Results of Proficiency Test
Caustic Soda (Sodium Hydroxide solution)
September 2020

Organized 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 2020/2021 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 need of the scope 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 38 laboratories in 20 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the 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 organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two samples of Caustic Soda: 1x 0.5L bottle labelled #20165 with a low salt content and 1x 0.25L bottle labelled #20166 with a high salt content.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for the 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 a regular basis by sending out questionnaires.

2.2 PROTOCOL

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). 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

A batch of approximately 50L of Caustic Soda was purchased from a local third party and was made positive on Iron. After homogenization 56 HDPE bottles of 0.5L were filled and labelled #20165. The homogeneity of the subsamples was checked by determination of Density at 20°C in accordance with ASTM D4052 and Alkalinity as NaOH in accordance with ASTM E291 on 4 stratified randomly selected subsamples.

	Density at 20°C in kg/L	Alkalinity as NaOH in %M/M
sample #20165-1	1.5244	49.95
sample #20165-2	1.5244	50.02
sample #20165-3	1.5244	50.01
sample #20165-4	1.5244	50.00

Table 1: homogeneity test results of subsamples #20165

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 ISO13528, Annex B2 in the next table.

	Density at 20°C in kg/L	Alkalinity as NaOH in %M/M
r (observed)	0.00000	0.09
reference test method	ISO12185:96	ASTM E291:18
0.3 x R (reference test method)	0.00015	0.21

Table 2: evaluation of the repeatabilities of subsamples #20165

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

For the second sample the same batch of Caustic Soda was taken and approximately 25L was made positive on Sodium Chloride, Sodium Chlorate and Sodium Sulfate. After homogenization 53 HDPE bottles of 0.25L were filled and labelled #20166. The homogeneity of the subsamples was checked by determination of Sodium Chlorate by an in-house test method on 3 stratified randomly selected subsamples.

	Sodium Chlorate as $NaClO_3$ in %M/M
sample #20166-1	0.094
sample #20166-2	0.094
sample #20166-3	0.094

Table 3: homogeneity test results of subsamples #20166

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility estimated from the Horwitz equation in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Sodium Chlorate as $NaClO_3$ in %M/M
r (observed)	0.0000
reference method	Horwitz
0.3 x R (reference method)	0.0045

Table 4: evaluation of the repeatability of subsamples #20166

The calculated repeatability was in agreement with 0.3 times the reproducibility estimated from the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample labelled #20165 and one sample labelled #20166 were sent on August 26, 2020. 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 ANALYZES

The participants were requested to determine on the low salt sample #20165: Alkalinity as NaOH, Appearance, Density at 20°C, Iron as Fe, Sodium Chloride as NaCl, Sodium Chlorate as $NaClO_3$ and Sodium Sulfate as Na_2SO_4.
On the high salt sample #20166 it was requested to determine: Sodium Chloride as NaCl, Sodium Chlorate as $NaClO_3$ and Sodium Sulfate 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 test results, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

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

3 RESULTS

During five weeks after sample dispatch, the test results of the participants 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 reanalyzes). Additional or corrected test results are used for data analysis and the 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 data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO5725 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.
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:

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

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 some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. When considering the test results of the two samples together one participant reported test results after the final reporting date and nine participants did not report any test results. Not all participants were able to report all tests requested. Finally, 29 reporting laboratories submitted 164 numerical test results. Observed were 15 outlying test results, which is 9.1%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all 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 test methods, which were used by the various laboratories, were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the reported test results in appendix 1. The abbreviations, used in these tables, are explained in appendix 3.

Unfortunately, a suitable reference test method, providing the precision data, is not available for all determinations. For these tests the calculated reproducibility was compared against the estimated reproducibility calculated from the Horwitz equation.

In the iis PT reports ASTM test methods are referred to with a number (e.g. E291) and an added designation for the year that the test method was adopted or revised (e.g. E291:18).

Sample #20165, low salt

Alkalinity as NaOH: This determination was not problematic. Two statistical outliers were observed. The observed reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM E291:18.

Appearance: This determination was not problematic. Almost all reporting participants, except one, agreed about the appearance of the sample as Pass (bright, clear and free from suspended matter).

Density at 20°C: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ISO12185:96.

Iron: This determination was not problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM E291:18.

Sodium Chloride: 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 E1787:16 nor with the requirements of ASTM E291:18.

Sodium Chlorate: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM E1787:16.

Sodium Sulfate: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM E1787:16.

Sample #20166, high salt

Sodium Chloride: This determination may be problematic for a number of laboratories. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM E291:18.

Sodium Chlorate: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the estimated reproducibility calculated from the Horwitz equation.

Sodium Sulfate: 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 E291:18.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method or as declared by the estimated target reproducibility using the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM test methods) or estimated using the Horwitz equation are presented in the next two tables.

Parameter	unit	n	average	2.8 * sd	R(target)
Alkalinity as NaOH	%M/M	25	49.88	0.49	0.70
Appearance		22	Pass	n.a.	n.a.
Density at 20°C	kg/L	24	1.5240	0.0010	0.0005
Iron as Fe	mg/kg	22	2.8	0.6	0.8
Sodium Chloride as NaCl	mg/kg	22	47.2	23.4	12.9
Sodium Chlorate as $NaClO_3$	mg/kg	10	2.6	3.5	5.6
Sodium Sulfate as Na_2SO_4	mg/kg	10	15.6	27.2	30.8

Table 5: reproducibilities of tests on sample #20165

Parameter	unit	n	average	2.8 * sd	R(target)
Sodium Chloride as NaCl	%M/M	16	0.851	0.048	0.08
Sodium Chlorate as $NaClO_3$	%M/M	11	0.095	0.015	0.015
Sodium Sulfate as Na_2SO_4	%M/M	9	0.012	0.014	0.010

Table 6: reproducibilities of tests on sample #20166

Without further statistical calculations, it can be concluded that for most tests there is a good compliance of the group of participating laboratories with the reference test method/target. The tests that are problematic have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2020 WITH PREVIOUS PTS

	September 2020	September 2018	September 2016	September 2014	September 2012
Number of reporting laboratories	29	38	30	26	25
Number of test results	164	181	175	150	145
Number of statistical outliers	15	13	17	10	13
Percentage of statistical outliers	9.1%	7.2%	9.7%	6.7%	9.0%

Table 7: 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.

	September 2020	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 Sulfate as Na_2SO_4	+	+	+	(--)	-

	September 2020	September 2018	September 2016	September 2014	September 2012
High Salt Caustic Soda					
Sodium Chloride as NaCl	+	+	-	-	+
Sodium Chlorate as $NaClO_3$	+/-	--	+/-	+	--
Sodium Sulfate as Na_2SO_4	-	-	-	(-)	+

Table 8: comparison determinations against the reference test method

For results between brackets the average was below the application range of the reference method

The following performance categories were used:

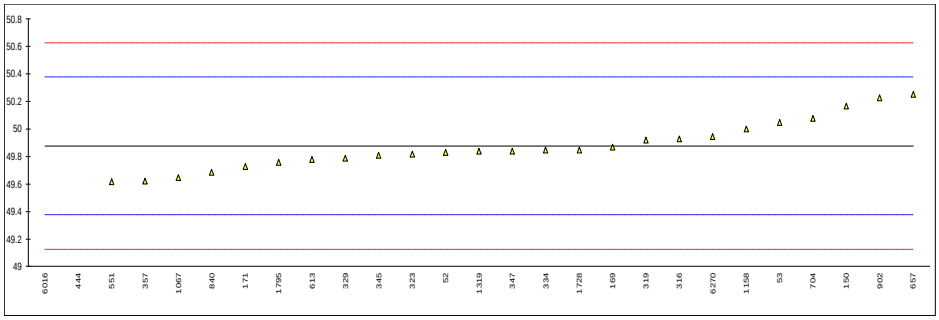
++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/- : group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.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

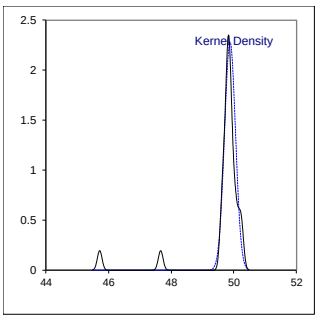
APPENDIX 1

Determination of Alkalinity as NaOH on sample #20165; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	E291	49.83		-0.19	
53	E291	50.05		0.69	
150	E291	50.17		1.17	
159		-----		-----	
169	E291	49.87		-0.03	
171	E291	49.73		-0.59	
316	INH-041	49.93		0.21	
319	INH-726	49.92		0.17	
323	E291	49.82		-0.23	
329	E291	49.79		-0.35	
334	E291	49.85		-0.11	
338		-----		-----	
345	E291	49.81		-0.27	
347	E291	49.84		-0.15	
348		-----		-----	
357	E291	49.622		-1.02	
391		-----		-----	
444	E291	47.66	C,R(0.01)	-8.87	first reported 50.57
551	E291	49.62		-1.03	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613	E291	49.78		-0.39	
657	E291	50.2531		1.50	
704	E291	50.08		0.81	
840	E291	49.688		-0.76	
902	E291	50.23		1.41	
1067	E291	49.65		-0.91	
1158	E291	50.00		0.49	
1264		-----		-----	
1319		49.839		-0.15	
1508		-----		-----	
1510		-----		-----	
1728		49.85		-0.11	
1795	In house	49.760		-0.47	
6016		45.711	R(0.01)	-16.66	
6262		-----		-----	
6270	JIS K1200-2	49.948		0.28	

normality	OK
n	25
outliers	2
mean (n)	49.877
st.dev. (n)	0.1747
R(calc.)	0.489
st.dev.(E291:18)	0.25
R(E291:18)	0.70





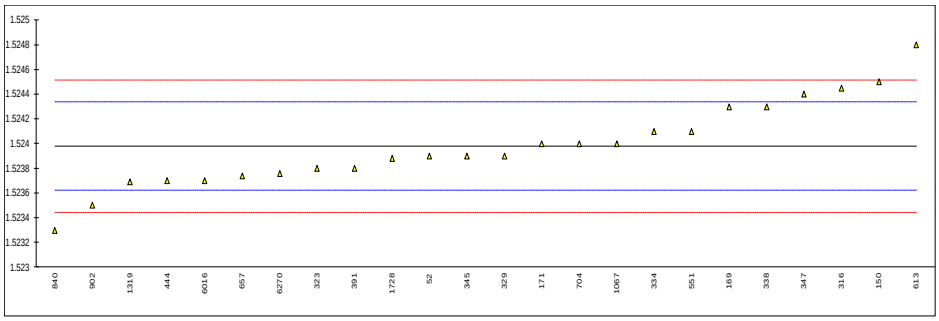
Determination of Appearance on sample #20165;

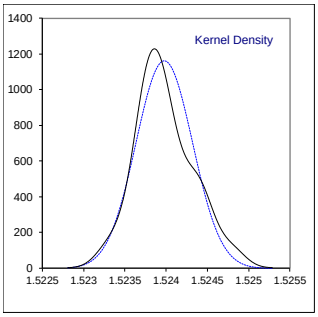
lab	method	value	mark	z(targ)	remarks
52	E2680	Pass		-----	
53		-----		-----	
150	Visual	C&B		-----	
159		-----		-----	
169	Visual	BCFSM		-----	
171	E2680	Clear without any suspended matter		-----	
316		-----		-----	
319	Visual	Clear		-----	
323	D4176	clear & bright		-----	
329	Visual	clear & bright		-----	
334	Visual	clear and bright		-----	
338		-----		-----	
345	E2680	PASS		-----	
347	E2680	Pass		-----	
348		-----		-----	
357	E2680	Pass		-----	
391	Visual	C&B		-----	
444	E2680	Pass		-----	
551	Visual	Pass		-----	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613	D2090	C&C		-----	
657	E2680	Pass		-----	
704	Visual	Clear and bright		-----	
840	E2680	Pass		-----	
902	E2680	Pass		-----	
1067	Visual	Some Particles Present		-----	
1158		-----		-----	
1264		-----		-----	
1319	Visual	Clear liquid		-----	
1508		-----		-----	
1510		-----		-----	
1728	Visual	CLEAR		-----	
1795		-----		-----	
6016		-----		-----	
6262		-----		-----	
6270	Visual	Clear Liquid		-----	
	n	22			
	mean (n)	Pass (Clear & Bright)			

Determination of Density at 20°C on sample #20165; results in kg/L

lab	method	value	mark	z(targ)	remarks
52	D4052	1.5239		-0.45	
53		-----		-----	
150	D4052	1.5245		2.91	
159		-----		-----	
169	D4052	1.5243		1.79	
171	D4052	1.5240		0.11	
316	INH-009	1.52445		2.63	
319		-----		-----	
323	D4052	1.5238		-1.01	
329	D4052	1.5239		-0.45	
334	ISO12185	1.5241		0.67	
338	ISO12185	1.5243	C	1.79	first reported 1.5277
345	D4052	1.5239		-0.45	
347	D4052	1.5244		2.35	
348		-----		-----	
357		-----		-----	
391	ISO12185	1.5238		-1.01	
444	D4052	1.5237		-1.57	
551	D4052	1.5241		0.67	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613	D4052	1.5248	C	4.59	first reported 1.5253
657	D4052	1.52374		-1.34	
704	D4052	1.524		0.11	
840	D4052	1.5233		-3.81	
902	ISO12185	1.5235		-2.69	
1067	ISO12185	1.5240		0.11	
1158		-----		-----	
1264		-----		-----	
1319		1.52369		-1.62	
1508		-----		-----	
1510		-----		-----	
1728	D4052	1.52388		-0.56	
1795		-----		-----	
6016	D4052	1.5237	C	-1.57	reported 1523.7 kg/L
6262		-----		-----	
6270	JIS K1200-1	1.52376		-1.23	

normality	OK
n	24
outliers	0
mean (n)	1.52398
st.dev. (n)	0.000343
R(calc.)	0.00096
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

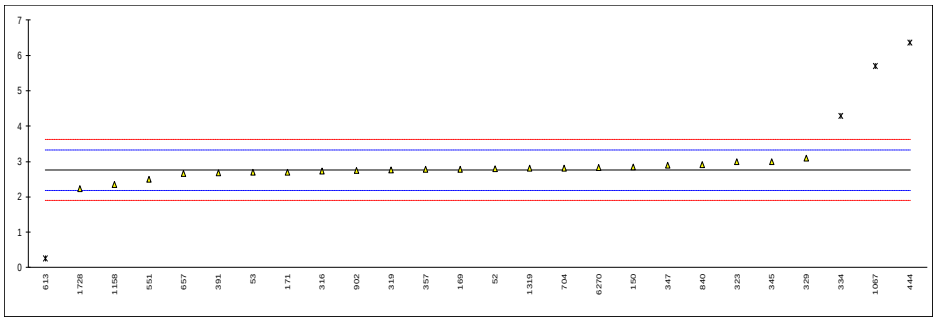


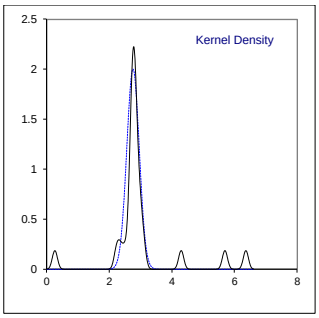


Determination of Iron as Fe on sample #20165; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	E291	2.8		0.15	
53	E291	2.7		-0.20	
150	E291	2.85	C	0.32	first reported 1.84
159		-----		-----	
169	E291	2.789		0.11	
171	E291	2.7		-0.20	
316	INH-043	2.73		-0.10	
319	INH-104	2.77		0.04	
323	E291	3.0		0.85	
329	E291	3.1		1.20	
334	E291	4.3	C,R(0.01)	5.40	first reported 0.4
338		-----		-----	
345	E291	3.0		0.85	
347	E291	2.9		0.50	
348		-----		-----	
357	E291	2.78		0.08	
391	E291	2.69		-0.24	
444	E291	6.37	C,R(0.01)	12.64	first reported 1.12
551	E291	2.5		-0.90	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613	E291	0.257	C,R(0.01)	-8.76	first reported 0.229
657	E291	2.6666		-0.32	
704	E291	2.82		0.22	
840	E291	2.91		0.53	
902	E291	2.75		-0.03	
1067	E291	5.7	R(0.01)	10.30	
1158	In house	2.35		-1.43	
1264		-----		-----	
1319		2.811		0.19	
1508		-----		-----	
1510		-----		-----	
1728	E291	2.23	C	-1.85	first reported 1.5
1795		-----		-----	
6016		-----		-----	
6262		-----		-----	
6270	JIS K1200-6	2.83		0.25	

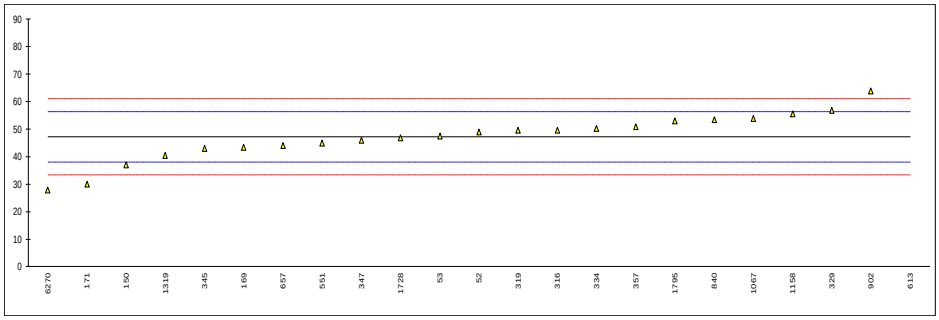
normality	suspect
n	22
outliers	4
mean (n)	2.758
st.dev. (n)	0.1992
R(calc.)	0.558
st.dev.(E291:18)	0.2857
R(E291:18)	0.800

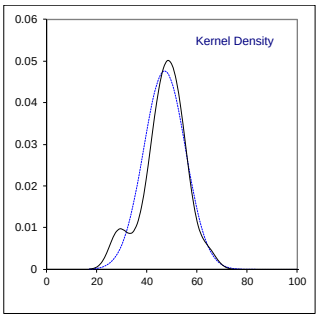




Determination of Sodium Chloride as NaCl on sample #20165; results in mg/kg

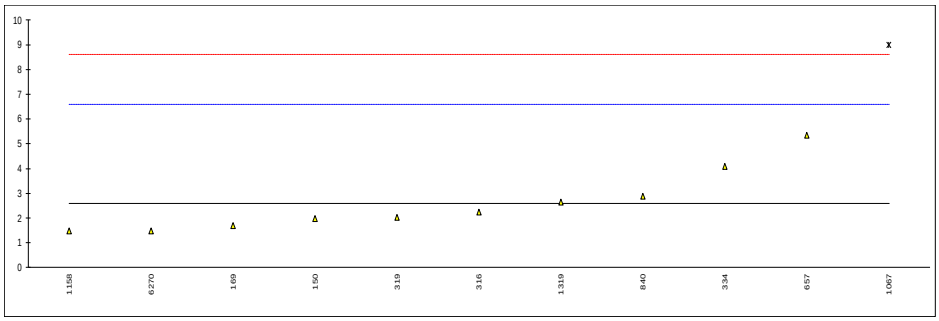
lab	method	value	mark	z(targ)	remarks
52	INH-802-PON-194	49		0.39	
53	INH-802Q-PON-002	47.47		0.06	
150	E1787	37		-2.22	
159		-----		-----	
169	E1787	43.5		-0.81	
171	E291	30		-3.74	
316	INH-044	49.75		0.55	
319	INH-239	49.66		0.53	
323		-----		-----	
329	INH-S/004	57		2.12	
334	E1787	50.3		0.67	
338		-----		-----	
345	E291	43		-0.91	
347	E291	46.1		-0.24	
348		-----		-----	
357	E291	51		0.82	
391		-----		-----	
444		-----		-----	
551	E291	45		-0.48	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613	E291	450	C,R(0.01)	87.43	first reported 0.045 mg/kg
657	E291	44.0322		-0.69	
704		-----		-----	
840	ISO6227	53.6		1.39	
902	E1787	64		3.64	
1067	E291	54		1.47	
1158	E291	55.7		1.84	
1264		-----		-----	
1319		40.561		-1.44	
1508		-----		-----	
1510		-----		-----	
1728		47	C	-0.05	first reported 78
1795	E291	53.0		1.26	
6016		-----		-----	
6262		-----		-----	
6270	JIS K1200-3-2	28.0		-4.17	
	normality	OK			
	n	22			
	outliers	1			
	mean (n)	47.212			
	st.dev. (n)	8.3704			
	R(calc.)	23.437			
	st.dev.(E1787:16)	4.6071			
	R(E1787:16)	12.9			
Compare:					
	R(E291:18)	15			

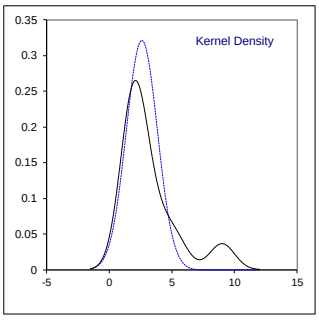




Determination of Sodium Chlorate as $NaClO_3$ on sample #20165; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	INH-802-PON-37	<20		-----	
53		-----		-----	
150	E1787	2.0		-0.30	
159		-----		-----	
169	INH-061112	1.7		-0.45	
171	INH-061112	<10		-----	
316	INH-075	2.25		-0.17	
319	INH-888	2.04		-0.28	
323	E1787	<10		-----	
329	INH-T010	<10		-----	
334	E1787	4.1		0.75	
338		-----		-----	
345		-----		-----	
347		-----		-----	
348		-----		-----	
357		-----		-----	
391		-----		-----	
444		-----		-----	
551	NBR9851	<10		-----	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613		-----		-----	
657	INH-0134	5.3450		1.37	
704		-----		-----	
840	INH-061112	2.9		0.15	
902		-----		-----	
1067	In house	9	G(0.01)	3.20	
1158	In house	1.5		-0.55	
1264		-----		-----	
1319		2.662		0.03	
1508		-----		-----	
1510		-----		-----	
1728		-----		-----	
1795		-----		-----	
6016		-----		-----	
6262		-----		-----	
6270	In house	1.5		-0.55	
	normality	not OK			
	n	10			
	outliers	1			
	mean (n)	2.600			
	st.dev. (n)	1.2430			
	R(calc.)	3.480			
	st.dev.(E1787:16)	2			
	R(E1787:16)	5.6			

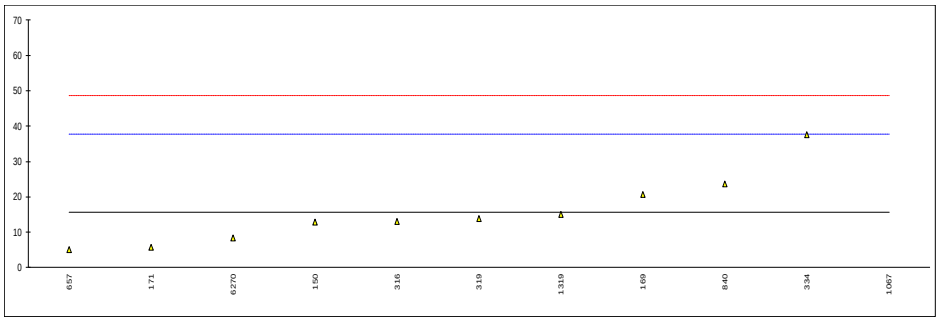


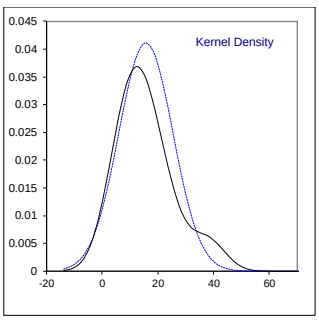


Determination of Sodium Sulfate as Na_2SO_4 on sample #20165; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	E291	<20		-----	
53		-----		-----	
150	E1787	13		-0.24	
159		-----		-----	
169	E1787	20.7		0.46	
171	E291	5.76		-0.90	
316	INH-073	13.1		-0.23	
319	INH-862	13.9		-0.16	
323	E1787	<10		-----	
329	INH-S/023	<10		-----	
334	E1787	37.6		2.00	
338		-----		-----	
345		-----		-----	
347		-----		-----	
348		-----		-----	
357		-----		-----	
391		-----		-----	
444		-----		-----	
551	NBR15132	<10		-----	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613		-----		-----	
657	E291	5.1262		-0.96	
704		-----		-----	
840	E291	23.7	C	0.73	first reported 47.3
902		-----		-----	
1067	E291	796	G(0.01)	70.94	
1158		-----		-----	
1264		-----		-----	
1319		15.063		-0.05	
1508		-----		-----	
1510		-----		-----	
1728		-----		-----	
1795		-----		-----	
6016		-----		-----	
6262		-----		-----	
6270	JIS K1200-4	8.5		-0.65	

normality	not OK
n	10
outliers	1
mean (n)	15.645
st.dev. (n)	9.7110
R(calc.)	27.191
st.dev.(E1787:16)	11
R(E1787:16)	30.8

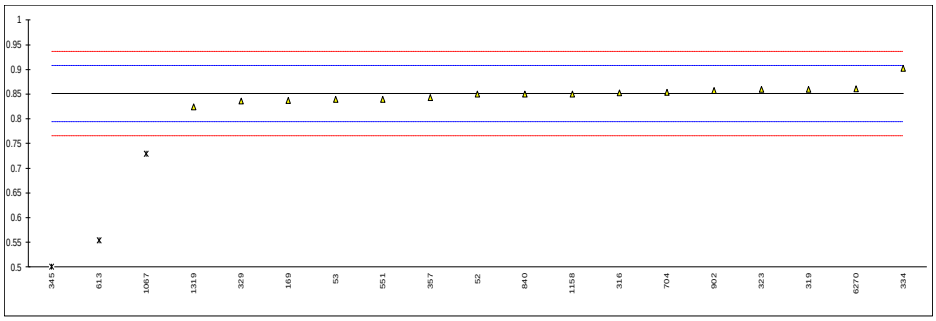


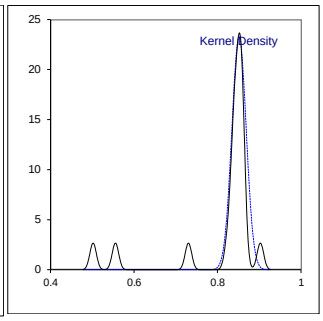


Determination of Sodium Chloride as NaCl on sample #20166; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	E291	0.85		-0.04	
53	E291	0.84		-0.39	
150		-----		-----	
159		-----		-----	
169	E291	0.8376		-0.48	
171		-----		-----	
316	INH-044	0.8533		0.07	
319	INH-269	0.8600		0.31	
323	INH-S/004	0.86		0.31	
329	E291	0.836		-0.53	
334	E291	0.9024		1.79	
338		-----		-----	
345	E291	0.5015	C,D(0.01)	-12.24	first reported 0.9515
347		-----		-----	
348		-----		-----	
357	E291	0.843		-0.29	
391		-----		-----	
444		-----		-----	
551	E291	0.84		-0.39	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613	E291	0.555	C,D(0.01)	-10.37	first reported 0.209
657		-----		-----	
704	E291	0.854		0.10	
840	E291	0.8510		-0.01	
902	E1787	0.8572		0.21	
1067	E291	0.73	D(0.01)	-4.24	
1158	E291	0.851		-0.01	
1264		-----		-----	
1319	E291	0.824		-0.95	
1508		-----		-----	
1510		-----		-----	
1728		-----		-----	
1795		-----		-----	
6016		-----		-----	
6262		-----		-----	
6270	JIS K1200-3-2	0.861		0.34	

normality	not OK
n	16
outliers	3
mean (n)	0.8513
st.dev. (n)	0.01712
R(calc.)	0.0479
st.dev.(E291:18)	0.02857
R(E291:18)	0.08

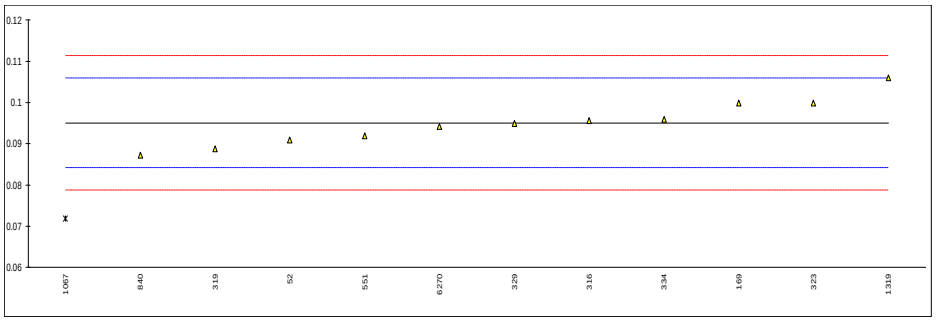


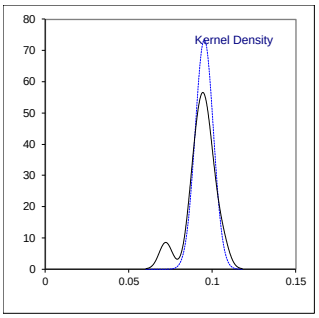


Determination of Sodium Chlorate as $NaClO_3$ on sample #20166; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	INH-802-PON-37	0.091		-0.76	
53		-----		-----	
150		-----		-----	
159		-----		-----	
169	INH-061112	0.10	C	0.90	first reported 0.00828
171		-----		-----	
316	INH-075	0.0957		0.11	
319	INH-888	0.08886		-1.15	
323	INH-T/010	0.10		0.90	
329	INH-T010	0.095		-0.02	
334	E1787	0.0960		0.17	
338		-----		-----	
345		-----		-----	
347		-----		-----	
348		-----		-----	
357		-----		-----	
391		-----		-----	
444		-----		-----	
551	NBR9851	0.092		-0.57	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613		-----		-----	
657		-----		-----	
704		-----		-----	
840	INH-061112	0.08731		-1.44	
902		-----		-----	
1067	In house	0.072	G(0.05)	-4.26	
1158		-----		-----	
1264		-----		-----	
1319	In house	0.106		2.01	
1508		-----		-----	
1510		-----		-----	
1728		-----		-----	
1795		-----		-----	
6016		-----		-----	
6262		-----		-----	
6270	In house	0.0942		-0.17	

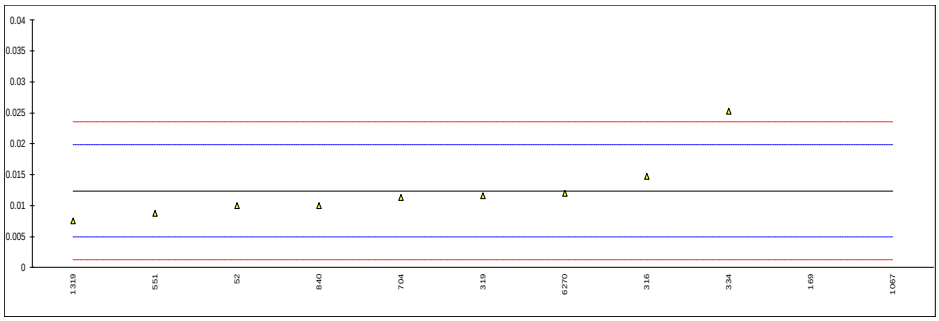
normality	OK
n	11
outliers	1
mean (n)	0.0951
st.dev. (n)	0.00543
R(calc.)	0.0152
st.dev.(Horwitz)	0.00542
R(Horwitz)	0.0152

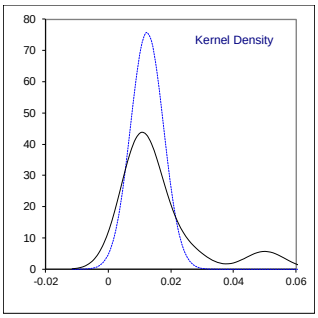




Determination of Sodium Sulfate as Na_2SO_4 on sample #20166; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	E291	0.010		-0.64	
53		-----		-----	
150		-----		-----	
159		-----		-----	
169	E291	0.05006	G(0.01)	10.13	
171		-----		-----	
316	INH-073	0.01477		0.64	
319	INH-862	0.01168		-0.19	
323		-----		-----	
329		-----		-----	
334	E291	0.0253		3.47	
338		-----		-----	
345		-----		-----	
347		-----		-----	
348		-----		-----	
357		-----		-----	
391		-----		-----	
444		-----		-----	
551	NBR15132	0.0088		-0.97	
554		-----		-----	
557		-----		-----	
562		-----		-----	
613		-----		-----	
657		-----		-----	
704	GOST2263	0.0114		-0.27	
840	E291	0.0100		-0.64	
902		-----		-----	
1067	E291	0.400	G(0.01)	104.24	
1158		-----		-----	
1264		-----		-----	
1319	E291	0.0076		-1.29	
1508		-----		-----	
1510		-----		-----	
1728		-----		-----	
1795		-----		-----	
6016		-----		-----	
6262		-----		-----	
6270	In house	0.0120		-0.11	
	normality	not OK			
	n	9			
	outliers	2			
	mean (n)	0.0124			
	st.dev. (n)	0.00526			
	R(calc.)	0.0147			
	st.dev.(E291:18)	0.00372			
	R(E291:18)	0.0104			





APPENDIX 2

Number of participants per country

1 lab in	AUSTRALIA
3 labs in	BELGIUM
3 labs in	BRAZIL
2 labs in	CANADA
1 lab in	CHILE
1 lab in	FINLAND
2 labs in	FRANCE
1 lab in	ITALY
2 labs in	JAPAN
1 lab in	KAZAKHSTAN
3 labs in	NETHERLANDS
3 labs in	ROMANIA
2 labs in	SAUDI ARABIA
1 lab in	SINGAPORE
3 labs in	SPAIN
1 lab in	TURKEY
1 lab in	UKRAINE
2 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	= possibly an error in calculations
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

Literature

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2 ASTM E178:02
3 ASTM E1301:95 (2003)
4 ISO5725:86
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9 IP367:96
10 DIN38402 T41/42
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12 J.N. Miller, Analyst, 118, 455, (1993)
13 ASTM E346:03e1
14 Analytical Methods Committee, Technical brief, No 4, January 2001
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