Results of Proficiency Test
Styrene
October 2010

CONTENTS

Appendices:

1   INTRODUCTION

Since 1995, the Institute for Interlaboratory Studies organizes a proficiency test for the analysis of Styrene. As part of the annual proficiency test program of 2010/2011, the Institute decided to continue this proficiency test on Styrene.
In this interlaboratory study 38 laboratories in 18 different countries have participated. See appendix 3 for a list of participants in alphabetical country order. In this report the results of the proficiency test are presented and discussed.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. It was decided to send one sample regular Styrene (1*500 mL, labelled #1058).
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% confidentially of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2   PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (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 95 amber glass bottles of 500 mL (labelled #1058).

The homogeneity of the subsamples #1058 was checked by determination of Density @ 20°C in accordance with ASTM D4052:02e1, water in accordance with ASTM E1064:05 and Aldehydes in accordance with ASTM D2119:03 on 8 stratified random selected samples.

	Density @20°C in kg/L	Aldehydes in mg/kg	Water in mg/kg
sample #1058-1	0.90620	39	130
sample #1058-2	0.90620	45	130
sample #1058-3	0.90620	42	130
sample #1058-4	0.90620	43	130
sample #1058-5	0.90630	43	130
sample #1058-6	0.90630	41	130
sample #1058-7	0.90630	43	130
sample #1058-8	0.90630	42	130

Table 1: homogeneity test results of subsamples #1058

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:

	Density @20°C in kg/L	Aldehydes in mg/kg	Water in mg/kg
r (sample #1058)	0.00015	4.9	0
Ref. method	ASTM D4052:02	ASTM D2119:03	ASTM E1064:05
0.3 x R (ref. method)	0.00015	5.1	6

Table 2: repeatabilities of subsamples #1058 evaluated against method requirements

Each calculated repeatability was equal or less than 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 #1058) was sent on September 8, 2010.

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 #1058: Aldehydes as benzaldehyde, Appearance, Colour Pt/Co, Inhibitor, Density @ 20°C, Peroxide as H_2O_2, 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, and a detailed questionnaire about the details of the Aldehyde determination 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(0.01) 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.

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 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.

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:

$\qquad$ | z | < 1 good
$\quad$ 1 < | z | < 2 satisfactory
$\quad$ 2 < | z | < 3 questionable
$\quad$ 3 < | z | unsatisfactory

4   EVALUATION

In this proficiency test some problems were encountered with the despatch of the samples to the laboratories in: Brazil, Kuwait and USA. Due to custom clearance problems, several laboratories did receive the samples near or after the final reporting date. Eight laboratories reported test results after the final reporting date.

Not all participants were able to report results for all the requested tests. Finally, 35 participants did report 496 test results. Observed were 30 outlying results, which is 6.1%. 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 (tables 3 and 4).

Not all original data sets proved to have a normal distribution. Not normal distributions were found for the following determinations: density, m-&p-ethyltoluenes and phenylacetylene.

In these cases the statistical evaluation should be used with due care.

Aldehydes as benzaldehyde:	This determination was problematic as in previous years. Only one statistical outlier was observed and one false negative result was reported (the latter remarkably by the laboratory that did report the presence of 20.5 mg/kg benzaldehyde). However, the observed reproducibility, after rejection of the statistical outlier, is not at all in agreement with the requirements of ASTM D2119:09. The participants were requested to report some critical details of the Aldehydes determination (see appendix 2). Several things can be noticed from the reported details, for example the large range of blank values (upto 9 ml) and the use of Thymol Blue in stead of the required Sodium salt of Thymol Blue (and the need of caustic to dissolve the Thymol blue). In the previous round it became clear that results, with use of the purchased sodium salt, may be significantly lower than when Thymol Blue is used to prepare the indicator solution. This may explain for the high results of laboratories 913 and 786.
Appearance:	No analytical problems were observed. All participants agreed about the appearance of sample #1058, which is bright, clear and free of suspended matter. The uniformity of reporting can be improved. A new standardized method is available for Appearance since 2009, being ASTM E2680. According this method the appearance should be reported as 'pass' (or 'fail').
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 D1209:05e1.

Inhibitor: This determination may be problematic. Only one statistical outlier was 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. Only 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.

Peroxides: 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 requirements of ASTM D2340:09.

Polymers: No significant conclusions were drawn as the polymer concentration was below the application range of the test method D2121:07. Only five laboratories reported a test result for polymers above 1 mg/kg.

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 at all 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, except one, agreed that less than 10 mg/kg of benzene was present.

Ethylbenzene: Several analytical problems have been observed. Three statistical outliers were detected and two false negative test results were reported. However, the calculated reproducibility, after rejection of the statistical outliers, is in good agreement with the requirements of ASTM D5135:07.

m-& p-Xylenes: No significant conclusions were drawn as the concentration m- & p-Xylenes was below the application range of the test method ASTM D5135:07. Only four laboratories reported a test result for m- & p- Xylenes above 10 mg/kg.

Cumene: This determination may be somewhat problematic. Only one statistical outlier was observed and one false negative test result was reported (probably mixed up with the result for o-Xylene). However, the observed reproducibility, after rejection of the statistical outlier, is not in full agreement with the requirements of ASTM D5135:07.

o-Xylene: No significant conclusions were drawn as the concentration o-Xylene was below the application range of the test method ASTM D5135:07. Only two laboratories reported a test result for o-Xylene above 10 mg/kg, of which one probably was mixed up with the test result for Cumene.

n-Prop.benzene: Several analytical problems have been observed. Four statistical outliers were detected. However, the calculated reproducibility, after rejection of the statistical outliers, is in good agreement with the requirements of ASTM D5135:07.

m-& p-Ethyltol.: This determination may be problematic. Two statistical outliers were observed and one false negative test result was reported. Also, the observed reproducibility, after rejection of the statistical outliers, is not at all in agreement with the requirements of ASTM D5135:07.

α-Methylstyrene: The determination of this component was problematic. Four statistical outliers were observed. And the calculated reproducibility, after rejection of the statistical outliers, is not at all in agreement with the requirements of ASTM D5135:07.

1,2-Diethylbenz.: Only eight laboratories reported a test result for this component. Three results were 'less than' the detection limit, while the other 5 results were clearly positive (from 11 upto 144 mg/kg). Because the actual 1,2-diethylbenzene concentration was unknown, no significant conclusions were drawn. The fact that 1,2-diethylbenzene may co-elute with α-Methylstyrene may be the reason for the wide range in test results.

Phenylacetylene: This determination may be problematic. No statistical outliers were observed, but two false negative test results were reported. The observed reproducibility is not in agreement with the requirements estimated from the Horwitz equation.

3,4-Methylstyr.: This determination may be problematic. No statistical outliers were observed. However, the observed reproducibility is not at all in agreement with the requirements estimated from the Horwitz equation.

Benzaldehyde: This determination was problematic. No statistical outliers were observed. However, one false negative result was reported and the observed

reproducibility is not at all in agreement with the requirement estimated from the Horwitz equation. The reported test results seem to be divided bimodally with 9 results around 20 mg/kg and 9 around 30 mg/kg.

Nonaromatics: Due to the low concentration of non aromatics no significant conclusions were drawn. All participants, except one, agreed that less than 10 mg/kg of non-aromatics was present.

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	24	42.55	42.75	17.01
Appearance		27	pass	n.a.	n.a.
Colour		31	7.6	4.5	7.0
Inhibitor	mg/kg	33	11.90	2.86	2.38
Density @20°C	kg/L	27	0.90626	0.00014	0.00050
Peroxides as H$_2$O$_2$	mg/kg	29	17.71	15.47	13.00
Polymers	mg/kg	19	0.77	1.33	(1.00)
Organic Chlorides	mg/kg	5	0.31	0.94	(0.18)
Sulphur	mg/kg	8	0.34	1.00	(0.12)
Water	mg/kg	30	139.6	31.9	20.7
Purity	%M/M	29	99.921	0.021	0.033
Benzene	mg/kg	3	(0.10)	(0.42)	(0.06)
Ethylbenzene	mg/kg	27	58.08	9.71	16.59
m- & p-Xylenes	mg/kg	11	(5.52)	(3.92)	(0.31)
Cumene	mg/kg	26	130.7	18.69	16.34
o-Xylene	mg/kg	17	(5.59)	(7.77)	(7.83)
n-Propylbenzene	mg/kg	23	57.89	7.02	14.47
m- & p-Ethyltoluenes	mg/kg	18	24.26	17.72	8.66
α-Methylstyrene	mg/kg	22	395.87	85.07	56.55
1,2-diethylbenzene	mg/kg	4	26.9	32.8	7.3
Phenylacetylene	mg/kg	22	21.89	14.28	6.16
3/4-methylstyrenes	mg/kg	7	17.89	22.67	7.34
Benzaldehyde	mg/kg	18	25.68	17.83	7.06
Nonaromatics	mg/kg	5	(4.18)	(9.44)	unknown

Table 3: reproducibilities of sample #1058

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

4.3   COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2010 WITH PREVIOUS PTS

	October 2010	October 2009	October 2008	October 2007
Number of reporting labs	35	33	33	33
Number of Results reported	496	613	708	553
Statistical outliers	30	44	37	26
Percentage outliers	6.1%	7.2%	5.2%	4.7%

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 the following table:

Determination	October 2010	October 2009		October 2008		October 2007
Aldehydes	--	--	--	--		--
Colour	++	++		++		+/-
Inhibitor	-	+/-		+/-		--
Density	++	+		++		++
Peroxides as H_2O_2	-	-		+		--
Polymer	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.	--	-	+	n.e.
o-Xylene	n.e.	n.e.	n.e.	++	++	++
n-Propylbenzene	++	n.e.	n.e.	+	+	++
m+p-Ethyltoluenes	--	n.e.	n.e.	--	--	--
α-Methylstyrene	-	-	--	-	+	+
Phenylacetylene	--	n.e.	n.e.	+	+	++
Benzaldehyde	--	--	--	--	--	+/-

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 #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D2119	47.0		0.73	
169		-----		-----	
171	D2119	23.4		-3.15	
174	D2119	23.5		-3.13	
176		-----		-----	
311	D2119	30		-2.06	
323	D2119	44		0.24	
329	D2119	40		-0.42	
333		-----		-----	
343		-----		-----	
347		-----		-----	
357	D2119	35		-1.24	
395	D2119	74.0		5.17	
396		-----		-----	
446	D2119	46		0.57	
551		-----		-----	
613		-----		-----	
786	D2119	71.4		4.75	
860	D2119	59		2.71	
862	D2119	60		2.87	
863	D2119	50		1.23	
865	D2119	62		3.20	
868	D2119	49		1.06	
869	D2119	52		1.56	
902		-----		-----	
913	D2119	112.2	G(0.05)	11.46	
1067	D2119	27.8		-2.43	
1085		-----		-----	
1117	D2119	29.5		-2.15	
1193	D2119	45.3		0.45	
1351	D2119	30.42		-2.00	
1429		-----		-----	
1509	D2119	37		-0.91	
1515	D2119	39		-0.58	
1921	D2119	17.5		-4.12	
7007	D2119	28.3		-2.34	
9005	D2119	0.00	ex, C	-7.00	zero is not a real result; first reported 0.001
9008		-----		-----	

normality	OK		
n	24		
outliers	2		
mean (n)	42.55		
st.dev. (n)	15.269		
R(calc.)	42.75		
R(D2119:09)	17.02		

Determination of Appearance on sample #1058;

lab	method	value	mark	z(targ)	remarks
150	E2680	PASS		-----	
169		-----		-----	
171	E2680	C&F		-----	
174	E2680	C&F		-----	
176		-----		-----	
311	E2680	PASS		-----	
323	E2680	PASS		-----	
329	E2680	PASS		-----	
333	E2680	C&B		-----	
343		-----		-----	
347	E2680	PASS		-----	
357	E2680	PASS		-----	
395	E2680	PASS		-----	
396	E2680	PASS		-----	
446	VISUAL	CFFMIS		-----	
551		-----		-----	
613		-----		-----	
786	E2680	PASS		-----	
860	E2680	PASS		-----	
862	VISUAL	C&B		-----	
863	VISUAL	CFFSM		-----	
865	E2680	PASS		-----	
868	VISUAL	C&B		-----	
869	E2680	PASS		-----	
902	E2680	PASS		-----	
913	E2680	CFFSM		-----	
1067	E2680	PASS		-----	
1085		-----		-----	
1117	D4176	PASS		-----	
1193		-----		-----	
1351		-----		-----	
1429	E2680	C&B		-----	
1509	E2680	CFFSM		-----	
1515	D2340	C&B		-----	
1921		-----		-----	
7007		-----		-----	
9005	E2680	PASS		-----	
9008		-----		-----	
	n	27			
	mean (n)	PASS			

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 #1058;

lab	method	value	mark	z(targ)	remarks
150	D5386	7	C	-0.26	first reported 0
169	D5386	8		0.14	
171	D1209	9		0.54	
174	D1209	9		0.54	
176		-----		-----	
311	D1209	5		-1.06	
323	D1209	10		0.94	
329	D1209	10		0.94	
333	D1209	6		-0.66	
343		-----		-----	
347	D1209	7		-0.26	
357	D1209	5		-1.06	
395	D5386	7		-0.26	
396	D1209	7		-0.26	
446	D1209	<5		<-1.06	
551		-----		-----	
613	D5386	7		-0.26	
786	D1209	8		0.14	
860	D1209	8		0.14	
862	D1209	7		-0.26	
863	D1209	10		0.94	
865	D1209	7		-0.26	
868	D5386	7		-0.26	
869	D1209	8		0.14	
902	D5386	6		-0.66	
913	D5386	5		-1.06	
1067	D1209	5		-1.06	
1085		-----		-----	
1117	D1209	9		0.54	
1193	D1209	9		0.54	
1351	D1209	6		-0.66	
1429	D6045	8		0.14	
1509	D1209	10		0.94	
1515	D1209	9		0.54	
1921	D1209	10		0.94	
7007		-----		-----	
9005	D5386	8	C	0.14	first reported 22
9008		-----		-----	

normality	OK
n	31
outliers	0
mean (n)	7.65
st.dev. (n)	1.603
R(calc.)	4.49
R(D1209:05e1)	7.00

Determination of Inhibitor (p-TBC) on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D4590	12.4		0.59	
169	D4590	11		-1.06	
171	D4590	12.3		0.47	
174	D4590	12.2		0.35	
176		-----		-----	
311	D4590	11		-1.06	
323	D4590	11		-1.06	
329	D4590	13		1.29	
333	D4590	12		0.12	
343		-----		-----	
347	D4590	13	C	1.29	first reported 15
357	D4590	13.6		2.00	
395	D4590	13.2		1.53	
396	D4590	13.3		1.65	
446	D4590	13		1.29	
551		-----		-----	
613	D4590	10.8	C	-1.29	first reported 17.9
786	D4590	11.4		-0.59	
860	D4590	11.9		0.00	
862	D4590	11.2		-0.82	
863	D4590	12.1		0.24	
865	D4590	11.2		-0.82	
868	D4590	11.9		0.00	
869	D4590	11.9		0.00	
902	D4590	12.85		1.12	
913	D4590	9.7		-2.59	
1067	D4590	11.8		-0.12	
1085	D4590	10.28		-1.91	
1117	D4590	12.89		1.17	
1193	D4590	10.6		-1.53	
1351	D4590	11.10		-0.94	
1429	D4590	21.291	G(0.01)	11.05	
1509	D4590	12.3		0.47	
1515	D4590	13.91		2.37	
1921	D4590	10.8		-1.29	
7007	D4590	12.0		0.12	
9005	D4590	11.06		-0.99	
9008		-----		-----	

normality	OK	
n	33	
outliers	1	
mean (n)	11.90	
st.dev. (n)	1.022	
R(calc.)	2.86	
R(D4590:09)	2.38	
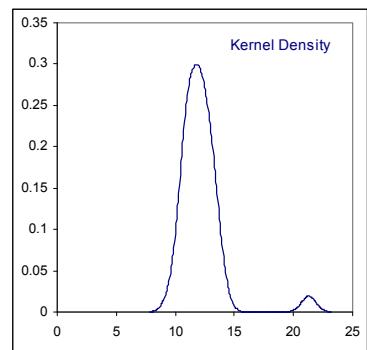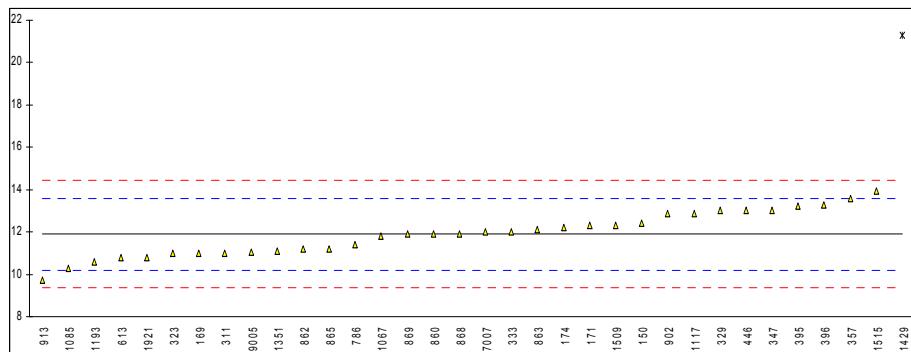
Determination of Density at 20°C on sample #1058; results in kg/L

lab	method	value	mark	z(targ)	remarks
150	D4052	0.9063		0.24	
169		-----		-----	
171	D4052	0.9062		-0.32	
174	D4052	0.9062		-0.32	
176		-----		-----	
311	D4052	0.9063		0.24	
323	D4052	0.9062		-0.32	
329	D4052	0.9063		0.24	
333		-----		-----	
343	D4052	0.90624		-0.10	
347	D4052	0.9063		0.24	
357	D4052	0.9062		-0.32	
395	D4052	0.9062		-0.32	
396		-----		-----	
446	D4052	0.9062		-0.32	
551		-----		-----	
613	D4052	0.90623		-0.15	
786	D4052	0.9062		-0.32	
860	D4052	0.90623		-0.15	
862	D4052	0.90628		0.13	
863	D4052	0.90631		0.29	
865	D4052	0.9063		0.24	
868	D4052	0.90634		0.46	
869	D4052	0.90625		-0.04	
902	D4052	0.90629		0.18	
913	D4052	0.9063		0.24	
1067	D4052	0.9063		0.24	
1085		-----		-----	
1117	D4052	0.9062		-0.32	
1193	D4052	0.90629	U	0.18	reported 906.29
1351		-----		-----	
1429	D4052	0.9078	G(0.01)	8.64	
1509	D4052	0.9063		0.24	
1515		-----		-----	
1921	D4052	0.90630		0.24	
7007		-----		-----	
9005	D4052	0.9062		-0.32	
9008		-----		-----	

normality	not OK
n	27
outliers	1
mean (n)	0.90626
st.dev. (n)	0.000048
R(calc.)	0.00014
R(D4052:02e1)	0.00050

Determination of Peroxides as H_2O_2 on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D2340	17.2		-0.11	
169		-----		-----	
171	D2340	21.8		0.88	
174	D2340	20.3		0.56	
176		-----		-----	
311	D2340	23		1.14	
323	D2340	14		-0.80	
329	D2340	13		-1.02	
333	D2340	30		2.65	
343	D2340	10.96		-1.45	
347		-----		-----	
357	D2340	20.6		0.62	
395		-----		-----	
396		-----		-----	
446	D2340	16.9		-0.18	
551		-----		-----	
613	D2340	28.458		2.31	
786	D2340	24.8		1.53	
860	D2340	15		-0.58	
862	D2340	18.7		0.21	
863	D2340	14.3		-0.74	
865	D2340	19.6		0.41	
868	D2340	14.0		-0.80	
869	D2340	14.2		-0.76	
902	D2340	18.95		0.27	
913	D2340	22.5		1.03	
1067	D2340	7.7		-2.16	
1085		-----		-----	
1117	D2340	18.2		0.10	
1193	D2340	8.0		-2.09	
1351	D2340	20.14		0.52	
1429	D2340	51	G(0.01)	7.17	
1509	D2340	13.3		-0.95	
1515	D2340	14.35		-0.72	
1921	D2340	16.4		-0.28	
7007	D2340	12.1		-1.21	
9005	D2340	25.25		1.62	
9008		-----		-----	

normality	OK
n	29
outliers	1
mean (n)	17.71
st.dev. (n)	5.524
R(calc.)	15.47
R(D2340:09)	13.00

Kernel Density
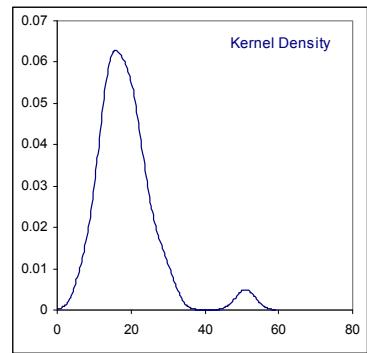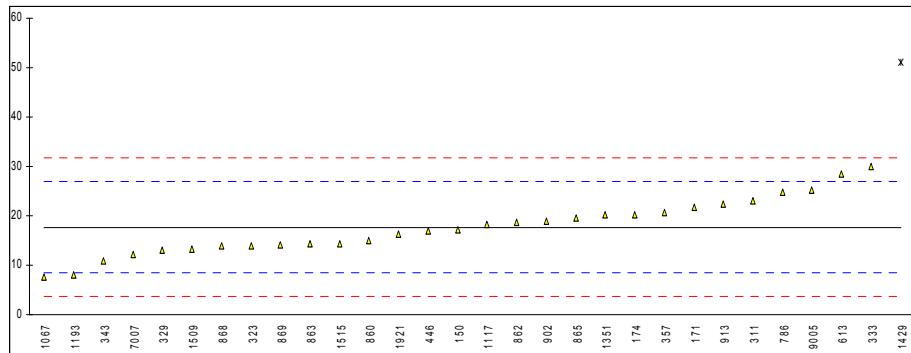
Determination of Polymers on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D2121	<1		-----	
169	D2121	<1		-----	
171	D2121	0.3		-----	
174	D2121	0.2		-----	
176		-----		-----	
311	D2121	<1		-----	
323	D2121	<1		-----	
329	D2121	1		-----	
333	D2121	<1		-----	
343		-----		-----	
347	D2121	<1		-----	
357	D2121	<1		-----	
395	D2121	<1		-----	
396	D2121	<1		-----	
446	D2121	1		-----	
551		-----		-----	
613	D2121	<1		-----	
786	D2121	0.76		-----	
860	D2121	0.1		-----	
862	D2121	<1		-----	
863	D2121	1.1		-----	
865	D2121	0.2		-----	
868	D2121	0.9		-----	
869	D2121	0.8		-----	
902	D2121	0.8		-----	
913	D2121	<1.0		-----	
1067	D2121	<0.5		-----	
1085		-----		-----	
1117	D2121	0.4		-----	
1193	D2121	1.25	C	-----	first reported 1.75
1351	D2121	1.16		-----	
1429	D2121	0.4521		-----	
1509	D2121	0.7		-----	
1515	D2121	0.20539		-----	
1921	D2121	1.43		-----	
7007	D2121	<1		-----	
9005	D2121	1.82		-----	
9008		-----		-----	

normality	OK	
n	19	
outliers	0	
mean (n)	(0.77)	
st.dev. (n)	(0.473)	
R(calc.)	(1.33)	
R(D2121:07)	(1)	application range 1 – 15 mg/kg
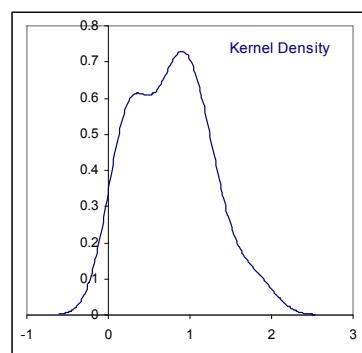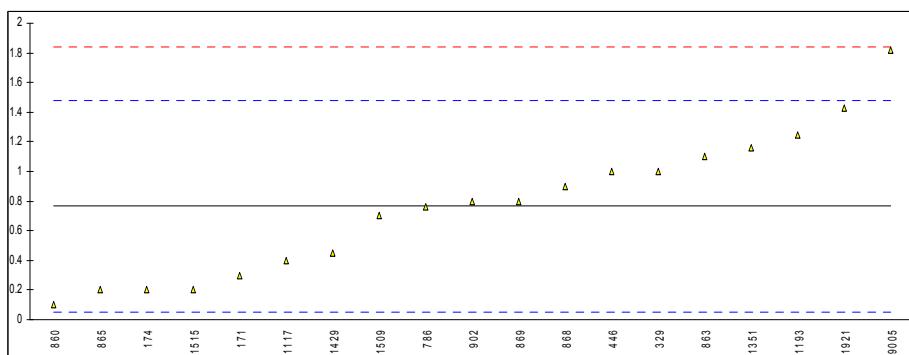
Determination of Organic Chloride on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D7359	<1		-----	
169		-----		-----	
171	D5808	<1		-----	
174		-----		-----	
176		-----		-----	
311	D5808	<1		-----	
323	UOP779	<1		-----	
329	UOP779	<1		-----	
333		<0.3		-----	
343		-----		-----	
347		-----		-----	
357	D5808	<1		-----	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786		-----		-----	
860		-----		-----	
862	D5808	0.1		-----	
863		-----		-----	
865	D5808	<1		-----	
868	D5808	<1		-----	
869	D5808	<1		-----	
902		-----		-----	
913		-----		-----	
1067		0.16		-----	
1085		-----		-----	
1117		-----		-----	
1193		-----		-----	
1351		-----		-----	
1429		0.9		-----	
1509	D5808	0.12		-----	
1515		-----		-----	
1921		-----		-----	
7007		-----		-----	
9005	D5808	0.288		-----	
9008		-----		-----	

normality	n.a.
n	5
outliers	0
mean (n)	(0.31)
st.dev. (n)	(0.336)
R(calc.)	(0.94)
R(Horwitz)	(0.17)
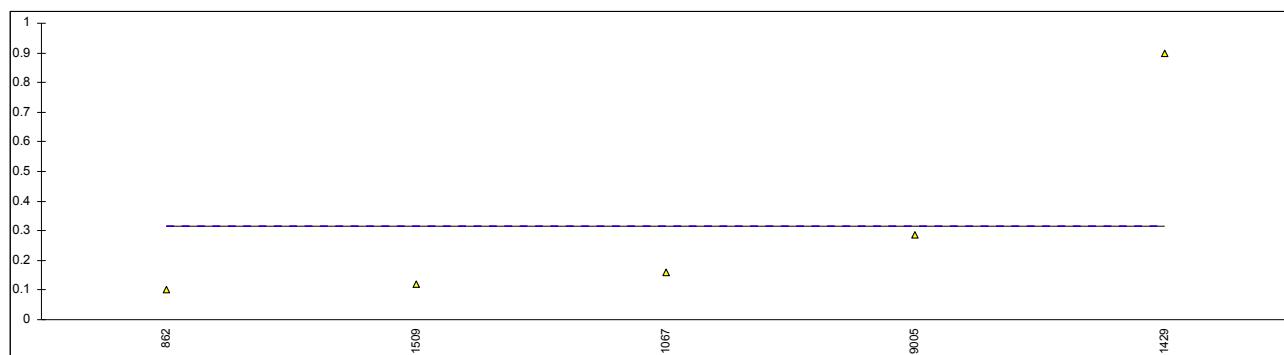
Determination of Sulphur on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5453	<1.0		-----	
169		-----		-----	
171	D5453	<0.5		-----	
174		-----		-----	
176		-----		-----	
311	D3961	<0.5		-----	
323	D5453	0.7		-----	
329	D3661	1		-----	
333	D5453	<1		-----	
343		-----		-----	
347	D5453	0.4		-----	
357	D5453	<1		-----	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786		-----		-----	
860		-----		-----	
862	D5453	0.1		-----	
863	D5453	<1.0		-----	
865	INH-253	<0.5		-----	
868	D3120	<1		-----	
869	D3120	<1		-----	
902		-----		-----	
913		-----		-----	
1067	D5453	0.39		-----	
1085		-----		-----	
1117	D5453	0.07		-----	
1193		-----		-----	
1351		-----		-----	
1429	D5453	<1		-----	
1509	D5453	0.06		-----	
1515		-----		-----	
1921		-----		-----	
7007	D5453	<1		-----	
9005	D5453	0		-----	
9008		-----		-----	

normality	OK
n	8
outliers	0
mean (n)	(0.34)
st.dev. (n)	(0.358)
R(calc.)	(1.00)
R(D5453:09)	(0.26)

application range 1 – 8000 mg/kg
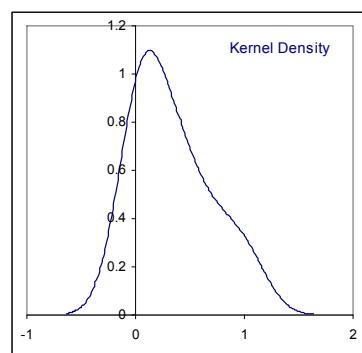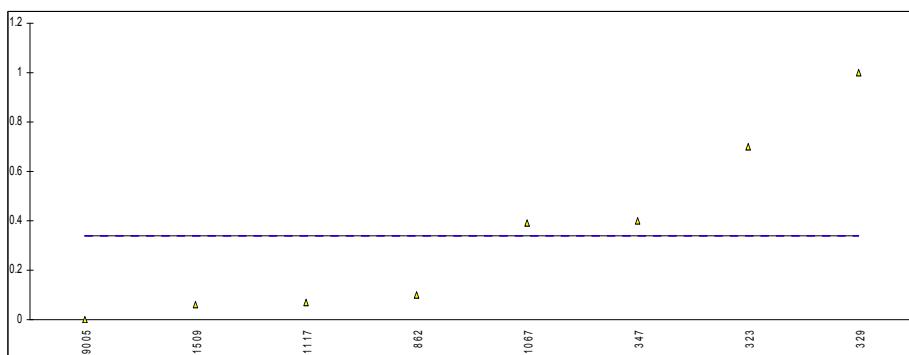
Determination of Water on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	E1064	142		0.30	
169		-----		-----	
171	E1064	145		0.68	
174	E1064	151		1.44	
176		-----		-----	
311	E1064	130		-1.21	
323	E1064	135		-0.58	
329	E203	131		-1.08	
333	E1064	114		-3.23	
343	E1064	142.7	C	0.39	first reported 194.4
347	E1064	142		0.30	
357	E1064	129		-1.34	
395	E1064	146.67		0.89	
396		-----		-----	
446	E203	165		3.21	
551		-----		-----	
613	E203	143.5		0.49	
786	E1064	197.6	G(0.01)	7.32	
860	E1064	138		-0.20	
862	E1064	140		0.05	
863	E1064	134		-0.71	
865	E1064	137		-0.33	
868	E1064	134		-0.71	
869	E1064	134		-0.71	
902	E1064	141.6		0.25	
913		-----		-----	
1067	E1064	134		-0.71	
1085		-----		-----	
1117	D4672	144.0		0.56	
1193	E1064	132.8	C	-0.86	first reported 109.2
1351	E1064	129		-1.34	
1429	E1064	157		2.20	
1509	E1064	144		0.56	
1515	E1064	167		3.46	
1921	E1064	144.2		0.58	
7007	UOP481	117.3		-2.81	
9005	E1064	142.9		0.42	
9008		-----		-----	

normality	OK
n	30
outliers	1
mean (n)	139.59
st.dev. (n)	11.394
R(calc.)	31.90
R(E1064:05)	22.19
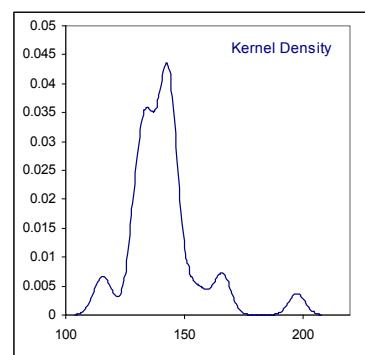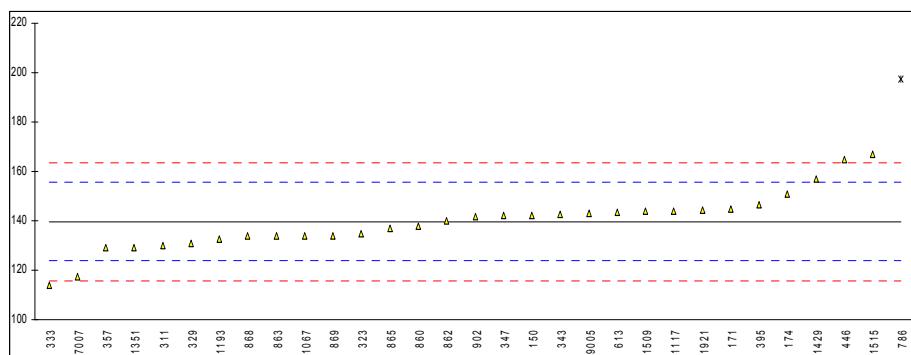
Determination of Purity on sample #1058; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	D5135	99.91		-0.95	
169	D5135	99.87	G(0.05)	-4.34	
171	D5135	99.914		-0.61	
174	D5135	99.926		0.41	
176		-----		-----	
311	D5135	99.918		-0.27	
323	D5135	99.92		-0.10	
329	D5135	99.91		-0.95	
333	D5135	99.94		1.60	
343	D5135	99.917		-0.35	
347	D5135	99.929		0.67	
357	D5135	99.92		-0.10	
395		-----		-----	
396		-----		-----	
446	D5135	99.99	G(0.01)	5.84	
551		-----		-----	
613	D5135	99.926		0.41	
786	D5135	99.929		0.67	
860	D5135	99.924		0.24	
862	D5135	99.923		0.16	
863	D5135	99.929		0.67	
865	D5135	99.926		0.41	
868	D5135	99.921		-0.01	
869	D5135	99.921		-0.01	
902	D5135	99.919		-0.18	
913	D5135	99.93		0.75	
1067	D5135	99.91		-0.95	
1085		-----		-----	
1117	D5135	99.93		0.75	
1193	D5135	99.914		-0.61	
1351	D5135	99.922		0.07	
1429	D5135	99.91		-0.95	
1509	D5135	99.916		-0.44	
1515	in house	99.9192		-0.17	
1921	D5135	99.915		-0.52	
7007	D5135	99.953	G(0.01)	2.70	
9005	D5135	99.926		0.41	
9008		-----		-----	

normality	OK	
n	29	
outliers	3	
mean (n)	99.921	
st.dev. (n)	0.0073	
R(calc.)	0.021	
R(D5135:07)	0.033	
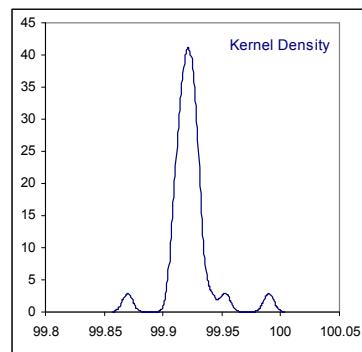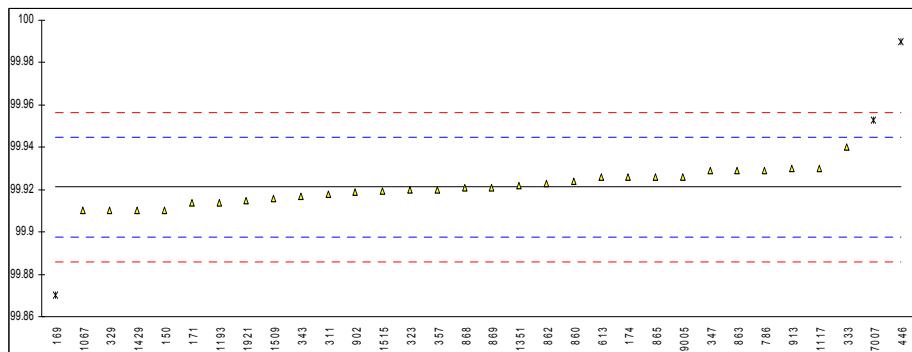
Determination of Benzene on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	<1		-----	
169	D5135	<1		-----	
171	D5135	<1		-----	
174	D5135	<1		-----	
176		-----		-----	
311	SPI222	<1		-----	
323	INH-189	<1		-----	
329		-----		-----	
333		-----		-----	
343	INH-1456	<1		-----	
347		-----		-----	
357	D5135	<1		-----	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786	INH-002	<1		-----	
860	D5135	<10		-----	
862	D5135	<10		-----	
863	D5135	<10		-----	
865	D5135	<10		-----	
868	D5135	<10		-----	
869	D5135	<10		-----	
902	INH-83	<1		-----	
913		-----		-----	
1067	GC	<1		-----	
1085	in house	0.0		-----	
1117	INH-2922	0.27		-----	
1193		-----		-----	
1351		-----		-----	
1429		<1		-----	
1509	in house	<0.5		-----	
1515	in house	<0.5		-----	
1921		-----		-----	
7007	D5135	23.8	G(0.01)	-----	false positive?
9005	D4534	0.015		-----	
9008		-----		-----	
	normality	n.a.			
	n	3			
	outliers	1			
	mean (n)	(0.095)			
	st.dev. (n)	(0.1517)			
	R(calc.)	(0.425)			
	R(Horwitz)	(0.061)			

Determination of Ethylbenzene on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	58		-0.01	
169	D5135	<1		<-9.63	false negative?
171	D5135	51.9		-1.04	
174	D5135	54		-0.69	
176		-----		-----	
311	D5135	61		0.49	
323	D5135	55		-0.52	
329	D5135	50		-1.36	
333	D5135	60		0.32	
343	D5135	53.70		-0.74	
347	D5135	60		0.32	
357	D5135	62		0.66	
395	D5135	64.68		1.11	
396	D5135	57		-0.18	
446	D5135	7	G(0.01)	-8.62	
551		-----		-----	
613		-----		-----	
786	D5135	58.72		0.11	
860	D5135	58		-0.01	
862	D5135	57		-0.18	
863	D5135	57		-0.18	
865	D5135	60		0.32	
868	D5135	59		0.15	
869	D5135	61		0.49	
902	INH-83	59		0.15	
913		-----		-----	
1067	GC	60		0.32	
1085	in house	39.50	G(0.01)	-3.14	
1117	D5135	54		-0.69	
1193	D5135	56.7		-0.23	
1351	D5135	58.1		0.00	
1429	D5135	65		1.17	
1509	D5135	59		0.15	
1515	in house	<2		<-9.46	false negative?
1921		-----		-----	
7007	D5135	36.1	G(0.01)	-3.71	
9005	D5135	58.4		0.05	
9008		-----		-----	

normality	OK	
n	27	
outliers	3	
mean (n)	58.08	
st.dev. (n)	3.467	
R(calc.)	9.71	
R(D5135:07)	16.59	
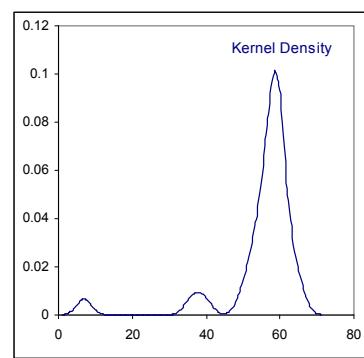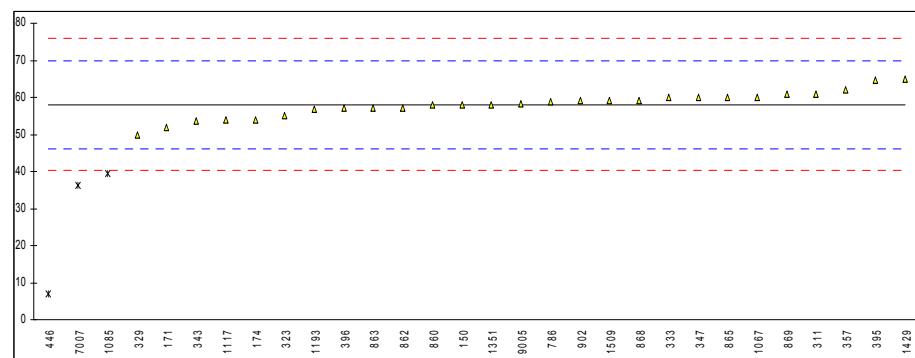
Determination of m- & p-Xylenes on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	6		-----	
169	D5135	62	G(0.01)	-----	
171	D5135	4.9		-----	
174	D5135	6		-----	
176		-----		-----	
311	D5135	<10		-----	
323	D5135	<10		-----	
329	D5135	<10		-----	
333		-----		-----	
343	D5135	6.39		-----	
347		-----		-----	
357	D5135	<10		-----	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786	D5135	5.06		-----	
860	D5135	6.2		-----	
862	D5135	8		-----	
863	D5135	<10		-----	
865	D5135	<10		-----	
868	D5135	<10		-----	
869	D5135	<10		-----	
902	INH-83	<10		-----	
913		-----		-----	
1067	GC	12.5	DG(0.05)	-----	
1085		-----		-----	
1117	D5135	<10		-----	
1193		-----		-----	
1351		-----		-----	
1429	D5135	11	DG(0.05)	-----	
1509	D5135	6		-----	
1515	in house	5		-----	
1921	D5135	19	G(0.05)	-----	
7007	D5135	2.3		-----	
9005	D5135	4.9		-----	
9008		-----		-----	

normality	OK
n	11
outliers	4
mean (n)	(5.52)
st.dev. (n)	(1.403)
R(calc.)	(3.93)
R(D5135:07)	(0.31)

application range 10-10000 mg/kg
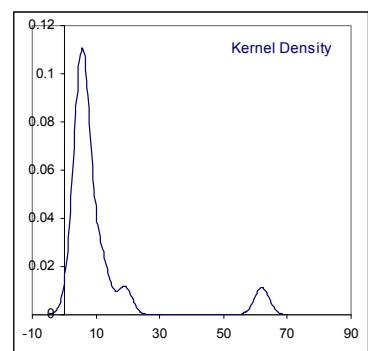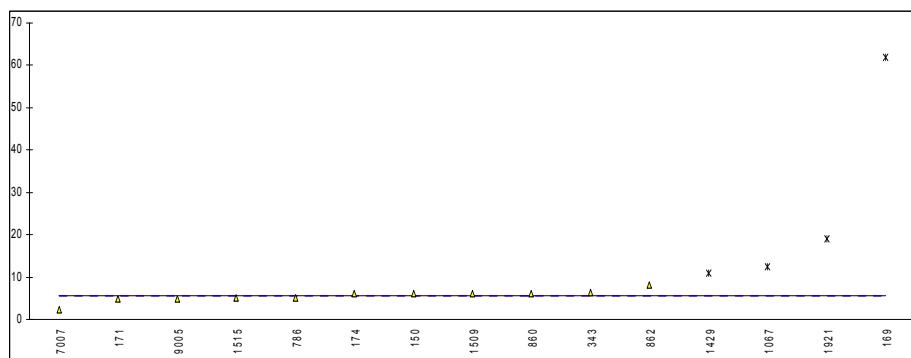
Determination of Cumene on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	130		-0.13	
169	D5135	<1		<-22.23	result probably mixed-up with o-Xylene result
171	D5135	135.8		0.87	
174	D5135	135		0.73	
176		-----		-----	
311	D5135	138		1.24	
323	D5135	125		-0.98	
329	D5135	140		1.59	
333	D5135	130		-0.13	
343	D5135	119.81	C	-1.87	first reported 114.32
347	D5135	120		-1.84	
357	D5135	141		1.76	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786	D5135	129.63		-0.19	
860	D5135	128		-0.47	
862	D5135	131		0.05	
863	D5135	130		-0.13	
865	D5135	135		0.73	
868	D5135	129		-0.30	
869	D5135	136		0.90	
902	INH-83	139.6		1.52	
913		-----		-----	
1067	GC	133		0.39	
1085	in house	113.13		-3.02	
1117	D5135	122	C	-1.50	first reported 64
1193		-----		-----	
1351		-----		-----	
1429	D5135	128		-0.47	
1509	D5135	133		0.39	
1515	in house	134		0.56	
1921	D5135	130		-0.13	
7007	D5135	78	G(0.01)	-9.04	
9005	D5135	133.2		0.42	
9008		-----		-----	

normality	OK	
n	26	
outliers	1	
mean (n)	130.74	
st.dev. (n)	6.677	
R(calc.)	18.69	
R(D5135:07)	16.34	
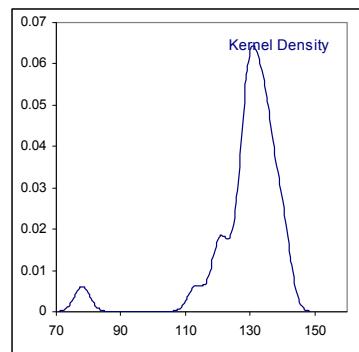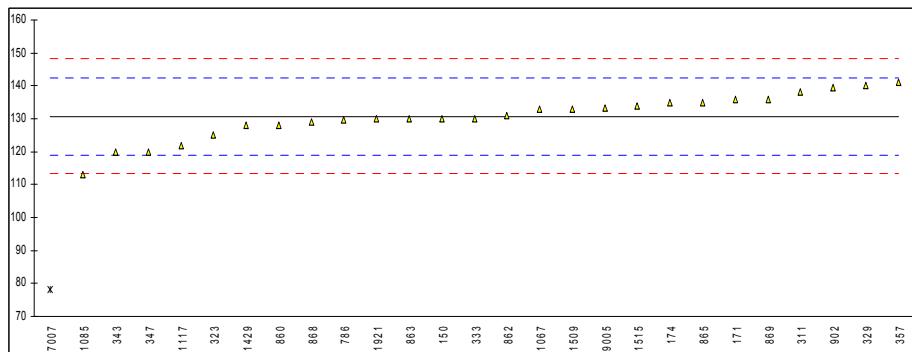
Determination of o-Xylene on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	6		-----	
169	D5135	143	G(0.01)	-----	result probably mixed-up with Cumene result
171	D5135	5.3		-----	
174	D5135	6		-----	
176		-----		-----	
311	D5135	9		-----	
323	D5135	<10		-----	
329	D5135	<10		-----	
333		-----		-----	
343	D5135	5.56		-----	
347		-----		-----	
357	D5135	<10		-----	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786	D5135	5.16		-----	
860	D5135	6		-----	
862	D5135	6.1		-----	
863	D5135	<10		-----	
865	D5135	6		-----	
868	D5135	<10		-----	
869	D5135	<10		-----	
902	INH-83	<10		-----	
913		-----		-----	
1067	GC	6.6		-----	
1085	in house	0.05		-----	
1117	D5135	6		-----	
1193		-----		-----	
1351		-----		-----	
1429	D5135	<1		-----	
1509	D5135	7		-----	
1515	in house	4		-----	
1921	D5135	12		-----	
7007	D5135	4.3		-----	
9005	D5135	0		-----	
9008		-----		-----	

normality	OK
n	17
outliers	1
mean (n)	(5.59)
st.dev. (n)	(2.775)
R(calc.)	(7.77)
R(D5135:07)	(7.83) application range 10-10000 mg/kg
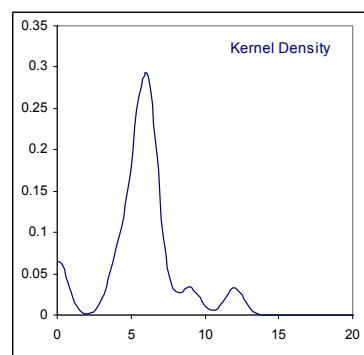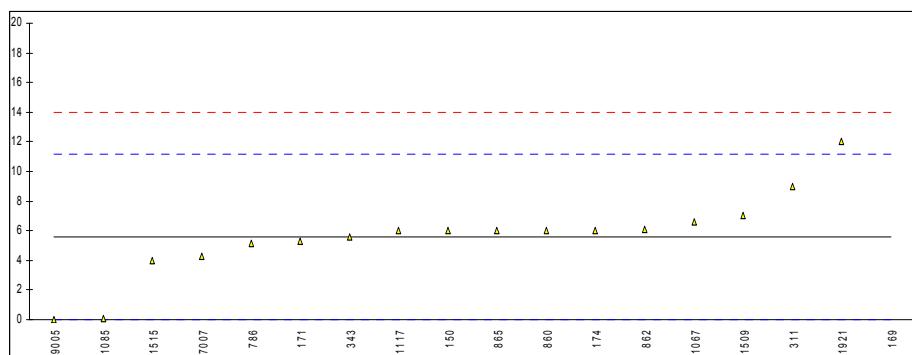
Determination of n-Propylbenzene on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	57		-0.17	
169	D5135	67	G(0.05)	1.76	
171	D5135	54.0		-0.75	
174	D5135	52		-1.14	
176		-----		-----	
311	D5135	60		0.41	
323	D5135	56		-0.37	
329	D5135	60		0.41	
333		-----		-----	
343	D5135	55.82	C	-0.40	first reported 11.92
347	D5135	58		0.02	
357	D5135	63		0.99	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786	D5135	57.53		-0.07	
860	D5135	57		-0.17	
862	D5135	59		0.21	
863	D5135	57		-0.17	
865	D5135	58		0.02	
868	D5135	59		0.21	
869	D5135	60		0.41	
902	INH-83	56.6		-0.25	
913		-----		-----	
1067	GC	60		0.41	
1085	in house	43.65	G(0.01)	-2.76	
1117	D5135	56	C	-0.37	first reported 19
1193		-----		-----	
1351		-----		-----	
1429	D5135	40	G(0.01)	-3.46	
1509	D5135	58	C	0.02	first reported 80
1515	in house	62		0.79	
1921	D5135	60		0.41	
7007	D5135	34.6	G(0.05)	-4.51	
9005	D5135	55.6		-0.44	
9008		-----		-----	

normality	OK	
n	23	
outliers	4	
mean (n)	57.89	
st.dev. (n)	2.508	
R(calc.)	7.02	
R(D5135:07)	14.47	
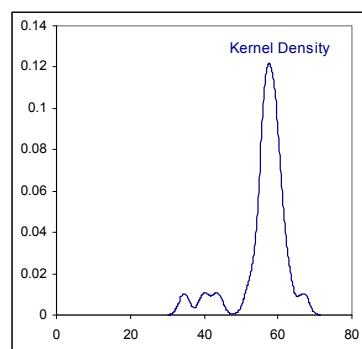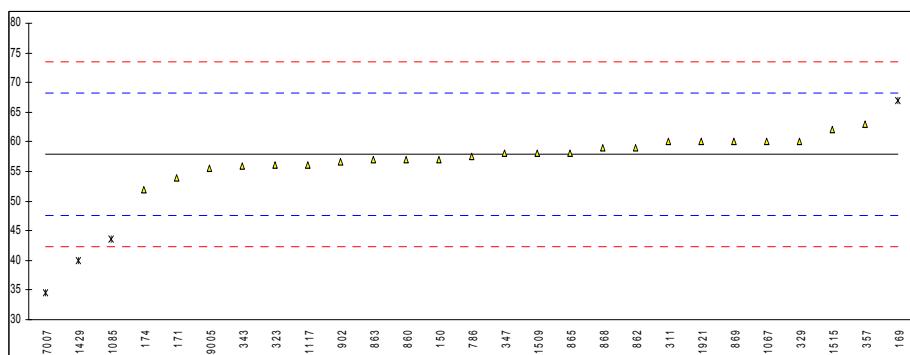
Determination of m- & p-Ethyltoluenes on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	23		-0.41	
169		-----		-----	
171	D5135	25.6		0.43	
174		-----		-----	
176		-----		-----	
311	D5135	28		1.21	
323	D5135	24		-0.08	
329	D5135	30		1.85	
333		-----		-----	
343	D5135	23.18	C	-0.35	first reported 63.45
347	D5135	28		1.21	
357	D5135	25		0.24	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786	D5135	22.71		-0.50	
860	D5135	23		-0.41	
862	D5135	25		0.24	
863	D5135	12		-3.96	
865		-----		-----	
868	D5135	25		0.24	
869		-----		-----	
902		-----		-----	
913		-----		-----	
1067	GC	32		2.50	
1085	in house	6.08	DG(0.05)	-5.88	
1117	D5135	18		-2.02	
1193		-----		-----	
1351		-----		-----	
1429	D5135	<10		<-4.61	false negative?
1509	D5135	25		0.24	
1515	in house	37		4.12	
1921		-----		-----	
7007	D5135	10.2		-4.54	
9005	D5135	6.0	DG(0.05)	-5.90	
9008		-----		-----	

normality	not OK
n	18
outliers	2
mean (n)	24.26
st.dev. (n)	6.330
R(calc.)	17.72
R(D5135:07)	8.66
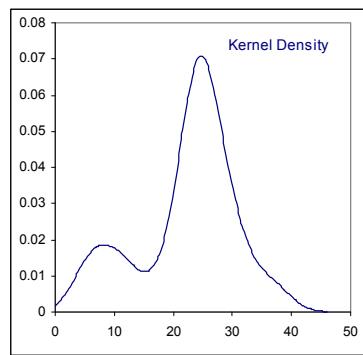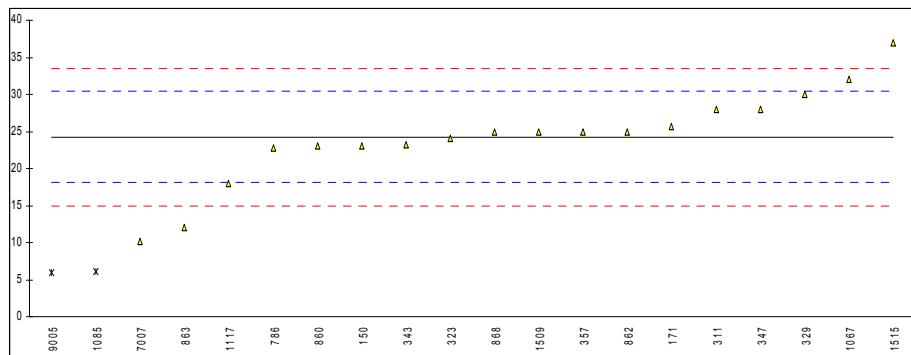
Determination of alpha-Methylstyrene on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	374		-1.08	
169	D5135	40	G(0.05)	-17.62	
171	D5135	414.6		0.93	
174	D5135	409		0.65	
176		-----		-----	
311	D5135	358		-1.88	
323	D5135	411		0.75	
329	D5135	450		2.68	
333	D5135	320		-3.76	
343	D5135	356.10		-1.97	
347	D5135	388		-0.39	
357	D5135	433		1.84	
395		-----		-----	
396		-----		-----	
446	D5135	52	G(0.01)	-17.03	
551		-----		-----	
613		-----		-----	
786	D5135	378.71		-0.85	
860	D5135	389		-0.34	
862		-----		-----	
863	D5135	369		-1.33	
865		-----		-----	
868	D5135	421		1.24	
869	D5135	389		-0.34	
902	INH-83	433		1.84	
913		-----		-----	
1067	GC	410		0.70	
1085	in house	239.00	G(0.05)	-7.77	
1117	D5135	375		-1.03	
1193		-----		-----	
1351		-----		-----	
1429	D5135	392		-0.19	
1509	D5135	424		1.39	
1515	in house	417		1.05	
1921		-----		-----	
7007	D5135	262.4	G(0.01)	-6.61	
9005	D5135	397.8		0.10	
9008		-----		-----	

normality	OK	
n	22	
outliers	4	
mean (n)	395.87	
st.dev. (n)	30.383	
R(calc.)	85.07	
R(D5135:07)	56.55	
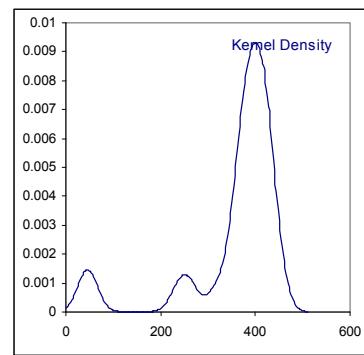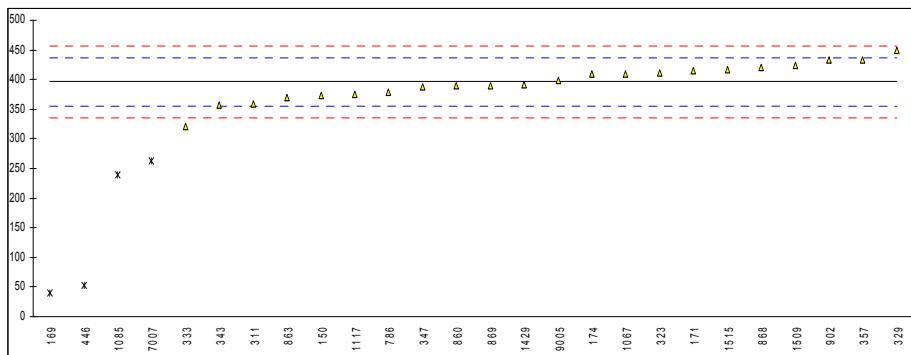
Determination of 1,2-diethylbenzene on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
169		-----		-----	
171	D5135	30.6		-----	
174	D5135	27		-----	
176		-----		-----	
311	D5135	39		-----	
323		-----		-----	
329		-----		-----	
333		-----		-----	
343		-----		-----	
347		-----		-----	
357		-----		-----	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786		-----		-----	
860		-----		-----	
862		-----		-----	
863		-----		-----	
865		-----		-----	
868		-----		-----	
869		-----		-----	
902		-----		-----	
913		-----		-----	
1067	GC	11		-----	
1085		-----		-----	
1117	D5135	<5		-----	
1193		-----		-----	
1351		-----		-----	
1429	D5135	144	G(0.05)	-----	
1509	in house	<5		-----	
1515	in house	<17		-----	
1921		-----		-----	
7007		-----		-----	
9005		-----		-----	
9008		-----		-----	

normality	n.a.
n	4
outliers	1
mean (n)	26.9
st.dev. (n)	11.73
R(calc.)	32.8
R(Horwitz)	7.3
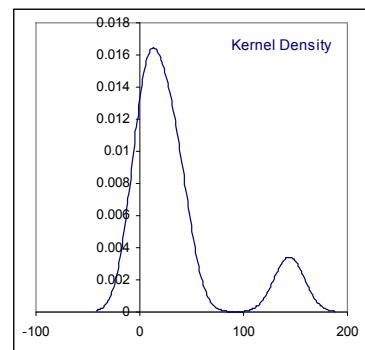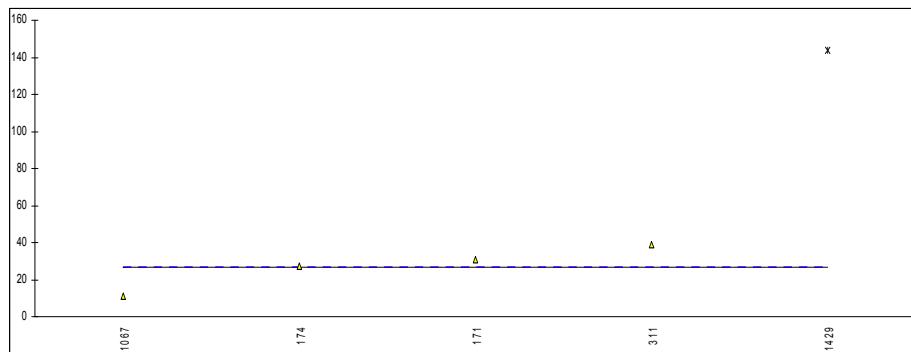
Determination of Phenylacetylene on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	21		-0.41	
169		-----		-----	
171	D5135	17.1		-2.18	
174	D5135	17.3	C	-2.09	first reported 14
176		-----		-----	
311	D5135	22		0.05	
323	D5135	18		-1.77	
329	D5135	20		-0.86	
333		20		-0.86	
343	D5135	<10		<-5.40	false negative?
347	D5135	30		3.68	
357	D5135	31		4.14	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786	D5135	25.97		1.85	
860	D5135	21		-0.41	
862	D5135	19		-1.31	
863	D5135	28		2.77	
865	D5135	22		0.05	
868		-----		-----	
869	D5135	30		3.68	
902	INH-83	20.3	C	-0.72	first reported 34.6
913		-----		-----	
1067	GC	20		-0.86	
1085		-----		-----	
1117	D5135	30		3.68	
1193		-----		-----	
1351		-----		-----	
1429	D5135	<1		<-9.50	false negative?
1509	in house	21		-0.41	
1515	in house	17		-2.22	
1921		-----		-----	
7007	D5135	12.1		-4.45	
9005	D5135	18.9		-1.36	
9008		-----		-----	

normality	not OK
n	22
outliers	0
mean (n)	21.89
st.dev. (n)	5.100
R(calc.)	14.28
R(Horwitz)	6.16
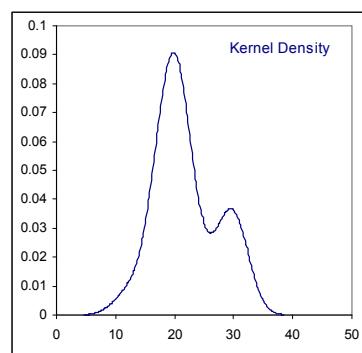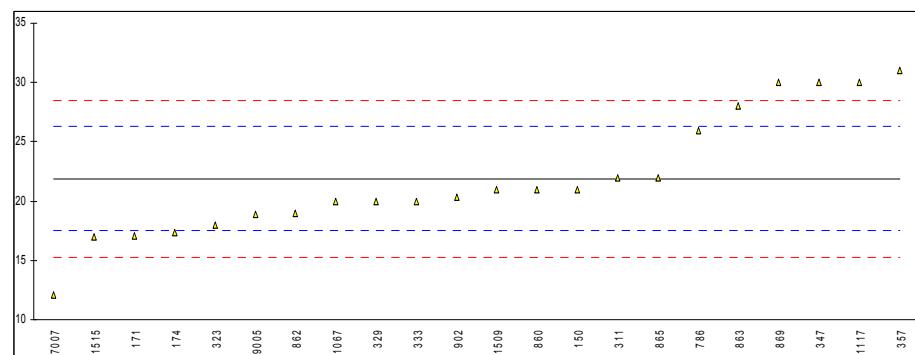
Determination of 3,4-methylstyrenes on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
169		-----		-----	
171		-----		-----	
174		-----		-----	
176		-----		-----	
311	D5135	17		-0.34	
323		-----		-----	
329		-----		-----	
333		-----		-----	
343	D5135	30.73	C	4.90	first reported 25.40
347		-----		-----	
357	D5135	15		-1.10	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786		-----		-----	
860		-----		-----	
862		-----		-----	
863		-----		-----	
865		-----		-----	
868		-----		-----	
869		-----		-----	
902		-----		-----	
913		-----		-----	
1067	GC	10		-3.01	
1085		-----		-----	
1117	D5135	<10		<-3.01	
1193		-----		-----	
1351		-----		-----	
1429		27		3.47	
1509	in house	16		-0.72	
1515		-----		-----	
1921		-----		-----	
7007		-----		-----	
9005	D5135	9.5		-3.20	
9008		-----		-----	

normality	OK	
n	7	
outliers	0	
mean (n)	17.89	
st.dev. (n)	8.095	
R(calc.)	22.67	
R(Horwitz)	7.34	
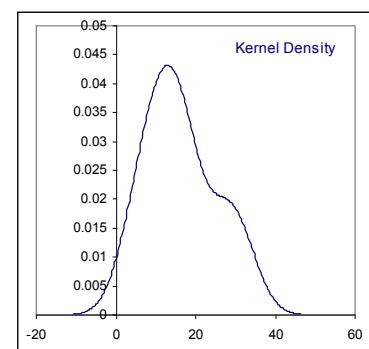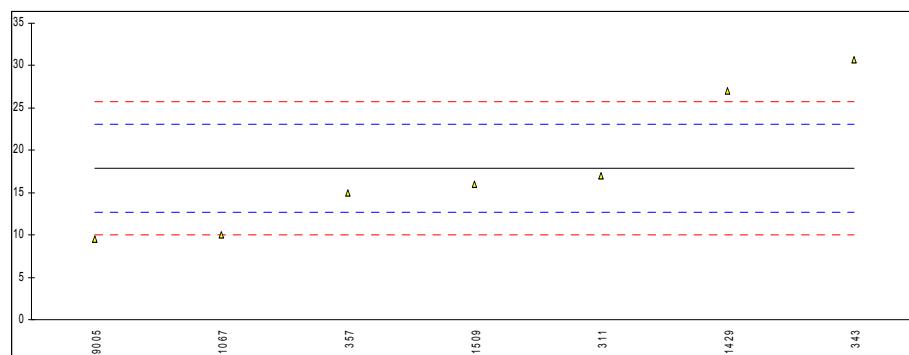
Determination of Benzaldehyde on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	31		2.11	
169		-----		-----	
171	D5135	21.6		-1.62	
174	D5135	23		-1.06	
176		-----		-----	
311	D5135	23		-1.06	
323	D5135	30		1.72	
329	D5135	20		-2.25	
333		-----		-----	
343		-----		-----	
347	D5135	32		2.51	
357	D5135	19		-2.65	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786	D5135	22.88		-1.11	
860	D5135	33		2.91	
862	D5135	26		0.13	
863		-----		-----	
865	D5135	30		1.72	
868		-----		-----	
869		-----		-----	
902	INH-83	29.2		1.40	
913		-----		-----	
1067	GC	31		2.11	
1085		-----		-----	
1117	D5135	14		-4.63	
1193		-----		-----	
1351		-----		-----	
1429		<1		<-9.79	false negative?
1509	D5135	38		4.89	
1515	in house	18		-3.05	
1921		-----		-----	
7007		-----		-----	
9005	D5135	20.5		-2.05	
9008		-----		-----	

normality	OK	
n	18	
outliers	0	
mean (n)	25.68	
st.dev. (n)	6.370	
R(calc.)	17.83	
R(Horwitz)	7.06	
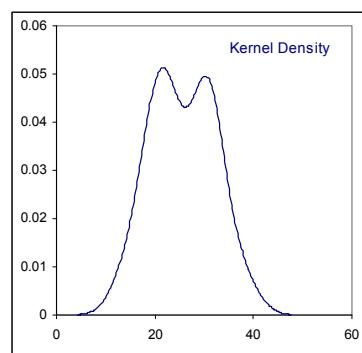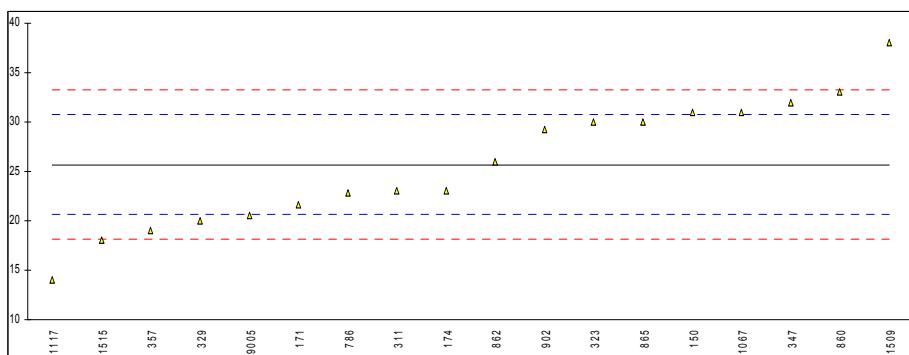
Determination of Nonaromatics on sample #1058; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	<10		-----	
169		-----		-----	
171		-----		-----	
174		-----		-----	
176		-----		-----	
311	D5135	<20		-----	
323	D5135	<10		-----	
329	D5135	<10		-----	
333		-----		-----	
343		-----		-----	
347		-----		-----	
357	D5135	<10		-----	
395		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
613		-----		-----	
786		-----		-----	
860	D5135	<10		-----	
862	D5135	<10		-----	
863	D5135	<10		-----	
865	D5135	<10		-----	
868	D5135	<10		-----	
869	D5135	<10		-----	
902		-----		-----	
913		-----		-----	
1067	GC	10		-----	
1085	in house	1.90		-----	
1117	D5135	249	C,G(0.01)	-----	first reported 155
1193		-----		-----	
1351		-----		-----	
1429		<1		-----	
1509		-----		-----	
1515	in house	2		-----	
1921		-----		-----	
7007	D5135	4.1		-----	
9005	D5135	2.9		-----	
9008		-----		-----	

normality	n.a.
n	5
outliers	1
mean (n)	(4.18)
st.dev. (n)	(3.371)
R(calc.)	(9.44)
R(lit.)	n.a.

APPENDIX 2

Reported details Aldehydes determination

Lab	date1	date2	ready-to-use	manufacturer	name	date3
150	12-Oct	30-Sep	NO		Thymol Sulfonphtalen, Sodium	13-Aug
169						
171	05-Oct	05-Oct	NO	Aldrich	Thymol blue	05-Oct
174	03-Oct	03-Oct	NO			03-Oct
176						
311	25-Sep	22-Sep	NO	ACS Reagent	Thymol blue, Sodium salt	25-Sep
323	08-Oct	20-Sep	NO	Merck		20-Sep
329	06-Oct	03-Oct	NO			05-Oct
333						
343						
347						
357	27-Sep	27-Sep	NO			05-Oct
395	01-Oct	01-Oct	NO	Merck	Thymol blue, sodium salt	01-Oct
396						
446	04-Oct	21-Sep	NO		Thymol Sulfonphtalein	06-Aug
551						
613						
786	05-Oct	04-Oct	NO	Vekton	Thymol blue	28-Sep
860	22-Sep	10-Sep	NO	Sigma-Aldrich	Thymol blue, Sodium salt	16-Jul
862	27-Sep	21-Sep	NO	Sigma-Aldrich	Thymol blue, Sodium salt	21-Sep
863	02-Oct	30-Sep	NO	Sigma-Aldrich	Thymol blue, Sodium salt	23-Dec
865	27-Sep	27-Sep	NO	Sigma-Aldrich	Thymol blue, Sodium salt	06-Sep
868	02-Oct	02-Oct	NO	Sigma-Aldrich	Thymol blue, Sodium salt	10-Sep
869	26-Sep	21-Sep	NO	Sigma-Aldrich	Thymol blue, Sodium salt	10-Aug
902						
913	12-Dec	12-Dec	NO	S.d Fine Chem	Thymol blue AR	09-Oct
1067	07-Oct	04-Oct	NO	Sigma-Aldrich	Thymol blue, Sodium salt	07-Oct
1085						
1117	17-Sep	05-Aug	NO			19-Jul
1193	25-Sep	20-Sep	NO	Guangdong Guanghua	Thymolsulphonephtalein	02-Sep
1351	26-Sep	26-Sep	NO	Sinopharm Chemical	Thymol blue	19-Sep
1429						
1509	03-Oct	29-Sep	NO	Merck	Thymol blue, Sodium salt	26-Sep
1515	16-Sep	15-Sep	YES	Alfa Aeser	Thymol blue, Sodium salt	
1921	10-Oct	08-Sep	YES	Sigma-Aldrich	Thymol blue, Sodium salt	10-Sep
7007	23-Oct	18-Oct	NO	Kanto Chemical Co	Thymol blue	18-Oct
9005	01-Oct	01-Oct	NO	Merck	Thymol blue, Sodium salt	01-Oct
9008						

Date 1   the date that the test was performed
Date 2   the preparation date of the Hydroxylamine Hydrochloride Solution
Date 3   the preparation date of the Indicator Solution

Lab	solvent	caustic	ind. amount	solv. Amount	sol. Amount	NaOH	HCl	NaOH step1	NaOH step2	blank	Normality NaOH sol.
150	Deionized water	NO	0.19	100	0.2		0.9	0.20	0.00	0.0	0.05
169											
171	Water	NO	0.1	100	3 drops	0.05	0.05	0.20	0.05	0.15	0.05
174	Methanol	NO	0.1001	100	0.2	0.15		0.1	0.1	0.1	0.05
176											
311	Water	NO	0.1	100	0.2			0.160	0.074	0.096	0.0492
323	Water	NO	0.1	100	0.2	0.1	0.05	0.300	0.100	0.182	0.0433
329	Deionized water	NO	0.1009	99.9832	5 drops		0.050	0.160	0.000	0.050	0.05004
333											
343											
347											
357	Water	NO	0.1	100	3 drops		0.1	0.16	0.04	0.06	0.05
395	Water	NO	0.1	100	0.2	0.14	0	0.24	0.18	0.10	0.04939
396											
446	Water	NO	0.1	100	0.2		0.05	0.2	0.1	0.1	0.049067
551											
613											
786	Water	YES	0.1	100	0.2		2 drops	0.26	0.125	0.08	0.05
860	Water	NO	0.10	100	0.2	0	0.02	0.32	0.10	0.17	0.05075
862	Water	NO	0.1	100	0.2	0.00	0.02	0.40	0.30	0.45 (!)	0.05088
863	Water	NO	0.1002	100	0.2	0.05	0.09	0.21	0.04	0.07	0.05950
865	Water	NO	0.1	100	0.2	0	0.03	0.23	0.06	0.03	0.05093
868	Water	NO	0.1	100	0.2	0	0.01	0.20	0.01	0.01	0.05059
869	Water	NO	0.1	100	0.2	0.00	0.05	0.26	0.10	0.10	0.04345
902											
913	Deionized water	NO	0.10	100	0.20		0.05	0.35	0.20	0.10	0.0533
1067	Water	NO	0.100	100	0.2		<0.01	0.177\ 0.159	0.009\ 0.02	0.070\ 0.069	0.0507
1085											
1117	Deionized water	YES	0.1	100	0.2			0.509	0.264	0.258	0.0101
1193	Water	YES	0.1025	250	0.20	3.5		0.175	0.050	0.050	0.05495
1351	Water	YES	0.1	100	0.2	0.05		0.30	0.30	0.90 (!)	0.00991
1429											
1509	Water	NO	0.1	100	0.2			1.044	0.020	0.227	0.0094
1515					0.2	0		1.45	0.00	0.5	0.01000
1921	Water	NO	0.1022	100	0.2		8 (!)	0.20	0.05	0.18	0.04950
7007	Deionized water	NO	0.1	100	2 drops			0.42	0.1	0.4	0.05
9005	Water	NO	0.1020	100	0.2	0.1		0.05	0	0.1	0.0507
9008											

APPENDIX 3

Number of participants per country

  1 lab in AUSTRALIA

2 labs in BELGIUM

  1 lab in BRAZIL

  1 lab in CANADA

  1 lab in FINLAND

  1 lab in FRANCE

  1 lab in INDIA

  1 lab in IRAN

2 labs in ITALY

2 labs in KUWAIT

9 labs in P.R. of CHINA

  1 lab in RUSSIA

  1 lab in SINGAPORE

2 labs in SPAIN

4 labs in THE NETHERLANDS

  1 lab in TURKEY

5 labs in U.S.A.

2 labs in UNITED KINGDOM

APPENDIX 4

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
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11   P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
12   J.N. Miller, Analyst, <u>118</u>, 455, (1993)
13   Analytical Methods Committee Technical brief, No4 January 2001.
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