Results of Proficiency Test
Caustic Soda
September 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2012, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Caustic Soda every other year. During the annual proficiency testing program 2018/2019, it was decided to continue the round robin for the analysis of Caustic Soda.
Depending on the production process a number of Caustic Soda grades are available on the market. To fulfil the scope, in this proficiency test two different samples were prepared: one with a low concentration Chloride (low salt) and one with a relatively high concentration Chloride (high salt).

In this interlaboratory study, 42 laboratories in 25 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2018 proficiency test on Caustic Soda are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
Sample #18155 was a low NaCl Caustic Soda. Sample #18156 was the same Caustic spiked with Sodium Chloride, Sodium Chlorate and Sodium Sulfate.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for the statistical evaluations.

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 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The necessary 50 litre bulk Caustic Soda was provided by a third party. From this batch, after homogenizing, 60 HDPE bottles of 0.5 litre were filled and labelled #18155.

The homogeneity of the subsamples #18155 was checked by determination of Density at 20°C in accordance with ASTM D4052 and Total Alkalinity as NaOH in accordance with ASTM E291 on 4 stratified randomly selected samples.

	Density at 20°C in kg/L	*Total Alkalinity as NaOH in %M/M*
sample #18155-1	1.52833	50.51
sample #18155-2	1.52837	50.48
sample #18155-3	1.52836	50.51
sample #18155-4	1.52836	50.50

Table 1: homogeneity test results of subsamples #18155

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility 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	*Total Alkalinity as NaOH in %M/M*
r (observed)	0.00005	0.04
reference test method	ISO12185:96	ASTM E291:18
0.3 x R (ref. test method)	0.00015	0.21

Table 2: evaluation of the repeatabilities of the subsamples #18155

The remaining bulk material of 17 L (approx. 25 kg) was spiked with the components listed in table 3:

Component	*Amount in g*
Sodium Chloride	229
Sodium Chlorate	25.8
Sodium Sulfate	7.8

Table 3: components that were added to bulk material for sample #18156

After homogenisation, this batch was divided over 52 HDPE bottles of 0.25 L and labelled #18156. The homogeneity of the subsamples #18156 was checked by determination of Sodium Chlorate and Sodium Sulphate in accordance with ASTM E291 on 4 stratified randomly selected samples.

	Sodium Chlorate as $NaClO_3$ in %M/M	*Sodium Sulphate as Na_2SO_4 in %M/M*
sample #18156-1	0.1065	0.0260
sample #18156-2	0.1055	0.0260
sample #18156-3	0.1070	0.0260
sample #18156-4	0.1065	0.0260

Table 4: homogeneity test results of subsamples #18156

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

	Sodium Chlorate as $NaClO_3$ in %M/M	*Sodium Sulphate as Na_2SO_4 in %M/M*
r (observed)	0.0018	0.0000
reference	Horwitz	ASTM E291:18
0.3 x R (reference)	0.0050	0.0066

Table 5: evaluation of the repeatabilities of the subsamples #18156

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples #18155 and #18156 were assumed.

To the participants 1x0.5L sample labelled #18155 and 1x0.25L sample labelled #18156 were sent on August 15, 2018. An MSDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Caustic Soda, packed in the HDPE 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 Alkalinity as NaOH, Appearance, Density at 20°C, Iron as Fe, Sodium Chloride as NaCl, Sodium Chlorate as $NaClO_3$ and Sodium Sulphate as Na_2SO_4 on the low salt sample #18155. On the high salt sample #18156 was requested to determine Sodium Chloride as NaCl, Sodium Chlorate as $NaClO_3$ and Sodium Sulphate as Na_2SO_4.

It was explicitly 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. It was also requested not to report 'less than' results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

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

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.
Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.

Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018' (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a dataset does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

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

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1, was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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 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. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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

The z-scores were calculated according to:

$z_{(target)}$ = (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 interlaboratory study, problems with sample dispatch were encountered due to custom clearance for the participants in Brazil. One participant reported test results after the final reporting date. Four participants did not report any result at all. Not all participants were able to report all requested parameters. In total 38 participants reported 181 numerical results. Observed were 13 outlying test results, which is 7.2% of the total of numerical test results. 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, see also paragraph 3.1.

4.1 EVALUATION PER SAMPLE AND PER TEST

In this section, the reported test results are discussed per sample and per test. The specified test methods and requirements were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3. In this section, the results are discussed per test.

Sample #18155

Alkalinity: This determination was problematic. One statistical outlier was observed. The observed reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM E291:18.

Appearance: All reporting laboratories, except two, agreed about the appearance of the sample #18155, which was Pass (bright, clear and free from suspended matter).

Density at 20°C: This determination was problematic. Two statistical outliers were observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ISO12185:96.

Iron: This determination was very problematic at the low level of 0.6 mg/kg. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier was not at all in agreement with the requirements of ASTM E291:18.
It was decided not to calculate z-scores because the average of the group is below the precision range of the used test method.

Sodium Chloride: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM E1787:16.

Sodium Chlorate: This determination was not problematic at the low level of 2.3 mg/kg. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM E1787:16.

Sodium Sulphate: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM E1787:16.

Sample #18156

Sodium Chloride: This determination was not problematic at the high level of 0.84%M/M. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM E291:18. This sample was spiked with Sodium Chloride. The recovery of 94% may be good (($0.8423_{(avg.)} - 0.056_{(avg. of\ \#18155)}$) / $0.8947_{(added\ amount)}$ * 100%).

Sodium Chlorate: This determination was problematic at the high level of 0.093%M/M. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the target reproducibility estimated from the Horwitz equation.
This sample was spiked with Sodium Chlorate. The recovery of 92% is good (($0.0933_{(avg.)} - 0.0002_{(avg. of\ \#18155)}$) / $0.1009_{(added\ amount)}$ * 100%).

Sodium Sulphate: This determination was problematic at the high level of 0.0111%M/M. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM E291:18. This sample was spiked with Sodium Sulphate. The recovery of 32% is marginal ((0.0111$_{(avg.)}$ – 0.0012$_{(avg. of #18155)}$) / 0.0305$_{(added amount)}$ * 100%).

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 number of significant test results,the average result, the calculated reproducibility (2.8*standard deviation) and reproducibility derived from literature reference methods (in casu ASTM, EN standards) are presented in the next tables.

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (lit)*
Alkalinity as NaOH	%M/M	35	50.21	0.80	0.70
Appearance	---	25	Pass	n.a.	n.a.
Density at 20°C	kg/L	26	1.5283	0.0009	0.0005
Iron as Fe	mg/kg	28	0.62	(1.07)*	(0.18)*
Sodium Chloride as NaCl	mg/kg	23	55.9	17.7	12.9
Sodium Chlorate as $NaClO_3$	mg/kg	10	2.3	3.3	5.6
Sodium Sulphate as Na_2SO_4	mg/kg	12	12.2	17.2	30.8

Table 6: Reproducibilities of tests for sample #18155

*) Results between brackets are outside of the precision range of the method.

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (lit)*
Sodium Chloride as NaCl	%M/M	22	0.842	0.052	0.08
Sodium Chlorate as $NaClO_3$	%M/M	11	0.093	0.027	0.011
Sodium Sulphate as Na_2SO_4	%M/M	15	0.011	0.015	0.009

Table 7: Reproducibilities of tests for sample #18156

Without further statistical calculations, it can be concluded that for several tests there is not a good compliance of the group of participating laboratories with the relevant standards. The tests that are problematic have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2018 WITH PREVIOUS PTS

	September 2018	*September 2016*	*September 2014*	*September 2012*
Number of reporting labs	38	30	26	25
Number of results reported	181	175	150	145
Statistical outliers	13	17	10	13
Percentage outliers	7.2%	9.7%	6.7%	9.0%

Table 8: 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 the following table:

	September 2018	*September 2016*	*September 2014*	*September 2012*
Low Salt Caustic Soda:				
Alkalinity as NaOH	-	++	+	+
Density at 20°C	--	-	-	-
Iron as Fe	(--)	-	+/-	-
Sodium Chloride as NaCl	-	+/-	--	-
Sodium Chlorate as $NaClO_3$	+	++	(--)	(--)
Sodium Sulphate as Na_2SO_4	+	+	(--)	-
High Salt Caustic Soda:				
Sodium Chloride as NaCl	+	-	-	+
Sodium Chlorate as $NaClO_3$	--	+/-	+	--
Sodium Sulphate as Na_2SO_4	-	-	(-)	+

Table 9: comparison determinations against the standard

() the average was below the application range of the reference method

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

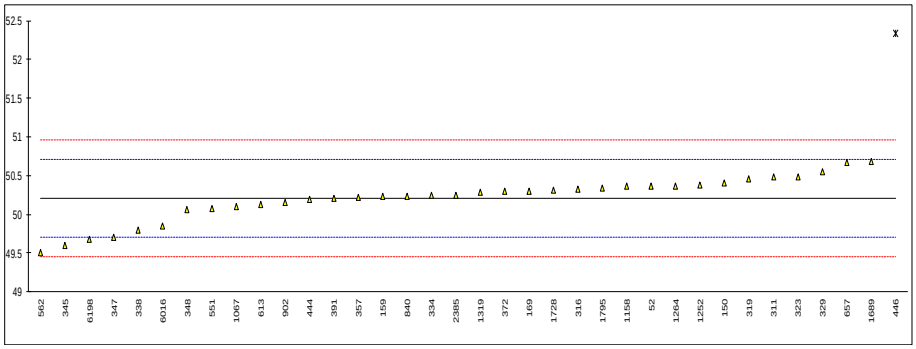
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

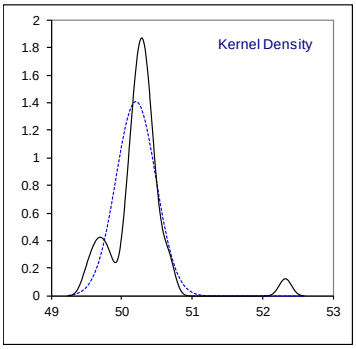
APPENDIX 1

Determination of Alkalinity as NaOH on sample #18155; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	E291	50.37		0.65	
150	E291	50.41		0.81	
159	E291	50.23		0.09	
169	E291	50.30		0.37	
171		-----		-----	
311	E291	50.48		1.09	
316	INH-041	50.33	C	0.49	first reported: 38.971
319	INH-726	50.46		1.01	
323	E291	50.48		1.09	
329	E291	50.55		1.37	
334	E291	50.24		0.13	
338	E291	49.80		-1.63	
345	E291	49.6		-2.43	
347	D501A	49.70		-2.03	
348	E291	50.0605		-0.59	
357	E291	50.217		0.04	
372	E291	50.30		0.37	
391	E291	50.2		-0.03	
444	E291	50.19		-0.07	
446	E291	52.33	R(0.01)	8.49	
541		-----		-----	
551	E291	50.07		-0.55	
554		-----		-----	
557		-----		-----	
562	INH-480	49.51		-2.79	
613	E291	50.13		-0.31	
657	E291	50.67		1.85	
840	E291	50.233		0.10	
902	E291	50.15		-0.23	
1067	E291	50.1		-0.43	
1158	E291	50.368		0.64	
1252	E291	50.38		0.69	
1264	E291	50.37		0.65	
1319	JIS K1200-2	50.283		0.30	
1373		-----		-----	
1656		-----		-----	
1689	E291	50.68		1.89	
1728		50.31		0.41	
1795	INH-3068	50.343		0.54	
2385	E291	50.245		0.15	
6016	UOP209	49.844		-1.46	
6198	GB/T4348.1	49.68		-2.11	

normality	OK
n	35
outliers	1
mean (n)	50.208
st.dev. (n)	0.2838
R(calc.)	0.795
st.dev.(E291:18)	0.25
R(E291:18)	0.70





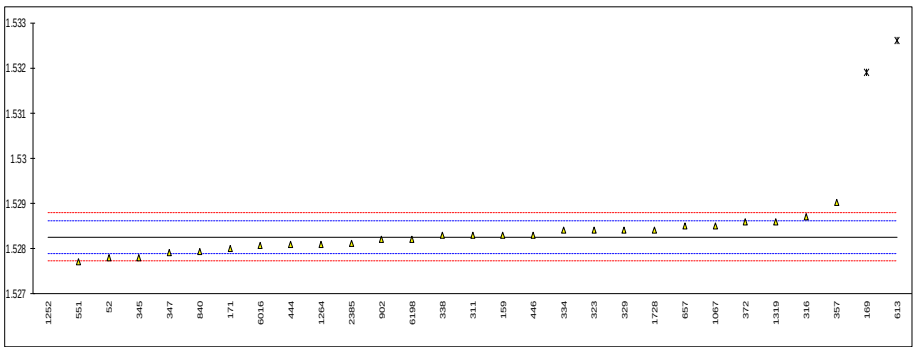
Determination of Appearance on sample #18155;

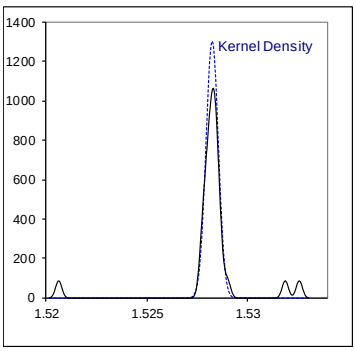
lab	method	value	mark	z(targ)	remarks
52	D4176	Pass		-----	
150	E2680	Fail		-----	
159	D4176	clear		-----	
169	D4176	CBFSM		-----	
171	E2680	Pass		-----	
311	E2680	pass		-----	
316	Visual	Clear		-----	
319		-----		-----	
323	E2680	clear & bright		-----	
329	Visual	clear		-----	
334	Visual	characteristique		-----	
338	Visual	Clear & bright FFSM		-----	
345	Visual	pass		-----	
347		-----		-----	
348		-----		-----	
357	E2680	Pass		-----	
372	E2680	Pass		-----	
391		-----		-----	
444	E2680	Pass		-----	
446	E2680	PASS		-----	
541		-----		-----	
551	Visual	Pass		-----	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613	D2090	C&C		-----	
657	E2680	Pass		-----	
840	E2680	Pass		-----	
902	E2680	Pass		-----	
1067	Visual	Bright and Clear		-----	
1158		-----		-----	
1252	Visual	Clear		-----	
1264	Visual	Clear & bright		-----	
1319	Visual	Clear liquid		-----	
1373		-----		-----	
1656		-----		-----	
1689		-----		-----	
1728	Visual	CLEAR		-----	
1795		-----		-----	
2385	Visual	clear, colourless		-----	
6016		-----		-----	
6198	D4176	Fail		-----	several black particulates found in the sample
	n	25 / 2			
	mean (n)	Pass/Clear / Fail			

Determination of Density at 20°C on sample #18155; results in kg/L

lab	method	value	mark	z(targ)	remarks
52	D4052	1.5278		-2.55	
150		-----		-----	
159	D4052	1.5283		0.25	
169	D4052	1.5319	R(0.01)	20.41	
171	D4052	1.528		-1.43	
311	D4052	1.5283		0.25	
316	INH-009	1.5287	C	2.49	first reported: 1.5316
319		-----		-----	
323	D4052	1.5284		0.81	
329	D4052	1.5284		0.81	
334	ISO12185	1.5284		0.81	
338	ISO12185	1.5283		0.25	
345	D4052	1.5278		-2.55	
347	D4052	1.5279		-1.99	
348		-----		-----	
357	D4052	1.52902		4.28	
372	ISO12185	1.5286		1.93	
391		-----		-----	
444	D4052	1.5281		-0.87	
446	D4052	1.5283	C	0.25	first reported: 1.531
541		-----		-----	
551	D4052	1.5277		-3.11	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613	D4052	1.5326	ex	24.33	reported to have tested at 15°C instead of 20°C
657	D4052	1.52849		1.31	
840	D4052	1.52794		-1.77	
902	D4052	1.5282		-0.31	
1067	ISO12185	1.5285		1.37	
1158		-----		-----	
1252	ISO12185	1.5206	R(0.01)	-42.87	
1264	D4052	1.5281		-0.87	
1319	ISO12185	1.5286		1.93	
1373		-----		-----	
1656		-----		-----	
1689		-----		-----	
1728	D4052	1.52842		0.92	
1795		-----		-----	
2385	ISO12185	1.52812		-0.76	
6016	D4052	1.52807	C	-1.04	first reported: 4.5269
6198	D4052	1.5282		-0.31	

normality	OK
n	26
outliers	2 (+1ex)
mean (n)	1.52826
st.dev. (n)	0.000307
R(calc.)	0.00086
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005



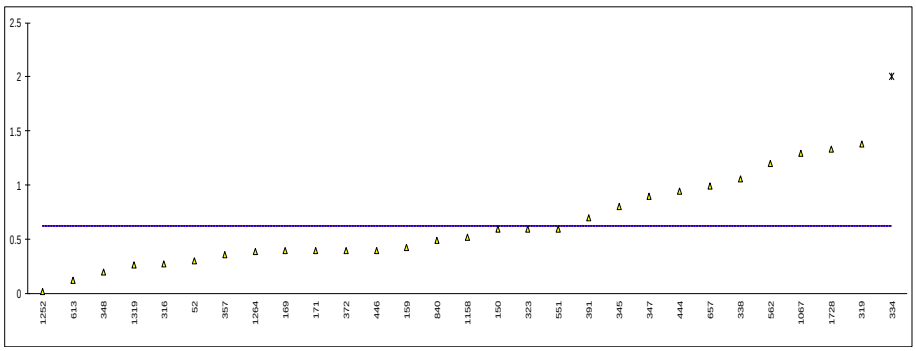


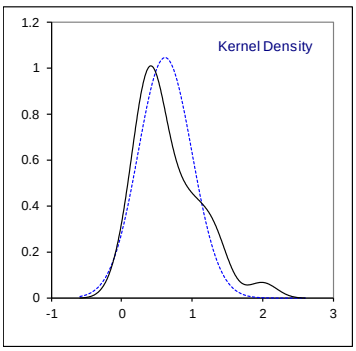
Determination of Iron as Fe on sample #18155; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	E291	0.3		-----	
150	E291	0.6		-----	
159	E291	0.43		-----	
169	E291	0.398819		-----	
171	E291	0.4		-----	
311	E291	<0.01		-----	
316	INH-043	0.28		-----	
319	INH-104	1.38		-----	
323	E291	0.6		-----	
329		-----		-----	
334	E291	2.005	R(0.05)	-----	
338	E291	1.0615		-----	
345	E291	0.8		-----	
347	E291	0.9		-----	
348	E291	0.20		-----	
357	E291	0.36		-----	
372	E291	0.4		-----	
391	E291	0.7		-----	
444	E291	0.943		-----	
446	E291	0.4		-----	
541		-----		-----	
551	E291	0.6		-----	
554		-----		-----	
557		-----		-----	
562	E291	1.2		-----	
613	E291	0.12		-----	
657	E291	0.9932		-----	
840	E291	0.49		-----	
902		-----		-----	
1067	E291	1.3		-----	
1158	INH-3068	0.52		-----	
1252	E291	0.019		-----	
1264	E291	0.39		-----	
1319	JIS K1200-6	0.27		-----	
1373		-----		-----	
1656		-----		-----	
1689		-----		-----	
1728	E291	1.333		-----	
1795		-----		-----	
2385	E291	<0,5		-----	
6016		-----		-----	
6198		-----		-----	

normality	OK
n	28
outliers	1
mean (n)	0.621
st.dev. (n)	0.3824
R(calc.)	1.071
st.dev.(E291:18)	(0.0643)
R(E291:18)	(0.180)

Application range R(E291:18) = 4-30 mg/kg



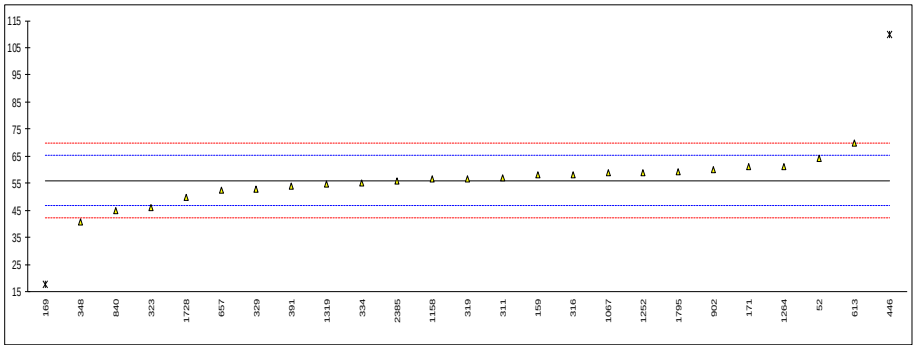


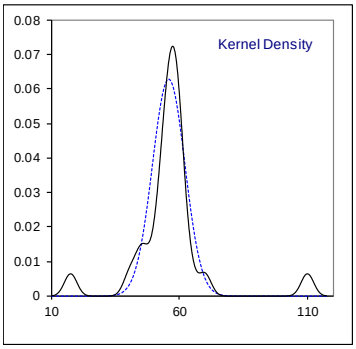
Determination of Sodium Chloride as NaCl on sample #18155; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	In house	64		1.75	
150		-----		-----	
159	In house	58		0.45	
169	E1787	17.7045	R(0.01)	-8.30	
171	E291	61		1.10	
311	INH-554	57		0.23	
316	INH-044	58.1		0.47	
319	INH-269	56.7		0.17	
323	E1787	46	C	-2.16	first reported: 32
329	E291	53	C	-0.64	first reported: 32
334	E1787	55	C	-0.20	first reported: 76.4
338		-----		-----	
345		-----		-----	
347		-----		-----	
348	E291	40.8004		-3.29	
357	E291	< 100		-----	
372	E291	<100		-----	
391	E1787	54		-0.42	
444		-----		-----	
446	E291	110	R(0.01)	11.73	
541		-----		-----	
551	E291	<100		-----	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613	E291	70		3.05	
657	E291	52.38		-0.77	
840	ISO6227	45.0		-2.37	
902	E1787	60	C	0.88	first reported: 36
1067	E291	59		0.66	
1158	E291	56.6		0.14	
1252	E1787	59		0.66	
1264	E291	61		1.10	
1319	JIS K1200-3-2	54.9		-0.23	
1373		-----		-----	
1656		-----		-----	
1689		-----		-----	
1728		50		-1.29	
1795	E291	59.41		0.75	
2385	E1787	55.7		-0.05	
6016		-----		-----	
6198		-----		-----	

normality	suspect
n	23
outliers	2
mean (n)	55.939
st.dev. (n)	6.3326
R(calc.)	17.731
st.dev.(E1787:16)	4.6071
R(E1787:16)	12.9

Compare R(E291:18) = 15

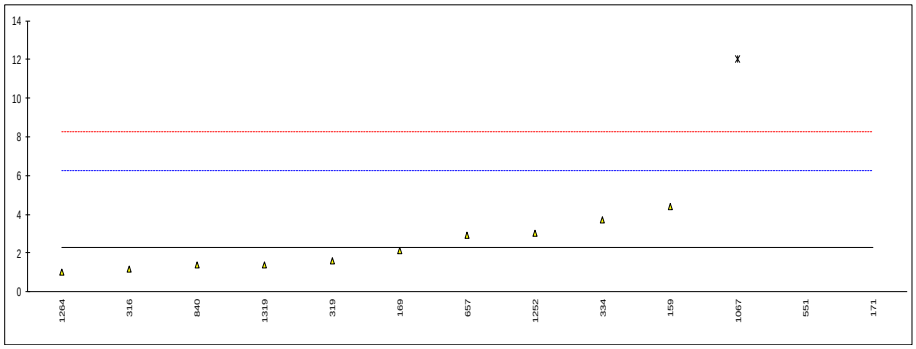


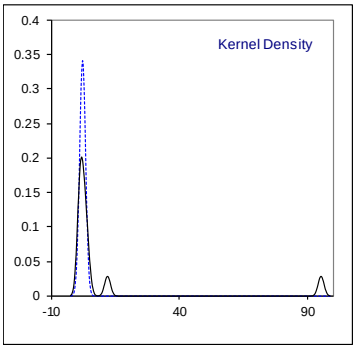


Determination of Sodium Chlorate as $NaClO_3$ on sample #18155; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	In house	<10		-----	
150		-----		-----	
159	D2202	4.4		1.06	
169	E291	2.1		-0.09	
171	INH-1112	43330	G(0.01)	21663.86	
311		-----		-----	
316	INH-075	1.2		-0.54	
319	INH-888	1.60		-0.34	
323		-----		-----	
329		-----		-----	
334		3.7		0.71	
338		-----		-----	
345		-----		-----	
347		-----		-----	
348		-----		-----	
357		-----		-----	
372		-----		-----	
391		-----		-----	
444		-----		-----	
446		-----		-----	
541		-----		-----	
551	NBR9851	95.4	G(0.01)	46.56	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613		-----		-----	
657	INH-134	2.927		0.33	
840	INH-61112	1.38		-0.45	
902		-----		-----	
1067	E291	12	G(0.01)	4.86	
1158		-----		-----	
1252		3		0.36	
1264	In house	1.0		-0.64	
1319	In house	1.4		-0.44	
1373		-----		-----	
1656		-----		-----	
1689		-----		-----	
1728		-----		-----	
1795		-----		-----	
2385		-----		-----	
6016		-----		-----	
6198		-----		-----	

normality	OK
n	10
outliers	3
mean (n)	2.271
st.dev. (n)	1.1705
R(calc.)	3.277
st.dev.(E1787:16)	2
R(E1787:16)	5.6

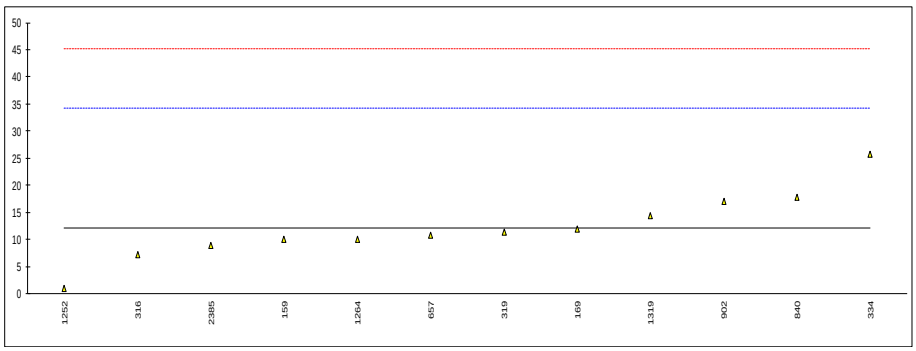


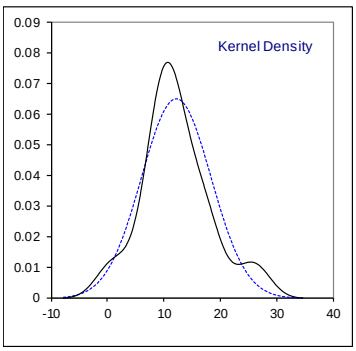


Determination of Sodium Sulphate as Na_2SO_4 on sample #18155; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	E291	<20		-----	
150		-----		-----	
159	In house	10		-0.20	
169	E1787	11.940		-0.02	
171		-----		-----	
311	E291	<40		-----	
316	INH-073	7.269		-0.45	
319	INH-862	11.4		-0.07	
323	E1787	<10		-----	
329		-----		-----	
334	E1787	25.8		1.24	
338		-----		-----	
345		-----		-----	
347		-----		-----	
348		-----		-----	
357		-----		-----	
372	E291	<40		-----	
391		-----		-----	
444		-----		-----	
446		-----		-----	
541		-----		-----	
551	NBR15132	<10		-----	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613		-----		-----	
657	E291	10.79		-0.13	
840	E291	17.8		0.51	
902	E1787	17	C	0.44	first reported: 12
1067	E291	< 40		-----	
1158		-----		-----	
1252	E1787	1		-1.02	
1264	E291	10.0		-0.20	
1319	JIS K1200-4	14.4		0.20	
1373		-----		-----	
1656		-----		-----	
1689		-----		-----	
1728		-----		-----	
1795		-----		-----	
2385	E1787	8.9		-0.30	
6016		-----		-----	
6198		-----		-----	

normality	suspect
n	12
outliers	0
mean (n)	12.192
st.dev. (n)	6.1587
R(calc.)	17.244
st.dev.(E1787:16)	11
R(E1787:16)	30.8

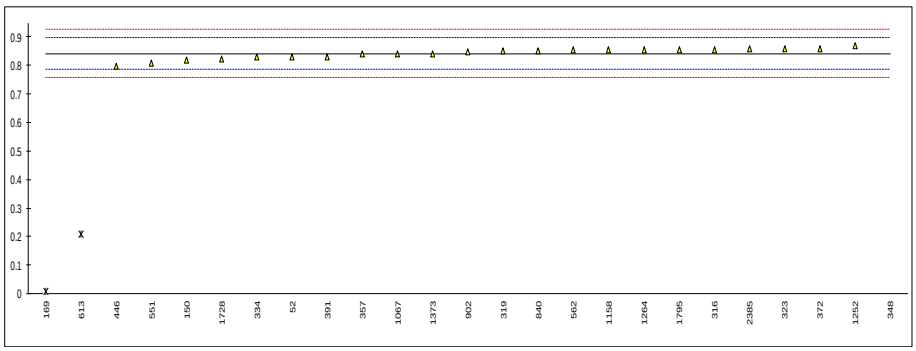


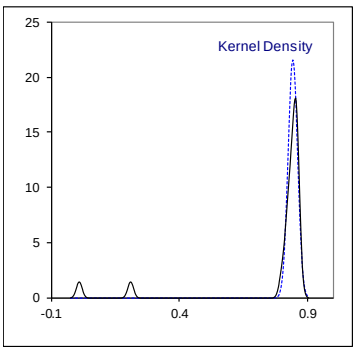


Determination of Sodium Chloride as NaCl on sample #18156; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	E291	0.83		-0.43	
150	E1787	0.8193		-0.80	
159		-----		-----	
169	E291	0.0096	R(0.01)	-29.14	
171		-----		-----	
311		-----		-----	
316	INH-044	0.856		0.48	
319	INH-269	0.852		0.34	
323	E291	0.86		0.62	
329		-----		-----	
334	E291	0.8286	C	-0.48	first reported: 8286.4%M/M
338		-----		-----	
345		-----		-----	
347		-----		-----	
348	E291	12.9726	R(0.01)	424.56	
357	E291	0.839		-0.11	
372	E291	0.86		0.62	
391	E291	0.8307		-0.41	
444		-----		-----	
446	E291	0.798		-1.55	
541		-----		-----	
551	E291	0.81		-1.13	
554		-----		-----	
557		-----		-----	
562	INH-632	0.854	C	0.41	first reported: 0.77
613	E291	0.21	R(0.01)	-22.13	
657		-----		-----	
840	E291	0.852		0.34	
902	E1787	0.846	C	0.13	first reported: 0.513
1067	E291	0.839		-0.11	
1158	E291	0.855		0.45	
1252	E291	0.87		0.97	
1264	E291	0.855		0.45	
1319		-----		-----	
1373	INH-005	0.84		-0.08	
1656		-----		-----	
1689		-----		-----	
1728		0.821		-0.74	
1795	E291	0.855		0.45	
2385	E291	0.8594	C	0.60	reported: 8594%M/M (probably a unit error?)
6016		-----		-----	
6198		-----		-----	

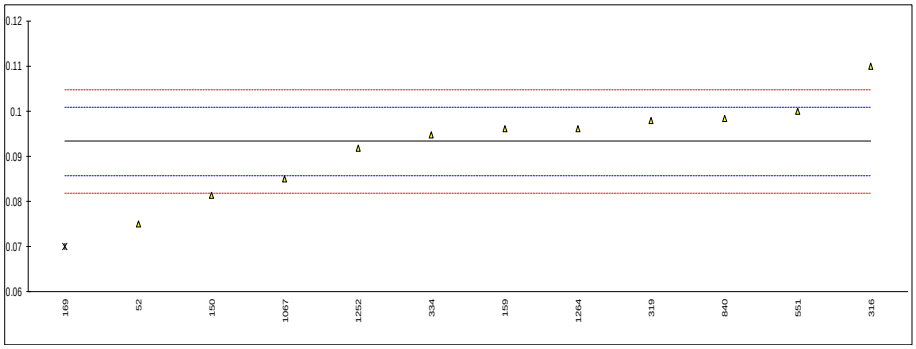
normality	OK		
n	22		
outliers	3	spike:	
mean (n)	0.8423	0.8947	See §4.1
st.dev. (n)	0.01850		
R(calc.)	0.0518		
st.dev.(E291:18)	0.02857		
R(E291:18)	0.08		

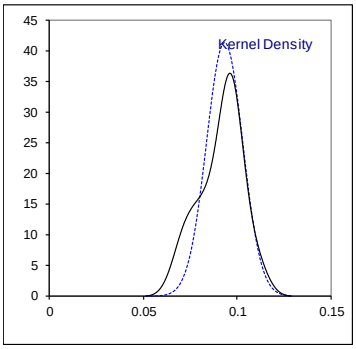




Determination of Sodium Chlorate as $NaClO_3$ on sample #18156; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	In house	0.075		-4.77	
150	E1787	0.0813		-3.13	
159	D2022	0.096		0.71	
169	E291	0.07	G(0.05)	-6.08	
171		-----		-----	
311		-----		-----	
316	INH-075	0.110		4.37	
319	INH-888	0.09785		1.20	
323		-----		-----	
329		-----		-----	
334		0.0948	C	0.40	first reported: 948%M/M
338		-----		-----	
345		-----		-----	
347		-----		-----	
348		-----		-----	
357		-----		-----	
372		-----		-----	
391		-----		-----	
444		-----		-----	
446		-----		-----	
541		-----		-----	
551	NBR9851	0.100		1.76	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613		-----		-----	
657		-----		-----	
840	INH-61112	0.09827		1.31	
902		-----		-----	
1067	E291	0.085		-2.16	
1158		-----		-----	
1252		0.0917		-0.41	
1264	In house	0.0960		0.71	
1319		-----		-----	
1373		-----		-----	
1656		-----		-----	
1689		-----		-----	
1728		-----		-----	
1795		-----		-----	
2385		-----		-----	
6016		-----		-----	
6198		-----		-----	
	normality	OK			
	n	11			
	outliers	1	spike:		
	mean (n)	0.0933	0.1009	See §4.1	
	st.dev. (n)	0.00968			
	R(calc.)	0.0271			
	st.dev.(Horwitz)	0.00383			
	R(Horwitz)	0.0107			

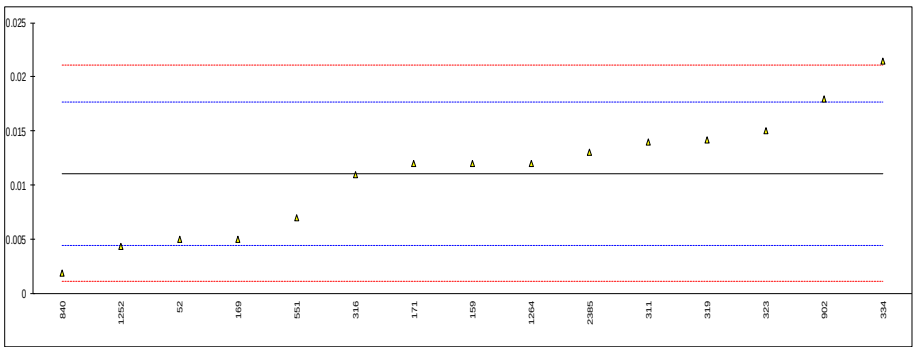


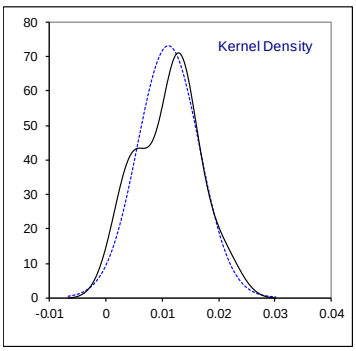


Determination of Sodium Sulphate as Na_2SO_4 on sample #18156; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	E291	0.005		-1.83	
150		-----		-----	
159	In house	0.012		0.28	
169	E1787	0.00505523		-1.81	reported: 50.5523%M/M (probably a unit error?)
171	E291	0.012		0.28	
311	E291	0.014		0.88	
316	INH-073	0.011		-0.02	
319	INH-862	0.01421		0.94	
323	E291	0.015		1.18	
329		-----		-----	
334	E291	0.0215	C	3.14	first reported: 214.5%M/M
338		-----		-----	
345		-----		-----	
347		-----		-----	
348		-----		-----	
357		-----		-----	
372	E291	<0.004		-----	
391		-----		-----	
444		-----		-----	
446		-----		-----	
541		-----		-----	
551	NBR15132	0.007		-1.23	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613		-----		-----	
657		-----		-----	
840	E291	0.0019		-2.76	
902	E1787	0.018	C	2.08	first reported: 0.012
1067	E291	< 0.004		-----	
1158		-----		-----	
1252	E291	0.0044		-2.01	
1264	E291	0.0120		0.28	
1319		-----		-----	
1373		-----		-----	
1656		-----		-----	
1689		-----		-----	
1728		-----		-----	
1795		-----		-----	
2385	E291	0.01304	C	0.59	reported: 130.4%M/M (probably a unit error?)
6016		-----		-----	
6198		-----		-----	

normality	OK		
n	15		
outliers	0	spike:	
mean (n)	0.0111	0.0305	See §4.1
st.dev. (n)	0.00545		
R(calc.)	0.0153		
st.dev.(E291:18)	0.00332		
R(E291:18)	0.0093		





APPENDIX 2

Number of participants per country

1 lab in ARGENTINA
1 lab in AUSTRALIA
2 labs in BELGIUM
3 labs in BRAZIL
1 lab in CANADA
1 lab in CHILE
2 labs in CHINA, People's Republic
1 lab in ESTONIA
1 lab in FINLAND
2 labs in FRANCE
1 lab in GERMANY
1 lab in ITALY
1 lab in JAPAN
1 lab in KAZAKHSTAN
4 labs in NETHERLANDS
1 lab in PORTUGAL
3 labs in ROMANIA
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
3 labs in SPAIN
1 lab in TURKEY
1 lab in UNITED ARAB EMIRATES
3 labs in UNITED KINGDOM
4 labs in UNITED STATES OF AMERICA
1 lab in VIETNAM

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
E = probably an error in calculations
U = test result probably reported in a different unit
W = test result withdrawn on request of participant
ex = test result excluded from statistical evaluation
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported
SDS = Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ASTM E178:02
3 ASTM E1301:95 (2003)
4 ISO 5725:86
5 ISO 5725, parts 1-6:94
6 ISO 13528:05
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 367:96
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 ASTM E346:03e1
14 Analytical Methods Committee Technical brief, No 4 January 2001.
15 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, Analyst 2002, 127, 1359-1364
16 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), 165-172, (1983)