Institute for
Interlaboratory Studies

Results of Proficiency Test
Caustic Soda (Sodium Hydroxide solution)
September 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: Mrs. E.R. Montenij-Bos
Correctors: ing. R.J. Starink & ing. A. Ouwerkerk
Approved by: ing. A.S. Noordman-de Neef

Report: iis22C07

November 2022

CONTENTS

1 INTRODUCTION

Since 2012 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Caustic Soda every other year. During the annual proficiency testing program 2022/2023 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 salt concentration and one with a high salt concentration.

In this interlaboratory study 33 laboratories in 21 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the Caustic Soda proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the 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 0.5 L PE-bottle with a low salt content labelled #22165 and 1x 0.25 L PE-bottle with a high salt content labelled #22166.
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 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 50 liters of Caustic Soda was obtained from a local supplier and was spiked with Iron Chloride. After homogenization 55 HDPE bottles of 0.5 L were filled and labelled #22165.
The homogeneity of the subsamples was checked by determination of Alkalinity as NaOH in accordance with ASTM E291 on 4 stratified randomly selected subsamples.

	Alkalinity as NaOH in %M/M
sample #22165-1	49.70
sample #22165-2	49.70
sample #22165-3	49.69
sample #22165-4	49.69

Table 1: homogeneity test results of subsamples #22165

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

	Alkalinity as NaOH in %M/M
r (observed)	0.02
reference test method	ASTM E291:18
0.3 x R (reference test method)	0.21

Table 2: evaluation of the repeatability of subsamples #22165

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of approximately 15 liters of Caustic Soda was obtained from a local supplier and spiked with Sodium Chloride, Sodium Chlorate and Sodium Sulfate. After homogenization 55 HDPE bottles of 0.25 L were filled and labelled #22166.
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 #22166-1	0.0922
sample #22166-2	0.0903
sample #22166-3	0.0940

Table 3: homogeneity test results of subsamples #22166

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility calculated with 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)	0.0052
reference method	Horwitz
0.3 x R (reference method)	0.0044

Table 4: evaluation of the repeatability of subsamples #22166

The calculated repeatability is almost in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation. When compared to the observed reproducibilities over iis PTs from 2012 to 2020 the RSD of the test results mentioned in table 3 are in agreement with 0.3 times the average RSD from previous iis PTs. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one 0.5 L bottle of Caustic Soda labelled #22165 and one 0.25 L bottle of Caustic Soda labelled #22166 were sent on August 24, 2022. An SDS 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 #22165: 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 #22166 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 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 reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, 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:

$z_{(target)}$ = (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. 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 some problems were encountered with the dispatch of the samples. No participants reported test results after the final reporting date but nine participants did not report any test results. Not all participants were able to report all tests requested.
In total 24 participants reported 134 numerical test results. Observed were 9 outlying test results, which is 6.7%. In proficiency tests 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 original data 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 with 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 #22165

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

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

Density at 20 °C: 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 ISO12185:96.

Iron as Fe: This determination was not problematic. Two 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 as NaCl: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM E1787:16 nor with the requirements of ASTM E291:18.

Sodium Chlorate as $NaClO_3$: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM E1787:16.

Sodium Sulfate as Na_2SO_4: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM E1787:16.

sample #22166

Sodium Chloride as NaCl: This determination was not problematic. Two 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 as $NaClO_3$: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated from the Horwitz equation.

Sodium Sulfate as Na_2SO_4: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full 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 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 reference methods are presented in the next two tables.

Parameter	unit	n	average	2.8 * sd	R(lit)
Alkalinity as NaOH	%M/M	22	49.58	0.64	0.7
Appearance		18	Pass (C&B)	n.a.	n.a.
Density at 20 °C	kg/L	15	1.5214	0.0010	0.0005
Iron as Fe	mg/kg	18	2.6	0.6	0.8
Sodium Chloride as NaCl	mg/kg	20	38.3	28.7	12.9
Sodium Chlorate as $NaClO_3$	mg/kg	6	2.4	3.9	5.6
Sodium Sulfate as Na_2SO_4	mg/kg	9	18.0	35.9	30.8

Table 5: reproducibilities of tests on sample #22165

Parameter	unit	n	average	2.8 * sd	R(lit)
Sodium Chloride as NaCl	%M/M	15	0.857	0.043	0.08
Sodium Chlorate as $NaClO_3$	%M/M	10	0.092	0.012	0.015
Sodium Sulfate as Na_2SO_4	%M/M	10	0.017	0.015	0.014

Table 6: reproducibilities of tests on sample #22166

Without further statistical calculations it can be concluded that for many tests there is a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2022 WITH PREVIOUS PTS

	September 2022	September 2020	September 2018	September 2016	September 2014
Number of reporting laboratories	24	29	38	30	26
Number of test results	134	164	181	175	150
Number of statistical outliers	9	15	13	17	10
Percentage of statistical outliers	6.7%	9.1%	7.2%	9.7%	6.7%

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 to the requirements of the reference test methods. The conclusions are given in the following two tables.

	September 2022	September 2020	September 2018	September 2016	September 2014
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	-	+	+	+	(--)

Table 8: comparison determinations to the reference test methods on sample #22165

	September 2022	September 2020	September 2018	September 2016	September 2014
Sodium Chloride as NaCl	+	+	+	-	-
Sodium Chlorate as $NaClO_3$	+	+/-	--	+/-	+
Sodium Sulfate as Na_2SO_4	+/-	-	-	-	(-)

Table 9: comparison determinations to the reference test methods on sample #22166

For tables 8 and 9: results between brackets the average was below the application range of the reference test method

The following performance categories were used:

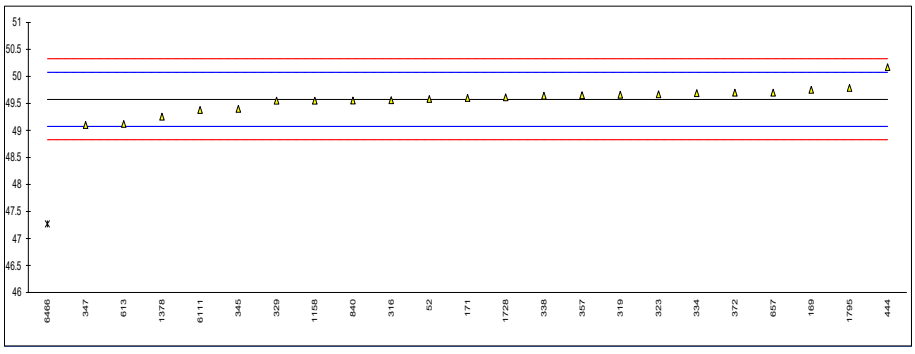
++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/- : group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.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

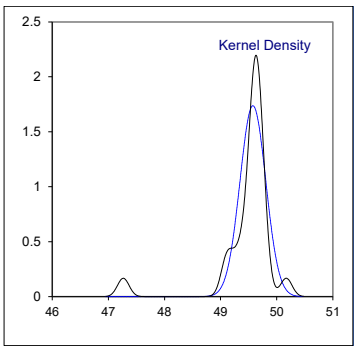
APPENDIX 1

Determination of Alkalinity as NaOH on sample #22165; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	E291	49.58		0.02	
53		-----		-----	
150		-----		-----	
159		-----		-----	
169	E291	49.75		0.70	
171	E291	49.60		0.10	
316	INH-041	49.56		-0.06	
319	INH-726	49.659		0.33	
323	E291	49.67		0.38	
329	E291	49.55		-0.10	
334	E291	49.69		0.46	
338	ISO979	49.64		0.26	
345	E291	49.40		-0.70	
347	D501	49.1		-1.90	
357	E291	49.650		0.30	
372	E291	49.70		0.50	
444	E291	50.17	C	2.38	first reported 51.72
551		-----		-----	
554		-----		-----	
557		-----		-----	
613		49.12		-1.82	
657	E291	49.7	C	0.50	first reported 47.19
704		-----		-----	
840	INH-3795	49.554		-0.09	
902		-----		-----	
1158	E291	49.55		-0.10	
1264		-----		-----	
1378	E291	49.2546		-1.29	
1728		49.61		0.14	
1795	In house	49.7856		0.84	
6111	E291	49.3796		-0.79	
6421		-----		-----	
6466	ISO979	47.2674375	R(0.01)	-9.23	

normality	suspect
n	22
outliers	1
mean (n)	49.576
st.dev. (n)	0.2296
R(calc.)	0.643
st.dev.(E291:18)	0.25
R(E291:18)	0.7





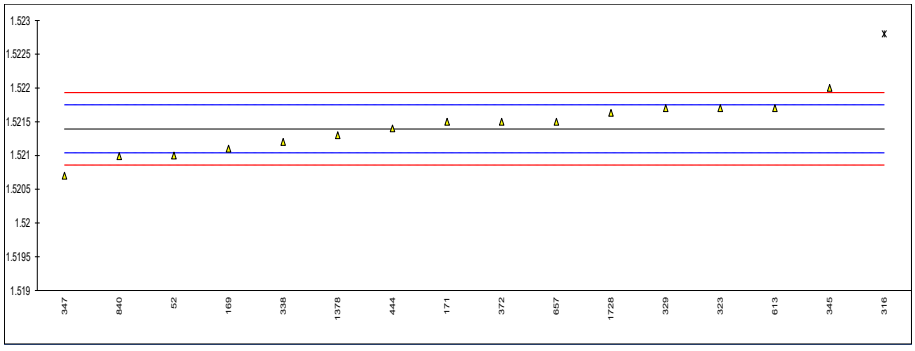
Determination of Appearance on sample #22165;

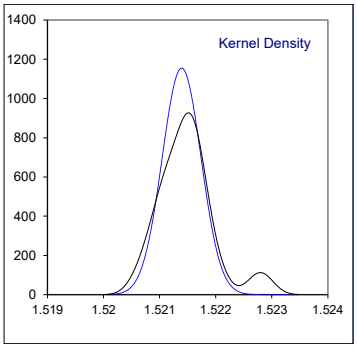
lab	method	value	mark	z(targ)	remarks
52	E2680	Pass		-----	
53		-----		-----	
150		-----		-----	
159		-----		-----	
169	Visual	CB&FSM		-----	
171	E2680	Clear and Free		-----	
316	Visual	CLEAR		-----	
319		-----		-----	
323	Visual	C&B		-----	
329	Visual	clear & bright		-----	
334	Visual	clear and bright SMS		-----	
338	Visual	clear & bright		-----	
345	E2680	PASS		-----	
347	E2680	Pass		-----	
357	E2680	Pass		-----	
372	E2680	pass		-----	
444	E2680	Pass		-----	
551		-----		-----	
554		-----		-----	
557		-----		-----	
613	D2090	C&C		-----	
657	E2680	clear & bright		-----	
704		-----		-----	
840	E2680	Pass		-----	
902		-----		-----	
1158		-----		-----	
1264		-----		-----	
1378	Visual	Clear		-----	
1728	Visual	CLEAR		-----	
1795		-----		-----	
6111		-----		-----	
6421		-----		-----	
6466		-----		-----	
	n	18			
	mean (n)	Pass (Clear & Bright)			

Determination of Density at 20 °C on sample #22165; results in kg/L

lab	method	value	mark	z(targ)	remarks
52	D4052	1.521	C	-2.21	first reported 1.520
53		-----		-----	
150		-----		-----	
159		-----		-----	
169	D4052	1.5211	C	-1.65	first reported 1522.7
171	D4052	1.5215		0.59	
316	INH-009	1.5228	G(0.05)	7.87	
319		-----		-----	
323	D4052	1.5217		1.71	
329	D4052	1.5217		1.71	
334	ISO12185	>1.100		-----	
338	ISO12185	1.5212		-1.09	
345	D4052	1.522		3.39	
347	D4052	1.5207		-3.89	
357		-----		-----	
372	ISO12185	1.5215		0.59	
444	D4052	1.5214		0.03	
551		-----		-----	
554		-----		-----	
557		-----		-----	
613	D4052	1.5217		1.71	
657	D4052	1.5215		0.59	
704		-----		-----	
840	D4052	1.52099		-2.27	
902		-----		-----	
1158		-----		-----	
1264		-----		-----	
1378	D4052	1.5213		-0.53	
1728	ISO12185	1.52163		1.32	
1795		-----		-----	
6111		-----		-----	
6421		-----		-----	
6466		-----		-----	

normality	OK
n	15
outliers	1
mean (n)	1.52139
st.dev. (n)	0.000345
R(calc.)	0.00097
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

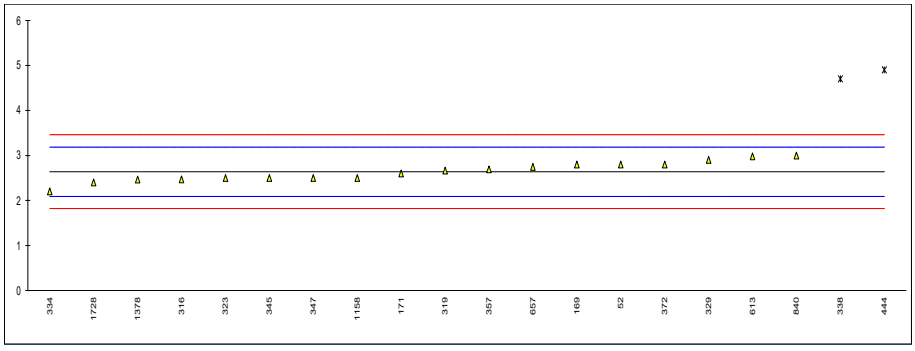


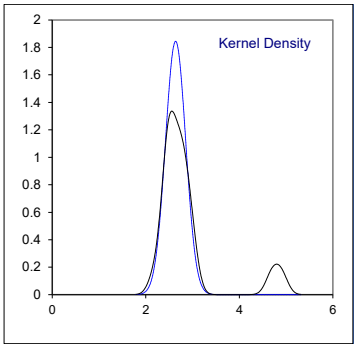


Determination of Iron as Fe on sample #22165; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	E291	2.8		0.59	
53		-----		-----	
150		-----		-----	
159		-----		-----	
169	E291	2.8		0.59	
171	E291	2.6		-0.15	
316	INH-043	2.47		-0.62	
319	INH-104	2.666		0.10	
323	E291	2.5		-0.51	
329	E291	2.9		0.95	
334	E291	2.2		-1.61	
338	E291	4.70	R(0.01)	7.54	
345	E291	2.5		-0.51	
347	E291	2.5		-0.51	
357	E291	2.69		0.18	
372	E291	2.8		0.59	
444	E291	4.9	C,R(0.01)	8.27	first reported 5.1
551		-----		-----	
554		-----		-----	
557		-----		-----	
613		2.98	C	1.24	first reported 1.811
657	E291	2.7449		0.38	
704		-----		-----	
840	INH-3797	3.0		1.32	
902		-----		-----	
1158	In house	2.50		-0.51	
1264		-----		-----	
1378	E291	2.4647		-0.64	
1728	E291	2.4	C	-0.88	first reported 1.33
1795		-----		-----	
6111		-----		-----	
6421		-----		-----	
6466		-----		-----	

normality	OK
n	18
outliers	2
mean (n)	2.640
st.dev. (n)	0.2162
R(calc.)	0.605
st.dev.(E291:18)	0.2734
R(E291:18)	0.766

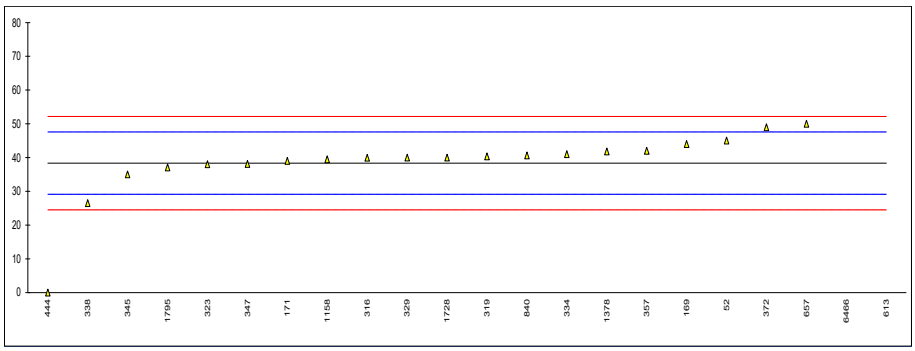


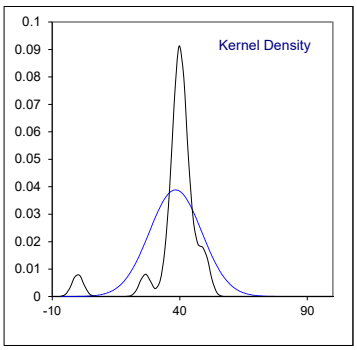


Determination of Sodium Chloride as NaCl on sample #22165; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	INH-061112	45		1.44	
53		-----		-----	
150		-----		-----	
159		-----		-----	
169	E1787	44		1.23	
171	E291	39	C	0.14	first reported 60
316	INH-044	39.9579		0.35	
319	INH-269	40.32		0.43	
323	INH-009	38		-0.07	
329	E291	40		0.36	
334	E1787	41		0.58	
338	E291	26.5		-2.57	
345	E291	35		-0.73	
347	E291	38.1		-0.05	
357	E291	42		0.79	
372	E291	49		2.31	
444	E291	0		-8.32	
551		-----		-----	
554		-----		-----	
557		-----		-----	
613	E291	460	C,R(0.01)	91.52	first reported 0.085 %M/M
657	E291	50	C	2.53	first reported 81.77
704		-----		-----	
840	ISO6227	40.6		0.49	
902		-----		-----	
1158	E291	39.5		0.25	
1264		-----		-----	
1378	In house	41.8172		0.75	
1728		40		0.36	
1795	E291	37.09		-0.27	
6111		-----		-----	
6421		-----		-----	
6466	EN896	237.7708	C,R(0.01)	43.29	first reported 130.6667

	normality	not OK
	n	20
	outliers	2
	mean (n)	38.344
	st.dev. (n)	10.2611
	R(calc.)	28.731
	st.dev.(E1787:16)	4.6071
	R(E1787:16)	12.9
Compare		
	R(E291:18)	15

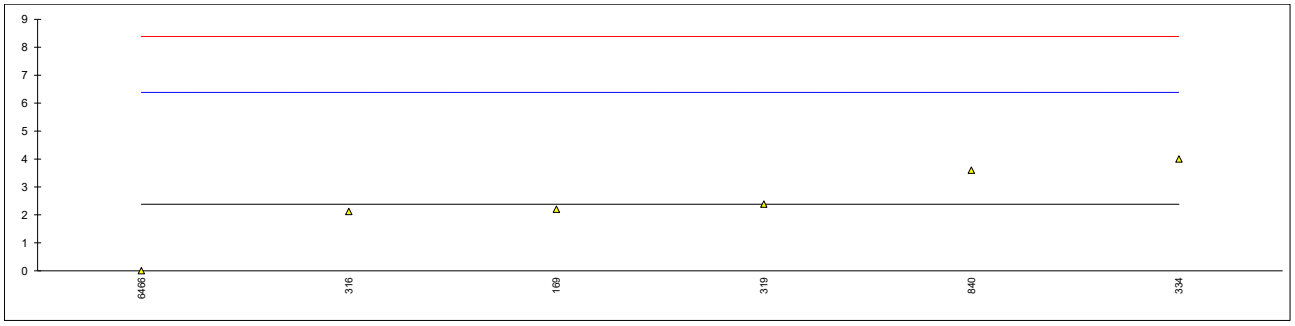




Determination of Sodium Chlorate as $NaClO_3$ on sample #22165; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	INH-016	<20		-----	
53		-----		-----	
150		-----		-----	
159		-----		-----	
169	INH-061112	2.2		-0.09	
171	E291	<10	C	-----	first reported 14
316	INH-075	2.11885		-0.13	
319	INH-888	2.381		0.00	
323		-----		-----	
329	INH-010	<10		-----	
334	E1787	4.0		0.81	
338		-----		-----	
345		-----		-----	
347		-----		-----	
357		-----		-----	
372		-----		-----	
444		-----		-----	
551		-----		-----	
554		-----		-----	
557		-----		-----	
613		-----		-----	
657	INH-134	<5	C	-----	first reported 17.27
704		-----		-----	
840	INH-061112	3.6		0.61	
902		-----		-----	
1158		-----		-----	
1264		-----		-----	
1378		-----		-----	
1728		-----		-----	
1795		-----		-----	
6111		-----		-----	
6421		-----		-----	
6466	EN896	0		-1.19	

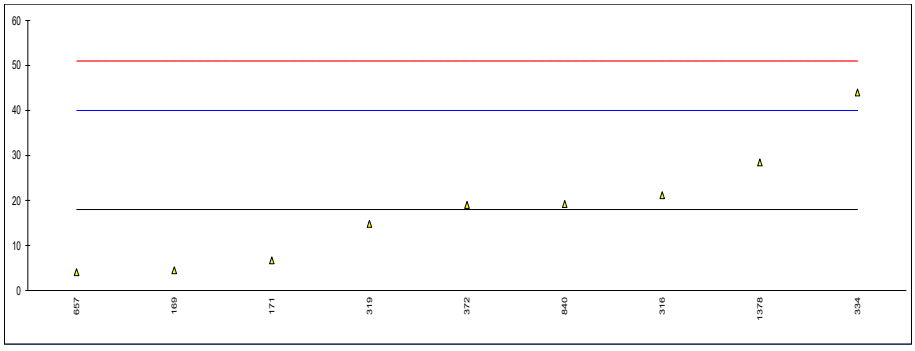
normality	unknown
n	6
outliers	0
mean (n)	2.383
st.dev. (n)	1.4055
R(calc.)	3.936
st.dev.(E1787:16)	2
R(E1787:16)	5.6

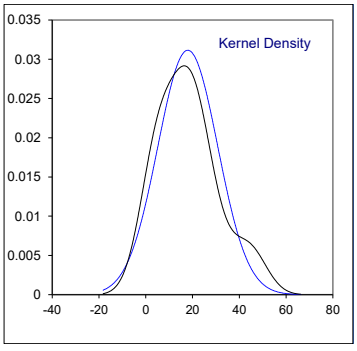


Determination of Sodium Sulfate as Na_2SO_4 on sample #22165; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	E291	<20		-----	
53		-----		-----	
150		-----		-----	
159		-----		-----	
169	E1787	4.5		-1.23	
171	E291	6.7		-1.03	
316	INH-073	21.164		0.29	
319	INH-862	14.78		-0.29	
323	INH-008	< 10		-----	
329	INH-008	<10		-----	
334	E1787	44		2.36	
338		-----		-----	
345		-----		-----	
347		-----		-----	
357		-----		-----	
372	E291	19		0.09	
444		-----		-----	
551		-----		-----	
554		-----		-----	
557		-----		-----	
613		-----		-----	
657	E291	4.056		-1.27	
704		-----		-----	
840	E1787	19.2		0.11	
902		-----		-----	
1158		-----		-----	
1264		-----		-----	
1378	In house	28.4666		0.95	
1728		-----		-----	
1795		-----		-----	
6111		-----		-----	
6421		-----		-----	
6466		-----		-----	

normality	OK
n	9
outliers	0
mean (n)	17.985
st.dev. (n)	12.8099
R(calc.)	35.868
st.dev.(E1787:16)	11
R(E1787:16)	30.8

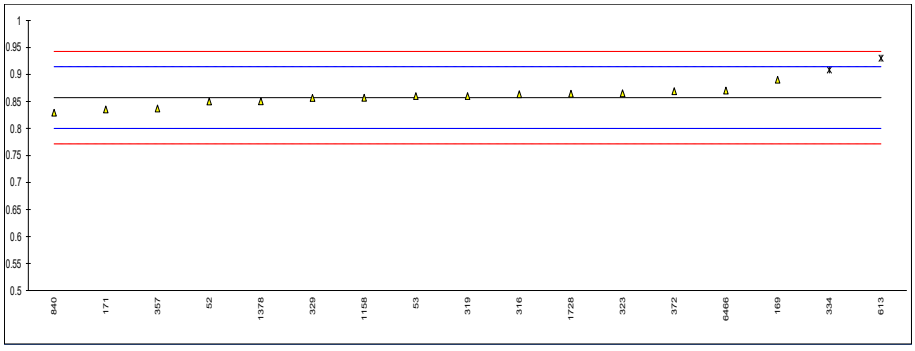


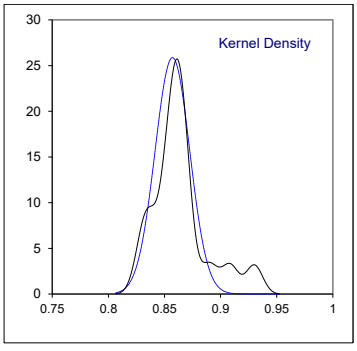


Determination of Sodium Chloride as NaCl on sample #22166; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	E291	0.85		-0.25	
53	E291	0.86		0.10	
150		-----		-----	
159		-----		-----	
169	E291	0.89		1.15	
171	E291	0.8352	C	-0.77	first reported 0.9762
316	INH-044	0.8632019		0.21	
319	INH-269	0.8600		0.10	
323	E291	0.865		0.28	
329	E291	0.8565		-0.02	
334	E1787	0.9083	DG(0.05)	1.79	
338		-----		-----	
345		-----		-----	
347		-----		-----	
357	E291	0.837		-0.70	
372	E291	0.869		0.42	
444		-----		-----	
551		-----		-----	
554		-----		-----	
557		-----		-----	
613	E291	0.93	C,DG(0.05)	2.55	first reported 1.045
657		-----		-----	
704		-----		-----	
840	INH-3796	0.829		-0.98	
902		-----		-----	
1158	E291	0.857		0.00	
1264		-----		-----	
1378	E291	0.8502	C	-0.24	first reported 0.7958
1728		0.864		0.24	
1795		-----		-----	
6111		-----		-----	
6421		-----		-----	
6466	EN896	0.870144	C	0.46	first reported 0.515577778

normality	OK
n	15
outliers	2
mean (n)	0.8571
st.dev. (n)	0.01542
R(calc.)	0.0432
st.dev.(E291:18)	0.02857
R(E291:18)	0.08

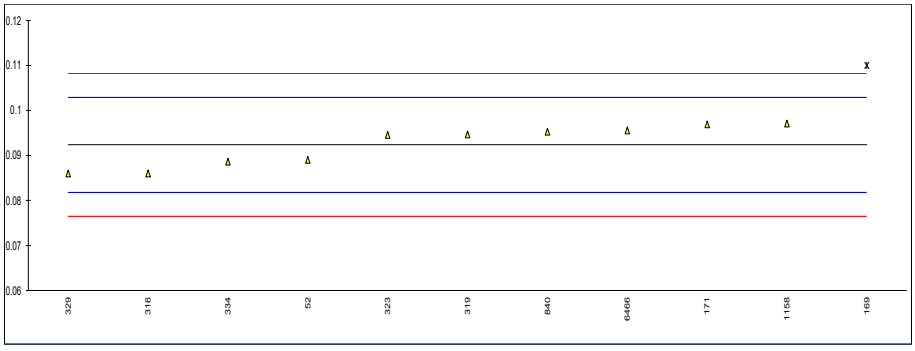


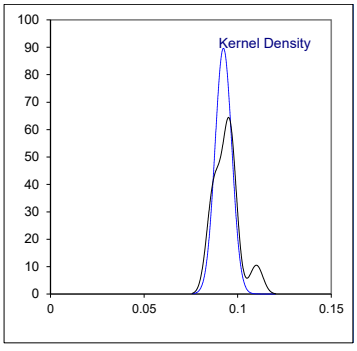


Determination of Sodium Chlorate as $NaClO_3$ on sample #22166; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	INH-016	0.089		-0.64	
53		-----		-----	
150		-----		-----	
159		-----		-----	
169	INH-061112	0.11	C,G(0.05)	3.33	first reported 0.08
171	E291	0.0969		0.86	
316	INH-075	0.0860127		-1.20	
319	INH-888	0.09468		0.44	
323	INH-010	0.09455		0.41	
329	INH-010	0.086		-1.20	
334	E1787	0.0886		-0.71	
338		-----		-----	
345		-----		-----	
347		-----		-----	
357		-----		-----	
372		-----		-----	
444		-----		-----	
551		-----		-----	
554		-----		-----	
557		-----		-----	
613		-----		-----	
657		-----		-----	
704		-----		-----	
840	INH-061112	0.0953		0.55	
902		-----		-----	
1158	In house	0.0971		0.89	
1264		-----		-----	
1378		-----		-----	
1728		-----		-----	
1795		-----		-----	
6111		-----		-----	
6421		-----		-----	
6466	EN896	0.09555558		0.60	

normality	OK
n	10
outliers	1
mean (n)	0.0924
st.dev. (n)	0.00445
R(calc.)	0.0125
st.dev.(Horwitz)	0.00529
R(Horwitz)	0.0148

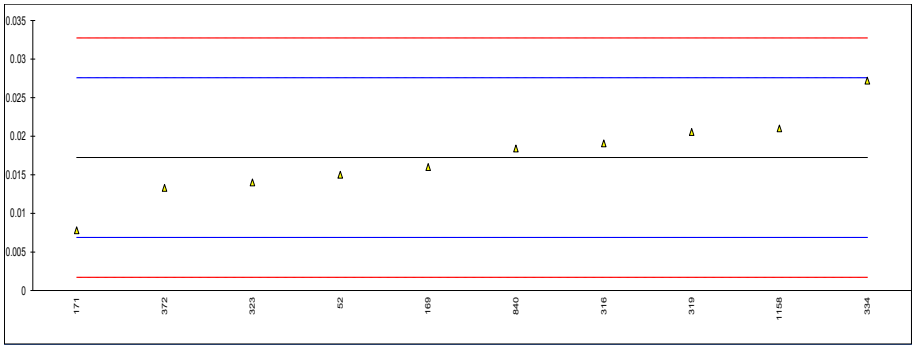


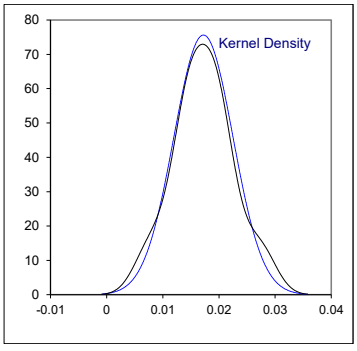


Determination of Sodium Sulfate as Na_2SO_4 on sample #22166; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	E291	0.015		-0.43	
53		-----		-----	
150		-----		-----	
159		-----		-----	
169	E291	0.016		-0.24	
171	E291	0.0078		-1.82	
316	INH-073	0.019	C	0.36	reported 190.652 %M/M
319	INH-862	0.02053		0.64	
323	INH-008	0.014		-0.62	
329		-----		-----	
334	E1787	0.0272		1.93	
338		-----		-----	
345		-----		-----	
347		-----		-----	
357		-----		-----	
372	E291	0.0133		-0.76	
444		-----		-----	
551		-----		-----	
554		-----		-----	
557		-----		-----	
613		-----		-----	
657		-----		-----	
704		-----		-----	
840	E291	0.0184		0.23	
902		-----		-----	
1158	In house	0.021		0.73	
1264		-----		-----	
1378		-----		-----	
1728		-----		-----	
1795		-----		-----	
6111		-----		-----	
6421		-----		-----	
6466		-----		-----	

normality	OK
n	10
outliers	0
mean (n)	0.017
st.dev. (n)	0.0053
R(calc.)	0.015
st.dev.(E291:18)	0.0052
R(E291:18)	0.014





APPENDIX 2

Number of participants per country

1 lab in AUSTRALIA
1 lab in AZERBAIJAN
2 labs in BELGIUM
3 labs in BRAZIL
2 labs in CANADA
1 lab in ESTONIA
1 lab in FINLAND
2 labs in FRANCE
1 lab in GREECE
1 lab in KUWAIT
2 labs in NETHERLANDS
1 lab in PORTUGAL
3 labs in ROMANIA
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
2 labs in SPAIN
1 lab in TURKEY
1 lab in UKRAINE
1 lab 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	= calculation difference between reported test result and result calculated by iis
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
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
8 J.N. Miller, Analyst, 118, 455, (1993)
9 Analytical Methods Committee, Technical Brief, No 4, January 2001
10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)