913	D2340	28.4		0.44	
1067		----		----	
1085		----		----	
1117	D2340	23.5		-0.61	
1264	D2340	21	C	-1.15	first reported:7.9
1429	D2340	3.4		-4.94	
1509	D2340	26.7		0.08	
1515	D2340	9.80		-3.56	
1823	D2340	29.3		0.64	
1866		----		----	
9008	D2340	17.1		-1.99	
normality		OK			
n		24			
outliers		0			
mean (n)		26.33			
st.dev. (n)		9.299			
R(calc.)		26.04			
R(D2340:09)		13.00			

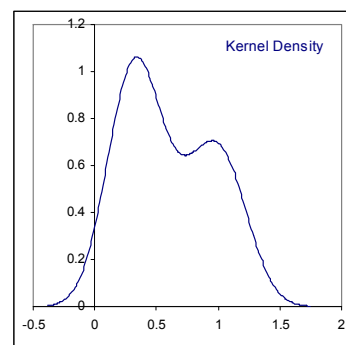
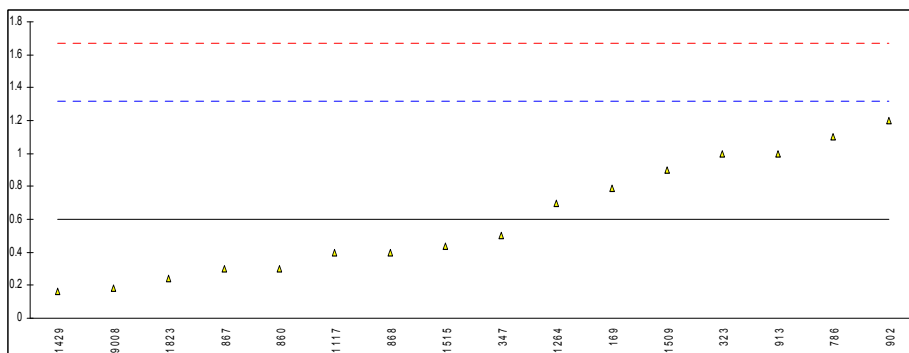


Determination of Polymers on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D2121	<1		----	
169	D2121	0.7893		0.53	
171	D2121	<1		----	
174	D2121	<1		----	
311	D2121	<1		----	
323	D2121	1		1.12	
333	D2121	<1		----	
342	D2121	<1		----	
343		----		----	
347	D2121	0.5		-0.28	
357	D2121	<1		----	
396	D2121	<1		----	
446	D2121	<1		----	
551		----		----	
557		----		----	
613		----		----	
786	D2121	1.1		1.40	
860	D2121	0.3		-0.84	
862	D2121	<1		----	
867	D2121	0.3		-0.84	
868	D2121	0.4	C	-0.56	first reported 1.4
902	D2121	1.2		1.68	
913	D2121	1.0		1.12	
1067		----		----	
1085		----		----	
1117	D2121	0.4		-0.56	
1264	D2121	0.7		0.28	
1429	D2121	0.16		-1.23	
1509	D2121	0.9		0.84	
1515	D2121	0.44		-0.45	
1823	D2121	0.24		-1.01	
1866		----		----	
9008	D2121	0.18		-1.18	

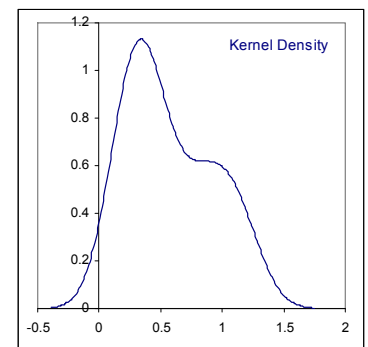
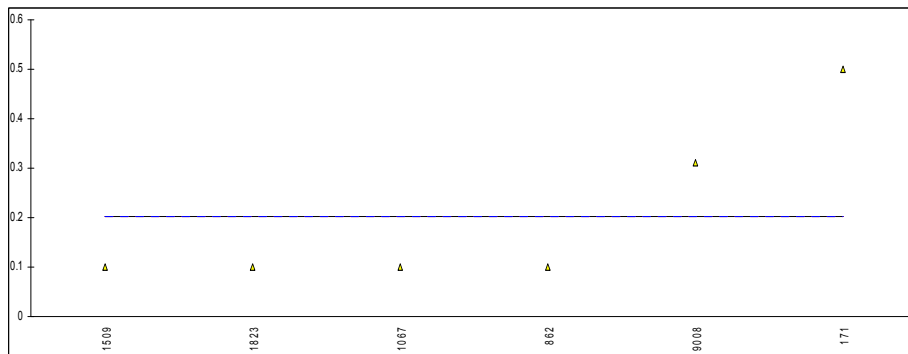
normality OK
n 16
outliers 0
mean (n) 0.600
st.dev. (n) 0.3522
R(calc.) 0.986
R(D2121:07) 1.000

Application range 1-15 mg/kg



Determination of Organic Chloride on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D7359	<1		----	
169		----		----	
171	D5808	0.5		----	
174		----		----	
311	D5808	<1		----	
323	UOP779	<1		----	
333		----		----	
342		----		----	
343		----		----	
347		----		----	
357	D5808	<1		----	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613		----		----	
786		----		----	
860		----		----	
862	D5808	0.1		----	
867		----		----	
868	D5808	<1		----	
902		----		----	
913		----		----	
1067	UOP779	0.1		----	
1085		----		----	
1117		----		----	
1264		----		----	
1429	D5808	<1		----	
1509	D5808	0.1		----	
1515		----		----	
1823	INH-2296	0.10		----	
1866		----		----	
9008		0.31		----	
	normality	not OK			
	n	6			
	outliers	n.a			
	mean (n)	0.202			
	st.dev. (n)	0.1686			
	R(calc.)	0.472			
	R(Horwitz)	(0.115)			

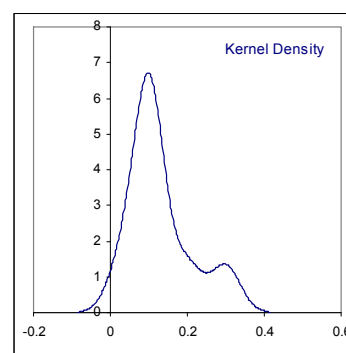
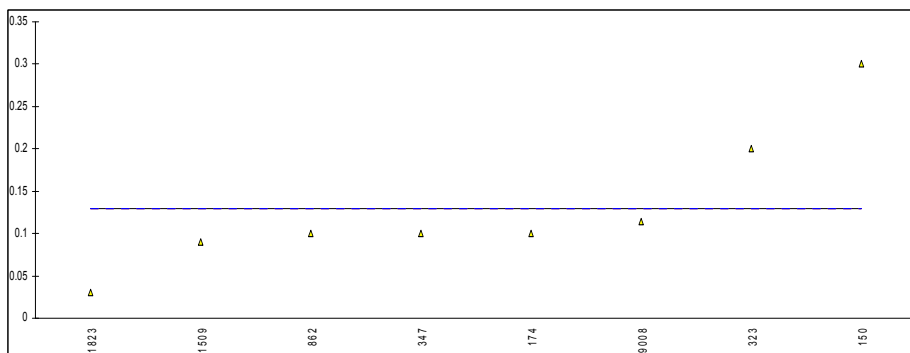


Determination of Sulphur on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5453	0.3		----	
169				----	
171	D5453	<1		----	
174	D5453	0.1		----	
311	D5453	<1		----	
323	D5453	0.2		----	
333	D5453	<1		----	
342				----	
343				----	
347	D5453	0.10		----	
357	D5453	<0.5		----	
396				----	
446				----	
551				----	
557				----	
613				----	
786				----	
860				----	
862	D5453	0.1		----	
867				----	
868	D3120	<1		----	
902				----	
913				----	
1067	D5453	<0.1		----	
1085				----	
1117	D5453	<0.1		----	
1264				----	
1429	D5453	<1		----	
1509	D5453	0.09		----	
1515				----	
1823	D5453	0.03		----	
1866				----	
9008	D5453	0.114		----	

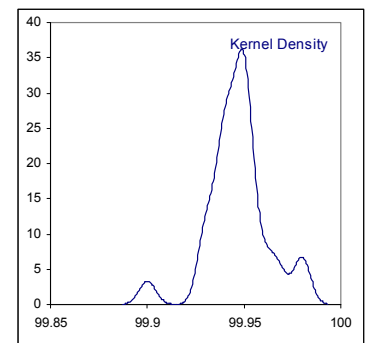
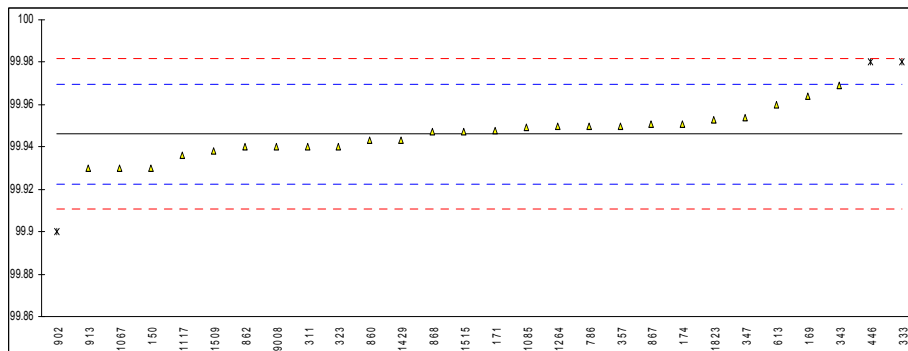
normality not OK
n 8
outliers n.a
mean (n) 0.129
st.dev. (n) 0.0831
R(calc.) 0.233
R(D5453:09) (0.125)

application range 1 – 8000 mg/kg



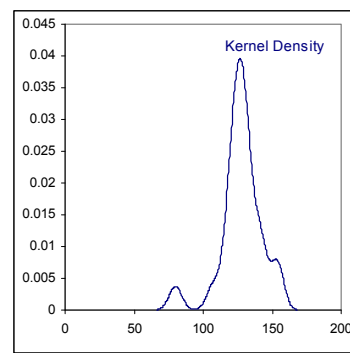
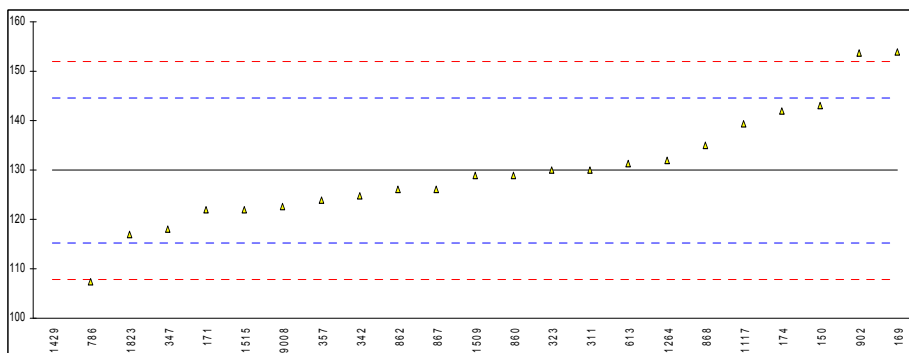
Determination of Purity on sample #11070; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	D5135	99.93		-1.37	
169	D5135	99.964		1.51	
171	D5135	99.948		0.16	
174	D5135	99.951		0.41	
311	D5135	99.94		-0.52	
323	D5135	99.94		-0.52	
333	D5135	99.98	DG(0.05)	2.87	
342		----		----	
343	D5135	99.969		1.94	
347	D5135	99.954		0.67	
357	D5135	99.95		0.33	
396		----		----	
446	D5135	99.98	DG(0.05)	2.87	
551		----		----	
557		----		----	
613	D5135	99.96		1.18	
786	D5135	99.95		0.33	
860	D5135	99.943		-0.27	
862	D5135	99.94		-0.52	
867	D5135	99.951		0.41	
868	D5135	99.947		0.07	
902	D5135	99.90	G(0.05)	-3.92	
913	D5135	99.93		-1.37	
1067	GC	99.93		-1.37	
1085	in house	99.9494	C	0.28	first reported:99.4945
1117	D5135	99.936		-0.86	
1264	D5135	99.95		0.33	
1429	D5135	99.943		-0.27	
1509	D5135	99.938		-0.69	
1515	In House	99.9470		0.07	
1823	D5135	99.953		0.58	
1866		----		----	
9008	D5135	99.94		-0.52	
	normality	OK			
	n	25			
	outliers	3			
	mean (n)	99.9462			
	st.dev. (n)	0.00994			
	R(calc.)	0.0278			
	R(D5135:07)	0.0330			



Determination of Water on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	E1064	143	C	1.78	first reported:149
169	E1064	154		3.27	
171	E203	122		-1.07	
174	E1064	142		1.64	
311	E1064	130		0.01	
323	E1064	130		0.01	
333		----		----	
342	E1064	124.7		-0.71	
343		----		----	
347	E1064	118		-1.61	
357	E1064	124		-0.80	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613	E1064	131.2		0.18	
786	E1064	107.3		-3.06	
860	E1064	129		-0.12	
862	E1064	126		-0.53	
867	E1064	126		-0.53	
868	E1064	135		0.69	
902	E1064	153.6		3.21	
913		----		----	
1067		----		----	
1085		----		----	
1117	D4672	139.4		1.29	
1264	E1064	132	C	0.28	first reported:249
1429	D1364	80	G(0.01)	-6.77	
1509	E1064	129		-0.12	
1515	E1064	122		-1.07	
1823	E1064	117		-1.75	
1866		----		----	
9008	E1064	122.7		-0.98	
normality		OK			
n		22			
outliers		1			
mean (n)		129.90			
st.dev. (n)		11.280			
R(calc.)		31.59			
R(E1064:05)		20.65			

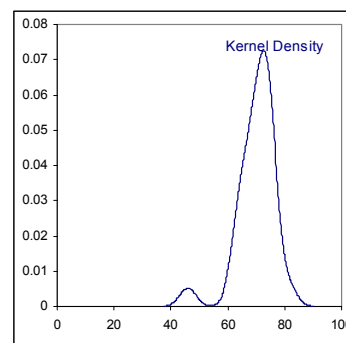
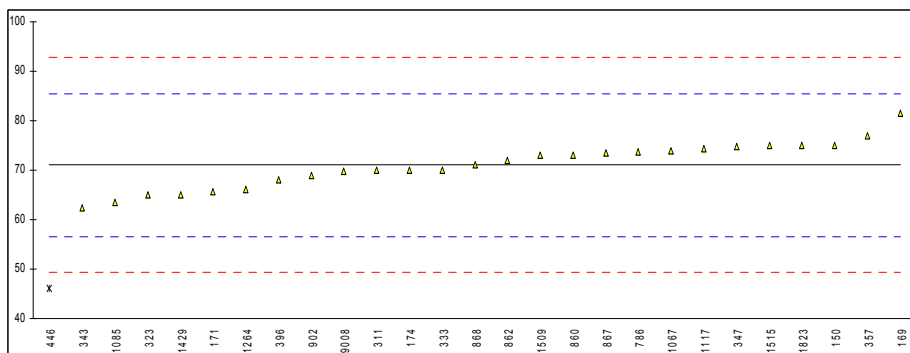


Determination of Benzene on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	<1		----	
169	D5135	<1		----	
171	D5135	<1		----	
174	D5135	<1		----	
311	INH-222	<1		----	
323	INH-189	<1		----	
333		----		----	
342		----		----	
343	In House	<1		----	
347		----		----	
357	D5135	<1		----	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613		----		----	
786	INH-1/2011	<1		----	
860	D5135	<10		----	
862		----		----	
867	D5135	<10		----	
868	D5135	<10		----	
902	INH-83	<1.0		----	
913		----		----	
1067		----		----	
1085	in house	0.0		----	
1117	In house	0.2		----	
1264	D5135	<1		----	
1429		<10		----	
1509	in house	<0.5		----	
1515	In house	<0.1		----	
1823	INH-2296	<0.5		----	
1866		----		----	
9008	D5135	0		----	
	normality	n.a			
	n	20			
	outliers	n.a			
	mean (n)	<10			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(Horwitz)	n.a			

Determination of Ethylbenzene on sample #11070; results in mg/kg

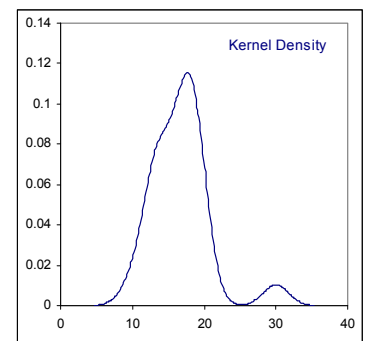
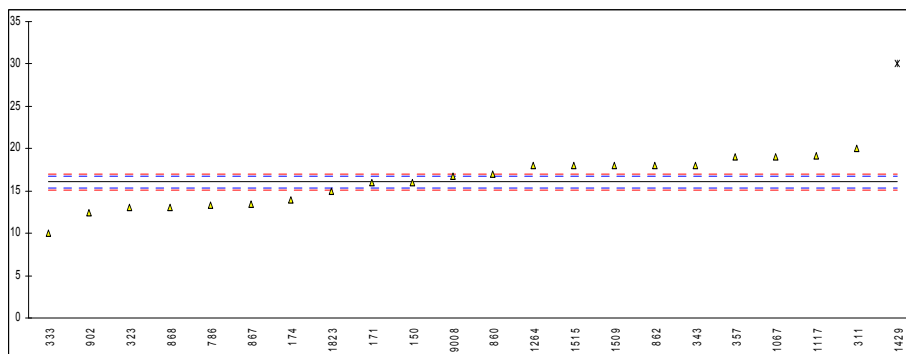
lab	method	value	mark	z(targ)	remarks
150	D5135	75		0.55	
169	D5135	81.5		1.44	
171	D5135	65.6		-0.75	
174	D5135	70		-0.14	
311	D5135	70		-0.14	
323	D5135	65		-0.83	
333	D5135	70		-0.14	
342		----		----	
343	D5135	62.429	C	-1.19	first reported: 52.472
347	D5135	74.7		0.51	
357	D5135	77		0.82	
396	D5135	68		-0.42	
446	D5135	46	U	-3.45	Different unit? reported:0.0046
551		----		----	
557		----		----	
613		----		----	
786	D5135	73.7		0.37	
860	D5135	73		0.27	
862	D5135	72		0.13	
867	D5135	73.4		0.33	
868	D5135	71		0.00	
902	INH-83	68.84		-0.30	
913		----		----	
1067	GC	74		0.41	
1085	in house	63.56		-1.03	
1117	D5135	74.3		0.45	
1264	D5135	66		-0.69	
1429	D5135	65		-0.83	
1509	D5135	73		0.27	
1515	in house	75		0.55	
1823	D5135	75		0.55	
1866		----		----	
9008	D5135	69.7		-0.18	
normality	OK				
n	26				
outliers	0				
mean (n)	71.03				
st.dev. (n)	4.575				
R(calc.)	12.81				
R(D5135:07)	20.29				



Determination of m- & p-Xylenes on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	16		-0.14	
169		----		----	
171	D5135	16.0		-0.14	
174	D5135	14	C	-6.37	first reported:10
311	D5135	20		12.33	
323	D5135	13		-9.49	
333	D5135	10		-18.84	
342		----		----	
343	D5135	18.00		6.10	
347		----		----	
357	D5135	19		9.21	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613		----		----	
786	D5135	13.3		-8.55	
860	D5135	17		2.98	
862	D5135	18		6.10	
867	D5135	13.4		-8.24	
868	D5135	13		-9.49	
902	INH-83	12.42		-11.29	
913		----		----	
1067	GC	19		9.21	
1085		----		----	
1117	D5135	19.1		9.52	
1264	D5135	18		6.10	
1429	D5135	30	G(0.01)	43.49	first reported: 21
1509	D5135	18		6.10	
1515	In House	18		6.10	
1823	D5135	15	C	-3.25	
1866		----		----	
9008	D5135	16.7		2.04	
normality		OK			
n		21			
outliers		1			
mean (n)		16.04			
st.dev. (n)		2.747			
R(calc.)		7.69			
R(D5135:07)		0.90			

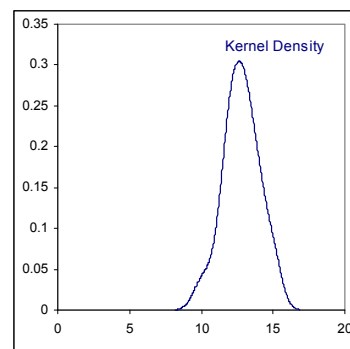
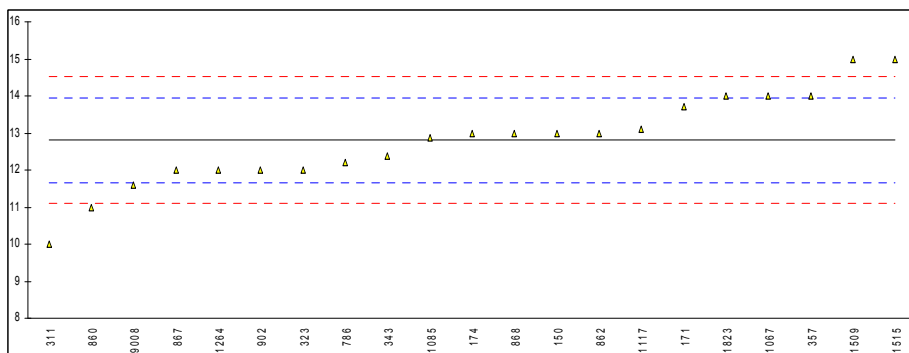
application range 10 -10000 mg/kg; Compare Horwitz: 6.69



Determination of Cumene on sample #11070; results in mg/kg

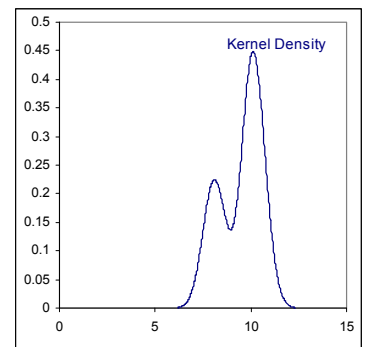
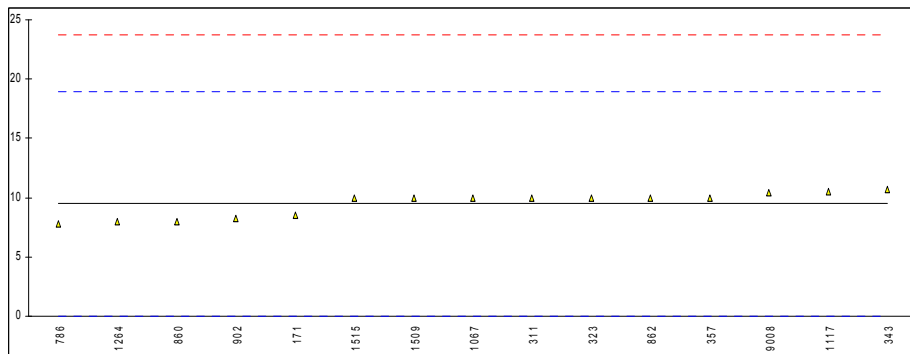
lab	method	value	mark	z(targ)	remarks
150	D5135	13		0.35	
169		----		----	
171	D5135	13.7		1.57	
174	D5135	13	C	0.35	first reported:<10
311	D5135	10	C	-4.90	first reported:20
323	D5135	12		-1.40	
333	D5135	<10		----	
342		----		----	
343	D5135	12.377		-0.74	
347		----		----	
357	D5135	14		2.10	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613		----		----	
786	D5135	12.2		-1.05	
860	D5135	11		-3.15	
862	D5135	13		0.35	
867	D5135	12	C	-1.40	first reported:<10
868	D5135	13	C	0.35	first reported:<10
902	INH-83	12		-1.40	
913		----		----	
1067	GC	14		2.10	
1085	in house	12.87		0.12	
1117	D5135	13.1		0.52	
1264	D5135	12		-1.40	
1429	D5135	<10		----	
1509	D5135	15		3.85	
1515	in house	15		3.85	
1823	D5135	14		2.10	
1866		----		----	
9008	D5135	11.6		-2.10	

normality OK
n 21
outliers 0
mean (n) 12.80
st.dev. (n) 1.239
R(calc.) 3.47
R(D5135:07) 1.60



Determination of o-Xylene on sample #11070; results in mg/kg

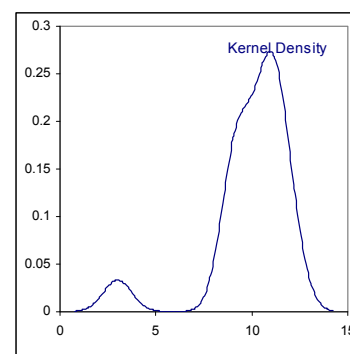
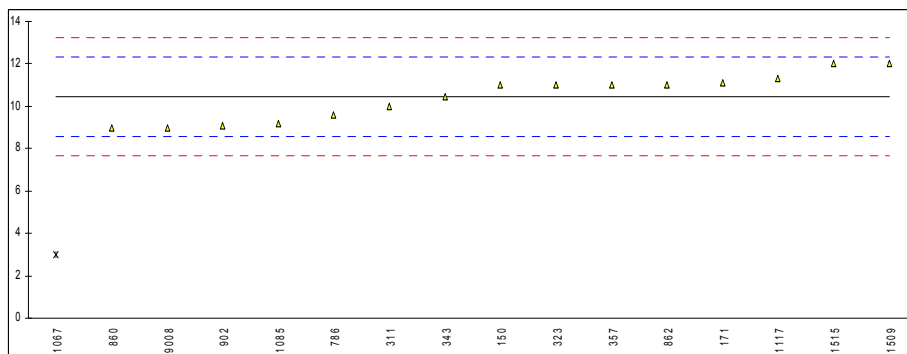
lab	method	value	mark	z(targ)	remarks
150	D5135	<10		----	
169		----		----	
171	D5135	8.5		-0.21	
174	D5135	<10		----	
311	D5135	10		0.11	
323	D5135	10		0.11	
333		----		----	
342		----		----	
343	D5135	10.67		0.25	
347		----		----	
357	D5135	10		0.11	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613		----		----	
786	D5135	7.8		-0.35	
860	D5135	8		-0.31	
862	D5135	10		0.11	
867	D5135	<10		----	
868	D5135	<10		----	
902	INH-83	8.26		-0.26	
913		----		----	
1067	GC	10		0.11	
1085		----		----	
1117	D5135	10.5		0.22	
1264	D5135	8		-0.31	
1429	D5135	<10		----	
1509	D5135	10		0.11	
1515	In House	10		0.11	
1823	D5135	<10		----	
1866		----		----	
9008	D5135	10.4		0.20	
normality		not OK			
n		15			
outliers		0			
mean (n)		9.47			
st.dev. (n)		1.030			
R(calc.)		2.88			
R(D5135:07)		13.27			



Determination of n-Propylbenzene on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	11		0.59	
169		----		----	
171	D5135	11.1		0.70	
174	D5135	<10		----	
311	D5135	10	C	-0.48	fisrt reported:20
323	D5135	11		0.59	
333		----		----	
342		----		----	
343	D5135	10.435		-0.02	
347		----		----	
357	D5135	11		0.59	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613		----		----	
786	D5135	9.6		-0.91	
860	D5135	9		-1.55	
862	D5135	11		0.59	
867	D5135	<10		----	
868	D5135	<10		----	
902	INH-83	9.1		-1.45	
913		----		----	
1067	GC	3	G(0.01)	-7.98	
1085	in house	9.20		-1.34	
1117	D5135	11.3		0.91	
1264		----		----	
1429	D5135	<10		----	
1509	D5135	12		1.66	
1515	in house	12		1.66	
1823	D5135	<10		----	
1866		----		----	
9008	D5135	9.0		-1.55	

normality not OK
n 15
outliers 1
mean (n) 10.45
st.dev. (n) 1.058
R(calc.) 2.96
R(D5135:07) 2.61

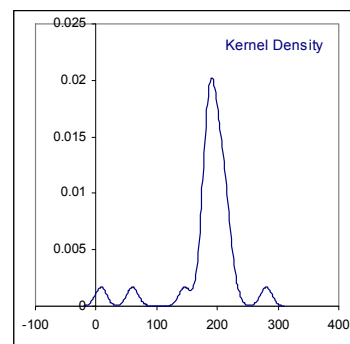
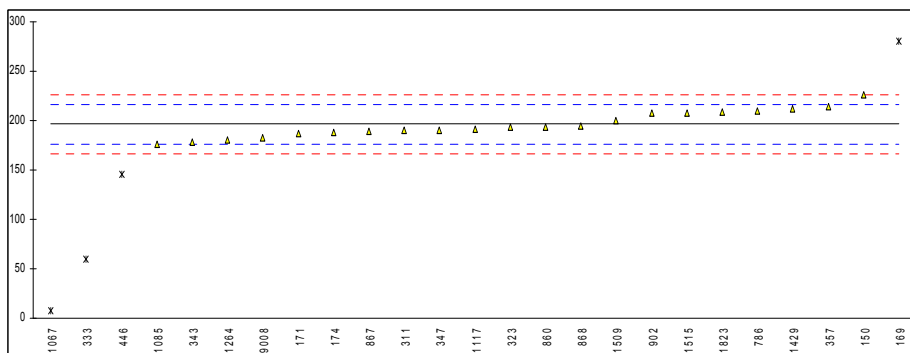


Determination of m- & p-Ethyltoluenes on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	<10		----	
169		----		----	
171	D5135	<1		----	
174	D5135	<10		----	
311	D5135	<10		----	
323	D5135	<10		----	
333		----		----	
342		----		----	
343	D5135	<10		----	
347		----		----	
357	D5135	<10		----	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613		----		----	
786	D5135	<10		----	
860	D5135	<10		----	
862	D5135	3.5		----	
867	D5135	<10		----	
868	D5135	<10		----	
902		----		----	
913		----		----	
1067		----		----	
1085		----		----	
1117	D5135	4.9		----	
1264		----		----	
1429	D5135	<10		----	
1509	D5135	<5		----	
1515	In House	<5		----	
1823	D5135	<10		----	
1866		----		----	
9008	D5135	1.2		----	
	normality	n.a			
	n	18			
	outliers	n.a			
	mean (n)	<10			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(D5135:07)	n.a			

Determination of alpha-Methylstyrene on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	226		2.98	
169	D5135	280.7	G(0.01)	8.44	
171	D5135	186.7		-0.95	
174	D5135	188		-0.82	
311	D5135	190		-0.62	
323	D5135	193		-0.32	
333	D5135	60	G(0.01)	-13.61	
342		----		----	
343	D5135	178.183	C	-1.80	first reported:154.367
347	D5135	190		-0.62	
357	D5135	214		1.78	
396		----		----	
446	D5135	146	U	-5.01	different unit? reported:0.0146
551		----		----	
557		----		----	
613		----		----	
786	D5135	210.2		1.40	
860	D5135	194		-0.22	
862		----		----	
867	D5135	189.1		-0.71	
868	D5135	195		-0.12	
902	INH-83	207.16		1.09	
913		----		----	
1067	GC	8	G(0.01)	-18.80	
1085	In House	175.97		-2.02	
1117	D5135	191.1		-0.51	
1264	D5135	180		-1.62	
1429	D5135	212		1.58	
1509	D5135	200		0.38	
1515	In House	208		1.18	
1823	D5135	209		1.28	
1866		----		----	
9008	D5135	182.8		-1.34	
normality		OK			
n		21			
outliers		3			
mean (n)		196.20			
st.dev. (n)		13.400			
R(calc.)		37.52			
R(D5135:07)		28.03			

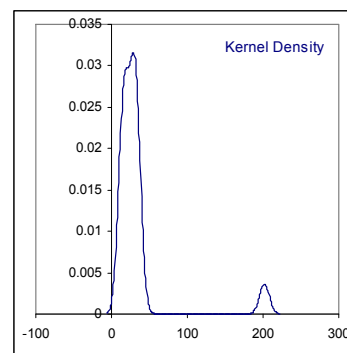
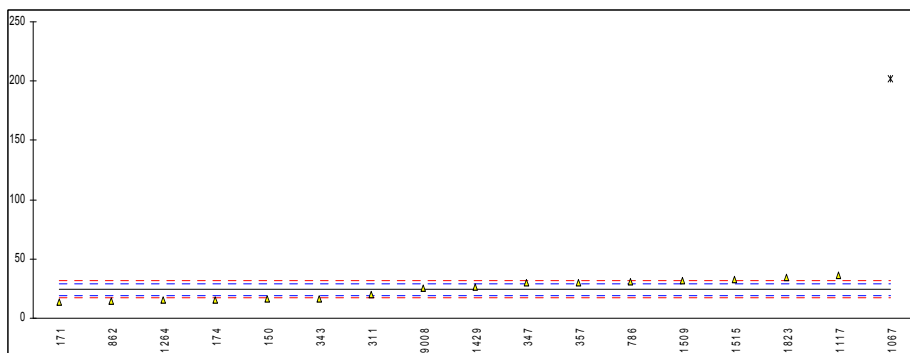


Determination of 1,2-diethylbenzene on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		----		----	
169		----		----	
171	D5135	14.4		----	
174		----		----	
311	INH-342	<10		----	
323		----		----	
333		----		----	
342		----		----	
343		----		----	
347		----		----	
357		----		----	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613		----		----	
786		----		----	
860		----		----	
862		----		----	
867		----		----	
868		----		----	
902		----		----	
913		----		----	
1067		----		----	
1085		----		----	
1117	D5135	14.4		----	
1264		----		----	
1429		----		----	
1509	in house	<5		----	false negative?
1515	in house	21		----	
1823	D5135	21		----	
1866		----		----	
9008		----		----	
	normality	n.a			
	n	4			
	outliers	n.a			
	mean (n)	17.7			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(Horwitz)	n.a			application range 10 -10000 mg/kg

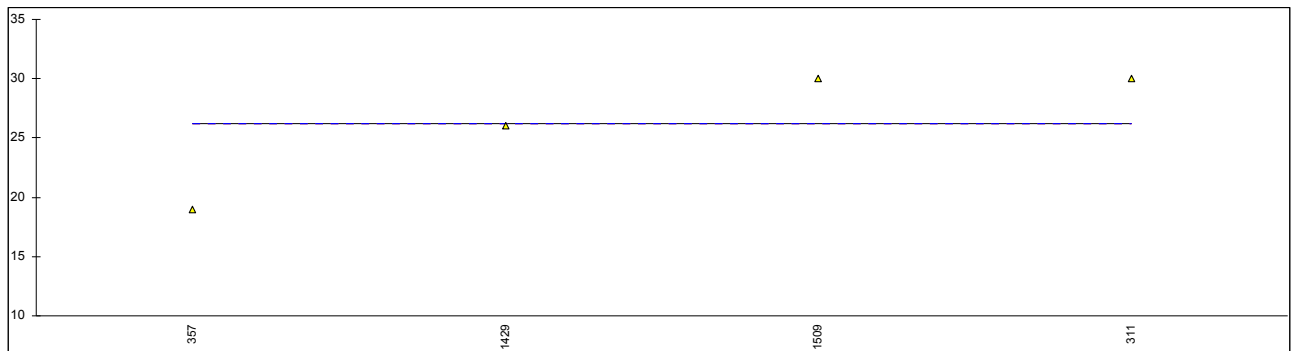
Determination of Phenylacetylene on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	16		-3.43	
169		----		----	
171	D5135	13.6		-4.43	
174	D5135	15		-3.85	
311	D5135	20		-1.77	
323	D5135	<10		< -5.88	false negative?
333		<10		< -5.88	false negative?
342		----		----	
343	D5135	16.418		-3.26	
347	D5135	29.6		2.23	
357	D5135	30		2.40	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613		----		----	
786	D5135	31.2		2.90	
860		----		----	
862	D5135	14.8		-3.93	
867		----		----	
868		----		----	
902		----		----	
913		----		----	
1067	GC	202	G(0.01)	74.04	
1085		----		----	
1117	D5135	35.9		4.85	
1264		15		-3.85	
1429		26		0.73	
1509	in house	32	C	3.23	first reported:7
1515	in house	33		3.65	
1823	D5135	34		4.06	
1866		----		----	
9008	D5135	25.4		0.48	
	normality	OK			
	n	16			
	outliers	1			
	mean (n)	24.24			
	st.dev. (n)	8.178			
	R(calc.)	22.90			
	R(Horwitz)	6.72			



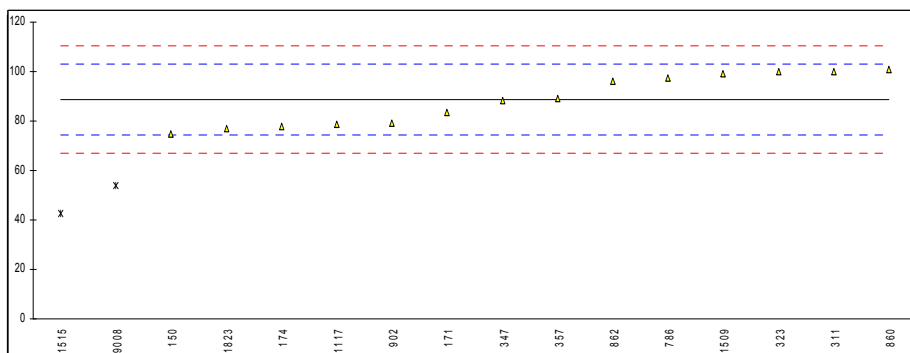
Determination of 3- & 4-methylstyrenes on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		----		----	
169		----		----	
171		----		----	
174		----		----	
311	INH-342	30		----	
323		----		----	
333		----		----	
342		----		----	
343		----		----	
347		----		----	
357	D5135	19		----	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613		----		----	
786	D5135	<10		----	
860		----		----	
862		----		----	
867		----		----	
868		----		----	
902		----		----	
913		----		----	
1067		----		----	
1085		----		----	
1117	D5135	<5		----	
1264		----		----	
1429		26		----	
1509	in house	30		----	
1515		----		----	
1823		----		----	
1866		----		----	
9008		----		----	
	normality	n.a			
	n	4			
	outliers	n.a			
	mean (n)	26.25			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(Horwitz)	n.a			



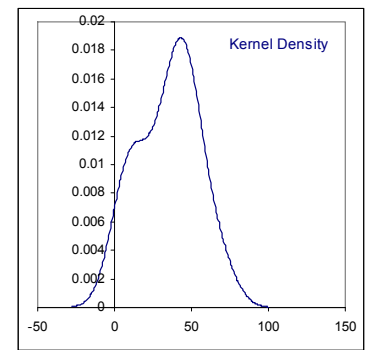
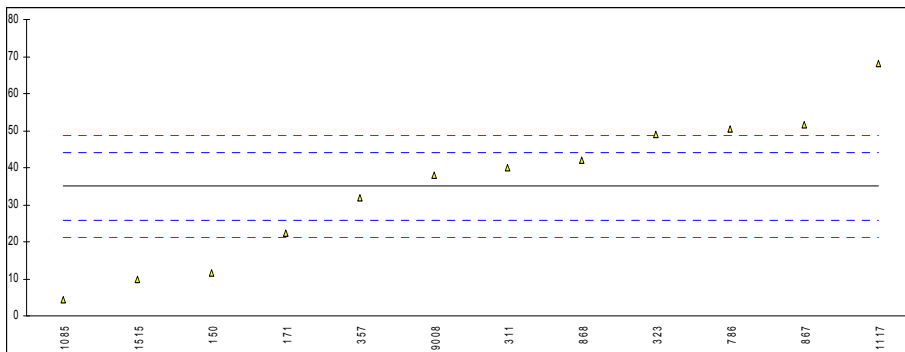
Determination of Benzaldehyde on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	75		-1.89	
169		----		----	
171	D5135	83.3		-0.74	
174	D5135	78	C	-1.48	first reported:14
311	INH-342	100		1.57	
323	D5135	100		1.57	
333		----		----	
342		----		----	
343		----		----	
347	D5135	88.2		-0.06	
357	D5135	89		0.05	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613		----		----	
786	D5135	97.3		1.20	
860	D5135	101		1.71	
862	D5135	96		1.02	
867		----		----	
868		----		----	
902	INH-83	78.94		-1.35	
913		----		----	
1067		----		----	
1085		----		----	
1117	D5135	78.5		-1.41	
1264		----		----	
1429		<10		<-10.16	false negative?
1509	D5135	99		1.43	
1515	in house	42.5	DG(0.05)	-6.39	
1823	D5135	77		-1.61	
1866		----		----	
9008	D5135	53.8	DG(0.05)	-4.83	
normality	OK				
n	14				
outliers	2				
mean (n)	88.66				
st.dev. (n)	10.030				
R(calc.)	28.08				
R(Horwitz)	20.22				



Determination of Nonaromatics on sample #11070; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	11.5		-5.06	
169		----		----	
171	D5135	22.4		-2.71	
174	D5135	<10		< -4.97	false negative?
311	INH-342	40		1.09	
323	D5135	49		3.04	
333		----		----	
342		----		----	
343		----		----	
347		----		----	
357	D5135	32		-0.63	
396		----		----	
446		----		----	
551		----		----	
557		----		----	
613		----		----	
786	D5135	50.4		3.34	
860		----		----	
862		----		----	
867		51.5		3.58	
868	D5135	42		1.52	
902		----		----	
913		----		----	
1067		----		----	
1085	in house	4.27		-6.62	
1117	D5135	68.1		7.16	
1264		----		----	
1429		<10		< -4.97	false negative?
1509		----		----	
1515	in house	10		-5.39	
1823	D5135	<10		< -4.97	false negative?
1866		----		----	
9008	D5135	38.1		0.68	
	normality	OK			
	n	12			
	outliers	0			
	mean (n)	34.94			
	st.dev. (n)	19.479			
	R(calc.)	54.54			
	R(Horwitz)	12.97			



APPENDIX 2

Number of participants per country

1 lab in AUSTRALIA
1 lab 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 KUWAIT
5 labs in P.R. of CHINA
1 lab in RUSSIA
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
3 labs in SPAIN
4 labs in THE NETHERLANDS
1 lab in TURKEY
1 lab in U.A.E.
4 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
Fr.	= first reported

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
- 2 ASTM E178-02
- 3 ASTM E1301-03
- 4 ISO13528-05
- 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/>).