

Results of Proficiency Test
Gas condensate
November 2015

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2008, the Institute for Interlaboratory Studies organizes a proficiency test for Gas condensate every year. During the annual proficiency testing program 2015/2016, it was decided to continue the round robin for the analysis of Gas condensate. In this interlaboratory study, 45 laboratories from 19 different countries have participated. See appendix 3 for the number of participating laboratories per country. In this report, the results of the 2015 Gas condensate proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. It was decided to send 1 sample of Gas condensate (1 * 0.5 L bottle labelled #15217). Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. 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 April 2014 (iis-protocol, version 3.3). This protocol is electronically available through the iis internet site 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, approximately 100 litre, was obtained from a participating laboratory. After homogenisation, 64 amber glass bottles of 0.5 litre were filled and labelled as sample #15217.

The homogeneity of the subsamples #15217 was checked by determination of Density at 15 °C in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Density at 15 °C in kg/m ³
Sample #15217-1	745.92
Sample #15217-2	745.87
Sample #15217-3	745.89
Sample #15217-4	745.86
Sample #15217-5	745.94
Sample #15217-6	745.92
Sample #15217-7	745.91
Sample #15217-8	745.90

Table 1: homogeneity test results of subsamples #15217

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 15 °C in kg/m ³
r sample	0.08
reference method	ASTM D4052:11
0.3xR(reference method)	0.61

Table 2: repeatability of subsamples #15217

The calculated repeatability was less than 0.3 times the reproducibility of the respective reference method. Therefore, homogeneity of the subsamples #15217 was assumed.

To each of the participating laboratories, 1 * 0.5 L bottle (labelled #15217) was sent on October 21, 2015.

2.5 STABILITY OF THE SAMPLES

The stability of Gas condensate, packed in the brown glass bottles, 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 #15217: Color Saybolt (Automated and Manual), Density at 15°C, Distillation (IBP, temperature at 5%, 10%, 50%, 90%, 95% recovered and FBP), Mercury, total Sulphur, Water by KF and Simulated Distillation.

To get maximum information for the statistical calculations, the participants were requested to report unrounded results and results below the usual lower reporting limits, where possible.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/.

A SDS and a form to confirm receipt of the samples were added to the samples.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The original data are tabulated per determination in the appendix 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 results at that moment. Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

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 April 2014 (iis-protocol, version 3.3).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<...>' or '>...>' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test 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 results should be used with due care.

According to ISO 5725 the original results per determination were submitted to Dixon's and/or 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 (ref. 14). 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 visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. 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. The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated in accordance with:

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

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

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

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

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

4 EVALUATION

In this proficiency test, problems were encountered during the transport of the samples to the laboratories in Malaysia. Some samples took an unexpected long time to reach the laboratory due to several problems with custom clearance and/or transport companies. Six participants reported test results after the final reporting date and seven laboratories did not report any test results at all. In total 38 laboratories reported 248 numerical results. Observed were 8 outlying results, which is 3.2%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section, the results are discussed per test.

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.

Color Saybolt: Both the automated version (ASTM D6045) and the manual version (ASTM D156) were evaluated. For the automated version 12 participants returned a test result and 6 participants returned a result for the manual version. This limited number of results may have some effect on the statistical evaluation. The automated determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D6045:12.

The manual determination was very problematic. No statistical outliers were observed. The calculated reproducibility is not at all in agreement with the requirements of ASTM D156:15.

Density at 15°C: This determination was problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D4052:11.

It should be taken into account that the reproducibility from ASTM D4052:11 is applicable to petroleum distillates and viscous oils only.

Therefore no precision data are stated in the 2011 version for Gas condensates. However, Gas condensates may contain relatively high concentrations of light ends and therefore should be treated as gasoline, i.e. cooling the sample prior to analysis to prevent loss of light ends.

Distillation: This determination may be problematic. In total three statistical outliers were observed. After rejection of the statistical outliers, the calculated reproducibilities of IBP, temperature at 95% recovered and FBP were in agreement with the requirements of the manual mode of ASTM D86:15. However, the temperatures at 5%, 10%, 50% en 90% were not in agreement with the requirements of the manual mode of ASTM D86:15. Since the temperature at 95% recovered and the FBP are close together, the reproducibility of the 95% recovered was also used for FBP. It should be noted that the scope of ASTM D86 does not include Gas condensates, but only products with a limited boiling range like distillate fuels, so the target reproducibilities as used in this report may not be applicable. The use of a simulated distillation determination may be more appropriate.

Mercury: The precision requirements of UOP938 (table 3b) are extremely strict and as they are 6 – 7 times more strict than the Horwitz estimate, these requirements will not be met easily. Also, the reproducibility of UOP938 is only available for very low concentrations (0.28 and 12.14 µg/L, table B3) and conversion and extrapolation up to 635 µg/kg will lead to extra uncertainty. Therefore, it was decided to use the Horwitz estimates for evaluation of the test results in this report. This determination may be problematic at a consensus of 635 µg Hg per kg. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated using the Horwitz equation.

Sulphur: 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 D5453:12.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D6304:07. It must be noted that the precision data of ASTM D4928 are not applicable at this low concentration (valid between 0.02 – 5.00%M/M).

Simulated Distillation: Only three participants reported a result for this determination. Therefore no significant conclusions were drawn.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average results of sample #15217, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM standards) are compared in the next table.

Parameter	unit	n	mean	2.8 * sd	R (lit)
Color Saybolt (Automated)		12	22.2	2.0	1.2
Color Saybolt (Manual)		6	21.3	4.6	2.0
Density at 15°C	kg/m ³	34	746.1	1.6	2.0
Initial Boiling Point	°C	14	33.4	7.6	14.5
5%-recovered	°C	14	62.8	10.1	7.3
10%-recovered	°C	12	74.4	5.4	3.9
50%-recovered	°C	14	132.1	5.7	4.9
90%-recovered	°C	14	259.4	19.8	6.8
95%-recovered	°C	6	301.2	8.4	12.9
Final Boiling Point	°C	14	305.4	10.7	12.9
Mercury as Hg	µg/kg	16	635	585	305
Sulphur	mg/kg	21	14.2	6.1	4.2
Water content by KF	mg/kg	30	32	36	136

Table 3: performance evaluation sample #15217

Without further statistical calculations it can be concluded from the overview given in table 3 that for almost all tests there is not a good compliance of the group of participants with the relevant standards. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2015 WITH THE PREVIOUS PTS

	<i>November 2015</i>	<i>November 2014</i>	<i>November 2013</i>	<i>November 2012</i>	<i>November 2011</i>
Number of reporting participants	38	36	36	38	41
Number of results reported	248	251	216	234	283
Number of statistical outliers	8	8	15	25	29
Percentage of statistical outliers	3.2%	3.2%	6.9%	10.7%	10.2%

Table 4: comparison with previous proficiency tests

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given the following table:

Determination	<i>November 2015</i>	<i>November 2014</i>	<i>November 2013</i>	<i>November 2012</i>	<i>November 2011</i>
Color Saybolt	--	--	-	--	--
Density at 15 °C	+	+	++	-	++
Distillation (ASTM D86)	-	--	--	--	--
Mercury as Hg	-	-	--	--	-
Sulphur	-	--	--	++	--
Water content by KF	++	++	++	-	-
Total vapour pressure	n.e.	n.e.	n.e.	-	-
DVPE acc. to ASTM D5191	n.e.	n.e.	n.e.	+/-	+

Table 5: comparison of the performance per determination against the target requirements

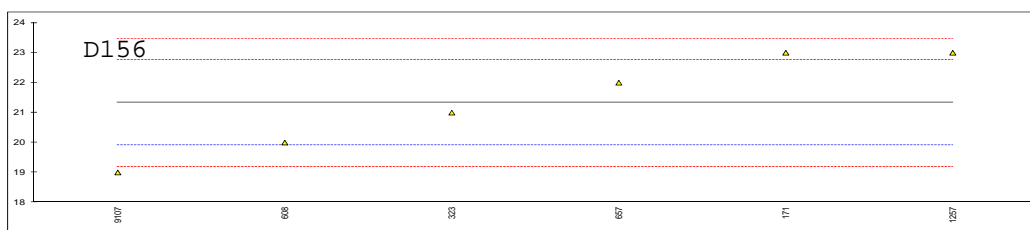
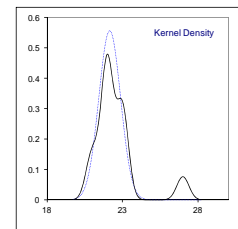
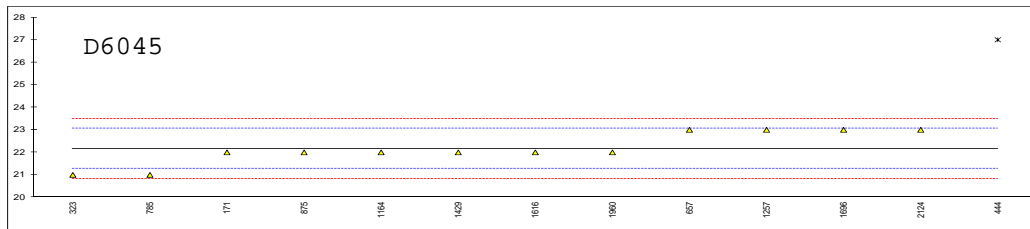
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

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

APPENDIX 1

Determination of Color Saybolt (automated and manual) on sample #15217;

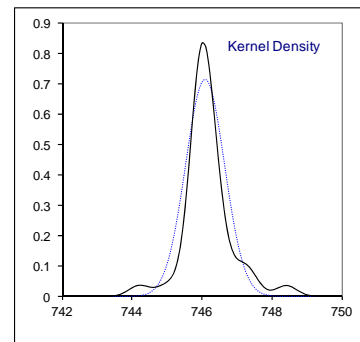
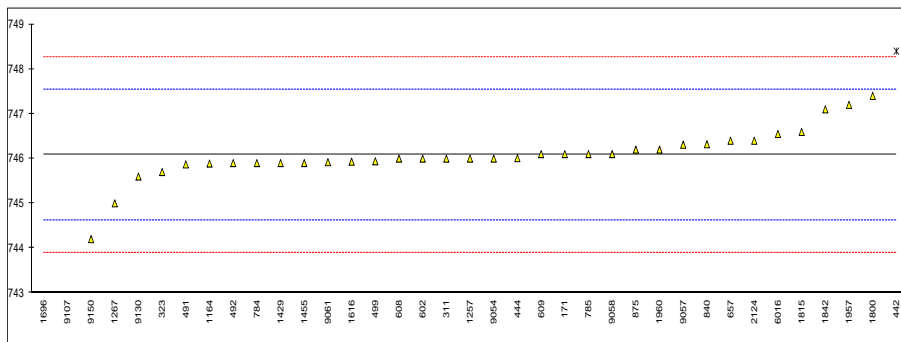
lab	method	value	mark	z(targ)	method	value	mark	z(targ)	remarks
171	D6045	22		-0.38	D156	23		2.33	
311		----		----		----		----	
323	D6045	21		-2.63	D156	21		-0.47	
442		----		----		----		----	
444	D6045	27	G(0.01)	10.91		----		----	
491		----		----		----		----	
492		----		----		----		----	
499		----		----		----		----	
602		----		----		----		----	
608		----		----	D156	20		-1.87	
609		----		----		----		----	
657	D6045	23	C	1.88	D156	22		0.93	first reported D6045: 25
784		----		----		----		----	
785	D6045	21		-2.63		----		----	
840		----		----		----		----	
875	D6045	22		-0.38		----		----	
1164	D6045	22		-0.38		----		----	
1214		----		----		----		----	
1257	D6045	23		1.88	D156	23		2.33	
1267		----		----		----		----	
1397		----		----		----		----	
1429	D6045	22		-0.38		----		----	
1455		----		----		----		----	
1616	D6045	22		-0.38		----		----	
1696	D6045	23		1.88		----		----	
1800		----		----		----		----	
1815		----		----		----		----	
1842		----		----		----		----	
1879		----		----		----		----	
1957		----		----		----		----	
1960	D6045	22		-0.38		----		----	
2124	D6045	23		1.88		----		----	
6009		----		----		----		----	
6016		----		----		----		----	
9050		----		----		----		----	
9054		----		----		----		----	
9055		----		----		----		----	
9056		----		----		----		----	
9057		----		----		----		----	
9058		----		----		----		----	
9061		----		----		----		----	
9107		----		----	D156	19.0		-3.27	
9108		----		----		----		----	
9130		----		----		----		----	
9150		----		----		----		----	
normality		OK			normality	unknown			
n		12			n	6			
outliers		1			outliers	0			
mean (n)		22.17			mean (n)	21.33			
st.dev. (n)		0.718			st.dev. (n)	1.633			
R(calc.)		2.01			R(calc.)	4.57			
R(D6045:12)		1.24			R(D156:15)	2.00			



Determination of Density at 15°C on sample #15217; results in kg/m³

lab	method	value	mark	z(targ)	remarks
171	D4052	746.1		0.03	
311	D4052	746.0		-0.11	
323	D4052	745.7		-0.52	
442	D4052	748.4	R(0.01)	3.19	
444	D4052	746.01		-0.10	
491	ISO12185	745.87	C	-0.29	first reported: 745.94
492	ISO12185	745.90	C	-0.25	first reported: 745.94
499	ISO12185	745.94		-0.19	
602	D1298	746.0		-0.11	
608	D4052	746.0		-0.11	
609	D5002	746.1		0.03	
657	D4052	746.4		0.44	
784	D4052	745.9		-0.25	
785	D4052	746.1		0.03	
840	D4052	746.32		0.33	
875	D4052	746.2		0.16	
1164	D4052	745.89		-0.26	
1214		----		----	
1257	D4052	746.0		-0.11	
1267	IP365	745		-1.49	
1397		----		----	
1429	D4052	745.9		-0.25	
1455	D4052	745.9		-0.25	
1616	D4052	745.93		-0.21	
1696	D1298	723.0	R(0.01)	-31.76	
1800	D4052	747.4		1.82	
1815	ISO12185	746.595		0.71	
1842	D4052	747.1		1.40	
1879		----		----	
1957	D4052	747.2	C	1.54	first reported: 748.2
1960	D4052	746.2		0.16	
2124	D4052	746.4		0.44	
6009		----		----	
6016	D4052	746.55		0.65	
9050		----		----	
9054	D4052	746.0		-0.11	
9055		----		----	
9056		----		----	
9057	D5002	746.31		0.32	
9058	D5002	746.1		0.03	
9061	D5002	745.92		-0.22	
9107	D4052	738.0	R(0.01)	-11.12	
9108		----		----	
9130	D4052	745.6		-0.66	
9150	D4052	744.2		-2.59	

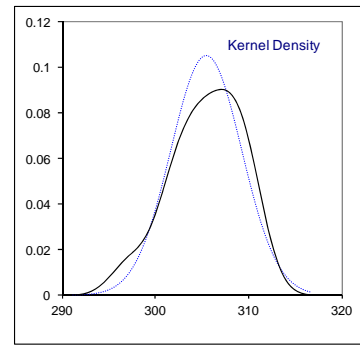
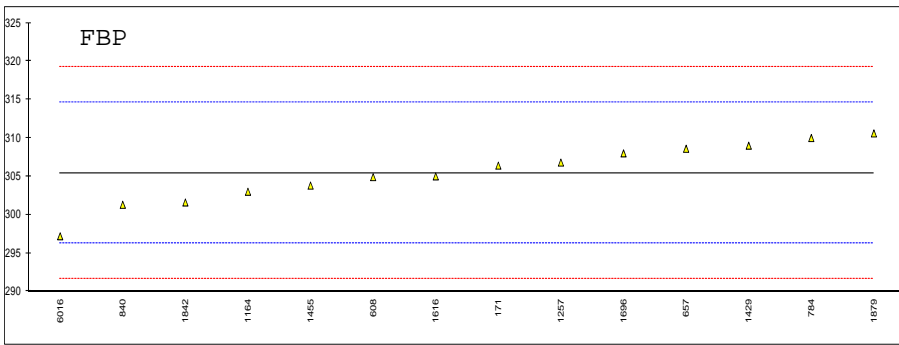
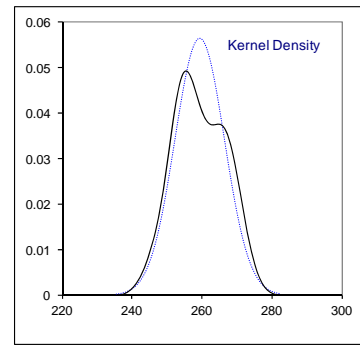
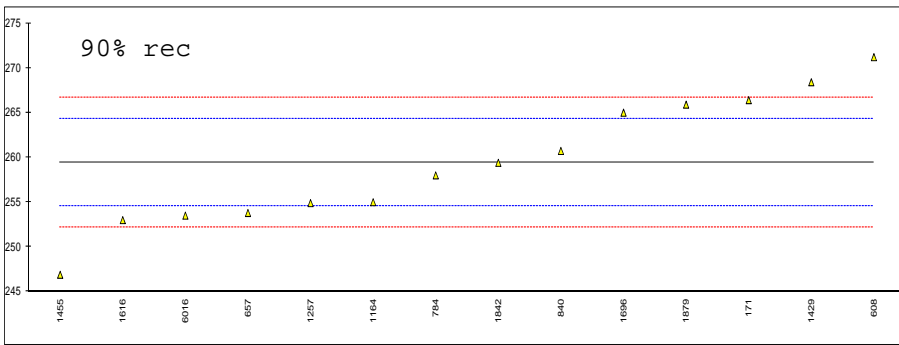
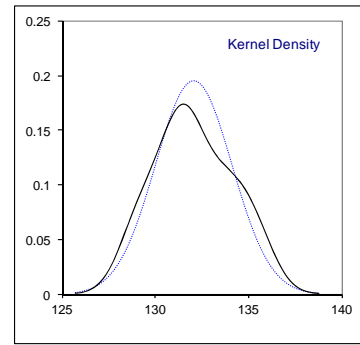
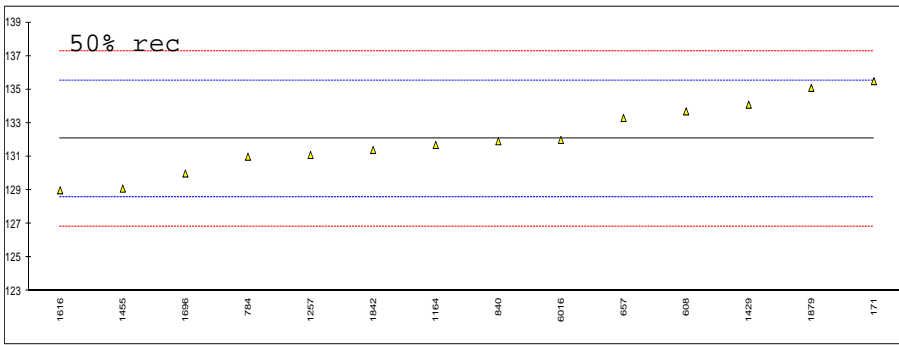
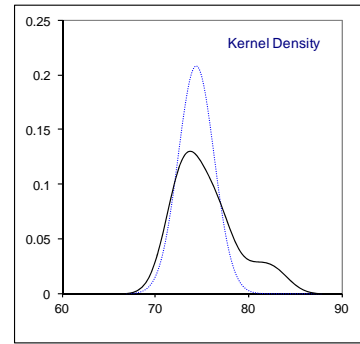
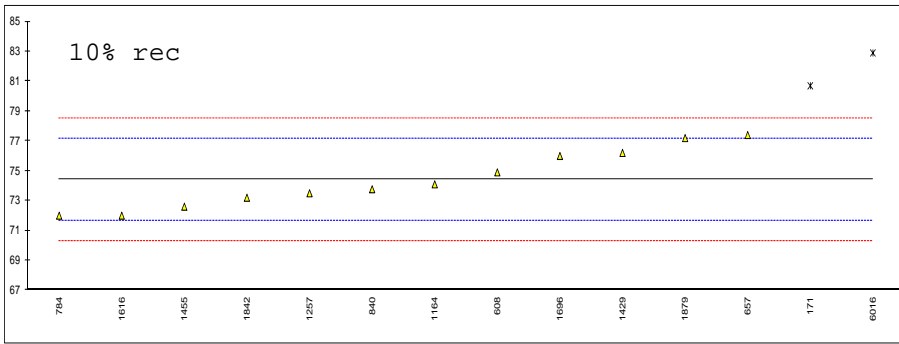
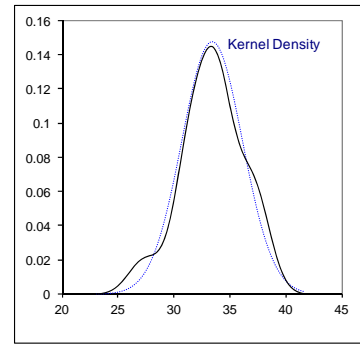
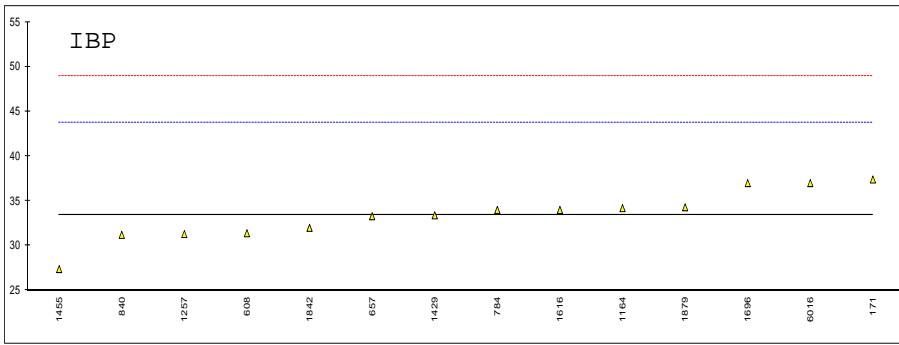
normality not OK
n 34
outliers 3
mean (n) 746.08
st.dev. (n) 0.558
R(calc.) 1.56
R(D4052:11) 2.03



Determination of Distillation on sample #15217; results in °C and %V/V

lab	method	mode	IBP	5% <i>rec</i>	10% <i>rec</i>	50% <i>rec</i>	90% <i>rec</i>	95% <i>rec</i>	FBP	loss	res
171	D86	Automated	37.4	69.1	80.7	135.5	266.4	----	306.4	3.9	1.2
311			----	----	----	----	----	----	----	----	----
323			----	----	----	----	----	----	----	----	----
442			----	----	----	----	----	----	----	----	----
444			----	----	----	----	----	----	----	----	----
491			----	----	----	----	----	----	----	----	----
492			----	----	----	----	----	----	----	----	----
499			----	----	----	----	----	----	----	----	----
602			----	----	----	----	----	----	----	----	----
608	D86	Automated	31.4	61.3	74.9	133.7	271.2	----	304.9	----	1.4
609			----	----	----	----	----	----	----	----	----
657	D86	Automated	33.3	66.1	77.4	133.3	253.8	298.2	308.6	1.3	2.5
784	D86	Manual	34.0	58.5	72.0	131.0	258.0	303.5	310.0	3.0	1.0
785			----	----	----	----	----	----	----	----	----
840	D86	Automated	31.22	61.10	73.77	131.92	260.73	----	301.30	5.1	1.4
875			----	----	----	----	----	----	----	----	----
1164	D86	Automated	34.2	61.4	74.1	131.7	255.0	302.1	303.0	1.6	3.0
1214			----	----	----	----	----	----	----	----	----
1257	D86	Automated	31.3	60.9	73.5	131.1	254.9	304.7	306.8	----	----
1267			----	----	----	----	----	----	----	----	----
1397			----	----	----	----	----	----	----	----	----
1429	D86	Automated	33.4	63.4	76.2	134.1	268.4	----	309.0	4.7	1.4
1455	D86	Automated	27.4	58.2	72.6	129.1	246.9	285.4	303.8	2.8	1.3
1616	D86	Manual	34.0	61.0	72.0	129.0	253.0	297.0	305.0	3.0	1.0
1696	D86	Manual	37.0	64.0	76.0	130.0	265.0	----	308.0	6.90	0.10
1800			----	----	----	----	----	----	----	----	----
1815		Automated	----	----	----	----	----	----	----	----	----
1842	D86	Automated	32.0	59.4	73.2	131.4	259.4	301.6	301.6	3.3	1.1
1879	INH-2177	Automated	34.3	65.1	77.2	135.1	265.9	----	310.6	4.0	1.4
1957			----	----	----	----	----	----	----	----	----
1960			----	----	----	----	----	----	----	----	----
2124			----	----	----	----	----	----	----	----	----
6009			----	----	----	----	----	----	----	----	----
6016	D86		37.0	69.6	82.9	132.0	253.5	----	297.2	3.8	1.3
9050			----	----	----	----	----	----	----	----	----
9054			----	----	----	----	----	----	----	----	----
9055			----	----	----	----	----	----	----	----	----
9056			----	----	----	----	----	----	----	----	----
9057			----	----	----	----	----	----	----	----	----
9058			----	----	----	----	----	----	----	----	----
9061			----	----	----	----	----	----	----	----	----
9107			----	----	----	----	----	----	----	----	----
9108			----	----	----	----	----	----	----	----	----
9130			----	----	----	----	----	----	----	----	----
9150			----	----	----	----	----	----	----	----	----
	normality		OK	OK	OK	OK	OK	unknown	OK		
	n		14	14	12	14	14	6	14		
	outliers		0	0	2	0	0	1	0		
	mean (n)		33.42	62.79	74.41	132.07	259.44	301.18	305.44		
	st.dev. (n)		2.706	3.606	1.916	2.047	7.069	3.005	3.806		
	R(calc.)		7.58	10.10	5.37	5.73	19.79	8.41	10.66		
	R(D86:15-M)		14.48	7.25	3.85	4.88	6.80	12.90	12.90		
	R(D86:15-A)		n.a.	n.a.	n.a.	n.a.	3.89	6.77	7.10		

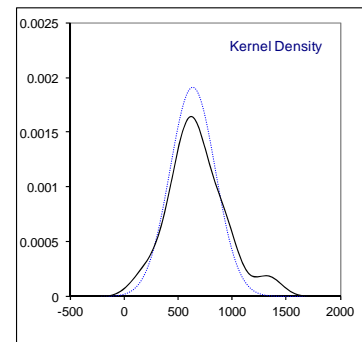
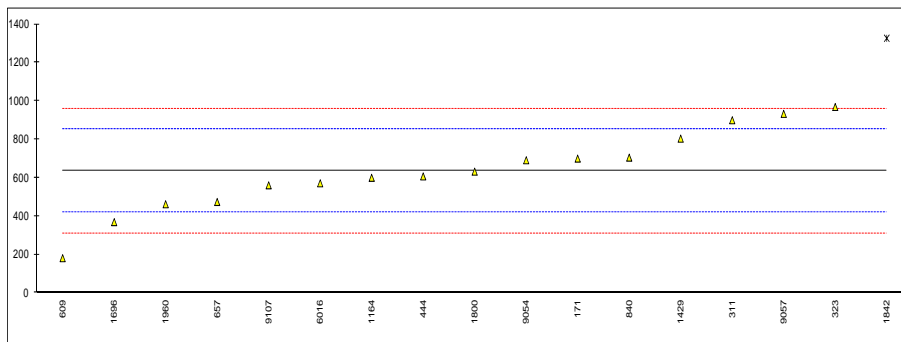
Bold and underlined test results are outliers according to Dixon/Grubbs/Rosner



Determination of Mercury as Hg on sample #15217; results in µg/kg

lab	method	value	mark	z(targ)	remarks
171	UOP938	700		0.60	
311	INH-001	900		2.44	
323	UOP938	969		3.07	
442		----		----	
444	UOP938	607.27		-0.25	
491		----		----	
492		----		----	
499		----		----	
602		----		----	
608		----		----	
609	UOP938	181.02		-4.17	
657	UOP938	474		-1.48	
784		----		----	
785		----		----	
840	INH-010	705.1		0.64	
875		----		----	
1164	UOP938	600		-0.32	
1214		----		----	
1257		----		----	
1267		----		----	
1397		----		----	
1429	in house	803.925		1.55	
1455		----		----	
1616		----		----	
1696	UOP938	369.0		-2.44	
1800	UOP938	631.85		-0.03	
1815		----		----	
1842	UOP938	1327	G(0.05)	6.36	
1879		----		----	
1957		----		----	
1960	UOP938	462		-1.59	
2124		----		----	
6009		----		----	
6016	UOP938	571.2		-0.59	
9050		----		----	
9054	UOP938	691.2994		0.52	
9055		----		----	
9056		----		----	
9057	in house	933		2.74	
9058		----		----	
9061		----		----	
9107	in house	560.8		-0.68	
9108		----		----	
9130		----		----	
9150		----		----	

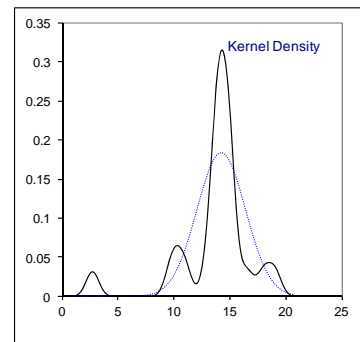
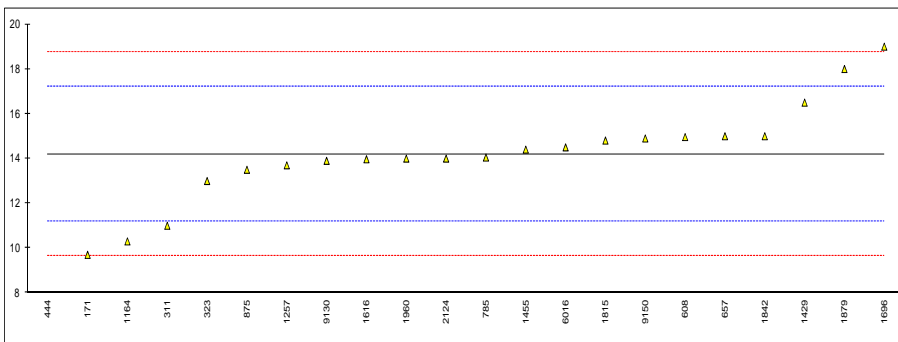
normality OK
n 16
outliers 1
mean (n) 634.967
st.dev. (n) 209.0794
R(calc.) 585.422
R(Horwitz) 304.592



Determination of Sulphur on sample #15217; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D5453	9.7		-2.97	
311	D5453	11		-2.11	
323	D5453	13		-0.79	
442		----		----	
444	D5453	2.69	R(0.01)	-7.60	
491		----		----	
492		----		----	
499		----		----	
602		----		----	
608	D5453	14.96		0.50	
609		----		----	
657	D5453	15		0.53	
784		----		----	
785	ISO20846	14.046		-0.10	
840	D4294	<16		----	
875	ISO20846	13.5		-0.46	
1164	D5453	10.3		-2.57	
1214		----		----	
1257	D5453	13.7		-0.33	
1267		----		----	
1397		----		----	
1429	ISO20846	16.5		1.52	
1455	D2622	14.4		0.13	
1616	D5453	13.97		-0.15	
1696	D5453	19.0		3.17	
1800		----		----	
1815	D5453	14.805		0.40	
1842	D5453	15		0.53	
1879	INH-51947	18	C	2.51	first reported: 23
1957		----		----	
1960	D5453	14		-0.13	
2124	D5453	14		-0.13	
6009		----		----	
6016	D5453	14.5		0.20	
9050		----		----	
9054		----		----	
9055		----		----	
9056		----		----	
9057		----		----	
9058		----		----	
9061		----		----	
9107		----		----	
9108		----		----	
9130	D5453	13.90		-0.20	
9150	D5453	14.9		0.46	

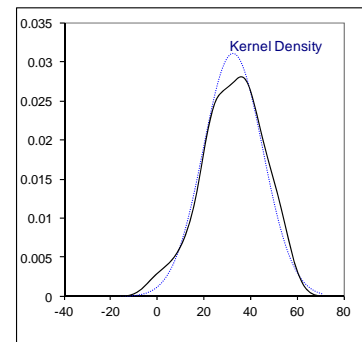
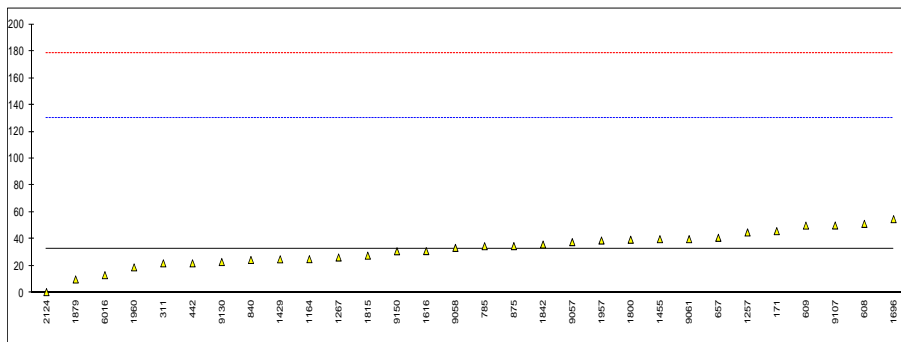
normality suspect
n 21
outliers 1
mean (n) 14.199
st.dev. (n) 2.16501
R(calc.) 6.062
R(D5453:12) 4.240



Determination of Water on sample #15217; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D6304-A	46		0.28	
311	D6304-A	22		-0.22	
323		----		----	
442	IP438	22		-0.22	
444		----		----	
491		----		----	
492		----		----	
499		----		----	
602		----		----	
608	D6304	51.4		0.39	
609	D4928	50.1		0.36	
657	D6304-A	41		0.17	
784		----		----	
785	D6304-A	34.76		0.05	
840	D6304-A	24.5		-0.16	
875	D6304-A	34.84		0.05	
1164	D6304-A	25.1		-0.15	
1214		----		----	
1257	D6304-A	45		0.26	
1267	D4928	26.36		-0.13	
1397		----		----	
1429	IP438	25		-0.15	
1455	ISO12937	40		0.15	
1616	UOP481	31.11		-0.03	
1696	D6304-A	54.9		0.46	
1800	D6304-A	39.6		0.15	
1815	ISO12937	27.73		-0.10	
1842	D6304-A	36		0.07	
1879	D4928	10		-0.46	
1957	D6304-A	39		0.13	
1960	D6304	19		-0.28	
2124	D6304-A	0.6		-0.65	
6009		----		----	
6016	D6304	13.2		-0.40	
9050		----		----	
9054		----		----	
9055		----		----	
9056		----		----	
9057	in house	37.8		0.11	
9058	in house	33.5		0.02	
9061	D4928	40		0.15	
9107	D6304	50.2		0.36	
9108		----		----	
9130	D6304-A	23		-0.19	
9150	D6304-A	31		-0.03	

normality OK
n 30
outliers 0
mean (n) 32.490
st.dev. (n) 12.8370
R(calc.) 35.944
R(D6304:07) 136.371



Determination of Simulated Distillation on sample #15217; results in °C

lab	method	IBP	5%rec	10%rec	50%rec	90%rec	95%rec	FBP
171	D2887	0.5	26.0	52.0	124.0	251.0	286.0	379.0
311		----	----	----	----	----	----	----
323		----	----	----	----	----	----	----
442		----	----	----	----	----	----	----
444		----	----	----	----	----	----	----
491		----	----	----	----	----	----	----
492		----	----	----	----	----	----	----
499		----	----	----	----	----	----	----
602		----	----	----	----	----	----	----
608	D2887	13.0	29.4	61.6	134.2	256.7	294.9	387.9
609		----	----	----	----	----	----	----
657		----	----	----	----	----	----	----
784		----	----	----	----	----	----	----
785		----	----	----	----	----	----	----
840		----	----	----	----	----	----	----
875		----	----	----	----	----	----	----
1164		----	----	----	----	----	----	----
1214		----	----	----	----	----	----	----
1257		----	----	----	----	----	----	----
1267		----	----	----	----	----	----	----
1397		----	----	----	----	----	----	----
1429		----	----	----	----	----	----	----
1455		----	----	----	----	----	----	----
1616		----	----	----	----	----	----	----
1696		----	----	----	----	----	----	----
1800		----	----	----	----	----	----	----
1815		----	----	----	----	----	----	----
1842		----	----	----	----	----	----	----
1879		----	----	----	----	----	----	----
1957		----	----	----	----	----	----	----
1960		----	----	----	----	----	----	----
2124		----	----	----	----	----	----	----
6009		----	----	----	----	----	----	----
6016	D2887	----	60.6	82.0	138.4	258.2	295.8	381.4
9050		----	----	----	----	----	----	----
9054		----	----	----	----	----	----	----
9055		----	----	----	----	----	----	----
9056		----	----	----	----	----	----	----
9057		----	----	----	----	----	----	----
9058		----	----	----	----	----	----	----
9061		----	----	----	----	----	----	----
9107		----	----	----	----	----	----	----
9108		----	----	----	----	----	----	----
9130		----	----	----	----	----	----	----
9150		----	----	----	----	----	----	----
	n	2	3	3	3	3	3	3
	mean (n)	6.8	38.7	65.2	132.2	255.3	292.2	382.8

APPENDIX 2:

Distillation z-scores

lab	IBP	5%	10%	50%	90%	95%	FBP	
171	D86	0.77	2.44	<u>4.58</u>	1.97	2.87	----	0.21
311		----	----	----	----	----	----	----
323		----	----	----	----	----	----	----
442		----	----	----	----	----	----	----
444		----	----	----	----	----	----	----
491		----	----	----	----	----	----	----
492		----	----	----	----	----	----	----
499		----	----	----	----	----	----	----
602		----	----	----	----	----	----	----
608	D86	-0.39	-0.58	0.36	0.94	4.84	----	-0.12
609		----	----	----	----	----	----	----
657	D86	-0.02	1.28	2.18	0.71	-2.32	-0.65	0.69
784	D86	0.11	-1.66	-1.75	-0.61	-0.59	0.50	0.99
785		----	----	----	----	----	----	----
840	D86	-0.43	-0.65	-0.46	-0.08	0.53	----	-0.90
875		----	----	----	----	----	----	----
1164	D86	0.15	-0.54	-0.22	-0.21	-1.83	0.20	-0.53
1214		----	----	----	----	----	----	----
1257	D86	-0.41	-0.73	-0.66	-0.55	-1.87	0.76	0.29
1267		----	----	----	----	----	----	----
1397		----	----	----	----	----	----	----
1429	D86	0.00	0.23	1.31	1.17	3.69	----	0.77
1455	D86	-1.16	-1.77	-1.31	-1.70	-5.16	<u>-3.42</u>	-0.36
1616	D86	0.11	-0.69	-1.75	-1.76	-2.65	-0.91	-0.10
1696	D86	0.69	0.47	1.16	-1.19	2.29	----	0.55
1800		----	----	----	----	----	----	----
1815		----	----	----	----	----	----	----
1842	D86	-0.28	-1.31	-0.88	-0.38	-0.02	0.09	-0.83
1879	INH-2177	0.17	0.89	2.03	1.74	2.66	----	1.12
1957		----	----	----	----	----	----	----
1960		----	----	----	----	----	----	----
2124		----	----	----	----	----	----	----
6009		----	----	----	----	----	----	----
6016	D86	0.69	2.63	<u>6.18</u>	-0.04	-2.45	----	-1.79
9050		----	----	----	----	----	----	----
9054		----	----	----	----	----	----	----
9055		----	----	----	----	----	----	----
9056		----	----	----	----	----	----	----
9057		----	----	----	----	----	----	----
9058		----	----	----	----	----	----	----
9061		----	----	----	----	----	----	----
9107		----	----	----	----	----	----	----
9108		----	----	----	----	----	----	----
9130		----	----	----	----	----	----	----
9150		----	----	----	----	----	----	----

Bold and underlined test results are outliers according to Dixon/Grubbs/Rosner

APPENDIX 3:

Number of participating laboratories per country

1 lab in ALGERIA
2 labs in AUSTRALIA
1 lab in BELGIUM
1 lab in CROATIA
3 labs in GERMANY
1 lab in INDONESIA
1 lab in KAZAKHSTAN
6 labs in MALAYSIA
4 labs in NETHERLANDS
2 labs in NORWAY
1 lab in OMAN
1 lab in POLAND
1 lab in QATAR
4 labs in RUSSIAN FEDERATION
1 lab in SINGAPORE
3 labs in UNITED ARAB EMIRATES
10 labs in UNITED KINGDOM
1 lab in UNITED STATES OF AMERICA
1 lab in VIETNAM

APPENDIX 4

Abbreviations:

C	= final result after checking of first reported suspect 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 outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= excluded from calculations
E	= error in calculations
n.a.	= not applicable
W	= withdrawn on request participant
U	= reported in deviating unit
SDS	= Safety Data Sheet

Literature:

- 1 i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
- 2 ASTM E178-02
- 3 ASTM E1301-03
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- 5 ISO 5725, parts 1-6, 1994
- 6 ISO 13528-05
- 7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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- 9 IP367/96
- 9 DIN 38402 T41/42
- 10 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 11 J.N. Miller, Analyst, 118, 455, (1993)
- 12 Analytical Methods Committee Technical Brief, No4 February 2001
- 13 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see <http://www.rsc.org/suppdata/an/b2/b205600n/>)
- 14 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)