

Results of Proficiency Test

Styrene

October 2017

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

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1 INTRODUCTION

Since 1995, the Institute for Interlaboratory Studies (iis) organizes a proficiency test for the analyses of Styrene. During the annual proficiency testing program 2017/2018, it was decided to continue the round robin for the analyses of Styrene in accordance with the latest applicable version of the product specification ASTM D2827.

In this interlaboratory study, 38 laboratories from 19 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2017 Styrene proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one 0.5 litre bottle with Styrene monomer (labelled #17190).

The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The necessary bulk material was obtained from a local Styrene producer. To approximately 38 kg of this batch, the components listed in table 1 were added:

<i>Component</i>	<i>Amount</i>
Benzene	1.63 g
Chloroform	83.2 mg
Dibenzothiophene	433.4 mg
Polystyrene	323 mg
Xylene (mixture)	1.54 g

Table 1: components added to the bulk materiaal

After homogenisation, 61 amber glass bottles of 0.5 L (labelled #17190) were filled. The homogeneity of subsamples of #17190 was checked by determination of Density at 20°C in accordance with ASTM D4052 and Chloride (Organic) in accordance with ASTM D5808 on stratified randomly selected samples.

	<i>Density at 20°C in kg/L</i>	<i>Chloride (Organic) in mg Cl/kg</i>
sample #17190-1	0.90622	
sample #17190-2	0.90628	2.12
sample #17190-3	0.90629	1.99
sample #17190-4	0.90628	2.03
sample #17190-5	0.90629	2.26
sample #17190-6	0.90629	2.04
sample #17190-7	0.90628	2.22
sample #17190-8	0.90625	1.98

Table 2: homogeneity test results of subsamples #17190

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

	<i>Density at 20°C in kg/L</i>	<i>Chloride (Organic) in mg Cl/kg</i>
r (observed)	0.00007	0.31
ref. test method	ISO 12185:96	ASTM D5808:09a
0.3 x R (ref. test method)	0.00015	0.39

Table 3: evaluation of the repeatabilities of subsamples #17190

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories, 1 bottle of 0.5 L (labelled #17190) was sent on September 20, 2017.

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 requested to determine on sample #17190: Aldehydes as benzaldehyde, Appearance, Chloride (Organic), Colour Pt/Co, Density at 20°C, Inhibitor as TBC, Peroxide as H₂O₂, Polymer, Sulphur, Water (coulometric KF titration), Purity and the Impurities by GC; Benzene, Toluene, Ethylbenzene, m- & p- Xylenes, Iso-propylbenzene (Cumene), o-Xylene, n-Propylbenzene, m- & p-Ethyltoluenes, α-Methylstyrene, 1,2-Diethylbenzene, sum α-Methylstyrene and 1,2-Diethylbenzene, Phenylacetylene, 3- & 4-Dimethylstyrenes, sum Phenylacetylene and 3- & 4-Dimethylstyrenes, Benzaldehyde and Nonaromatics.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.

Shortly after the deadline, the available test results were screened for suspect data. A test 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 reported test results (no reanalysis). Additional or corrected test results are used for

data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test 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, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

Finally, the reproducibilities were calculated from the standard deviations by multiplying them 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 test results are plotted. The corresponding laboratory numbers are on 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 reference test method. 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. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used. 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 whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

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

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

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

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the sample to the laboratory in Brazil. This laboratory received the sample late and reported after the final reporting date. Not all participants were able to report all requested parameters. Finally, 38 laboratories did report 524 numerical test results. Observed were 24 outlying test results, which is 4.6%. In proficiency studies outlier percentages of 3 - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care.

4.1 EVALUATION PER TEST

In this section, the reported test results are discussed per test. The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the reported data. The abbreviations, used in these tables, are listed in appendix 4.

In iis PT reports, ASTM test methods are referred to with a number (e.g. D1209) and an added designation for the year that the test method was adopted or revised (e.g. D1209:05). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1209:05(2011)). In the tables of Appendix 1 only the test method number and year of adoption or revision will be used.

Aldehydes as benzaldehyde: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D2119:09.

The large variation was also observed in previous years. A possible cause of this large variation could be the use of a different molarity of titrant than described in ASTM D2119 (eg 0.01N NaOH instead of 0.05N NaOH).

There is a suspicion that the concentration of 0.05N NaOH as described in ASTM D2119 is too strong for aldehyde concentrations <100 mg/kg and that this may result in a positive bias. Two laboratories reported to have used ASTM D7704 as test method. ASTM D7704 describes the use of 0.02N NaOH. One of these two laboratories reported not only the test results of the ASTM D7704 method but also the test results using ASTM D2119 with two different molarities of NaOH: ASTM D2119 – 0.05 N NaOH (146 mg/kg) and ASTM D2119 modified - 0.01 N NaOH (117 mg/kg); compare with ASTM D7704 - 0.02N (116 mg/kg).

None of the participants reported a test result lower than the test result of Benzaldehyde content by GC.

Appearance: Although not mentioned in ASTM D2827:13, the specification of Styrene; it is advised to use test method ASTM E2680:09(2015)e1 for the appearance determination. All participants agreed about the appearance of sample #17190 to be 'clear and bright' or 'pass'. Participants who used ASTM E2680 should report the appearance as 'pass' or as 'fail' dependent on the appearance of the product.

Chloride, Organic: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D5808:09a(2014).

The average recovery of Inorganic Chloride (theoretical increment of 1.95 mg Cl/kg) may be good: "less than 128%" (the actual blank Chloride content is unknown).

Colour Pt/Co: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5386:16 and with ASTM D1209:05(2011).

Density at 20°C: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO 12185:96 and with ASTM D4052:02e1).

Inhibitor as TBC: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D4590:13.

Peroxides as H₂O₂: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D2340:13.

Polymer: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D2121-A:16.
The average recovery of Polymer (theoretical increment of 8.49 mg Polymer/kg) may be good: "less than 129%" (the actual blank Polymer content is unknown).

Sulphur: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D5453:16e1.

The average recovery of Sulphur (theoretical increment of 1.98 mg Sulphur /kg) may be good: "less than 129%" (the actual blank Sulphur content is unknown).

Water, coulometric KF titration: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirement of ASTM E1064:16.

Purity by GC: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5135:16e1.

Benzene: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5135:16e1.
The average recovery of Benzene (theoretical increment of 42.78 mg Benzene/kg) may be good: "less than 127%" (the actual blank Benzene content is unknown).

The high level of Benzene (42.78 mg/kg) was not intended, but was caused by a human error during the spike process. The spike process is adapted to avoid this kind of errors in future.

Ethylbenzene: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5135:16e1.

o-Xylene: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D5135:16e1.

α -Methylstyrene: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D5135:16e1.

1,2-Diethylbenzene: Three laboratories reported a numeric test result and five laboratories reported a test value as "<10" or "N.D.". Therefore, no significant conclusions could be drawn.

Sum α-Methylstyrene & 1,2-Diethylbenzene: Only six laboratories reported a numerical test result for the sum of the two components. However, this determination may not be problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D5135:16e1.

The level of the sum of the two components is equal to the level of α-Methylstyrene as the level of 1,2-Diethylbenzene is below the detection limit.

Benzaldehyde: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D5135:16e1.

Nonaromatics: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5135:16e1.

Toluene, m- & p-Xylenes, Iso-Propylbenzene, n-Propylbenzene, m- & p-Ethyltoluene, Phenylacetylene, 3- & 4-Methylstyrenes, Sum Phenylacetylene and 3- & 4-Methylstyrenes: The majority of the participants reported a test result below the application range of ASTM D5135:16e1 (range impurity concentrations 0.001-1.00 %). Therefore, no significant conclusions could be drawn.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The target reproducibilities derived from reference test methods (in casu ASTM reference test methods) or previous proficiency tests are compared in the next table.

Parameter	unit	n	average	2.8 *sd	R (lit)
Aldehydes as benzaldehyde	mg/kg	28	132.4	86.7	53.0
Appearance		34	Pass	n.a.	n.a.
Chloride, Organic	mg/kg	9	2.5	0.8	1.3
Colour Pt/Co		33	9.9	2.7	6.1
Density at 20°C	kg/L	32	0.9063	0.0002	0.0005
Inhibitor as TBC	mg/kg	35	6.1	2.4	2.8
Peroxides as H ₂ O ₂	mg/kg	27	22.2	15.4	13
Polymer	mg/kg	30	11.0	5.6	9.7
Sulphur	mg/kg	24	2.6	1.1	1.2

Table 4a: reproducibilities of tests on sample #17190

Parameter	unit	n	average	2.8 *sd	R (lit)
Water, coulometric KF titration	mg/kg	33	154	20	44
Purity by GC	%M/M	34	99.96	0.02	0.03
Benzene	mg/kg	32	54.3	11.3	23.3
Ethylbenzene	mg/kg	32	87.2	9.1	18.5
o-Xylene	mg/kg	31	54.8	10.1	13.7
α-Methylstyrene	mg/kg	33	111	26	33
1,2-Diethylbenzene	mg/kg	8	<10	n.a.	n.a.
sum α-Methylstyrene & 1,2-Diethylbenzene	mg/kg	6	106	32	32
Benzaldehyde	mg/kg	16	101	28	38
Nonaromatics	mg/kg	9	17.6	14.5	20.5

Table 4b: reproducibilities of tests on sample #17190 (cont.)

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2017 WITH PREVIOUS PTS

	October 2017	October 2016	October 2015	September 2014	September 2013
Number of reporting labs	38	38	37	36	34
Number of test results reported	524	521	447	510	474
Number of statistical outliers	24	14	20	33	29
Percentage outliers	4.6%	2.7%	4.5%	6.5%	6.1%

Table 5: 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 reference test methods. The conclusions are given in the following table:

Determination	October 2017	October 2016	October 2015	September 2014	September 2013
Aldehydes as benzaldehyde	-	-	--	--	--
Chloride, Organic	+	+	n.e.	+	+/-
Colour Pt/Co	++	+	++	++	++
Density at 20°C	++	++	++	++	++
Inhibitor as TBC	+	+/-	+	--	--
Peroxides as H ₂ O ₂	-	-	-	--	--
Polymer	+	-	++	--	--
Sulphur	+/-	+	n.e.	+	++

Table 6a: comparison of overall performance against the requirements of the reference test method per parameter

Determination	October 2017	October 2016	October 2015	September 2014	September 2013
Water, coulometric KF titration	++	+	+	++	--
Purity by GC	+	+/-	+	(--)	++
Benzene	++	-	n.e.	--	--
Toluene	n.e.	n.e.	n.e.	--	n.e.
Ethylbenzene	++	+	++	++	++
m- & p-Xylenes	n.e.	n.e.	++	++	--
iso-Propylbenzene (Cumene)	n.e.	n.e.	-	--	n.e.
o-Xylene	+	n.e.	++	--	++
n-Propylbenzene	n.e.	+	(--)	--	+
m- & p-Ethyltoluenes	n.e.	+	(-)	++	n.e.
α -Methylstyrene	+	--	++	--	--
1,2-Diethylbenzene	n.e.	(--)	n.e.	(--)	n.e.
sum α -Methylstyrene & 1,2-Diethylbenzene	+/-	+/-	++	n.e.	n.e.
Phenylacetylene	n.e.	n.e.	n.e.	--	--
3- & 4-Methylstyrenes	n.e.	n.e.	n.e.	n.e.	--
sum Phenylacetylene & 3- & 4-Methylstyrenes	n.e.	+	n.e.	n.e.	n.e.
Benzaldehyde	+	-	-	--	--
Nonaromatics	+	--	n.e.	--	n.e.

Table 6b: comparison of overall performance against the requirements of the reference test method per parameter (cont.)

Between brackets is near or below the detection limit of the test method.

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

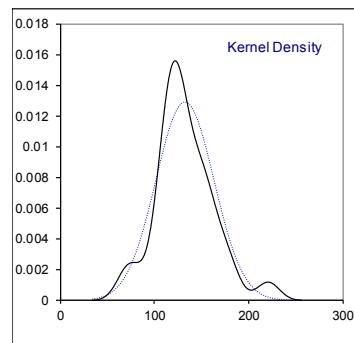
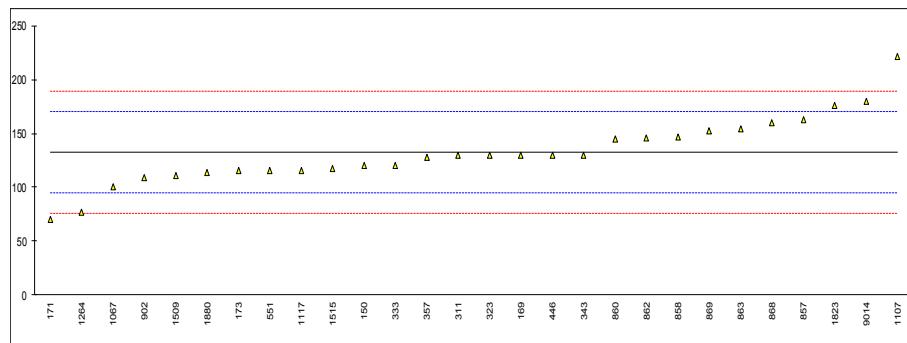
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Aldehydes as benzaldehyde on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D2119	120		-0.66	
169	D2119	130		-0.13	
171	D2119	70		-3.30	
173	D2119	116		-0.87	
273		----		-----	
311	D2119	130		-0.13	
323	D2119	130		-0.13	
333	D2119	120		-0.66	
343	D2119	130		-0.13	
347		----		-----	
357	D2119	128		-0.23	
446	D2119	130		-0.13	
551	D2119	116		-0.87	
613		----		-----	
857	D2119	163		1.62	
858	D2119	147		0.77	
860	D2119	145		0.66	
862	D2119	146		0.72	
863	D2119	154		1.14	
868	D2119	160		1.46	
869	D2119	152		1.03	
902	D2119	109		-1.24	
913		----		-----	
1067	D2119	100		-1.71	
1107	D2119	221		4.68	
1117	D7704	116		-0.87	reported also D2119: 0.05 mol/l NaOH = 146, 0.01 mol/l NaOH = 117 mg/kg
1135		----		-----	
1201		----		-----	
1264	D2119	77.0		-2.93	
1509	D2119	111.3		-1.12	
1515	D2119	117		-0.82	
1823	D2119	176		2.30	
1866		----		-----	
1880	D7704	114		-0.97	
6100		----		-----	
7014		----		-----	
9008		----		-----	
9014	D2119	180		2.51	
normality					
n		suspect			
outliers		28			
mean (n)		0			
st.dev. (n)		132.44			
R(calc.)		30.952			
st.dev.(D2119:09)		86.66			
R(D2119:09)		18.919			
R(D2119:09)		52.98			

Compare R(D7704:16) = 106 (at conc. 129 mg/kg)



Determination of Appearance on sample #17190;

lab	method	value	mark	z(targ)	remarks
150	D4176	Clear and Bright	-----		
169	Visual	Pass	-----		
171	E2680	Pass	-----		
173	D4176	Pass	-----		
273	Visual	Pass	-----		
311	E2680	pass	-----		
323	Visual	C&B	-----		
333		-----	-----		
343	E2680	PASS	-----		
347	E2680	PASS	-----		
357	E2680	Pass	-----		
446	E2680	Pass	-----		
551	E2680	Pass	-----		
613	E2680	Pass	-----		
857	E2680	Pass	-----		
858	E2680	Pass	-----		
860	E2680	Pass	-----		
862	E2680	pass	-----		
863	E2680	Pass	-----		
868	E2680	PASS	-----		
869	Visual	Clear and bright	-----		
902	E2680	Pass	-----		
913	E2680	Passes Test	-----		
1067	Visual	Clear	-----		
1107	E2680	passes	-----		
1117	D4176	pass	-----		
1135	Visual	CFSM	-----		
1201	Visual	Br&Cl	-----		
1264	E2680	Pass	-----		
1509	E2680	Clear & FFSM	-----		
1515	E2680	Pass	-----		
1823	D4176	Pass	-----		
1866		-----	-----		
1880	Visual	Pass	-----		
6100		-----	-----		
7014	Visual	Clear	-----		
9008	Visual	Clear liquid	-----		
9014		-----	-----		
n		34			
mean (n)		Pass			

25 labs reported Pass

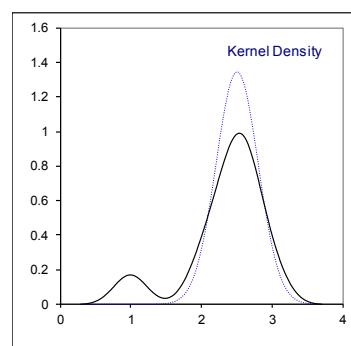
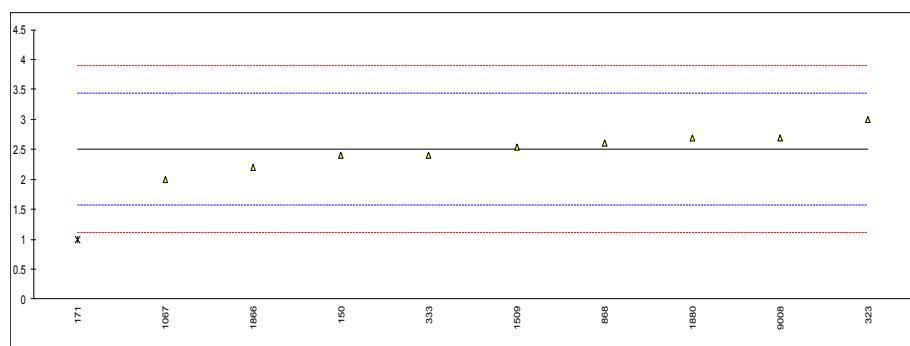
4 labs reported Clear and Bright

3 labs reported Clear

2 labs reported various descriptions indicating that the liquid is clear and free of suspended matter

Determination of Chloride, Organic on sample #17190; results in mg/kg

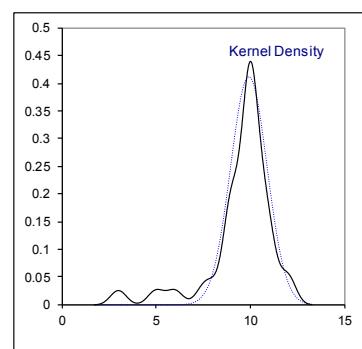
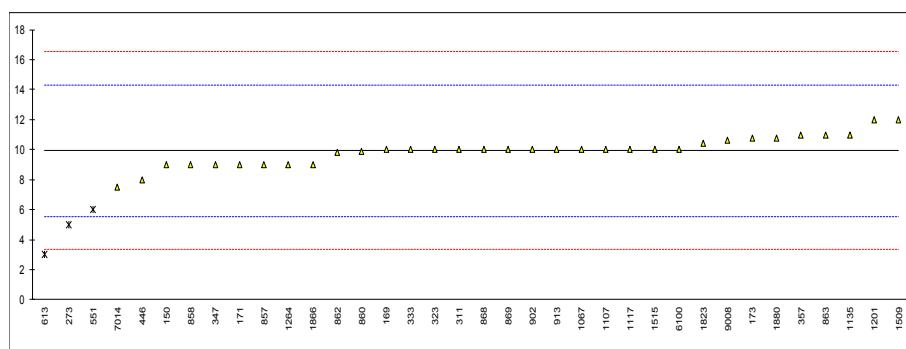
lab	method	value	mark	z(targ)	remarks
150	D7359	2.4		-0.22	
169		----		----	
171	D5808	1	D(0.05)	-3.24	
173		----		----	
273		----		----	
311	D5808	<1		<-3.24	false negative test result?
323	D5808	3		1.07	
333	D5808	2.4		-0.22	
343		----		----	
347		----		----	
357		----		----	
446		----		----	
551		----		----	
613		----		----	
857		----		----	
858		----		----	
860		----		----	
862		----		----	
863		----		----	
868	D5808	2.6		0.21	
869		----		----	
902		----		----	
913		----		----	
1067	UOP779	2		-1.09	
1107		----		----	
1117		----		----	
1135	D5808	<1		<-3.24	false negative test result?
1201		----		----	
1264		----		----	
1509	D5808	2.54		0.08	
1515		----		----	
1823		----		----	
1866	D5808	2.2		-0.66	
1880	D7359	2.7		0.42	
6100		----		----	
7014		----		----	
9008	D5808	2.7		0.42	
9014		----		----	
<hr/>					
normality					
n		OK			
outliers		n			
mean (n)		9			
st.dev. (n)		1			spike
R(calc.)		2.50			1.95 (recovery <128%)
st.dev.(D5808:09a)		0.296			
R(D5808:09a)		0.83			
R(D5808:09a)		0.464			
R(D5808:09a)		1.30			



Determination of Colour Pt/Co on sample #17190;

lab	method	value	mark	z(targ)	remarks
150	D5386	9		-0.43	
169	D5386	10.0		0.03	
171	D5386	9		-0.43	
173	D5386	10.8		0.40	
273	D1209	5	R(0.01)	-2.25	
311	D5386	10		0.03	
323	D5386	10		0.03	
333	D5386	10		0.03	
343		----		----	
347	D5386	9		-0.43	
357	D5386	11		0.49	
446	D5386	8		-0.88	
551	D1209	6	R(0.05)	-1.79	
613	D1209	3	R(0.01)	-3.16	
857	D5386	9		-0.43	
858	D5386	9		-0.43	
860	D5386	9.9		-0.02	
862	D5386	9.8		-0.06	
863	D1209	11		0.49	
868	D5386	10		0.03	
869	D1209	10		0.03	
902	D5386	10		0.03	
913	D5386	10		0.03	
1067	D1209	10		0.03	
1107	D5386	10.0		0.03	
1117	D1209	10		0.03	
1135	D5386	11		0.49	
1201	D5386	12		0.94	
1264	D1209	9		-0.43	
1509	D1209	12		0.94	
1515	D1209	10		0.03	
1823	D5386	10.4		0.21	
1866	D5386	9		-0.43	
1880	D5386	10.8		0.40	
6100	D5386	10		0.03	
7014	D1209	7.5		-1.11	
9008	D5386	10.6		0.30	
9014		----		----	
normality		OK			
n		33			
outliers		3			
mean (n)		9.93			
st.dev. (n)		0.967			
R(calc.)		2.71			
st.dev.(D5386:16)		2.191			
R(D5386:16)		6.14			

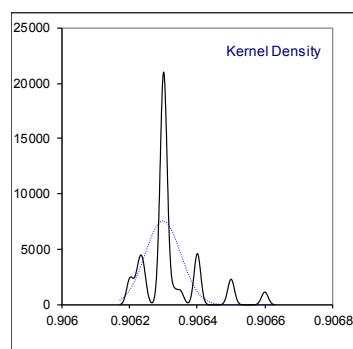
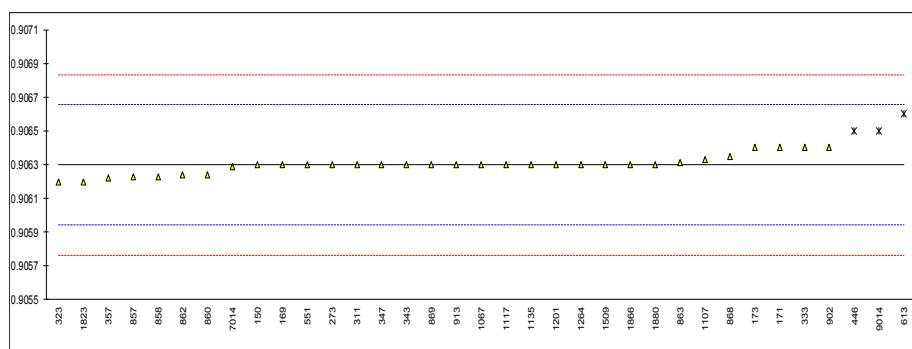
Compare R(D1209:05(2011)) = 7



Determination of Density at 20°C on sample #17190; results in kg/L

lab	method	value	mark	z(targ)	remarks
150	D4052	0.9063		0.01	
169	D4052	0.9063		0.01	
171	D4052	0.9064		0.57	
173	D4052	0.9064		0.57	
273	D4052	0.9063		0.01	
311	D4052	0.9063		0.01	
323	ISO12185	0.9062		-0.55	
333	ISO12185	0.9064		0.57	
343	D4052	0.9063		0.01	
347	D4052	0.9063		0.01	
357	D4052	0.90622		-0.44	
446	D4052	0.9065	C,R(0.05)	1.13	first reported: 0.4052
551	D4052	0.9063		0.01	
613	D4052	0.9066	R(0.01)	1.69	
857	D4052	0.90623		-0.38	
858	D4052	0.90623		-0.38	
860	D4052	0.90624		-0.32	
862	D4052	0.90624		-0.32	
863	D4052	0.90631		0.07	
868	D4052	0.90635		0.29	
869	D4052	0.90630		0.01	
902	D4052	0.9064		0.57	
913	D4052	0.9063		0.01	
1067	D4052	0.9063		0.01	
1107	D4052	0.90633		0.18	
1117	D4052	0.9063		0.01	
1135	ISO12185	0.9063		0.01	
1201	D4052	0.9063		0.01	
1264	D4052	0.9063		0.01	
1509	D4052	0.90630		0.01	
1515		-----		-----	
1823	D4052	0.9062		-0.55	
1866	D4052	0.9063		0.01	
1880	D4052	0.9063		0.01	
6100		-----		-----	
7014	D4052	0.90629		-0.04	
9008		-----		-----	
9014	D4052	0.9065	R(0.05)	1.13	
normality					
n		OK			
outliers		3			
mean (n)		0.90630			
st.dev. (n)		0.000053			
R(calc.)		0.00015			
st.dev.(ISO12185:96)		0.000179			
R(ISO12185:96)		0.0005			

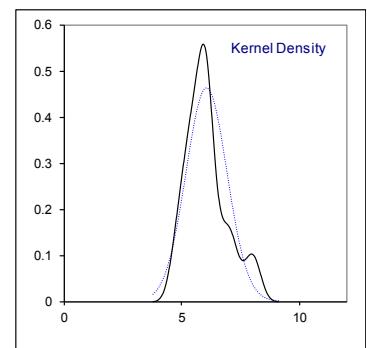
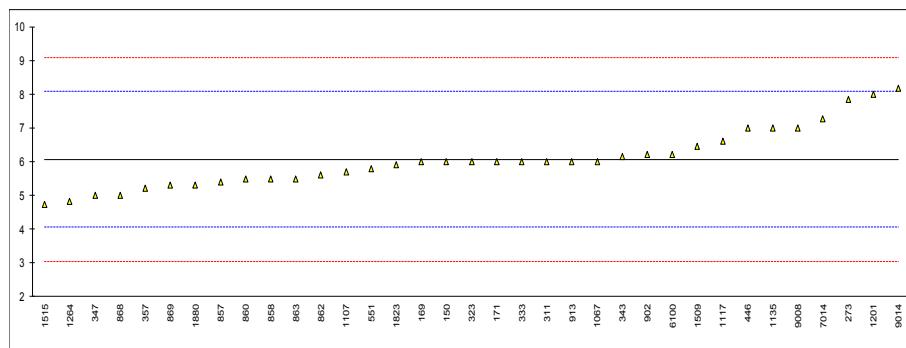
Compare R(D4052:02e1) = 0.00050



Determination of Inhibitor as TBC on sample #17190; results in mg/kg

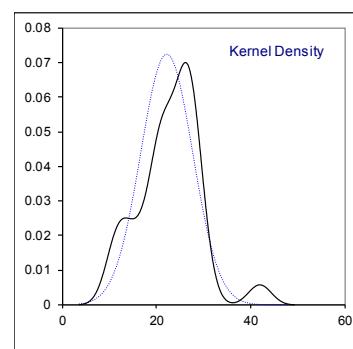
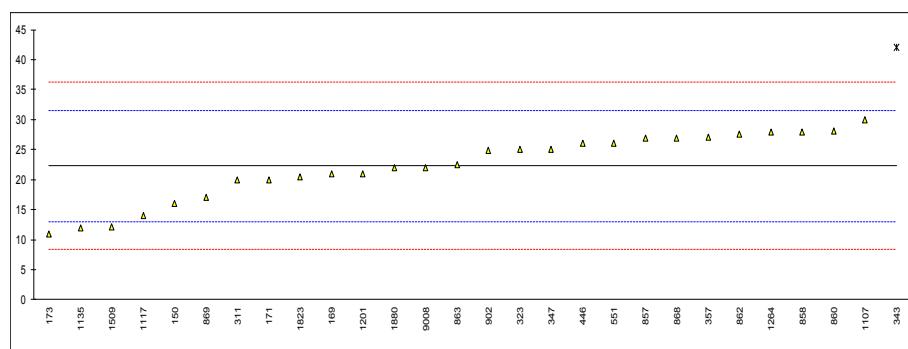
lab	method	value	mark	z(targ)	remarks
150	D4590	6		-0.06	
169	D4590	6.0		-0.06	
171	D4590	6		-0.06	
173		----		----	
273	D4590	7.83		1.76	
311	D4590	6		-0.06	
323	D4590	6		-0.06	
333	D4590	6		-0.06	
343	D4590	6.1518		0.09	
347	D4590	5		-1.06	
357	D4590	5.2		-0.86	
446	D4590	7		0.93	
551	D4590	5.8		-0.26	
613		----		----	
857	D4590	5.4		-0.66	
858	D4590	5.5		-0.56	
860	D4590	5.5		-0.56	
862	D4590	5.6		-0.46	
863	D4590	5.5		-0.56	
868	D4590	5.0		-1.06	
869	D4590	5.3		-0.76	
902	D4590	6.2		0.14	
913	D4590	6.0		-0.06	
1067	D4590	6		-0.06	
1107	D4590	5.7		-0.36	
1117	D4590	6.60		0.54	
1135	D4590	7		0.93	
1201	D4590	8.0		1.93	
1264	D4590	4.82		-1.24	
1509	D4590	6.44		0.38	
1515	D4590	4.73815		-1.32	
1823	D4590	5.924		-0.14	
1866		----		----	
1880	D4590	5.3		-0.76	
6100	D4590	6.2222		0.16	
7014	D4590	7.28		1.21	
9008	In house	7.0		0.93	
9014		8.16	C	2.09	first reported: 10.52

normality OK
n 35
outliers 0
mean (n) 6.062
st.dev. (n) 0.8608
R(calc.) 2.410
st.dev.(D4590:13) 1.0042
R(D4590:13) 2.812



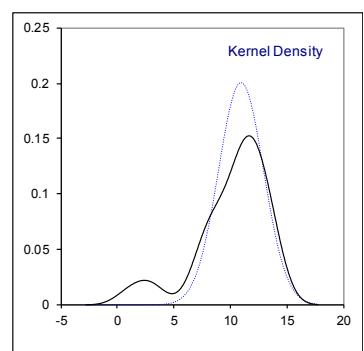
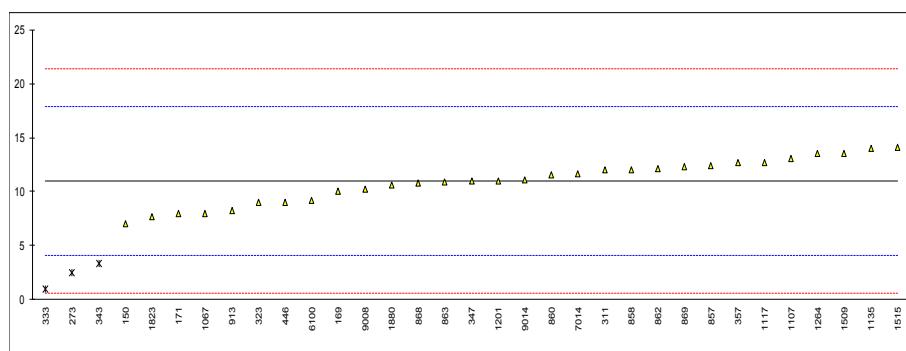
Determination of Peroxides as H₂O₂ on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D2340	16	C	-1.35	first reported: 51
169	D2340	21		-0.27	
171	D2340	20		-0.48	
173	D2340	11		-2.42	
273		----		----	
311	D2340	20		-0.48	
323	D2340	25		0.59	
333		----		----	
343	D2340	42	C,R(0.05)	4.25	first reported: 54
347	D2340	25		0.59	
357	D2340	27.1		1.04	
446	D2340	26		0.81	
551	D2340	26		0.81	
613		----		----	
857	D2340	27		1.02	
858	D2340	28		1.24	
860	D2340	28.1		1.26	
862	D2340	27.6		1.15	
863	D2340	22.5		0.05	
868	D2340	27.0		1.02	
869	D2340	17.0		-1.13	
902	D2340	24.9		0.57	
913		----		----	
1067		----		----	
1107	D2340	30		1.67	
1117	D2340	14.0		-1.78	
1135	D2340	12		-2.21	
1201	D2340	21		-0.27	
1264	D2340	27.90		1.22	
1509	D2340	12.2		-2.16	
1515		----		----	
1823	D2340	20.432		-0.39	
1866		----		----	
1880	D2340	22.0		-0.05	
6100		----		----	
7014		----		----	
9008	D2340	22		-0.05	
9014		----		----	
normality		OK			
n		27			
outliers		1			
mean (n)		22.249			
st.dev. (n)		5.5153			
R(calc.)		15.443			
st.dev.(D2340:13)		4.6429			
R(D2340:13)		13			



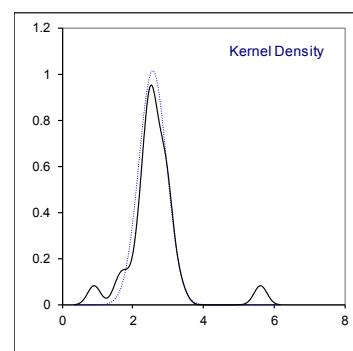
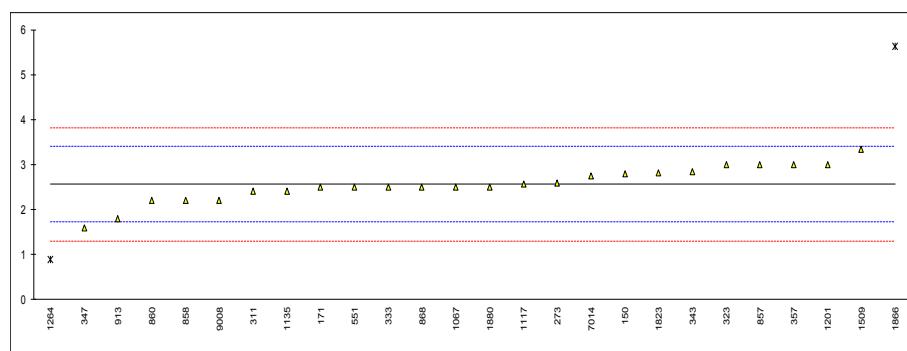
Determination of Polymer on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D2121-method A	7		-1.16	
169	D2121-method A	10.047		-0.27	
171	D2121-method A	8		-0.87	
173		----		----	
273	D2121-method A	2.47	R(0.05)	-2.47	
311	D2121-method A	12		0.29	
323	D2121-method A	9		-0.58	
333	D2121-method A	1	R(0.05)	-2.89	
343	D2121-method A	3.3371	R(0.05)	-2.22	
347	INH-CM09	11		0.00	
357	D2121-method A	12.7		0.50	
446	D2121-method A	9		-0.58	
551		----		----	
613		----		----	
857	D2121-method A	12.4		0.41	
858	D2121-method A	12		0.29	
860	D2121-method A	11.6		0.18	
862	D2121-method A	12.1		0.32	
863	D2121-method A	10.9		-0.03	
868	D2121-method A	10.8		-0.05	
869	D2121-method A	12.3		0.38	
902		----		----	
913	D2121-method A	8.28		-0.78	
1067	D2121-method A	8		-0.87	
1107	D2121-method A	13.1		0.61	
1117	D2121-method A	12.73		0.51	
1135	D2121-method A	14		0.87	
1201	D2121-method A	11		0.00	
1264	D2121-method A	13.56		0.75	
1509	D2121-method A	13.57		0.75	
1515	D2121-method A	14.0725		0.89	
1823	D2121-method A	7.67	C	-0.96	first reported: 22.392
1866		----		----	
1880	D2121-method A	10.6		-0.11	
6100	D2121-method A	9.1717		-0.53	
7014	D2121-method A	11.69		0.20	
9008	In house	10.2		-0.23	
9014	D2121-method A	11.11		0.04	
normality					
n					
outliers					
mean (n)					
st.dev. (n)					
R(calc.)					
st.dev.(D2121-A:16)					
R(D2121-A:16)					
spike					
8.49 (recovery <129%)					



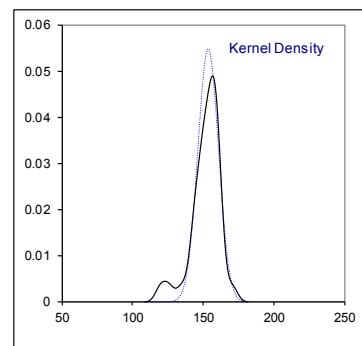
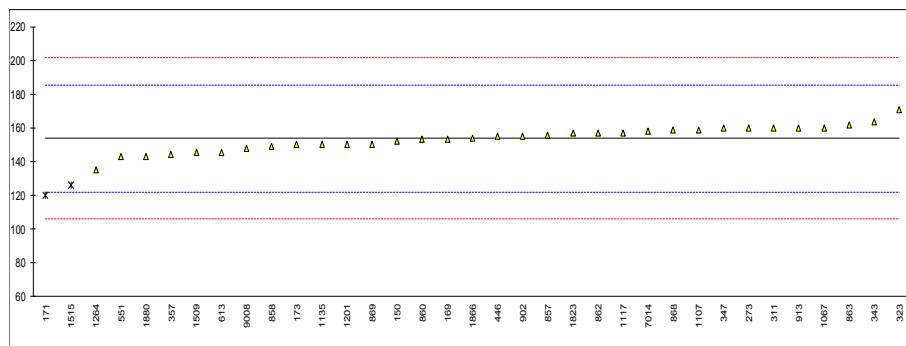
Determination of Sulphur on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5453	2.8		0.56	
169		----		----	
171	D5453	2.5		-0.15	
173		----		----	
273	D5453	2.60		0.09	
311	D5453	2.4		-0.39	
323	D5453	3		1.04	
333	D5453	2.5		-0.15	
343	D5453	2.84		0.66	
347	D5453	1.6		-2.30	
357	D5453	3.0		1.04	
446		----		----	
551	D5453	2.5		-0.15	
613		----		----	
857	D3120	3.0		1.04	
858	D5453	2.2		-0.87	
860	D3120	2.2		-0.87	
862		----		----	
863		----		----	
868	D5453	2.5		-0.15	
869		----		----	
902		----		----	
913	D5453	1.8		-1.82	
1067	D5453	2.5		-0.15	
1107		----		----	
1117	D5453	2.57		0.02	
1135	D5453	2.4		-0.39	
1201	D5453	3.0		1.04	
1264	D5453	0.9	C,R(0.05)	-3.97	first reported: 1.1
1509	D5453	3.33		1.83	
1515		----		----	
1823	D5453	2.82		0.61	
1866	D5453	5.62	R(0.01)	7.29	
1880	D5453	2.5		-0.15	
6100		----		----	
7014	D5453	2.76		0.47	
9008	D5453	2.2		-0.87	
9014		----		----	
normality		OK			
n		24			
outliers		2			<u>spike</u>
mean (n)		2.563			1.98 (recovery <129%)
st.dev. (n)		0.3941			
R(calc.)		1.104			
st.dev.(D5453:16e1)		0.4194			
R(D5453:16e1)		1.174			



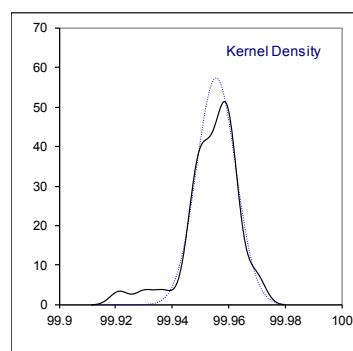
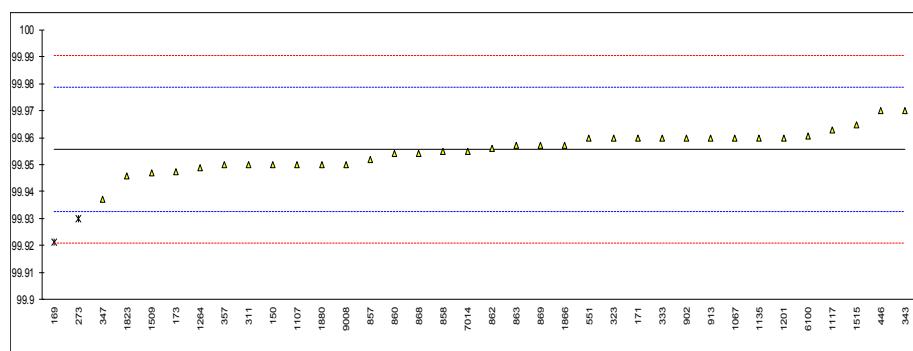
Determination of Water, coulometric KF titration on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	E1064	152		-0.11	
169	E1064	153		-0.05	
171	E1064	120	R(0.05)	-2.13	
173	D6304	150		-0.24	
273	E203	160		0.39	
311	E1064	160		0.39	
323	E1064	171		1.09	
333		-----		-----	
343	E1064	163.25		0.60	
347	E1064	160		0.39	
357	E1064	144		-0.61	
446	E1064	155		0.08	
551	E1064	143		-0.68	
613	E203	145.5		-0.52	
857	E1064	156		0.14	
858	E1064	149		-0.30	
860	E1064	153		-0.05	
862	E1064	157		0.20	
863	E1064	162		0.52	
868	E1064	159		0.33	
869	E1064	150.5		-0.21	
902	E1064	155		0.08	
913	E1064	160.0		0.39	
1067	E1064	160		0.39	
1107	E1064	159		0.33	
1117	E1064	157		0.20	
1135	E1064	150		-0.24	
1201	E1064	150		-0.24	
1264	E1064	135		-1.18	
1509	E1064	145.2		-0.54	
1515	E1064	126	R(0.05)	-1.75	
1823	E1064	156.62		0.18	
1866	E1064	154		0.02	
1880	E1064	143		-0.68	
6100		-----		-----	
7014	E1064	157.9		0.26	
9008	E1064	148		-0.36	
9014		-----		-----	
normality		OK			
n		33			
outliers		2			
mean (n)		153.757			
st.dev. (n)		7.2729			
R(calc.)		20.364			
st.dev.(E1064:16)		15.8730			
R(E1064:16)		44.444			



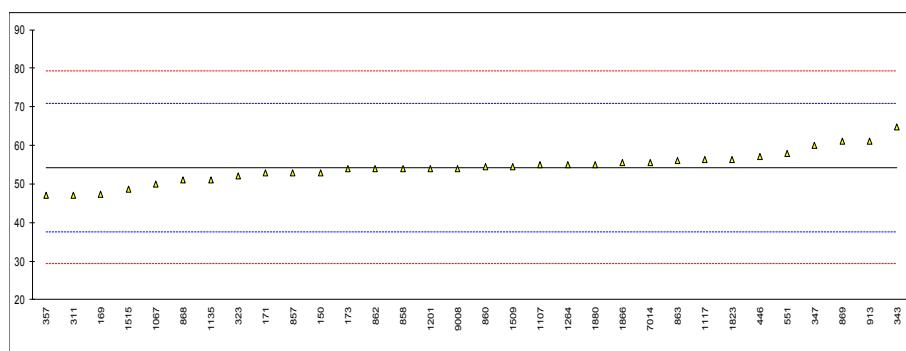
Determination of Purity by GC on sample #17190; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	D5135	99.95		-0.49	
169	D5135	99.92117	R(0.01)	-2.99	
171	D5135	99.96		0.38	
173	D5135	99.9473		-0.72	
273	D5135	99.93	R(0.05)	-2.22	
311	D5135	99.95		-0.49	
323	D5135	99.96		0.38	
333	D5135	99.96		0.38	
343	D5135	99.97		1.25	
347	D5135	99.937		-1.62	
357	D5135	99.950		-0.49	
446	D5135	99.97		1.25	
551	D5135	99.96		0.38	
613		-----		-----	
857	D5135	99.952		-0.31	
858	D5135	99.955		-0.05	
860	D5135	99.954		-0.14	
862	D5135	99.956		0.03	
863	D5135	99.957		0.12	
868	D5135	99.954		-0.14	
869	D5135	99.957		0.12	
902	D5135	99.96		0.38	
913	D5135	99.96		0.38	
1067	In house	99.96		0.38	
1107	D5135	99.95		-0.49	
1117	D5135	99.963		0.64	
1135	D5135	99.96		0.38	
1201	D5135	99.96		0.38	
1264	D5135	99.9488		-0.59	
1509	D5135	99.947		-0.75	
1515	D5135	99.9649		0.80	
1823	D5135	99.946	C	-0.84	first reported: 99.955
1866	D5135	99.957		0.12	
1880	D5135	99.950		-0.49	
6100	D5135	99.9606		0.43	
7014	D5135	99.955		-0.05	
9008	D5135	99.95		-0.49	
9014		-----		-----	
normality					
OK					
n					
34					
outliers					
2					
mean (n)					
99.9556					
st.dev. (n)					
0.00695					
R(calc.)					
0.0195					
st.dev.(D5135:16e1)					
0.01153					
R(D5135:16e1)					
0.0323					

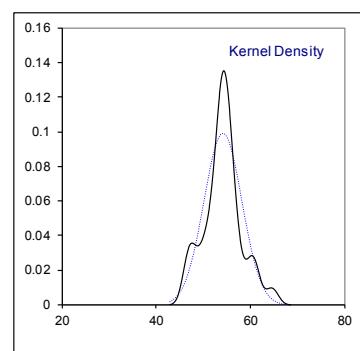


Determination of Benzene on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	53		-0.16	
169	D5135	47.4		-0.83	
171	D5135	53		-0.16	
173	D7504	54		-0.04	
273		----		----	
311	D6229	47.1		-0.87	
323	D5135	52		-0.28	
333		----		----	
343	INH-1456	64.75		1.25	
347	D5135	60		0.68	
357	D5135	47		-0.88	
446	D5135	57		0.32	
551	D5135	58		0.44	
613		----		----	
857	D5135	53		-0.16	
858	D5135	54		-0.04	
860	D5135	54.4		0.01	
862	D5135	54		-0.04	
863	D5135	56		0.20	
868	D5135	51		-0.40	
869	D5135	61		0.80	
902		----		----	
913	D5135	61.0		0.80	
1067	In house	50		-0.52	
1107	D5135	55		0.08	
1117	D5135	56.2		0.23	
1135	D5135	51		-0.40	
1201	D5135	54		-0.04	
1264	D5135	55.0		0.08	
1509	In house	54.4		0.01	reported: not within spec. method
1515	INH-2922	48.78		-0.67	reported: not within spec. method, also reported D5135: 50 mg/kg
1823		56.4	C	0.25	first reported: 56.1
1866	D5135	55.41		0.13	
1880	D4534	55		0.08	
6100		----		----	
7014	D5135	55.5		0.14	
9008	D5135	54		-0.04	
9014		----		----	
normality		OK			
n		32			
outliers		0			
mean (n)		54.32			
st.dev. (n)		4.023			
R(calc.)		11.27			
st.dev.(D5135:16e1)		8.315			
R(D5135:16e1)		23.28			

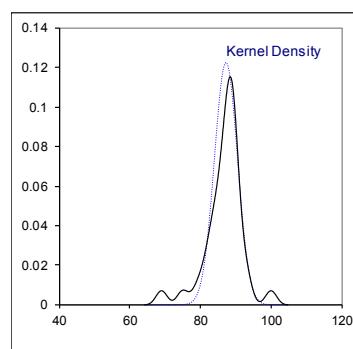
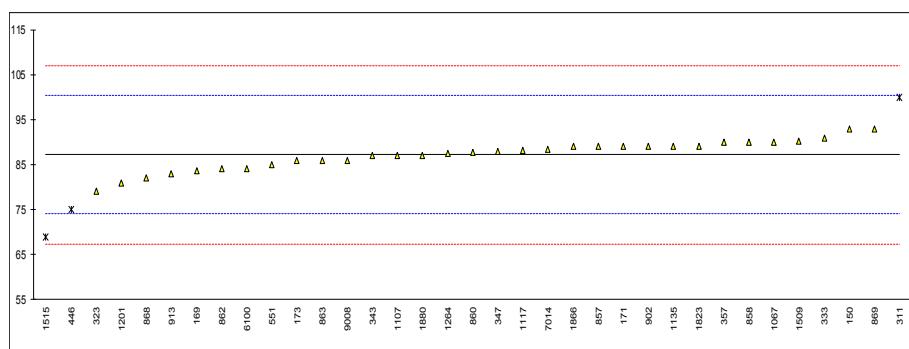


spike
42.78 (recovery <127%)



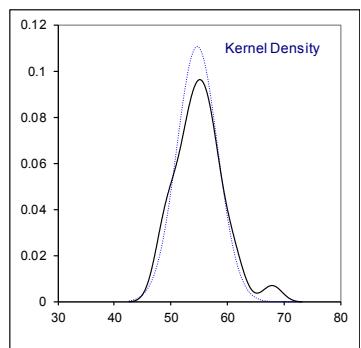
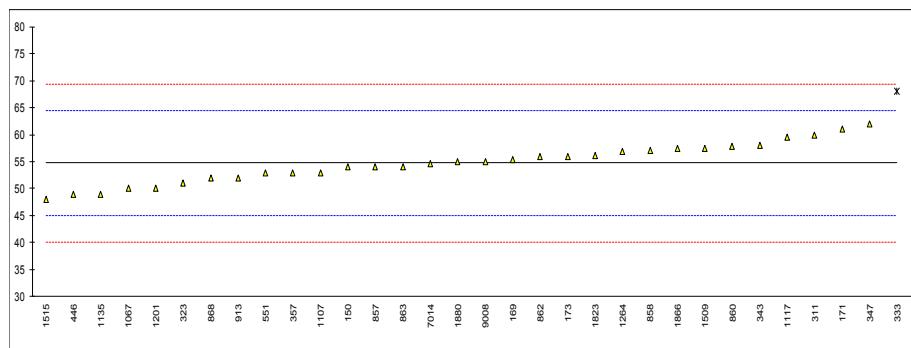
Determination of Ethylbenzene on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	93		0.87	
169	D5135	83.6		-0.55	
171	D5135	89		0.27	
173	D5135	86		-0.19	
273		----		----	
311	D5135	100	R(0.05)	1.93	
323	D5135	79		-1.25	
333	D5135	91		0.57	
343	D5135	87		-0.04	
347	D5135	88		0.11	
357	D5135	90		0.42	
446	D5135	75	R(0.05)	-1.86	
551	D5135	85		-0.34	
613		----		----	
857	D5135	89		0.27	
858	D5135	90		0.42	
860	D5135	87.8		0.08	
862	D5135	84		-0.49	
863	D5135	86		-0.19	
868	D5135	82		-0.79	
869	D5135	93		0.87	
902	D5135	89		0.27	
913	D5135	83.0		-0.64	
1067	In house	90		0.42	
1107	D5135	87		-0.04	
1117	D5135	88.2		0.14	
1135	D5135	89		0.27	
1201	D5135	81		-0.95	
1264	D5135	87.40		0.02	
1509	D5135	90.2		0.45	
1515	D5135	69	R(0.01)	-2.76	
1823	D5135	89.0	C	0.27	first reported: 89.6
1866	D5135	88.99		0.26	
1880	D5135	87.0		-0.04	
6100	D5135	84.2		-0.46	
7014	D5135	88.4		0.18	
9008	D5135	86		-0.19	
9014		----		----	
normality					
n		OK			
outliers		32			
mean (n)		3			
st.dev. (n)		87.24			
R(calc.)		3.257			
st.dev.(D5135:16e1)		9.12			
R(D5135:16e1)		6.599			
		18.48			



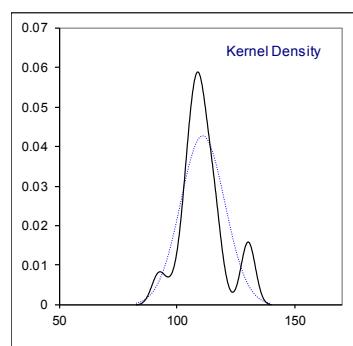
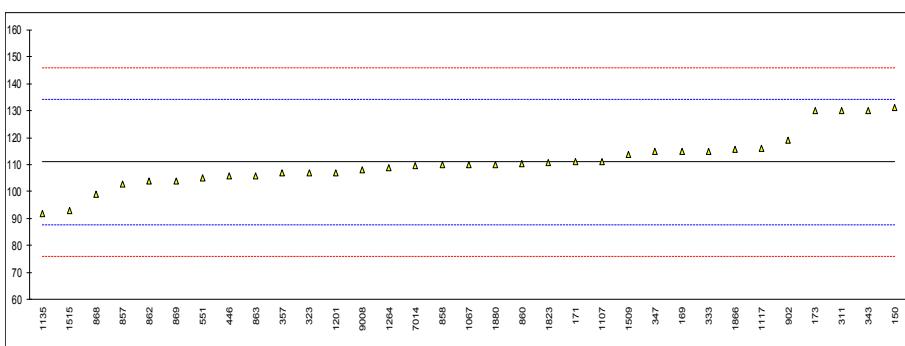
Determination of o-Xylene on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	54		-0.15	
169	D5135	55.3		0.11	
171	D5135	61		1.28	
173	D5135	56	C	0.26	first reported: <10
273		----		----	
311	D5135	60		1.07	
323	D5135	51		-0.77	
333	D5135	68	R(0.05)	2.71	
343	D5135	58		0.66	
347	D5135	62		1.48	
357	D5135	53		-0.36	
446	D5135	49		-1.18	
551	D5135	53		-0.36	
613		----		----	
857	D5135	54		-0.15	
858	D5135	57		0.46	
860	D5135	57.9		0.64	
862	D5135	56		0.26	
863	D5135	54		-0.15	
868	D5135	52		-0.56	
869		----		----	
902		----		----	
913	D5135	52.0		-0.56	
1067	In house	50		-0.97	
1107	D5135	53		-0.36	
1117	D5135	59.5		0.97	
1135	D5135	49		-1.18	
1201	D5135	50		-0.97	
1264	D5135	56.90		0.44	
1509	D5135	57.5		0.56	
1515	D5135	48		-1.38	
1823	D5135	56.2	C	0.30	first reported: 3.0
1866	D5135	57.39		0.54	
1880	D5135	55.0		0.05	
6100		----		----	
7014	D5135	54.6		-0.03	
9008	D5135	55		0.05	
9014		----		----	
normality		OK			
n		31			
outliers		1			
mean (n)		54.75			
st.dev. (n)		3.601			
R(calc.)		10.08			
st.dev.(D5135:16e1)		4.889			
R(D5135:16e1)		13.69			



Determination of α -Methylstyrene on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	131		1.72	
169	D5135	115.0		0.34	
171	D5135	111		0.00	
173	D5135	130		1.63	
273		----		----	
311	D5135	130		1.63	
323	D5135	107		-0.34	
333	D5135	115		0.34	
343	D5135	130		1.63	
347	D5135	115		0.34	
357	D5135	107		-0.34	
446	D5135	106		-0.43	
551	D5135	105		-0.52	
613		----		----	
857	D5135	103		-0.69	
858	D5135	110		-0.09	
860	D5135	110.2		-0.07	
862	D5135	104		-0.60	
863	D5135	106		-0.43	
868	D5135	99		-1.03	
869	D5135	104		-0.60	
902	D5135	119		0.69	
913		----		----	
1067	In house	110		-0.09	
1107	D5135	111		0.00	
1117	D5135	116.2		0.45	
1135	D5135	92		-1.63	
1201	D5135	107		-0.34	
1264	D5135	109.0		-0.17	
1509	D5135	113.9		0.25	
1515	D5135	93		-1.55	
1823	D5135	110.6	C	-0.04	first reported: 111.6
1866	D5135	115.69		0.40	
1880	D5135	110.0		-0.09	
6100		----		----	
7014	D5135	109.8		-0.10	
9008	D5135	108		-0.26	
9014		----		----	
normality					
n		OK			
outliers		33			
mean (n)		0			
st.dev. (n)		111.01			
R(calc.)		9.347			
st.dev.(D5135:16e1)		26.17			
R(D5135:16e1)		11.649			
		32.62			

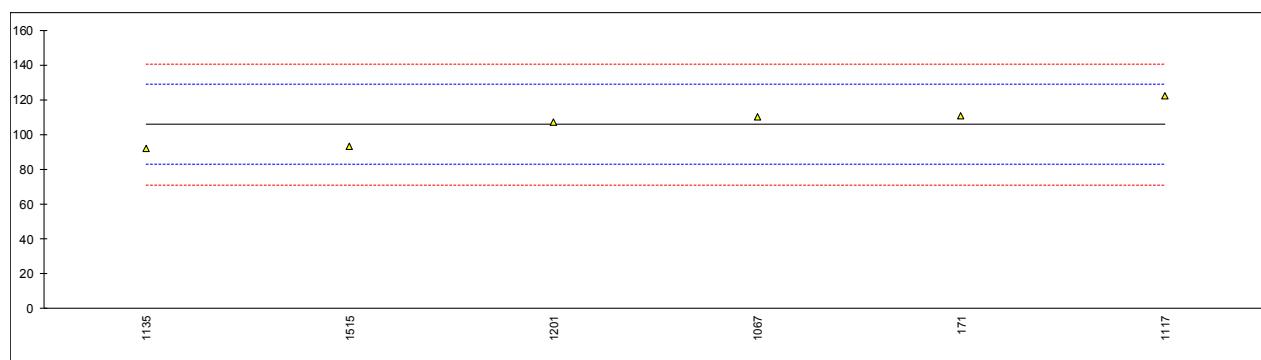


Determination of 1,2-Diethylbenzene on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		----		----	
169		----		----	
171	D5135	<10		----	
173		----		----	
273		----		----	
311		----		----	
323		----		----	
333		----		----	
343		----		----	
347		----		----	
357		----		----	
446		----		----	
551		----		----	
613		----		----	
857		----		----	
858		----		----	
860		----		----	
862		----		----	
863		----		----	
868		----		----	
869		----		----	
902		----		----	
913		----		----	
1067	In house	< 10		----	
1107		----		----	
1117	D5135	5.8		----	
1135	D5135	<10	C	----	first reported: 98
1201	D5135	0		----	
1264		----		----	
1509		----		----	
1515	D5135	0		----	
1823		----		----	
1866	D5135	<10		----	
1880		----		----	
6100		----		----	
7014		----		----	
9008	D5135	N.D.		----	
9014		----		----	
normality		n.a.			
n		8			
outliers		n.a.			
mean (n)		<10			
st.dev. (n)		n.a.			
R(calc.)		n.a.			
st.dev.(lit)		n.a.			
R(lit)		n.a.			

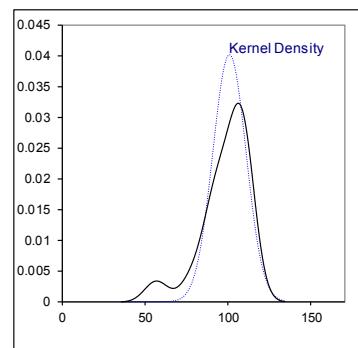
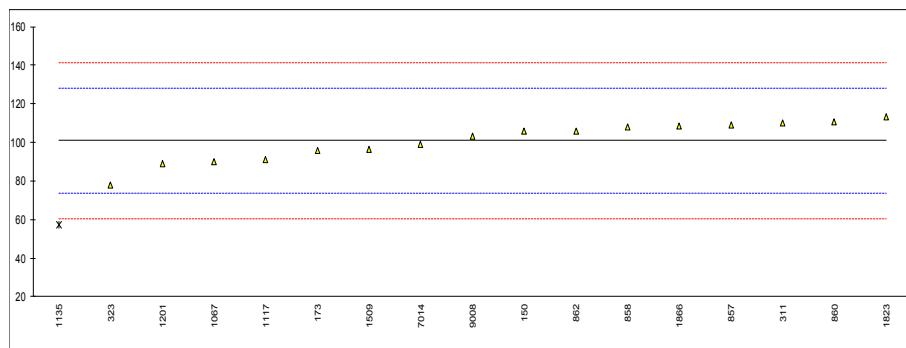
Determination of sum of α -Methylstyrene and 1,2-Diethylbenzene on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		----		----	
169		----		----	
171	D5135	111		0.45	
173		----		----	
273		----		----	
311		----		----	
323		----		----	
333		----		----	
343		----		----	
347		----		----	
357		----		----	
446		----		----	
551		----		----	
613		----		----	
857		----		----	
858		----		----	
860		----		----	
862		----		----	
863		----		----	
868		----		----	
869		----		----	
902		----		----	
913		----		----	
1067	In house	110		0.36	
1107		----		----	
1117	D5135	122.0		1.40	
1135	D5135	92	C	-1.20	first reported: 190
1201	D5135	107		0.10	
1264		----		----	
1509		----		----	
1515	D5135	93		-1.11	
1823		----		----	
1866		----		----	
1880		----		----	
6100		----		----	
7014		----		----	
9008		----		----	
9014		----		----	
normality		unknown			
n		6			
outliers		0			
mean (n)		105.83			
st.dev. (n)		11.514			
R(calc.)		32.24			
st.dev.(D5135:16e1)		11.554			
R(D5135:16e1)		32.35			



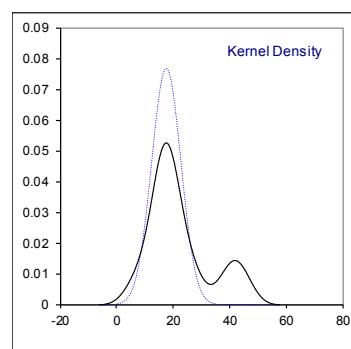
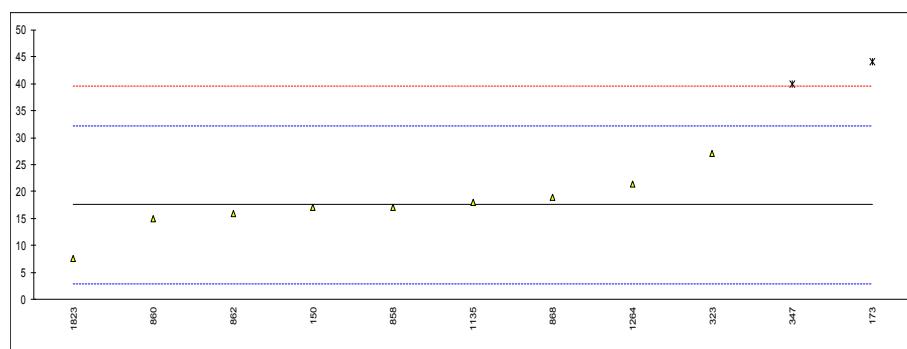
Determination of Benzaldehyde on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	106		0.38	
169		----		----	
171	D5135	<10		<-6.73	false negative test result?
173	D5135	96		-0.36	
273		----		----	
311	D5135	110		0.67	
323	D5135	78		-1.69	
333		----		----	
343		----		----	
347		----		----	
357		----		----	
446		----		----	
551		----		----	
613		----		----	
857	D5135	109		0.60	
858	D5135	108		0.53	
860	D5135	110.7		0.73	
862	D5135	106		0.38	
863		----		----	
868		----		----	
869		----		----	
902		----		----	
913		----		----	
1067	In house	90		-0.81	
1107		----		----	
1117	D5135	91.0		-0.73	
1135		57	D(0.05)	-3.25	
1201	D5135	89		-0.88	
1264		----		----	
1509	D5135	96.6		-0.32	
1515		----		----	
1823	D5135	113.1	C	0.90	first reported: 113.2
1866	D5135	108.55		0.57	
1880		----		----	
6100		----		----	
7014	D5135	99.2		-0.12	
9008	D5135	103		0.16	
9014		----		----	
normality					
n		OK			
n		16			
outliers		1			
mean (n)		100.88			
st.dev. (n)		9.956			
R(calc.)		27.88			
st.dev.(D5135:16e1)		13.511			
R(D5135:16e1)		37.83			



Determination of Nonaromatics on sample #17190; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	17		-0.08	
169		----		-----	
171	D5135	<10		-----	
173	D5135	44	DG(0.05)	3.61	
273		----		-----	
311	D5135	<10		-----	
323	D5135	27		1.29	
333		----		-----	
343		----		-----	
347	D5135	40	DG(0.05)	3.06	
357	D5135	<10		-----	
446	D5135	<10		-----	
551		----		-----	
613		----		-----	
857		----		-----	
858	D5135	17		-0.08	
860	D5135	15		-0.35	
862	D5135	16		-0.21	
863		----		-----	
868	D5135	19		0.20	
869		----		-----	
902		----		-----	
913		----		-----	
1067	In house	< 50		-----	
1107		----		-----	
1117		----		-----	
1135	D5135	18		0.06	
1201	D5135	<100		-----	
1264	D5135	21.5	C	0.54	first reported: 95
1509					-----
1515	D5135	<10		-----	
1823	D5135	7.6	C	-1.36	first reported: 68.0
1866		----		-----	
1880		----		-----	
6100		----		-----	
7014		----		-----	
9008		----		-----	
9014		----		-----	
normality					
n		not OK			
outliers		n			
mean (n)		9			
st.dev. (n)		2			
R(calc.)		17.57			
st.dev.(D5135:16e1)		5.193			
R(D5135:16e1)		14.54			
		7.319			
		20.49			



APPENDIX 2: Other reported test results; results in mg/kg

Lab	method	Toluene	m- & p-Xylenes	Iso-Propylbenzene	n-Propylbenzene	remarks
150	D5135	<10	----	<10	<10	
169	D5135	79.6 *)	4.4	----	2.8	*) false positive?
171	D5135	<10	7	5	<10	
173	D5135/D7504	<1	9	67 *)	<10	*) false positive?
273		----	----	----	----	
311	D5135/D7504	<10	<10	<10	<10	
323	D5135	<1	<10	<10	<10	
333	D5135	----	<10	<10	----	
343	D5135	----	<10	<10	<10	
347	D5135	----	<10	<10	<10	
357	D5135	----	7	4	<10	
446	D5135	37 *)	----	<10	<10	*) false positive?
551	D5135	<10	<10	<10	<10	
613		----	----	----	----	
857	D5135	<10	8	<10	<10	
858	D5135	<10	7	<10	<10	
860	D5135	<10	7.9	4.7	3.1	
862	D5135	<10	7.1	4.6	<10	
863	D5135	<10	<10	<10	<10	
868	D5135	<10	<10	<10	<10	
869	D5135	----	----	<10	----	
902		----	----	----	----	
913		----	----	----	----	
1067	In house	< 10	< 10	< 10	< 10	
1107	D5135	<1	6	4	----	
1117	D5135	4.9	8.6	4.1	2.8	
1135	D5135	<5	4	1	<10	
1201	D5135	<10	4	4	2	
1264	D5135	<10	<10	<10	<10	
1509	D5135	<5	5.0	<5	<5	
1515	D5135	0	7	3	2	
1823	D5135	N.D.	6.5 C	4.7 C	----	fr. 6.6, 4.8
1866	D5135	<10	<10	<10	<10	
1880	D5135	----	<10	<10	<10	
6100		----	----	----	----	
7014	D5135	<0.7	6.8	3.9	1.4	
9008	D5135	N.D.	11	<10	<10	
9014		----	----	----	----	

Other reported test results; results in mg/kg - continued

Lab	method	m- & p-Ethyltoluene	Phenylacetylene	3- & 4-Methylstyrenes	Sum Phenylacetylene and 3- & 4-Methylstyrenes	remarks
150	D5135	<10	<10	----	----	
169	D5135	----	0	----	----	
171	D5135	7	<10	<10	<10	
173	D5135	<10	<10	----	----	
273	----	----	----	----	----	
311	D5135	<10	<10	----	----	
323	D5135	----	<10	----	----	
333	----	----	----	----	----	
343	D5135	<10	----	----	----	
347	D5135	----	<10	----	----	
357	D5135	<10	<10	----	----	
446	D5135	----	<10	----	----	
551	D5135	----	<10	----	----	
613	----	----	----	----	----	
857	D5135	----	<10	----	----	
858	D5135	<10	<10	----	----	
860	D5135	<10	----	----	----	
862	D5135	<10	<10	----	----	
863	----	----	----	----	----	
868	D5135	<10	----	----	----	
869	D5135	----	<10	----	----	
902	----	----	----	----	----	
913	----	----	----	----	----	
1067	In house	< 20	< 10	< 20	< 30	
1107	D5135	----	----	----	----	
1117	D5135	3.9	<5	0	0	
1135	D5135	<20	<5	<10	<15	
1201	D5135	----	<10	0	0	
1264	D5135	11.4	<10	----	----	
1509	D5135	<5	<5	----	----	
1515	D5135	5	1 **)	2 **)	95 **)	**) note iis: sum differs from sum single values
1823	D5135	----	----	----	----	
1866	D5135	<10	<10	<10	<10	
1880	D5135	<10	<10	----	----	
6100	----	----	----	----	----	
7014	D5135	4.1	2.1	----	----	
9008	D5135	<10	<10	----	----	
9014	----	----	----	----	----	

APPENDIX 3

Number of participants per country

1 lab in AUSTRALIA
3 labs in BELGIUM
1 lab in BRAZIL
1 lab in CANADA
9 labs in CHINA, People's Republic
1 lab in FINLAND
1 lab in FRANCE
1 lab in INDIA
1 lab in IRAN, Islamic Republic of
2 labs in KUWAIT
4 labs in NETHERLANDS
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
1 lab in SOUTH AFRICA
2 labs in SPAIN
2 labs in TURKEY
1 lab in UNITED ARAB EMIRATES
1 lab in UNITED KINGDOM
4 labs in UNITED STATES OF AMERICA

APPENDIX 4

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
- 2 ASTM E178:02
- 3 ASTM E1301:03
- 4 ISO13528:15
- 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, No 4 January 2001.
- 14 P.J. Lothian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364 (2002)
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
- 16 Horwitz, R. Albert, J. AOAC Int. 79-3, 589 (1996)