Results of Proficiency Test
Styrene
October 2016

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

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Report: iis16C07

November 2016

CONTENTS

1 INTRODUCTION

Since 1995, the Institute for Interlaboratory Studies organizes a proficiency test for the analyses of Styrene. During the annual proficiency testing program 2016/2017, it was decided to organize again a round robin for the analyses of Styrene in accordance with the latest applicable version of the product specification ASTM D2827:13. In this interlaboratory study, 42 laboratories from 20 different countries did register for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2016 Styrene proficiency test are presented and discussed.
This report is also available as PDF file 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 #16200).
Participants were requested to report the test results using the indicated units and 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 proficiency test falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, version 3.3) of April 2014. This protocol can be downloaded from 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 36 kg of this batch, the components listed in table 1 were added:

Component	*Amount*
Chloroform	107 mg
Dibenzothiophene	400 mg
Benzene	79 mg
1,2 - Diethylbenzene	1.8 g
n-Propylbenzene	1.8 g
Polystyrene	358 mg

Table 1: components added to the bulk materiaal

After homogenisation in a pre-cleaned drum 68 amber glass bottles of 0.5 L (labelled #16200) were filled. The homogeneity of subsamples of #16200 was checked by determination of Density at 20°C in accordance with ASTM D4052 and α-Methyl Styrene in accordance with ASTM D5135 on 8 stratified randomly selected samples.

	Density at 20°C in kg/L	*α-Methyl Styrene in mg/kg*
sample #16200-1	0.90623	202
sample #16200-2	0.90624	199
sample #16200-3	0.90621	197
sample #16200-4	0.90621	199
sample #16200-5	0.90621	198
sample #16200-6	0.90625	199
sample #16200-7	0.90622	201
sample #16200-8	0.90625	200

Table 2: homogeneity test results of subsamples #16200

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

	Density at 20°C in kg/L	*α-Methyl Styrene in mg/kg*
r (observed)	0.00005	4.5
ref. test method	ISO 12185:96	ASTM D5135:14
0.3 x R (ref. test method)	0.00015	10.8

Table 3: evaluation of the repeatabilities of subsamples #16200

The calculated repeatabilities were less than 0.3 times the corresponding reproducibility of the reference test methods. Therefore homogeneity of the subsamples of #16200 was assumed.

To each of the participating laboratories, 1 bottle of 0.5 L (labelled #16200) was sent on September 21, 2016.

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 #16200: Aldehydes as benzaldehyde, Appearance, Chloride (Organic), Colour Pt/Co, Density at 20°C, Inhibitor as TBC, Peroxide as H_2O_2, 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 explicitely requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the results, but report as much significant figures as possible. Therefore to get comparable test results a detailed report form, on which the units were prescribed as well as the required reference test method and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The laboratories were also requested to confirm the sample receipt on the same data entry portal. A SDS was added to the sample.

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 April 2014 (iis-protocol, version 3.3).
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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the test results 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 of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.
When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.
The z-scores were calculated according to:

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

The $z_{(target)}$ 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 samples to the laboratories in Brazil, United Arab Emirates and India. These laboratories received the samples late or not at all. One participant reported test results after the final reporting date and four laboratories did not report any test results at all. Not all participants were able to report all requested parameters. Finally, 38 laboratories did report 521 numerical test results. Observed were 14 outlying test results, which is 2.7%. 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 reference reproducibilities used for the evaluation of the GC impurities were extrapolated from the reproducibilities as mentioned in ASTM D5135:14 (table 3 or 4 of the ASTM D5135 test method) or calculated using the Horwitz equation when no precision data was mentioned in ASTM D5135:14 or when $R_{D5135} << R_{Horwitz}$.
Please note: After closure of the PT a new version of ASTM D5135 was published in October 2016 and new reproducibilities were published. However, it is decided to use the reproducibilities of 2014 for the statistical evaluations as this PT took place from September 21 till October 28, 2016. The reproducibilities from ASTM D5135:16 are also mentioned in the test results tables of appendix 1.

In iis PT reports, ASTM test methods are referred to with a number (e.g. D5808) and an added designation for the year that the test method was adopted or revised (e.g. D5808:09a). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D5808:09a (2014)). In the tables of Appendix 1 only the test method number and year of adoption or revision will be used.

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, see also paragraph 3.1.

Aldehydes as benzaldehyde: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D2119:09. The large variation was also observed in previous PTs. In iis PT report of 2014 (iis14C07) two possible root causes are mentioned for this large variation. The first one: CO2-absorption during the standing period of 1 hour and secondly: the use of Thymol Blue in stead of the sodium salt of Thymol Blue (and the need of caustic to dissolve the Thymol blue). None of the participants reported a value lower than the Benzaldehyde content by GC.

Appearance: Allthough 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 #16200 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. 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 2.7 mg Cl/kg) may be acceptable: “less than 79%” (the actual blank Chloride content is unknown).

Colour Pt/Co: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D5386:10 and with ASTM D1209:05(2011).

Density at 20°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ISO 12185:96.

Inhibitor as TBC: This determination was not problematic. Two statisticals outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D4590:13.

Peroxides as H_2O_2: 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.

Polymers: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D2121-A:16.
The average recovery of Polymer (theoretical increment of 10.1 mg Polymer/kg) may be good: “less than 82%” (the actual blank Polymer content is unknown).

Sulphur: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D5453:16e1.
The average recovery of Sulphur (theoretical increment of 2.0 mg Sulphur /kg) may be very good: “less than 97%” (the actual blank Sulphur content is unknown).

Water, coulometric KF titration: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirement of ASTM E1064:16.

Purity by GC: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D5135:14.

Benzene: 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 D6229:06(2010).
The average recovery of Benzene (theoretical increment of 2.2 mg Benzene/kg) may be very good: “less than 98%” (the actual blank Benzene content is unknown). Ten participants reported a Benzene value “<10 mg/kg”. It should be noted that the Styrene specification D2827:13 mentions a Benzene content of 1 mg/kg maximum and thus reporting ‘<10 mg/kg’ is not in accordance with the D2827 specification.

Toluene: About 77% of the reporting participants reported a Toluene value “less than” and other reported test values were below or near the application range. Therefore no significant conclusions were drawn. One false positive test result was reported.

Ethylbenzene: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D5135:14.

m- & p-Xylenes: About 68% of the reporting participants reported a value “less than” and other reported test values were below or near the application range. Therefore no significant conclusions were drawn. No false positive test results were reported.

iso-Propylbenzene (Cumene): About 69% of the reporting participants reported a value “less than” and other reported test values were below or near the application range. Therefore no significant conclusions were drawn. No false positive test results were reported.

o-Xylene: About 74% of the reporting participants reported a value “less than” and other reported test values were below or near the application range. Therefore no significant conclusions were drawn. Two false positive test results were reported.

n-Propylbenzene: This determination was not problematic. Two statisticals outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5135:14. Two false negative test results were reported.
The average recovery of n-Propylbenzene (theoretical increment of 50.3 mg n-Propylbenzene /kg) may be acceptable: “less than 125%” (the actual blank n-Propylbenzene content is unknown).

m-& p-Ethyltoluenes: This determination was not problematic. One statistical outlier was observed and one false negative test result was reported. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5135:14.

α-Methylstyrene: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D5135:14.

1,2-Diethylbenzene: Only four laboratories reported a numerical test result for this component. Therefore no z-scores were calculated. Two false negative test results were reported.
Surprisingly, the average recovery based on 4 reported test results of 1,2-Diethylbenzene (theoretical increment of 50.5 mg 1,2-Diethylbenzene /kg) may be very good: “less than 95%” (the actual blank 1,2-Diethylbenzene content is unknown).

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

Phenylacetylene: About 64% of the reporting participants reported a value “less than” and other reported test values were below or near the application range. Therefore no significant conclusions were drawn. No false positive test results were reported.

3-&4-Methylstyrenes: Three laboratories reported a numeric test result and four reported a test value as “lower than”. Therefore, no significant conclusions could be drawn. One false positive test result was reported.

Sum Phenylacetylene & 3- & 4 -Methylstyrenes: Only six laboratories reported a numerical test result for the sum of the three components. However, this determination may not problematic. One statistical outlier was observed and one false negative test result was reported. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the estimated requirements using the Horwitz equation for three components.

Benzaldehyde: This determination may be problematic. No statistical outliers were observed and one false negative test result was reported. However, the calculated reproducibility is not in agreement with the estimated requirements using the Horwitz equation, but it is in agreement with the requirements of ASTM D5135:16.

Nonaromatics: This determination may be very problematic. No statistical outliers were observed and one false negative test result was reported. The calculated reproducibility is not in agreement with the estimated requirements using the Horwitz equation for nine components. The number of nonaromatic impurities is not known but expected to be many; hence the choice for nine components. The calculated reproducibility is also not in agreement with the requirements of ASTM D5135:16.

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) and the calculated reproducibilities of sample #16200 are compared in the next table.

Parameter	unit	n	average	2.8 *sd	R (lit)
Aldehydes as benzaldehyde	mg/kg	29	109.5	65.5	43.8
Appearance		34	Pass	n.a.	n.a.
Chloride, Organic	mg/kg	13	2.3	1.1	1.3
Colour Pt/Co		38	9.0	4.2	6.0
Density at 20°C	kg/L	33	0.9063	0.0002	0.0005
Inhibitor as TBC	mg/kg	33	9.2	3.2	3.1
Peroxides as H_2O_2	mg/kg	26	18.4	21.1	13.0
Polymers	mg/kg	34	8.2	9.4	7.9
Sulphur	mg/kg	25	1.9	0.7	1.0
Water, coulometric KF titration	mg/kg	35	161	25	45
Purity by GC	%M/M	34	99.94	0.03	0.03
Benzene	mg/kg	19	2.2	1.8	1.2
Toluene	mg/kg	22	<10	n.a.	n.a.
Ethylbenzene	mg/kg	33	56.6	12.2	16.2
m- & p-Xylenes	mg/kg	28	<10	n.a.	n.a.
iso-Propylbenzene (Cumene)	mg/kg	29	<10	n.a.	n.a.
o-Xylene	mg/kg	23	<10	n.a.	n.a.
n-Propylbenzene	mg/kg	22	62.8	11.3	15.7
m- & p-Ethyltoluenes	mg/kg	10	9.0	2.5	3.2
α-Methylstyrene	mg/kg	28	229	69	38
1,2-Diethylbenzene	mg/kg	4	47.9	53.3	(12.0)
sum α-Methylstyrene & 1,2-diethylbenzene	mg/kg	8	249	74	69
Phenylacetylene	mg/kg	22	<10	n.a.	n.a.
3- & 4-Methylstyrenes	mg/kg	6	<10	n.a.	n.a.
sum Phenylacetylene & 3- & 4-Methylstyrenes	mg/kg	5	10.6	5.3	5.8
Benzaldehyde	mg/kg	16	64.0	23.8	15.3
Nonaromatics	mg/kg	15	61.0	83.3	44.1

Table 4: comparison of the observed and target reproducibilities of sample #16200

Figure under R(lit) between brackets is based on very little test results

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2016 WITH PREVIOUS PTS

	October 2016	*October 2015*	*September 2014*	*September 2013*	*September 2012*
Number of reporting labs	38	37	36	34	38
Number of test results reported	521	447	510	474	552
Number of statistical outliers	14	20	33	29	30
Percentage outliers	2.7%	4.5%	6.5%	6.1%	5.4%

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 2016*	*October 2015*	*September 2014*	*September 2013*	*September 2012*
Aldehydes as benzaldehyde	-	--	--	--	--
Chloride, Organic	+	n.e.	+	+/-	++
Colour Pt/Co	+	++	++	++	+
Density at 20°C	++	++	++	++	++
Inhibitor as TBC	+/-	+	--	--	+
Peroxides as H_2O_2	-	-	--	--	--
Polymers	-	++	--	--	--
Sulphur	+	n.e.	+	++	+
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.	++	++	--	--
iso-Propylbenzene (Cumene)	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.	n.e.
Phenylacetylene	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.	n.e.

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

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:

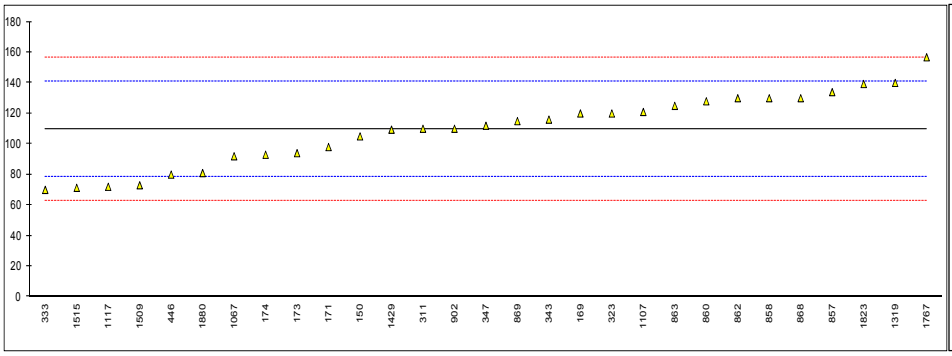
++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

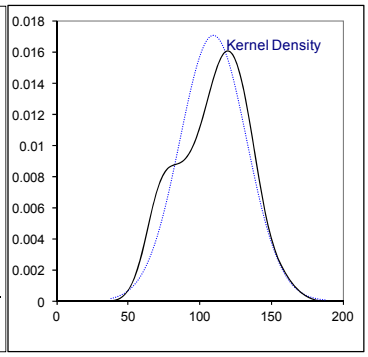
APPENDIX 1

Determination of Aldehydes as benzaldehyde on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D2119	105		-0.29	
169	D2119	120		0.67	
171	D2119	98		-0.74	
173	D2119	94		-0.99	
174	D2119	93		-1.06	
273		-----		-----	
311	D2119	110		0.03	
323	D2119	120		0.67	
333	D2119	70		-2.53	
343	D2119	116		0.41	
347	D2119	112		0.16	
396		-----		-----	
446	D2119	80		-1.89	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D2119	134		1.57	
858	D2119	130.0		1.31	
860	D2119	128		1.18	
862	D2119	130		1.31	
863	D2119	125		0.99	
868	D2119	130		1.31	
869	D2119	115		0.35	
902	D2119	110		0.03	
913		-----		-----	
1067	D2119	92		-1.12	
1107	D2119	121		0.73	
1117	D7704	72		-2.40	
1201		-----		-----	
1252		-----		-----	
1264		-----		-----	
1319		140		1.95	
1429		109.4		-0.01	
1509	D2119	73		-2.33	
1515	D2119	71.34		-2.44	
1767	D2119	156.8		3.02	
1823	D2119	139.3		1.90	
1866		-----		-----	
1880	D7704	81.0		-1.82	
6013		-----		-----	
7014		-----		-----	
9008	In house	-----	W	-----	first reported: 57.7

normality	OK
n	29
outliers	0
mean (n)	109.51
st.dev. (n)	23.406
R(calc.)	65.54
R(D2119:09)	43.80





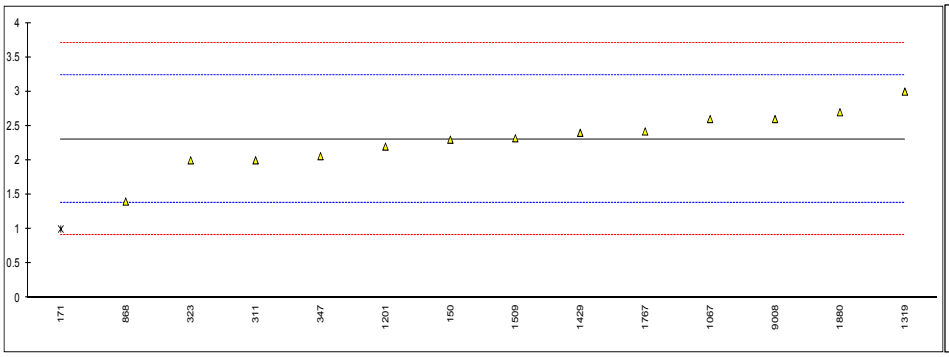
Determination of Appearance on sample #16200;

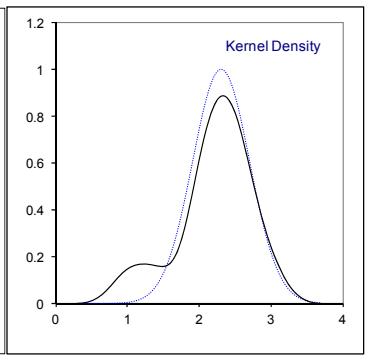
lab	method	value	mark	z(targ)	remarks
150	E2680	Pass		-----	
169	D4176	BC&FSM		-----	
171	E2680	Pass		-----	
173	D4176	Pass		-----	
174	E2680	CFSM		-----	
273	E2680	Pass		-----	
311	E2680	pass		-----	
323	Visual	clear & bright		-----	
333		-----		-----	
343	E2680	Pass		-----	
347	E2680	PASS		-----	
396	E2680	Pass		-----	
446	E2680	Pass		-----	
551		-----		-----	
557		-----		-----	
613	E2680	PASS		-----	
857	E2680	Pass		-----	
858		-----		-----	
860	Visual	Bright & Clear		-----	
862	E2680	Pass		-----	
863	E2680	Pass		-----	
868	E2680	pass		-----	
869	Visual	Clear and bright		-----	
902	E2680	PASS		-----	
913	E2680	CFSM		-----	
1067	E2680	Pass		-----	
1107	E2680	pass		-----	
1117	D4176	pass		-----	
1201	Visual	Clear and free		-----	
1252	Visual	PASS		-----	
1264		-----		-----	
1319		Clear & Bright		-----	
1429	E2680	Pass		-----	
1509	E2680	Clear & Free from suspended matter		-----	
1515	E2680	PASS		-----	
1767		-----		-----	
1823	Visual	Clear/FFSM/No free water		-----	
1866		-----		-----	
1880	Visual	Pass		-----	
6013		-----		-----	
7014	Visual	Clear		-----	
9008	Visual	Clear liquid		-----	
	n	34			
	outliers	0			
	mean (n)	Pass			

Determination of Chloride, Organic on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D7359	2.3		-0.02	
169		-----		-----	
171	D5808	1	D(0.05)	-2.82	
173		-----		-----	
174		-----		-----	
273		-----		-----	
311	D5808	2		-0.66	
323	D5808	2		-0.66	
333		-----		-----	
343		-----		-----	
347	D5808	2.06		-0.53	
396		-----		-----	
446		-----		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857		-----		-----	
858		-----		-----	
860		-----		-----	
862		-----		-----	
863		-----		-----	
868	D5808	1.4		-1.96	
869		-----		-----	
902		-----		-----	
913		-----		-----	
1067	UOP779	2.6		0.63	
1107		-----		-----	
1117		-----		-----	
1201	D5808	2.2		-0.23	
1252		-----		-----	
1264		-----		-----	
1319		3		1.49	
1429	D7359	2.4		0.20	
1509	D5808	2.321		0.03	
1515		-----		-----	
1767	D5808	2.42		0.24	
1823		-----		-----	
1866		-----		-----	
1880	D7359	2.7		0.84	
6013		-----		-----	
7014		-----		-----	
9008	D5808	2.6		0.63	

	normality	suspect	
	n	13	
	outliers	1	<u>spike</u>
	mean (n)	2.31	2.7 (recovery: <79%)
	st.dev. (n)	0.399	
	R(calc.)	1.12	
	R(D5808:09a)	1.30	



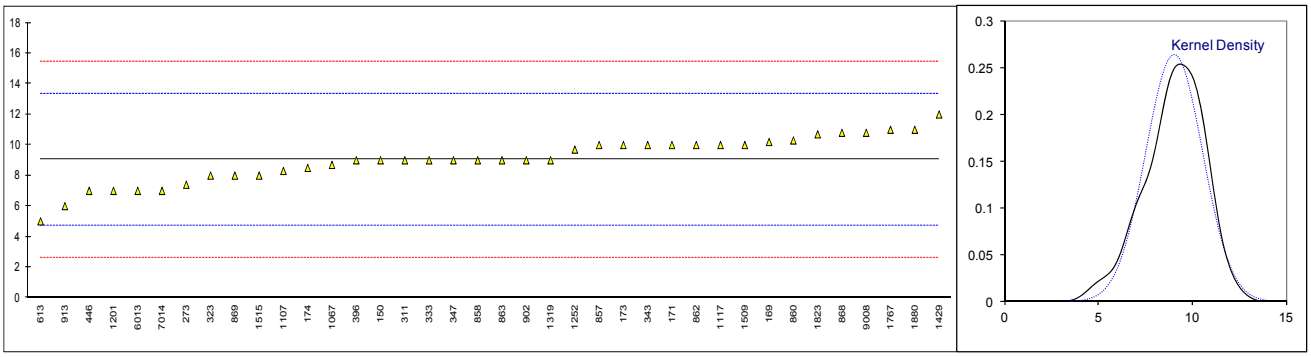


Determination of Colour Pt/Co on sample #16200;

lab	method	value	mark	z(targ)	remarks
150	D5386	9		-0.02	
169	D5386	10.2		0.54	
171	D1209	10		0.45	
173	D5386	10		0.45	
174	D5386	8.5		-0.25	
273	D1209	7.4		-0.76	
311	D5386	9		-0.02	
323	D5386	8		-0.48	
333	D5386	9		-0.02	
343	D5386	10		0.45	
347	D5386	9		-0.02	
396	D5386	9		-0.02	
446	D5386	7		-0.95	
551		-----		-----	
557		-----		-----	
613	D1209	5		-1.88	
857	D5386	10		0.45	
858	D5386	9		-0.02	
860	D5386	10.3		0.59	
862	D5386	10		0.45	
863	D1209	9		-0.02	
868	D5386	10.8		0.82	
869	D1209	8		-0.48	
902	D5386	9		-0.02	
913	D5386	6		-1.42	
1067	D5386	8.7		-0.16	
1107	D5386	8.3		-0.34	
1117	D1209	10		0.45	
1201	D5386	7		-0.95	
1252	D1209	9.70		0.31	
1264		-----		-----	
1319		9		-0.02	
1429	D5386	12		1.38	
1509	D1209	10		0.45	
1515	D1209	8		-0.48	
1767	D5386	11.0		0.92	
1823	D5386	10.7		0.78	
1866		-----		-----	
1880	D5386	11.0		0.92	
6013	D5386	7		-0.95	
7014	D1209	7		-0.95	
9008	D5386	10.8		0.82	

normality	OK
n	38
outliers	0
mean (n)	9.04
st.dev. (n)	1.510
R(calc.)	4.23
R(D5386:10)	6.00

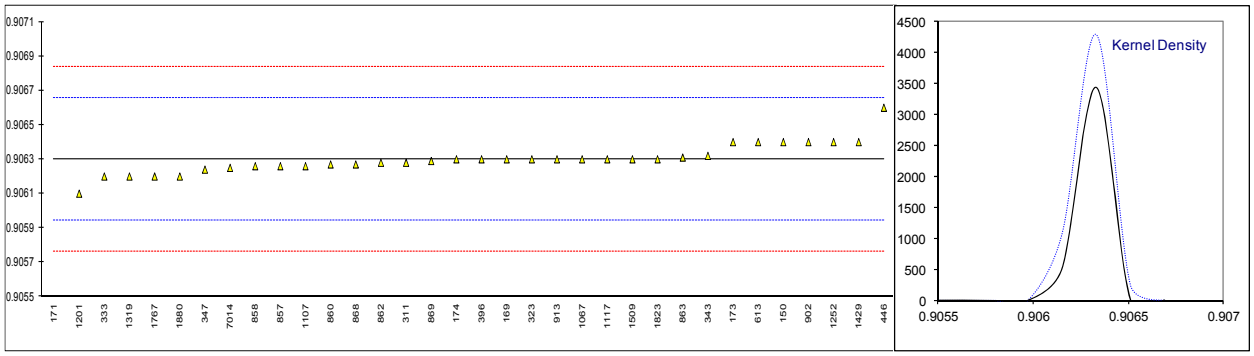
compare R(D1209:05(2011)) = 7



Determination of Density at 20°C on sample #16200; results in kg/L

lab	method	value	mark	z(targ)	remarks
150	D4052	0.9064		0.56	
169	D4052	0.9063		0.00	
171	D4052	0.8610	R(0.01)	-253.68	
173	D4052	0.9064		0.56	
174	D4052	0.9063		0.00	
273		-----		-----	
311	ISO12185	0.90628		-0.11	
323	D4052	0.9063		0.00	
333	ISO12185	0.9062		-0.56	
343	D4052	0.90632		0.11	
347	D4052	0.90624		-0.33	
396	D4052	0.9063		0.00	
446	D4052	0.9066		1.68	
551		-----		-----	
557		-----		-----	
613	D4052	0.9064		0.56	
857	D4052	0.90626		-0.22	
858	D4052	0.90626		-0.22	
860	D4052	0.90627		-0.17	
862	D4052	0.90628		-0.11	
863	D4052	0.90631		0.06	
868	D4052	0.90627		-0.17	
869	D4052	0.90629		-0.05	
902	D4052	0.9064		0.56	
913	D4052	0.9063		0.00	
1067	D4052	0.9063		0.00	
1107	D4052	0.90626		-0.22	
1117	D4052	0.9063		0.00	
1201	D4052	0.9061		-1.12	
1252	D4052	0.9064		0.56	
1264		-----		-----	
1319		0.9062		-0.56	
1429	D4052	0.9064		0.56	
1509	D4052	0.90630		0.00	
1515		-----		-----	
1767	D4052	0.9062		-0.56	
1823	D4052	0.9063		0.00	
1866		-----		-----	
1880	D4052	0.9062		-0.56	
6013		-----		-----	
7014	D4052	0.90625		-0.28	
9008		-----		-----	

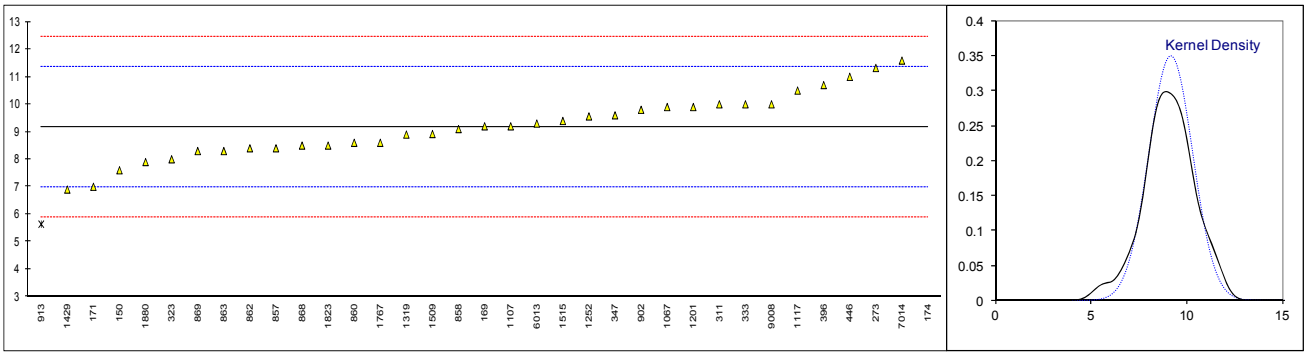
normality	not OK
n	33
outliers	1
mean (n)	0.90630
st.dev. (n)	0.000087
R(calc.)	0.00024
R(ISO12185:96)	0.00050



Determination of Inhibitor as TBC on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D4590	7.6		-1.44	
169	D4590	9.2		0.02	
171	D4590	7		-1.98	
173		-----		-----	
174	D4590	49.95	R(0.01)	37.15	
273	D4590	11.32	C	1.95	first reported: 13.7
311	D4590	10		0.75	
323	D4590	8		-1.07	
333	D4590	10		0.75	
343		-----		-----	
347	D4590	9.6		0.38	
396	D4590	10.7		1.39	
446	D4590	11		1.66	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D4590	8.4		-0.71	
858	D4590	9.1		-0.07	
860	D4590	8.6		-0.53	
862	D4590	8.4		-0.71	
863	D4590	8.3		-0.80	
868	D4590	8.5		-0.62	
869	D4590	8.3		-0.80	
902	D4590	9.8		0.57	
913	D4590	5.64	R(0.05)	-3.22	
1067	D4590	9.9		0.66	
1107	D4590	9.2		0.02	
1117	D4590	10.5		1.20	
1201	D4590	9.9		0.66	
1252	D4590	9.56		0.35	
1264		-----		-----	
1319		8.9		-0.25	
1429	D4590	6.9		-2.08	
1509	D4590	8.918		-0.24	
1515	D4590	9.393		0.20	
1767	D4590	8.60		-0.53	
1823	D4590	8.5		-0.62	
1866		-----		-----	
1880	D4590	7.9		-1.16	
6013	D4590	9.3		0.11	
7014	D4590	11.59		2.20	
9008	In house	10		0.75	

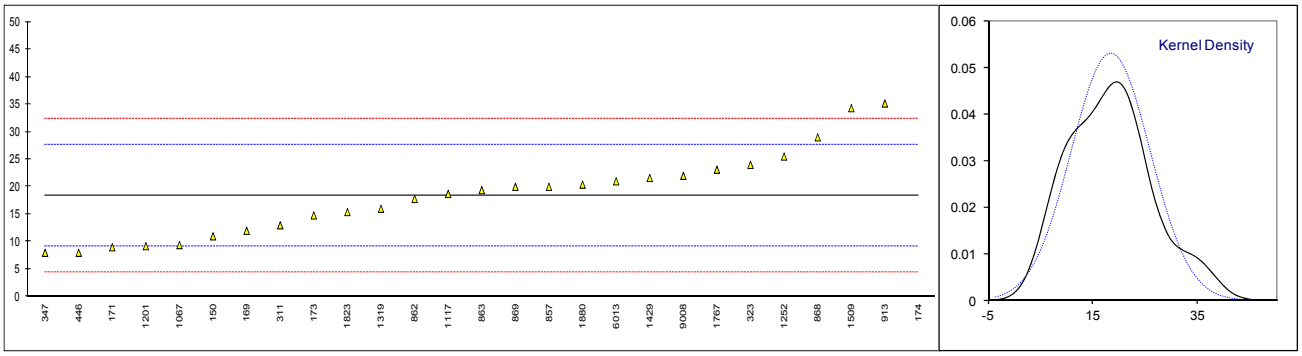
normality	OK
n	33
outliers	2
mean (n)	9.178
st.dev. (n)	1.1397
R(calc.)	3.191
R(D4590:13)	3.073



Determination of Peroxides as H_2O_2 on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D2340	11		-1.59	
169	D2340	12		-1.37	
171	D2340	9		-2.02	
173	D2340	14.8		-0.77	
174	D2340	92.19	R(0.01)	15.90	
273		-----		-----	
311	D2340	13		-1.16	
323	D2340	24		1.21	
333		-----		-----	
343		-----		-----	
347	D2340	8		-2.23	
396		-----		-----	
446	D2340	8		-2.23	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D2340	20		0.35	
858		-----		-----	
860		-----		-----	
862	D2340	17.8		-0.12	
863	D2340	19.4		0.22	
868	D2340	29.0		2.29	
869	D2340	20		0.35	
902		-----		-----	
913	D2340	35.15		3.61	
1067	D2340	9.4		-1.93	
1107		-----		-----	
1117	D2340	18.7		0.07	
1201	D2340	9.2		-1.98	
1252	D2340	25.50		1.53	
1264		-----		-----	
1319		16		-0.51	
1429	D2340	21.6		0.69	
1509	D2340	34.3		3.43	
1515		-----		-----	
1767	D2340	23.1		1.02	
1823	D2340	15.4		-0.64	
1866		-----		-----	
1880	D2340	20.4		0.44	
6013	D2340	21		0.57	
7014		-----		-----	
9008	D2340	22	C	0.78	first reported: 40

normality	OK
n	26
outliers	1
mean (n)	18.37
st.dev. (n)	7.518
R(calc.)	21.05
R(D2340:13)	13.00

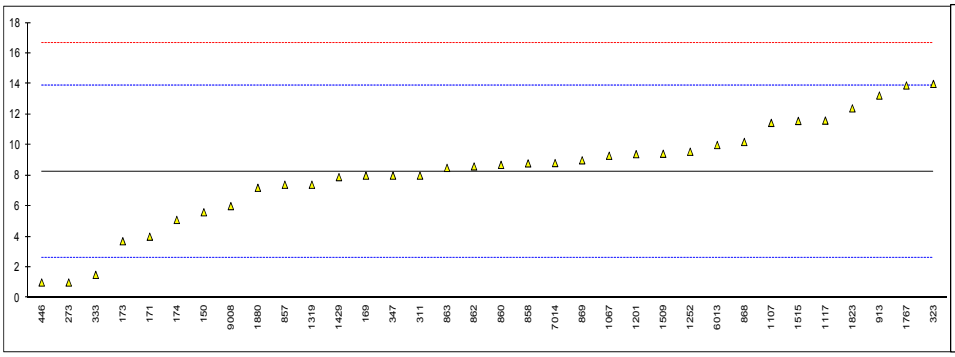


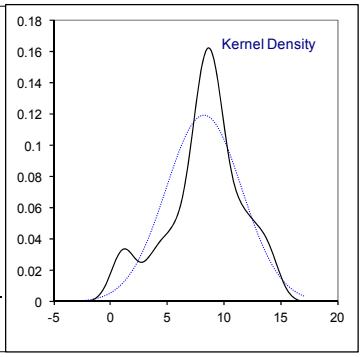
Determination of Polymers on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D2121-A	5.6		-0.94	
169	D2121-A	8		-0.09	
171	D2121-A	4		-1.51	
173	D2121-A	3.7		-1.61	
174	D2121-A	5.096		-1.12	
273	D2121-A	1.0		-2.57	
311	D2121-A	8		-0.09	
323	D2121-A	14		2.05	
333	D2121-A	1.5		-2.40	
343		-----		-----	
347	D2121-A	8		-0.09	
396		-----		-----	
446	D2121-A	1		-2.57	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D2121-A	7.4		-0.30	
858	D2121-A	8.8		0.20	
860	D2121-A	8.7		0.16	
862	D2121-A	8.6		0.13	
863	D2121-A	8.5		0.09	
868	D2121-A	10.2		0.70	
869	D2121-A	9.0		0.27	
902		-----		-----	
913	D2121-A	13.24		1.78	
1067	D2121-A	9.3		0.38	
1107	D2121-A	11.45		1.14	
1117	D2121-A	11.6		1.19	
1201	D2121-A	9.4		0.41	
1252	D2121-A	9.56		0.47	
1264		-----		-----	
1319		7.4		-0.30	
1429	D2121-A	7.9		-0.12	
1509	D2121-A	9.43		0.42	
1515	D2121-A	11.579		1.19	
1767	D2121-A	13.9		2.01	
1823	D2121-A	12.4		1.48	
1866		-----		-----	
1880	D2121-A	7.2		-0.37	
6013	D2121-A	10		0.62	
7014	D2121-A	8.82		0.20	
9008	D2121-A	6.0		-0.80	

	normality	OK
	n	34
	outliers	0
	mean (n)	8.24
	st.dev. (n)	3.339
	R(calc.)	9.35
	R(D2121-A:16)	7.88

spike
10.1 (recovery: <82%)

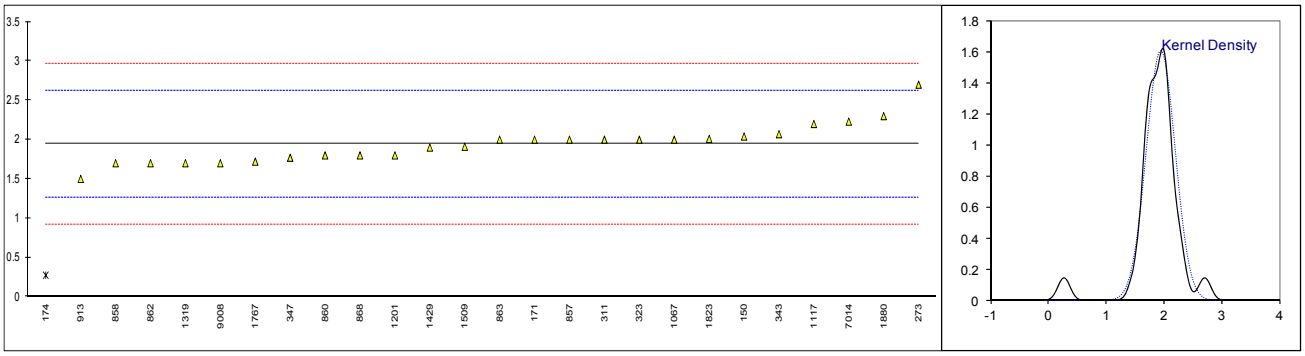




Determination of Sulphur on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5453	2.04		0.29	
169		-----		-----	
171	D5453	2.0		0.17	
173		-----		-----	
174	D5453	0.274	R(0.01)	-4.90	
273	D5453	2.7		2.23	
311	D5453	2.0		0.17	
323	D5453	2		0.17	
333		-----		-----	
343	D5453	2.07		0.38	
347	D5453	1.77		-0.51	
396		-----		-----	
446		-----		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D3120	2		0.17	
858	D5453	1.7		-0.71	
860	D3120	1.8		-0.42	
862	D5453	1.7		-0.71	
863	D5453	2.00		0.17	
868	D5453	1.8		-0.42	
869		-----		-----	
902		-----		-----	
913	D5453	1.50		-1.30	
1067	D5453	2.0		0.17	
1107		-----		-----	
1117	D5453	2.2		0.76	
1201	D5453	1.8		-0.42	
1252	D5453	<1		<-2.76	possibly a false negative test result?
1264		-----		-----	
1319		1.7		-0.71	
1429	D5453	1.9		-0.12	
1509	D5453	1.91		-0.09	
1515		-----		-----	
1767	D5453	1.72		-0.65	
1823	D5453	2.01		0.20	
1866		-----		-----	
1880	D5453	2.3		1.05	
6013		-----		-----	
7014	D5453	2.23		0.85	
9008	D5453	1.7		-0.71	

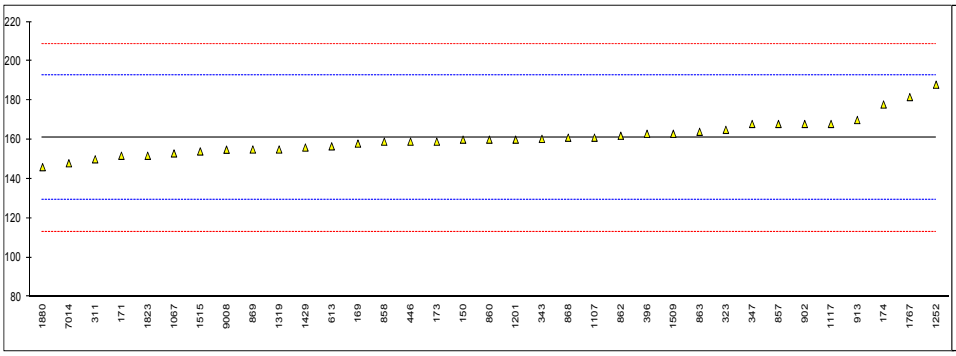
	normality	not OK	
	n	25	
	outliers	1	spike
	mean (n)	1.942	2.0 (recovery: <97%)
	st.dev. (n)	0.2479	
	R(calc.)	0.694	
	R(D5453:16e1)	0.954	

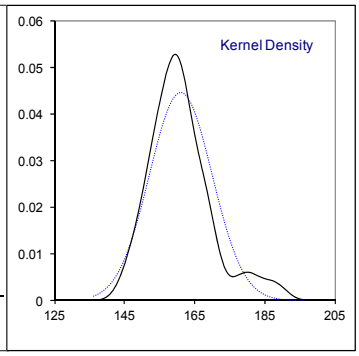


Determination of Water, coulometric KF titration on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D6304	160		-0.06	
169	E1064	158		-0.19	
171	E1064	151.8		-0.58	
173	D6304	159		-0.13	
174	E1064	177.9		1.06	
273		-----		-----	
311	E1064	150		-0.69	
323	E1064	165		0.25	
333		-----		-----	
343	E1064	160.4		-0.04	
347	E1064	168		0.44	
396	E1064	163		0.12	
446	E1064	159		-0.13	
551		-----		-----	
557		-----		-----	
613	E203	156.6		-0.28	
857	E1064	168		0.44	
858	E1064	159		-0.13	
860	E1064	160		-0.06	
862	E1064	162		0.06	
863	E1064	164		0.19	
868	E1064	161		0.00	
869	E1064	155		-0.38	
902	E1064	168		0.44	
913	E1064	170		0.56	
1067	E1064	153		-0.50	
1107	E1064	161		0.00	
1117	E1064	168		0.44	
1201	E1064	160		-0.06	
1252	E1064	188		1.69	
1264		-----		-----	
1319		155		-0.38	
1429	E1064	156		-0.32	
1509	E1064	163		0.12	
1515	E1064	154		-0.44	
1767	E1064	181.7		1.30	
1823	E1064	151.8		-0.58	
1866		-----		-----	
1880	D6304	146		-0.94	
6013		-----		-----	
7014	E1064	147.98		-0.82	
9008	E1064	154.9		-0.38	

normality	not OK
n	35
outliers	0
mean (n)	161.03
st.dev. (n)	8.960
R(calc.)	25.09
R(E1064:16)	44.61

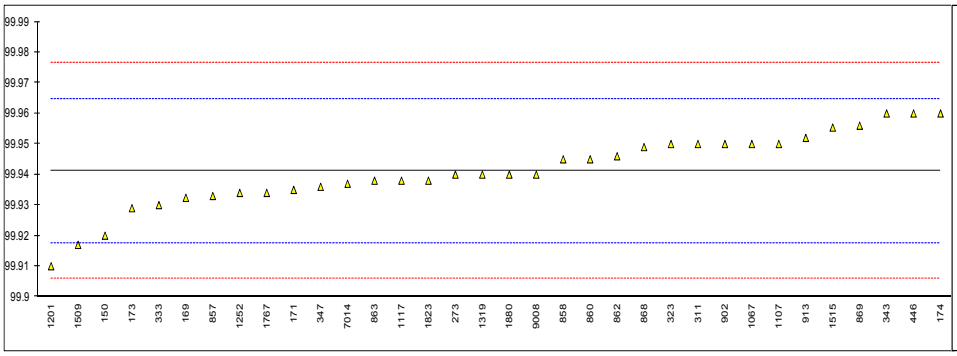


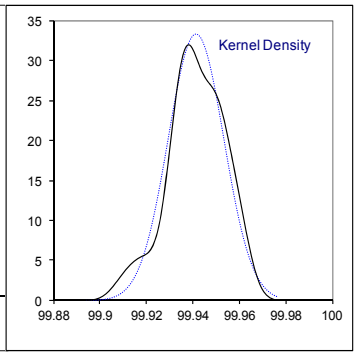


Determination of Purity by GC on sample #16200; results in %M/M

lab	method	value	mark	z(targ)	remarks
150	D5135	99.92		-1.79	
169	D5135	99.9324		-0.74	
171	D5135	99.935		-0.52	
173	D5135	99.929		-1.03	
174	D5135	99.96		1.60	
273	D5135	99.94	C	-0.10	first reported: 99.87
311	D5135	99.95		0.75	
323	D5135	99.95		0.75	
333	D5135	99.93		-0.95	
343	D5135	99.96		1.60	
347	D5135	99.936		-0.44	
396		-----		-----	
446	D5135	99.96		1.60	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D5135	99.933		-0.69	
858	D5135	99.945		0.33	
860	D5135	99.945		0.33	
862	D5135	99.946		0.41	
863	D5135	99.938		-0.27	
868	D5135	99.949		0.67	
869	D5135	99.956		1.26	
902	D5135	99.95		0.75	
913	D5135	99.952		0.92	
1067	D5135	99.95		0.75	
1107	D5135	99.95		0.75	
1117	D5135	99.938		-0.27	
1201	D5135	99.91		-2.64	
1252	D5135	99.934		-0.61	
1264		-----		-----	
1319		99.94		-0.10	
1429		-----		-----	
1509	D5135	99.917		-2.05	
1515	D7504	99.9554		1.21	
1767	D5135	99.934		-0.61	
1823	D5135	99.938		-0.27	
1866		-----		-----	
1880	D5135	99.940		-0.10	
6013		-----		-----	
7014	D5135	99.937		-0.35	
9008	D5135	99.94		-0.10	

	normality	OK	
	n	34	
	outliers	0	
	mean (n)	99.9411	
	st.dev. (n)	0.01199	
	R(calc.)	0.0336	
	R(D5135:14)	0.0330	compare R(D1535:16) = 0.0295

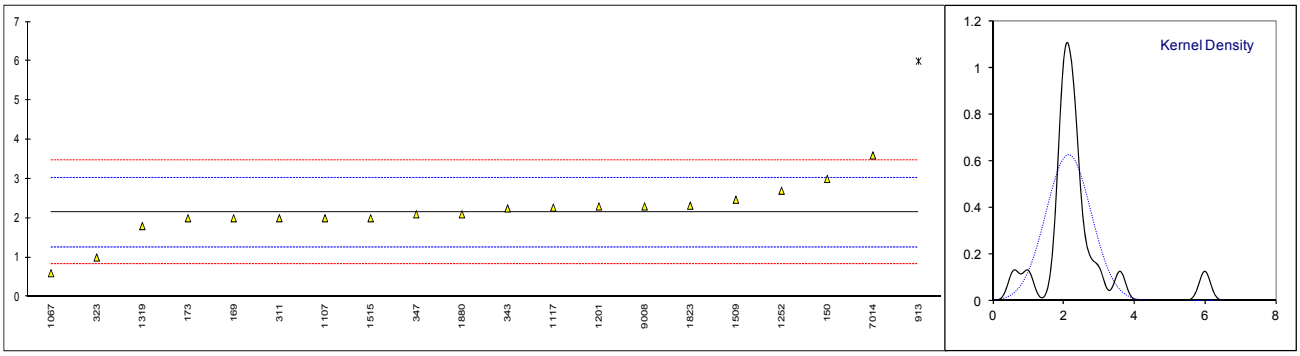




Determination of Benzene on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	3	C	1.93	first reported: 4
169	D5135	2		-0.34	
171		-----		-----	
173	D7504	2		-0.34	
174	D5135	<10		-----	
273		-----		-----	
311	D5135	2		-0.34	
323	D6229	1		-2.60	
333		-----		-----	
343		2.25		0.23	
347	INH-CM3015	2.1		-0.11	
396		-----		-----	
446	D5135	<10		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D5135	<10		-----	
858	D5135	<10		-----	
860	D5135	<10		-----	
862	D5135	<10	C	-----	first reported: 5
863	D5135	<10	C	-----	first reported: 4
868	D5135	<10		-----	
869	D5135	<10		-----	
902	INH-123	<10		-----	
913	D5135	6	R(0.01)	8.73	
1067		0.6		-3.51	
1107	D5135	2		-0.34	
1117	In house	2.27		0.28	
1201	In house	2.3		0.34	
1252	D5135	2.70		1.25	
1264		-----		-----	
1319		1.8		-0.79	
1429		-----		-----	
1509	In house	2.47		0.73	
1515	INH-2922	2	C	-0.34	first reported: 5.96
1767		-----		-----	
1823	D6229	2.32		0.39	
1866		-----		-----	
1880	D4534	2.1		-0.11	
6013		-----		-----	
7014	D5135	3.6		3.29	
9008	D6229	2.3		0.34	

	normality	not OK	
	n	19	
	outliers	1	spike
	mean (n)	2.15	2.2 (recovery: <98%)
	st.dev. (n)	0.637	
	R(calc.)	1.78	
	R(D6229:06)	1.23	compare R(Horwitz) = 0.86 and R(D1535:16) = 0.92



Determination of Toluene on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	15	D(0.05)	-----	possibly a false positive test result?
169		-----		-----	
171	D5135	<1		-----	
173	D7504	<1		-----	
174	D5135	<10		-----	
273		-----		-----	
311	D5135	<10		-----	
323	D5135	<1		-----	
333		-----		-----	
343		-----		-----	
347		-----		-----	
396		-----		-----	
446	D5135	<10		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D5135	<10		-----	
858	D5135	<10		-----	
860	D5135	<10		-----	
862	D5135	<1		-----	
863	D5135	<10		-----	
868	D5135	<10		-----	
869		-----		-----	
902		-----		-----	
913	D5135	nd		-----	
1067		-----		-----	
1107	D5135	<10		-----	
1117	D5135	2		-----	
1201	D5135	5		-----	
1252	D5135	3.50		-----	
1264		-----		-----	
1319		2.2		-----	
1429		-----		-----	
1509	D5135	<5		-----	
1515	D7504	<5		-----	
1767		-----		-----	
1823	D5135	<10		-----	
1866		-----		-----	
1880		-----		-----	
6013		-----		-----	
7014	D5135	0.9		-----	
9008		<1		-----	

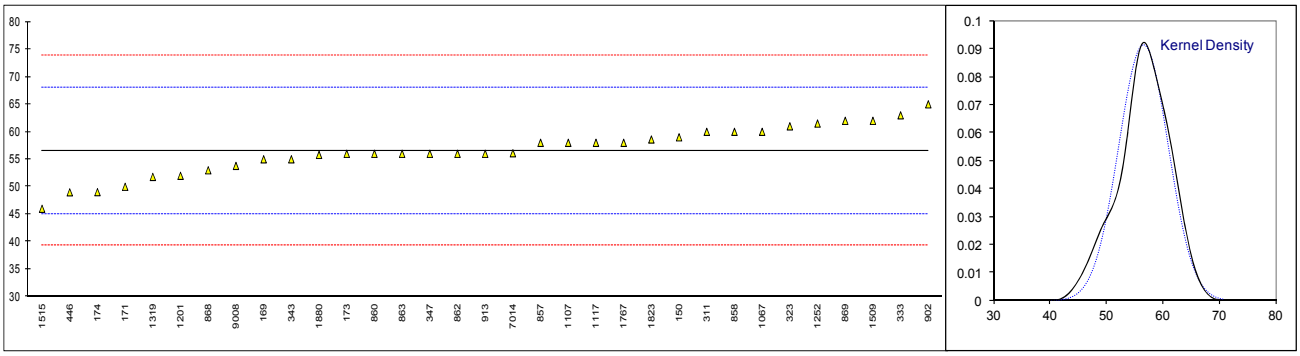
normality	n.a.
n	22
outliers	1
mean (n)	<10
st.dev. (n)	n.a.
R(calc.)	n.a.
R(lit)	n.a.

Determination of Ethylbenzene on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	59		0.42	
169	D5135	55		-0.27	
171	D5135	50		-1.14	
173	D5135	56		-0.10	
174	D5135	49.03		-1.31	
273		-----		-----	
311	D5135	60		0.60	
323	D5135	61		0.77	
333	D5135	63		1.11	
343	D5135	55		-0.27	
347	D5135	56		-0.10	
396		-----		-----	
446	D5135	49		-1.31	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D5135	58		0.25	
858	D5135	60		0.60	
860	D5135	56		-0.10	
862	D5135	56		-0.10	
863	D5135	56		-0.10	
868	D5135	53		-0.62	
869	D5135	62		0.94	
902	D5135	65		1.46	
913	D5135	56		-0.10	
1067	D5135	60		0.60	
1107	D5135	58		0.25	
1117	D5135	58		0.25	
1201	D5135	52		-0.79	
1252	D5135	61.5		0.86	
1264		-----		-----	
1319		51.8		-0.83	
1429		-----		-----	
1509	D5135	62		0.94	
1515	D7504	46		-1.83	
1767	D5135	58	C	0.25	first reported: 0.0058 mg/kg
1823	D5135	58.6		0.35	
1866		-----		-----	
1880	D5135	55.8		-0.13	
6013		-----		-----	
7014	D5135	56.1		-0.08	
9008	D5135	53.8		-0.48	

	normality	OK
	n	33
	outliers	0
	mean (n)	56.56
	st.dev. (n)	4.365
	R(calc.)	12.22
	R(D5135:14)	16.16

compare R(Horwitz) = 13.80 and R(D1535:16) = 10.81



Determination of m- & p-Xylenes on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	5		-----	
169	D5135	<10		-----	
171	D5135	<1		-----	
173	D5135	6		-----	
174	D5135	<10		-----	
273		-----		-----	
311	D5135	<10		-----	
323	D5135	<10		-----	
333		-----		-----	
343	D5135	<10		-----	
347	D5135	<10		-----	
396		-----		-----	
446	D5135	<10		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D5135	<10		-----	
858	D5135	<10		-----	
860	D5135	<10		-----	
862	D5135	<10		-----	
863	D5135	<10		-----	
868	D5135	<10		-----	
869		-----		-----	
902		-----		-----	
913	D5135	<5		-----	
1067		-----		-----	
1107	D5135	<10		-----	
1117	D5135	7		-----	
1201	D5135	1		-----	
1252	D5135	8.2		-----	
1264		-----		-----	
1319		Less than 4		-----	
1429		-----		-----	
1509	D5135	5		-----	
1515	D7504	5		-----	
1767		-----		-----	
1823	D5135	<10		-----	
1866		-----		-----	
1880	D5135	4.4		-----	
6013		-----		-----	
7014	D5135	1.7		-----	
9008	D5135	<10		-----	

normality	n.a.
n	28
outliers	0
mean (n)	<10
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D5135:14)	n.a.

Determination of iso-Propylbenzene (Cumene) on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	4		-----	
169	D5135	<10		-----	
171	D5135	<1		-----	
173	D5135	4		-----	
174	D5135	<10		-----	
273		-----		-----	
311	D5135	<10		-----	
323	D5135	<10		-----	
333	D5135	<10		-----	
343	D5135	<10		-----	
347	D5135	<10		-----	
396		-----		-----	
446	D5135	<10		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D5135	<10		-----	
858	D5135	<10		-----	
860	D5135	<10		-----	
862	D5135	3		-----	
863	D5135	<10		-----	
868	D5135	<10		-----	
869	D5135	<10		-----	
902		-----		-----	
913	D5135	nd		-----	
1067		-----		-----	
1107	D5135	<10		-----	
1117	D5135	3		-----	
1201	D5135	<10		-----	
1252	D5135	4.0		-----	
1264		-----		-----	
1319		5.0		-----	
1429		-----		-----	
1509	D5135	<5		-----	
1515	D7504	<5		-----	
1767		-----		-----	
1823	D5135	2.2		-----	
1866		-----		-----	
1880	D5135	3.3		-----	
6013		-----		-----	
7014	D5135	3.1		-----	
9008	D5135	<10		-----	
	normality	n.a.			
	n	29			
	outliers	0			
	mean (n)	<10			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

Determination of o-Xylene on sample #16200; results in mg/kg

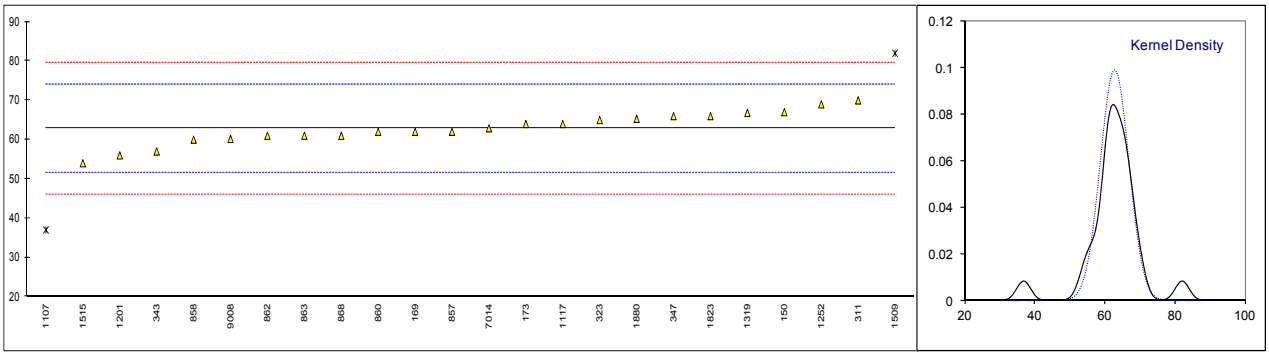
lab	method	value	mark	z(targ)	remarks
150	D5135	2		-----	
169	D5135	33	DG(0.05)	-----	possibly a false positive test result?
171	D5135	30	DG(0.05)	-----	possibly a false positive test result?
173	D5135	14		-----	
174	D5135	<10		-----	
273		-----		-----	
311	D5135	10		-----	
323	D5135	<10		-----	
333	D5135	15		-----	
343	D5135	<10		-----	
347	D5135	<10		-----	
396		-----		-----	
446	D5135	<10		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D5135	<10		-----	
858	D5135	<10		-----	
860	D5135	<10		-----	
862	D5135	<10		-----	
863	D5135	<10		-----	
868	D5135	<10		-----	
869		-----		-----	
902		-----		-----	
913	D5135	nd		-----	
1067		-----		-----	
1107	D5135	<10		-----	
1117	D5135	2		-----	
1201	D5135	<10		-----	
1252	D5135	2.5		-----	
1264		-----		-----	
1319		2.0		-----	
1429		-----		-----	
1509	D5135	<5		-----	
1515	D7504	<5		-----	
1767		-----		-----	
1823	D5135	<10		-----	
1866		-----		-----	
1880	D5135	1.4		-----	
6013		-----		-----	
7014	D5135	1.3		-----	
9008	D5135	<10		-----	

normality	n.a.
n	23
outliers	2
mean (n)	<10
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D5135:14)	n.a.

Determination of n-Propylbenzene on sample #16200; results in mg/kg

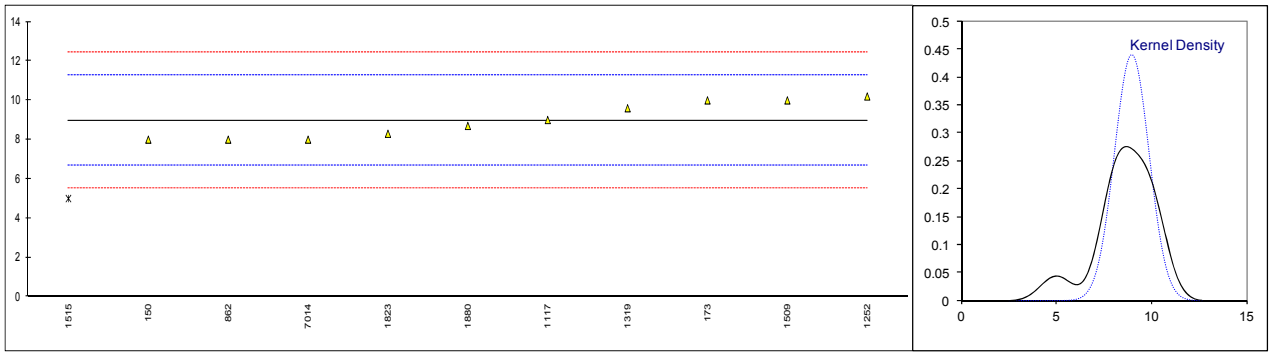
lab	method	value	mark	z(targ)	remarks
150	D5135	67		0.74	
169	D5135	62		-0.15	
171	D5135	<1		<-11.02	possibly a false negative test result?
173	D5135	64		0.21	
174	D5135	<10		<-9.42	possibly a false negative test result?
273		-----		-----	
311	D5135	70		1.28	
323	D5135	65		0.39	
333		-----		-----	
343	D5135	57		-1.04	
347	D5135	66		0.57	
396		-----		-----	
446		-----		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D5135	62		-0.15	
858	D5135	60		-0.50	
860	D5135	62		-0.15	
862	D5135	61		-0.33	
863	D5135	61		-0.33	
868	D5135	61		-0.33	
869		-----		-----	
902		-----		-----	
913		-----		-----	
1067		-----		-----	
1107	D5135	37	R(0.01)	-4.60	
1117	D5135	64		0.21	
1201	D5135	56		-1.22	
1252	D5135	69.0		1.10	
1264		-----		-----	
1319		66.8		0.71	
1429		-----		-----	
1509	D5135	82	R(0.01)	3.42	
1515	D7504	54		-1.57	
1767		-----		-----	
1823	D5135	66.0		0.57	
1866		-----		-----	
1880	D5135	65.3		0.44	
6013		-----		-----	
7014	D5135	62.9		0.01	
9008	D5135	60.2		-0.47	

	normality	OK	
	n	22	
	outliers	2	spike
	mean (n)	62.83	50.3 (recovery: < 125%)
	st.dev. (n)	4.035	
	R(calc.)	11.30	
	R(D5135:14)	15.71	compare R(Horwitz) = 15.09 and R(D1535:16) = R(D1535:14)



Determination of m- & p-Ethyltoluenes on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	8		-0.86	
169		-----		-----	
171	D5135	<1		<-6.96	possibly a false negative test result?
173	D5135	10		0.89	
174	D5135	<10		-----	
273		-----		-----	
311	D5135	<10		-----	
323		-----		-----	
333		-----		-----	
343	D5135	<10		-----	
347		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857		-----		-----	
858		-----		-----	
860		-----		-----	
862	D5135	8		-0.86	
863	D5135	<10		-----	
868	D5135	<10		-----	
869		-----		-----	
902		-----		-----	
913		-----		-----	
1067		-----		-----	
1107		-----		-----	
1117	D5135	9		0.02	
1201		-----		-----	
1252	D5135	10.2		1.07	
1264		-----		-----	
1319		9.6		0.54	
1429		-----		-----	
1509	D5135	10		0.89	
1515	D7504	5	D(0.05)	-3.47	
1767		-----		-----	
1823	D5135	8.3		-0.59	
1866		-----		-----	
1880	D5135	8.7		-0.24	
6013		-----		-----	
7014	D5135	8		-0.86	
9008	D5135	<10		-----	
	normality	OK			
	n	10			
	outliers	1			
	mean (n)	8.98			
	st.dev. (n)	0.905			
	R(calc.)	2.53			
	R(D5135:14)	3.21			compare R(Horwitz 2 comp) = 4.09 and R(D1535:16) = 4.53

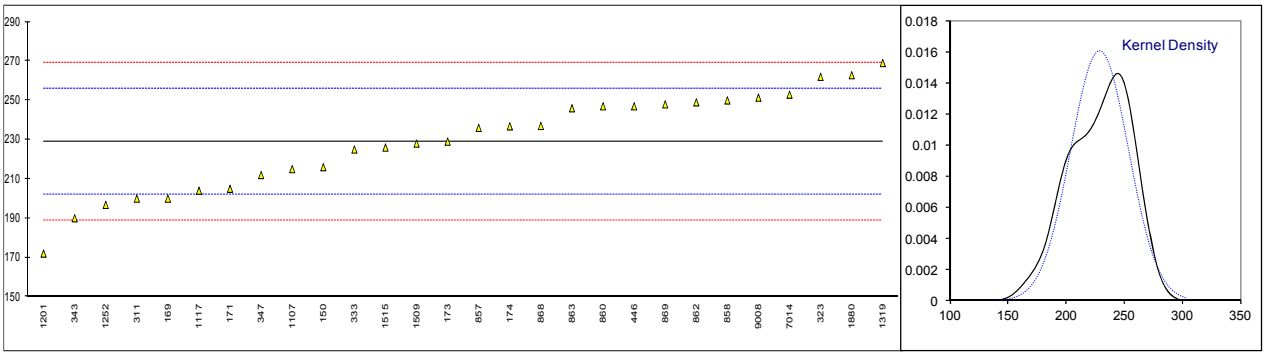


Determination of α-Methylstyrene on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	216		-0.97	
169	D5135	200		-2.17	
171	D5135	205		-1.79	
173	D5135	229		0.00	
174	D5135	236.8		0.58	
273		-----		-----	
311	D5135	200		-2.17	
323	D5135	262		2.45	
333	D5135	225		-0.30	
343	D5135	190		-2.91	
347	D5135	212		-1.27	
396		-----		-----	
446	D5135	247		1.34	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D5135	236		0.52	
858	D5135	250		1.56	
860	D5135	247		1.34	
862	D5135	249		1.49	
863	D5135	246		1.26	
868	D5135	237		0.59	
869	D5135	248		1.41	
902		-----		-----	
913		-----		-----	
1067		-----		-----	
1107	D5135	215		-1.05	
1117	D5135	204		-1.87	
1201	D5135	172		-4.25	
1252	D5135	196.8		-2.40	
1264		-----		-----	
1319		269		2.98	
1429		-----		-----	
1509	D5135	228		-0.08	
1515	D7504	226		-0.23	
1767		-----		-----	
1823		-----		-----	
1866		-----		-----	
1880	D5135	262.8		2.51	
6013		-----		-----	
7014	D5135	252.9		1.78	
9008	D5135	251.4		1.66	

normality	OK
n	28
outliers	0
mean (n)	229.06
st.dev. (n)	24.786
R(calc.)	69.40
R(D5135:14)	37.57

compare R(Horwitz) = 45.29 and R(D1535:16) = 38.67



Determination of 1,2-Diethylbenzene on sample #16200; results in mg/kg

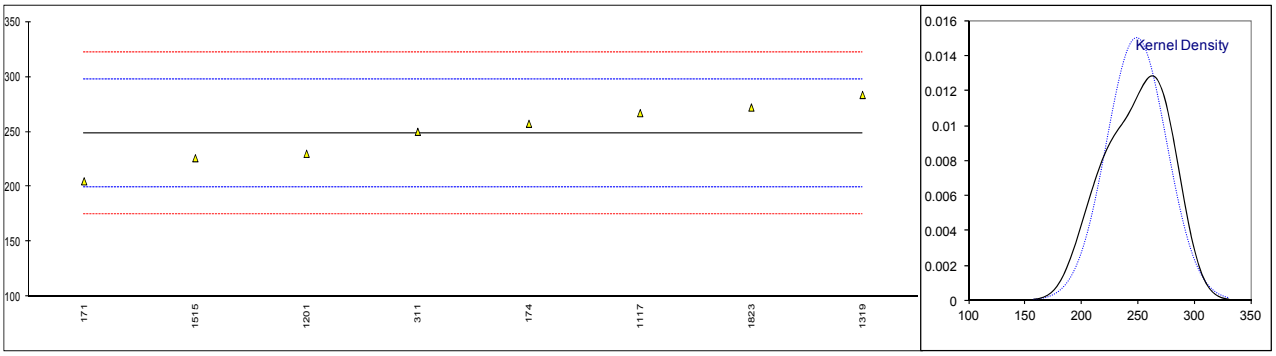
lab	method	value	mark	z(targ)	remarks
150		-----		-----	
169		-----		-----	
171	D5135	<1		-----	possibly a false negative test result?
173		-----		-----	
174	D5135	20.5		-----	
273		-----		-----	
311	D5135	50		-----	
323		-----		-----	
333		-----		-----	
343		-----		-----	
347		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857		-----		-----	
858		-----		-----	
860		-----		-----	
862		-----		-----	
863		-----		-----	
868		-----		-----	
869		-----		-----	
902		-----		-----	
913		-----		-----	
1067		-----		-----	
1107		-----		-----	
1117	D5135	63		-----	
1201	D5135	58		-----	
1252		-----		-----	
1264		-----		-----	
1319		-----		-----	
1429		-----		-----	
1509		-----		-----	
1515	D7504	<5		-----	possibly a false negative test result?
1767		-----		-----	
1823		-----		-----	
1866		-----		-----	
1880		-----		-----	
6013		-----		-----	
7014		-----		-----	
9008		-----		-----	

	normality	unknown	
	n	4	
	outliers	0	spike
	mean (n)	47.88	50.5 (recovery <95%)
	st.dev. (n)	19.019	
	R(calc.)	53.25	
	R(Horwitz)	(11.98)	

Determination of sum of α-Methylstyrene and 1,2-Diethylbenzene on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
169		-----		-----	
171	D5135	205		-1.79	
173		-----		-----	
174	D5135	257.3		0.34	
273		-----		-----	
311	D5135	250		0.05	
323		-----		-----	
333		-----		-----	
343		-----		-----	
347		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857		-----		-----	
858		-----		-----	
860		-----		-----	
862		-----		-----	
863		-----		-----	
868		-----		-----	
869		-----		-----	
902		-----		-----	
913		-----		-----	
1067		-----		-----	
1107		-----		-----	
1117	D5135	267		0.74	
1201	D5135	230		-0.77	
1252		-----		-----	
1264		-----		-----	
1319		283.5		1.41	
1429		-----		-----	
1509		-----		-----	
1515	D7504	226		-0.93	
1767		-----		-----	
1823	D5135	272.1		0.95	
1866		-----		-----	
1880		-----		-----	
6013		-----		-----	
7014		-----		-----	
9008		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	248.86
st.dev. (n)	26.575
R(calc.)	74.41
R(Horwitz 2 comp)	68.73



Determination of Phenylacetylene on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	6		-----	
169	D5135	<10		-----	
171	D5135	<1		-----	
173	D5135	5		-----	
174	D5135	<10		-----	
273		-----		-----	
311	D5135	10		-----	
323	D5135	<10		-----	
333	D5135	<10		-----	
343		-----		-----	
347	D5135	<10		-----	
396		-----		-----	
446	D5135	<10		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D5135	11		-----	
858	D5135	<10		-----	
860	D5135	<10		-----	
862	D5135	<10		-----	
863	D5135	9		-----	
868		-----		-----	
869	D5135	<10		-----	
902		-----		-----	
913		-----		-----	
1067		-----		-----	
1107	D5135	5		-----	
1117	D5135	12		-----	
1201	D5135	<10		-----	
1252	D5135	6.3		-----	
1264		-----		-----	
1319		3.4		-----	
1429		-----		-----	
1509	D5135	7		-----	
1515	D7504	<5		-----	
1767		-----		-----	
1823		-----		-----	
1866		-----		-----	
1880	D5135	11.1		-----	
6013		-----		-----	
7014	D5135	9.3		-----	
9008	D5135	<10		-----	
	normality	n.a.			
	n	22			
	outliers	0			
	mean (n)	<10			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

Determination of 3- & 4-Methylstyrenes on sample #16200; results in mg/kg

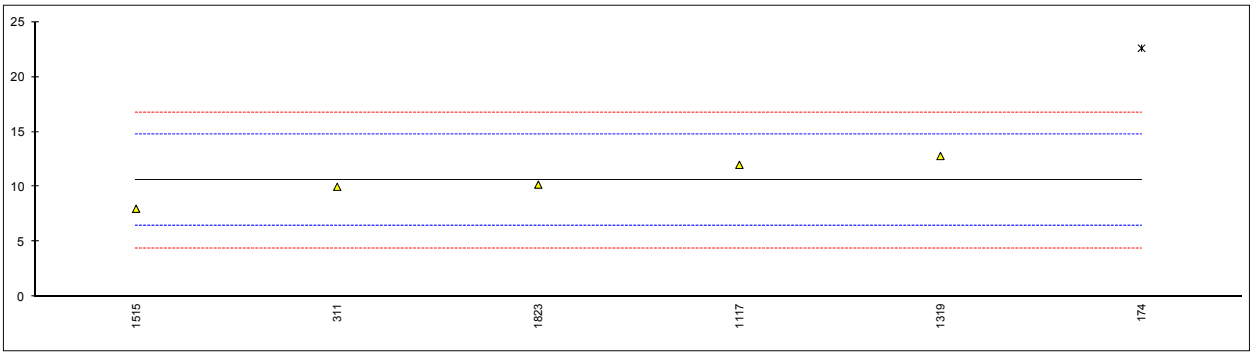
lab	method	value	mark	z(targ)	remarks
150		-----		-----	
169		-----		-----	
171	D5135	<1		-----	
173		-----		-----	
174	D5135	22.6		-----	possibly a false positive test result?
273		-----		-----	
311	D5135	<10		-----	
323		-----		-----	
333		-----		-----	
343		-----		-----	
347		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857		-----		-----	
858		-----		-----	
860		-----		-----	
862		-----		-----	
863		-----		-----	
868		-----		-----	
869		-----		-----	
902		-----		-----	
913		-----		-----	
1067		-----		-----	
1107		-----		-----	
1117	D5135	<5		-----	
1201	D5135	<10		-----	
1252		-----		-----	
1264		-----		-----	
1319		9.5		-----	
1429		-----		-----	
1509		-----		-----	
1515	D7504	8		-----	
1767		-----		-----	
1823		-----		-----	
1866		-----		-----	
1880		-----		-----	
6013		-----		-----	
7014		-----		-----	
9008		-----		-----	

normality	n.a.
n	6
outliers	0
mean (n)	<10
st.dev. (n)	n.a.
R(calc.)	n.a.
R(lit)	n.a.

Determination of sum Phenylacetylene and 3-&4-Methylstyrenes on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
169		-----		-----	
171	D5135	<1		<-4.66	possibly a false negative test result?
173		-----		-----	
174	D5135	22.6	D(0.05)	5.83	
273		-----		-----	
311	D5135	10		-0.29	
323		-----		-----	
333		-----		-----	
343		-----		-----	
347		-----		-----	
396		-----		-----	
446		-----		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857		-----		-----	
858		-----		-----	
860		-----		-----	
862		-----		-----	
863		-----		-----	
868		-----		-----	
869		-----		-----	
902		-----		-----	
913		-----		-----	
1067		-----		-----	
1107		-----		-----	
1117	D5135	12		0.68	
1201	D5135	<20		-----	
1252		-----		-----	
1264		-----		-----	
1319		12.8		1.07	
1429		-----		-----	
1509		-----		-----	
1515	D7504	8		-1.26	
1767		-----		-----	
1823	D5135	10.2		-0.19	
1866		-----		-----	
1880		-----		-----	
6013		-----		-----	
7014		-----		-----	
9008		-----		-----	

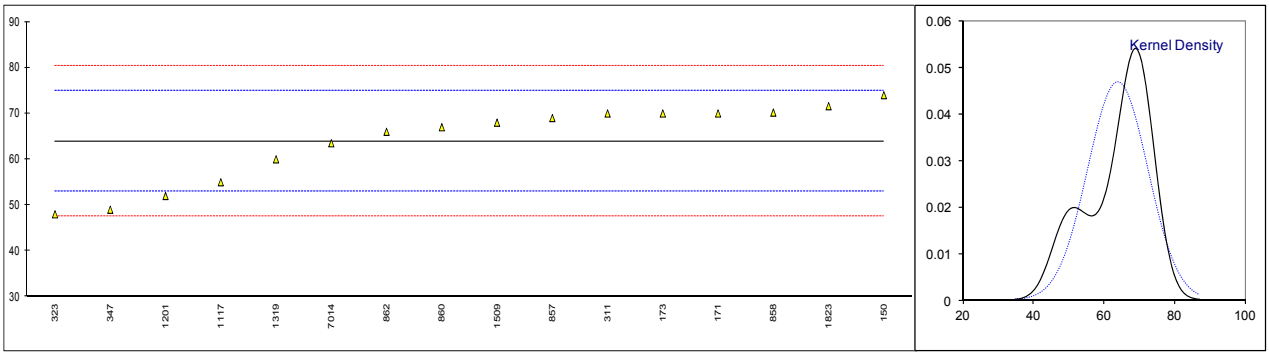
normality	unknown
n	5
outliers	1
mean (n)	10.60
st.dev. (n)	1.876
R(calc.)	5.25
R(Horwitz 3 comp)	5.77



Determination of Benzaldehyde on sample #16200; results in mg/kg

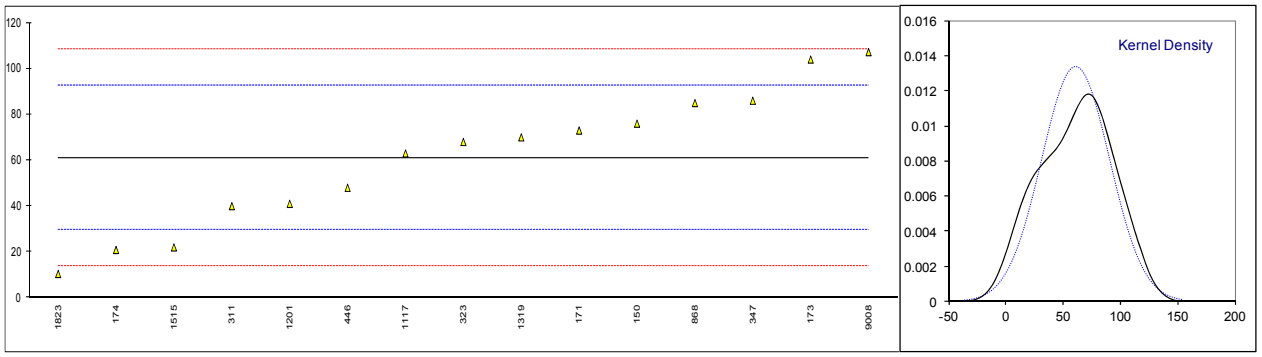
lab	method	value	mark	z(targ)	remarks
150	D5135	74		1.84	
169		-----		-----	
171	D5135	70		1.10	
173	D5135	70		1.10	
174	D5135	<10		<-9.86	possibly a false negative test result?
273		-----		-----	
311	D5135	70		1.10	
323	D5135	48		-2.92	
333		-----		-----	
343		-----		-----	
347	D5135	49		-2.73	
396		-----		-----	
446		-----		-----	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857	D5135	69		0.92	
858	D5135	70.2		1.14	
860	D5135	67		0.56	
862	D5135	66		0.37	
863		-----		-----	
868		-----		-----	
869		-----		-----	
902		-----		-----	
913		-----		-----	
1067		-----		-----	
1107		-----		-----	
1117	D5135	55		-1.64	
1201	D5135	52		-2.18	
1252		-----		-----	
1264		-----		-----	
1319		60.0		-0.72	
1429		-----		-----	
1509	D5135	68		0.74	
1515		-----		-----	
1767		-----		-----	
1823	D5135	71.6		1.40	
1866		-----		-----	
1880		-----		-----	
6013		-----		-----	
7014	D5135	63.5		-0.08	
9008		-----		-----	

normality	OK	
n	16	
outliers	0	
mean (n)	63.96	
st.dev. (n)	8.487	
R(calc.)	23.76	
R(Horwitz)	15.32	compare R(D5135:16) = 23.98



Determination of Nonaromatics on sample #16200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D5135	76		0.95	
169		-----		-----	
171	D5135	73		0.76	
173	D5135	104		2.73	
174	D5135	20.86		-2.54	
273		-----		-----	
311	D5135	40		-1.33	
323	D5135	68		0.45	
333		-----		-----	
343		-----		-----	
347	D5135	86		1.59	
396		-----		-----	
446	D5135	48		-0.82	
551		-----		-----	
557		-----		-----	
613		-----		-----	
857		-----		-----	
858		-----		-----	
860		-----		-----	
862		-----		-----	
863		-----		-----	
868	D5135	85		1.52	
869		-----		-----	
902		-----		-----	
913		-----		-----	
1067		-----		-----	
1107		-----		-----	
1117	D5135	63		0.13	
1201	D5135	41		-1.27	
1252	D5135	<1		<-3.80	possibly a false negative test result?
1264		-----		-----	
1319		70		0.57	
1429		-----		-----	
1509		-----		-----	
1515	D7504	22		-2.47	
1767		-----		-----	
1823	D5135	10.4		-3.21	
1866		-----		-----	
1880		-----		-----	
6013		-----		-----	
7014		-----		-----	
9008	D5135	107.2		2.93	
	normality	OK			
	n	15			
	outliers	0			
	mean (n)	60.96			
	st.dev. (n)	29.749			
	R(calc.)	83.30			
	R(Horwitz 9 comp)	44.14			compare R(D5135:16) = 71.13



APPENDIX 2

Number of participants per country

1 lab in	AUSTRALIA
2 labs in	BELGIUM
2 labs in	BRAZIL
1 lab in	CANADA
9 labs in	CHINA, People's Republic
1 lab in	FRANCE
1 lab in	INDIA
1 lab in	IRAN, Islamic Republic of
1 lab in	ITALY
1 lab in	JAPAN
2 labs in	KUWAIT
4 labs in	NETHERLANDS
2 labs 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
2 labs in	UNITED KINGDOM
5 labs in	UNITED STATES OF AMERICA

APPENDIX 3

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 calculations
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, April 2014
2 ASTM E178:02
3 ASTM E1301:03
4 ISO13528:15
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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.
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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)