Institute for
Interlaboratory Studies

Results of Proficiency Test
AdBlue (32% Urea solution)
May 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2017 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of AdBlue 32% Urea solution based on the latest version of ISO22241 part 1 every year. During the annual proficiency testing program 2021/2022 it was decided to continue the round robin for the analysis of AdBlue 32% Urea solution.

In this interlaboratory study 28 laboratories in 18 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the AdBlue 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 one sample AdBlue 32% Urea solution in a 1-liter HDPE wide-neck bottle labelled #22082.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the 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 40 liters of AdBlue 32% Urea solution was obtained from a local supplier. After homogenization 35 wide-neck HDPE bottles of 1 L were filled and labelled #22082.
The homogeneity of the subsamples was checked by determination of Density at 20 °C in accordance with ASTM D4052 on 4 stratified randomly selected subsamples.

	Density at 20 °C in kg/m^3
sample #22082-1	1088.67
sample #22082-2	1088.62
sample #22082-3	1088.70
sample #22082-4	1088.71

Table 1: homogeneity test results of subsamples #22082

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.

	Density at 20 °C in kg/m^3
r (observed)	0.11
reference test method	ISO12185:96
0.3 x R (reference test method)	0.15

Table 2: evaluation of the repeatability of subsamples #22082

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

To each of the participating laboratories one sample AdBlue 32% Urea solution labelled #22082 was sent on April 20, 2022. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of AdBlue 32% Urea solution 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: Aldehyde as Formaldehyde, Alkalinity as NH_3, Biuret, Density at 20 °C, Insoluble matter, Phosphate as PO_4, Refractive index at 20 °C, Urea content (by total Nitrogen and by refractive index) and Trace elements (Al, Ca, Cr, Cu, Fe, Mg, Ni, K, Na and Zn).

It was explicitly requested to treat the sample as if it was a routine sample 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 appendices 1 and 2 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 appendices 1 and 2. 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 result tables of 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, due to COVID-19 pandemic.
Two participants reported test results after the final reporting date and four other participants did not report any test results. Not all participants were able to report all tests requested.
In total 24 participants reported 174 numerical test results. Observed were 9 outlying test results, which is 5.2%. 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 TEST

In this section the reported test results are discussed 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 appendices 1 and 2. The abbreviations, used in these tables, are explained in appendix 4.

Aldehyde as Formaldehyde: This determination was not problematic. Two statistical outliers were observed. The consensus value (0.035 mg/kg) is far below the application range of ISO22241-2:19 (0.5 – 10 mg/kg). Therefore it is decided not to calculate z-scores.

Alkalinity as NH_3: This determination was not problematic. All reporting participants agreed on a test result of less than 0.1 %M/M. Therefore, no z-scores are calculated.

Biuret: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO22241-2:19.

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.

Insoluble matter: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO22241-2:19.

Phosphate as PO_4: This determination may be problematic. Two statistical outliers were observed. The mean was below the application range. Therefore, no z-scores are calculated.

Refractive index at 20 °C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO22241-2:19.

Urea content (by total Nitrogen): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO22241-2:19.

Urea content (by refractive index): 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 ISO22241-2:19.

Sodium as Na: This determination was problematic. No statistical outliers were observed. It was decided not to calculate z-scores for Sodium as Na due to the large difference between the calculated and reference reproducibility.

The majority of the participants agreed on a concentration near or below the limit of detection for the other trace elements mentioned in paragraph 2.6. Therefore, no z-scores are calculated. The reported test results of these elements are given in appendix 2.

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 table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Aldehyde as Formaldehyde	mg/kg	10	0.04	0.09	(0.46)
Alkalinity as NH_3	%M/M	22	<0.1	n.e.	n.e.
Biuret	%M/M	23	0.145	0.035	0.044
Density at 20 °C	kg/m^3	22	1088.8	0.7	0.5
Insoluble Matter	mg/kg	18	2.08	4.65	8.22
Phosphate as PO_4	mg/kg	10	0.08	0.23	(0.08)
Refractive index at 20 °C		22	1.3823	0.0003	0.0003
Urea content (by total Nitrogen)	%M/M	6	32.3	0.6	1.1
Urea content (by refractive index)	%M/M	21	32.1	0.3	0.2
Sodium as Na	mg/kg	16	0.19	0.22	(0.03)

Table 3: reproducibilities of tests on sample #22082

For results between brackets no z-scores are calculated.

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 MAY 2022 WITH PREVIOUS PTS

	May 2022	May 2021	May 2020	June 2019	June 2018
Number of reporting laboratories	24	16	18	18	14
Number of test results	174	121	161	192	146
Number of statistical outliers	9	7	8	7	2
Percentage of statistical outliers	5.2%	5.8%	5.0%	3.6%	1.4%

Table 4: 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 table.

	May 2022	May 2021	May 2020	June 2019	June 2018
Aldehyde as Formaldehyde	(++)	+/-	+/-	+/-	+/-
Alkalinity as NH_3	n.e.	n.e.	++	++	-
Biuret	+	+	++	+/-	-
Density at 20 °C	-	+	+	+/-	+/-
Insoluble Matter	+	+	+/-	+/-	(--)
Phosphate as PO_4	(--)	-	n.e.	n.e.	n.e.
Refractive index at 20 °C	+/-	+	+	++	++
Urea content (by total Nitrogen)	+	n.e.	+	n.e.	n.e.
Urea content (by refractive index)	-	+/-	+	-	++
Potassium as K	n.e.	--	n.e.	n.e.	n.e.
Sodium as Na	(--)	(--)	n.e.	n.e.	n.e.

Table 5: comparison determinations to the reference test methods

Result between brackets no z-scores are calculated.

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

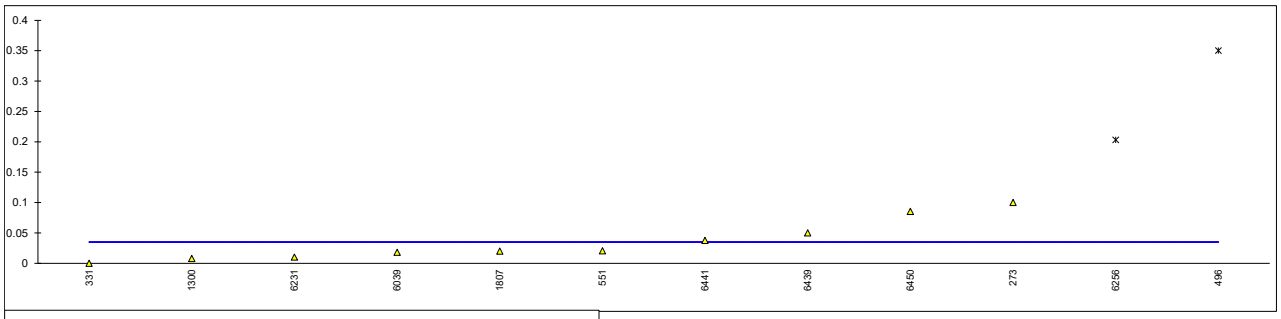
APPENDIX 1

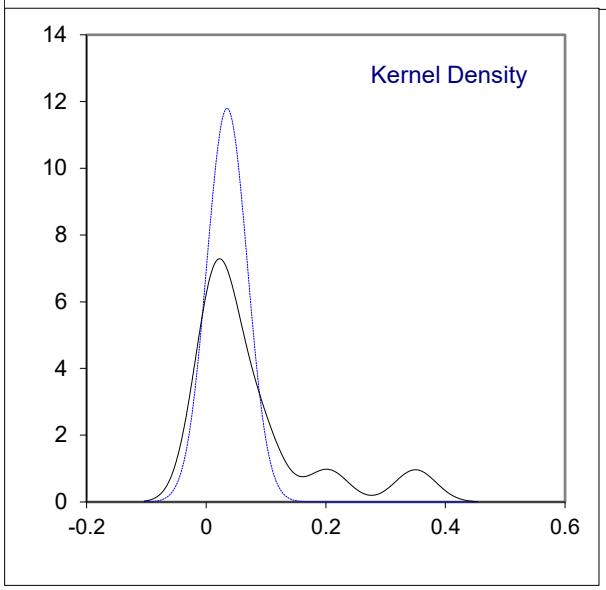
Determination of Aldehyde as Formaldehyde on sample #22082; result in mg/kg

lab	method	value	mark	z(targ)	remarks
273	ISO22241-2 Annex F	0.1		-----	
309	ISO22241-2 Annex F	<0.5		-----	
331	ISO22241-2 Annex F	0.0		-----	
334	ISO22241-2 Annex F	<0.5		-----	
398	ISO22241-2 Annex F	<0,5		-----	
405		-----		-----	
420		-----		-----	
455		-----		-----	
496	ISO22241-2 Annex F	0.35	G(0.01)	-----	
541	ISO22241-2 Annex F	<0.5		-----	
551	ISO22241-2 Annex F	0.02063		-----	
840	ISO22241-2 Annex F	<0.5		-----	
862		-----		-----	
863		-----		-----	
864		-----		-----	
1300	ISO22241-2 Annex F	0.008		-----	
1615	ISO22241-2 Annex F	<0.50		-----	
1656	ISO22241-2 Annex F	<0.5		-----	
1743	ISO22241-2 Annex F	<0.20		-----	
1807	ISO22241-2 Annex F	0.02		-----	
6039	ISO22241-2 Annex F	0.018		-----	
6139		-----		-----	
6146		-----		-----	
6231	ISO22241-2 Annex F	0.01		-----	
6256	ISO22241-2	0.203	G(0.05)	-----	
6439	ISO22241-2 Annex F	0.05		-----	
6441	ISO22241-2 Annex F	0.0377		-----	
6450	ISO22241-2 Annex F	0.0854		-----	

normality	suspect
n	10
outliers	2
mean (n)	0.035
st.dev. (n)	0.0338
R(calc.)	0.095
st.dev.(ISO22241-2:19)	(0.1657)
R(ISO22241-2:19)	(0.464)

Application range: 0.5 – 10 mg/kg





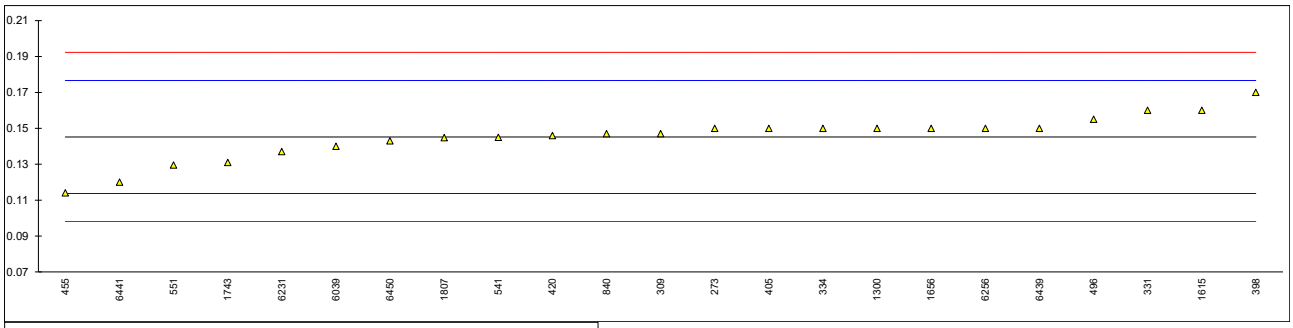
Determination of Alkalinity as NH_3 on sample #22082; result in %M/M

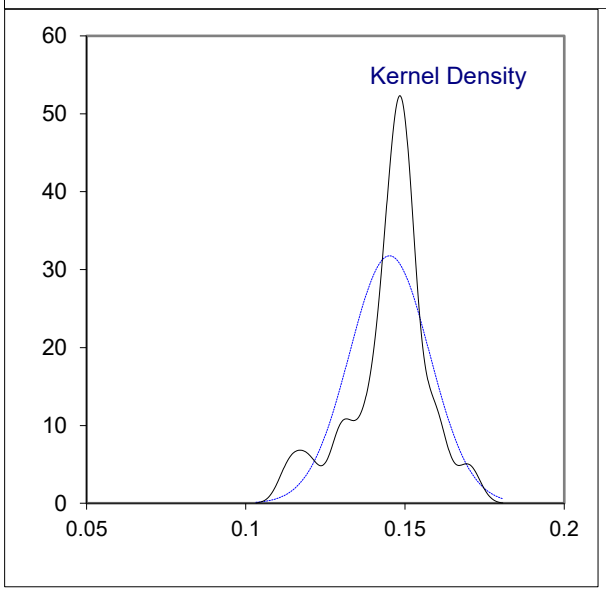
lab	method	value	mark	z(targ)	remarks
273	ISO22241-2 Annex D	0.001		-----	
309	ISO22241-2 Annex D	0.01		-----	
331	ISO22241-2 Annex D	0.0		-----	
334	ISO22241-2 Annex D	<0.10		-----	
398	ISO22241-2 Annex D	<0,1		-----	
405	ISO22241-2 Annex D	0.014		-----	
420	ISO22241-2 Annex D	0.01		-----	
455	ISO22241-2 Annex D	0.00870		-----	
496	ISO22241-2 Annex D	0.010		-----	
541	ISO22241-2 Annex D	<0.1		-----	
551	ISO22241-2 Annex D	<0.10		-----	
840	ISO22241-2 Annex D	0.01		-----	
862		-----		-----	
863		-----		-----	
864		-----		-----	
1300	ISO22241-2 Annex D	0.0091		-----	
1615	ISO22241-2 Annex D	0.011		-----	
1656	ISO22241-2 Annex D	<0.01		-----	
1743		-----		-----	
1807	ISO22241-2 Annex D	0.01		-----	
6039	ISO22241-2 Annex D	0.0114		-----	
6139		-----		-----	
6146		-----		-----	
6231	ISO22241-2 Annex D	0.009		-----	
6256	ISO22241-1	0.0136		-----	
6439	ISO22241-2 Annex D	0.00845		-----	
6441	ISO22241-2 Annex D	0.007	C	-----	first reported 0.07
6450	ISO22241-2 Annex D	0.0101		-----	
	n	22			
	mean (n)	<0.1			

Determination of Biuret on sample #22082; result in %M/M

lab	method	value	mark	z(targ)	remarks
273	ISO22241-2 Annex E	0.15		0.31	
309	ISO22241-2 Annex E	0.147		0.12	
331	ISO22241-2 Annex E	0.16		0.94	
334	ISO22241-2 Annex E	0.15		0.31	
398	ISO22241-2 Annex E	0.17		1.58	
405	ISO22241-2 Annex E	0.15		0.31	
420	ISO22241-2 Annex E	0.146		0.05	
455	ISO22241-2 Annex E	0.11400		-1.98	
496	ISO22241-2 Annex E	0.155		0.62	
541	ISO22241-2 Annex E	0.145		-0.01	
551	ISO22241-2 Annex E	0.1296		-0.99	
840	ISO22241-2 Annex E	0.147		0.12	
862		-----		-----	
863		-----		-----	
864		-----		-----	
1300	ISO22241-2 Annex E	0.150		0.31	
1615	ISO22241-2 Annex E	0.160		0.94	
1656	ISO22241-2 Annex E	0.15		0.31	
1743	ISO22241-2 Annex E	0.131		-0.90	
1807	ISO22241-2 Annex E	0.1448		-0.02	
6039	ISO22241-2 Annex E	0.14		-0.33	
6139		-----		-----	
6146		-----		-----	
6231	ISO22241-2 Annex E	0.137		-0.52	
6256	ISO22241-2	0.150		0.31	
6439	ISO22241-2 Annex E	0.15	C	0.31	first reported 0.115
6441	ISO22241-2 Annex E	0.120	C	-1.60	first reported 0.20
6450	ISO22241-2 Annex E	0.143		-0.14	

normality	suspect
n	23
outliers	0
mean (n)	0.1452
st.dev. (n)	0.01256
R(calc.)	0.0352
st.dev.(ISO22241-2:19)	0.01571
R(ISO22241-2:19)	0.044

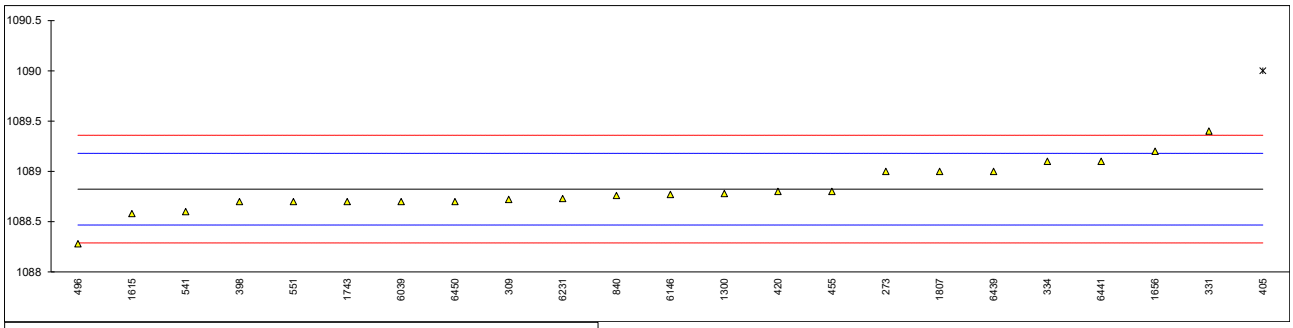


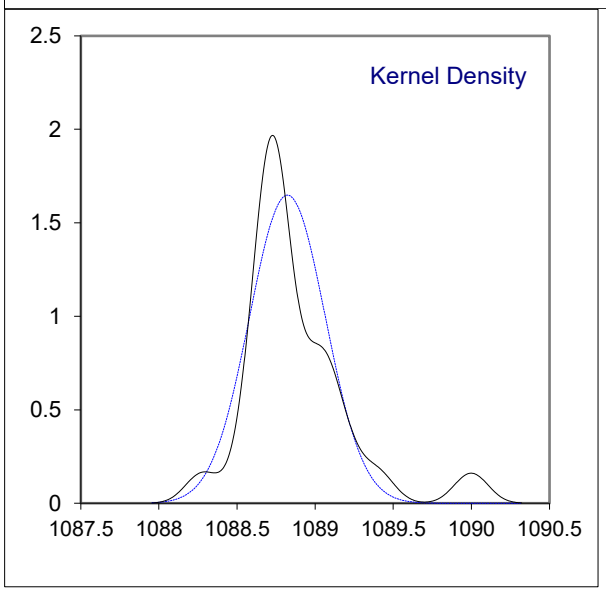


Determination of Density at 20 °C on sample #22082; result in kg/m^3

lab	method	value	mark	z(targ)	remarks
273	D4052	1089		0.99	
309	ISO12185	1088.72		-0.58	
331	ISO12185	1089.4	C	3.23	first reported 1087.8 kg/L
334	ISO12185	1089.1		1.55	
398	ISO12185	1088.70		-0.69	
405	ISO3675	1090	C,G(0.01)	6.59	first reported 1.091 kg/L
420	ISO12185	1088.8		-0.13	
455	ISO22241-2 Annex K	1088.80		-0.13	
496	ISO12185	1088.28	C	-3.04	first reported 1091.20 kg/L
541	ISO12185	1088.6		-1.25	
551	D4052	1088.7		-0.69	
840	ISO12185	1088.76		-0.36	
862		-----		-----	
863		-----		-----	
864		-----		-----	
1300	ISO12185	1088.78		-0.24	
1615	ISO12185	1088.58		-1.36	
1656	ISO12185	1089.2		2.11	
1743	ISO12185	1088.7		-0.69	
1807	ISO12185	1089.0		0.99	
6039	ISO12185	1088.7		-0.69	
6139		-----		-----	
6146	ISO12185	1088.77	C	-0.30	first reported 1.08877
6231	ISO12185	1088.73		-0.52	
6256		-----		-----	
6439	ISO3675	1089.0		0.99	
6441	ISO12185	1089.1		1.55	
6450	ISO12185	1088.7		-0.69	

normality	OK
n	22
outliers	1
mean (n)	1088.824
st.dev. (n)	0.24209
R(calc.)	0.6778
st.dev.(ISO12185:96)	0.17857
R(ISO12185:96)	0.5

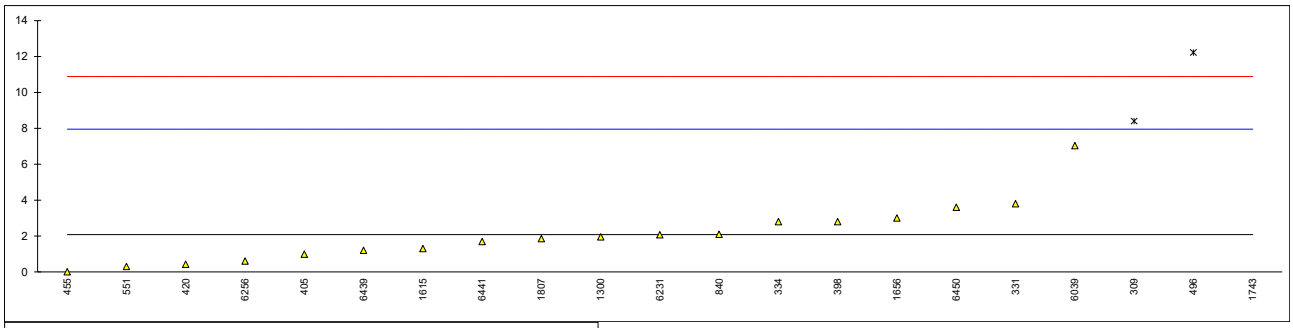


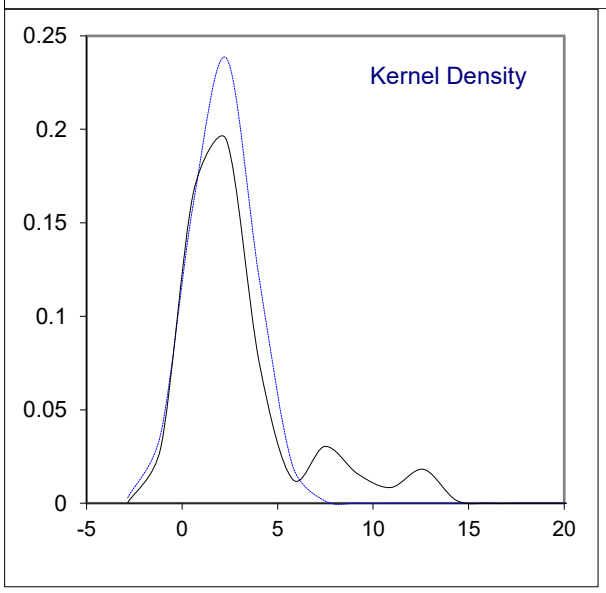


Determination of Insoluble matter on sample #22082; result in mg/kg

lab	method	value	mark	z(targ)	remarks
273	ISO22241-2 Annex G	<1		-----	
309	ISO22241-2 Annex G	8.4	G(0.05)	2.15	
331	ISO22241-2 Annex G	3.8		0.58	
334	ISO22241-2 Annex G	2.8		0.24	
398	ISO22241-2 Annex G	2.8		0.24	
405	ISO22241-2 Annex G	0.99		-0.37	
420	ISO22241-2 Annex G	0.43		-0.56	
455	ISO22241-2 Annex G	0.01000		-0.71	
496	ISO22241-2 Annex G	12.222	G(0.01)	3.45	
541	ISO22241-2 Annex G	<1.0		-----	
551	ISO22241-2 Annex G	0.3		-0.61	
840	ISO22241-2 Annex G	2.10		0.01	
862		-----		-----	
863		-----		-----	
864		-----		-----	
1300	ISO22241-2 Annex G	1.951		-0.05	
1615	ISO22241-2 Annex G	1.30		-0.27	
1656	ISO22241-2 Annex G	3		0.31	
1743	ISO22241-2 Annex G	433	C,G(0.01)	146.78	first reported 337
1807	ISO22241-2 Annex G	1.85		-0.08	
6039	ISO22241-2 Annex G	7.0333		1.69	
6139		-----		-----	
6146		-----		-----	
6231	ISO22241-2 Annex G	2.06		-0.01	
6256	ISO22241-2	0.606		-0.50	
6439	ISO22241-2 Annex G	1.2	C	-0.30	first reported 12
6441	ISO22241-2 Annex G	1.69	C	-0.13	first reported 16.9
6450	ISO22241-2 Annex G	3.6		0.52	

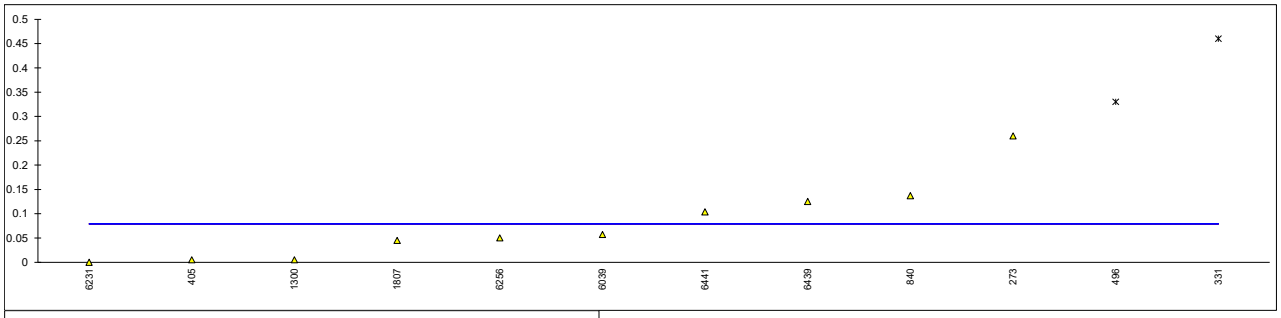
normality	not OK
n	18
outliers	3
mean (n)	2.0845
st.dev. (n)	1.65971
R(calc.)	4.6472
st.dev.(ISO22241-2:19)	2.93571
R(ISO22241-2:19)	8.220

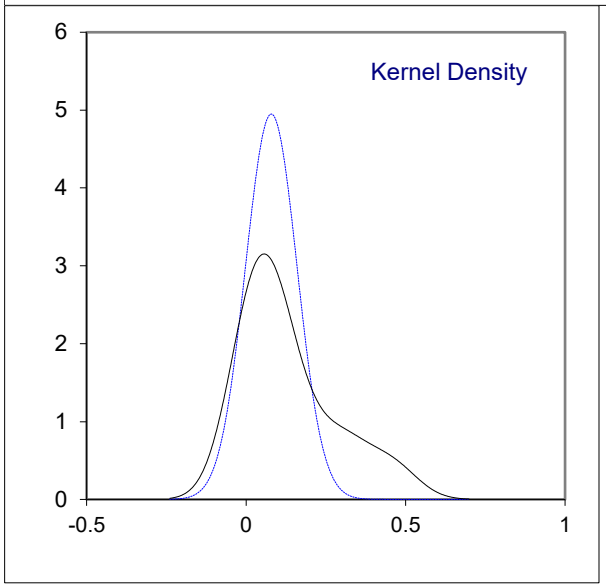




Determination of Phosphate as PO_4 on sample #22082; result in mg/kg

lab	method	value	mark	z(targ)	remarks
273	ISO22241-2 Annex H	0.26		-----	
309	ISO22241-2 Annex H	<0.25		-----	
331	ISO22241-2 Annex H	0.46	DG(0.05)	-----	
334	ISO22241-2 Annex H	<0.05		-----	
398	ISO22241-2 Annex H	<0,05		-----	
405	ISO22241-2 Annex H	0.005		-----	
420	ISO22241-2 Annex H	<0,05		-----	
455		-----		-----	
496	ISO22241-2 Annex H	0.33	DG(0.05)	-----	
541	ISO22241-2 Annex H	<0.05		-----	
551		-----		-----	
840	ISO22241-2 Annex H	0.137		-----	
862		-----		-----	
863		-----		-----	
864		-----		-----	
1300	ISO22241-2 Annex H	0.005		-----	
1615	ISO22241-2 Annex H	<0.05		-----	
1656		-----		-----	
1743		-----		-----	
1807	In house	0.045		-----	
6039	ISO22241-2 Annex H	0.057		-----	
6139		-----		-----	
6146		-----		-----	
6231	ISO22421-2 Annex I	0		-----	
6256	ISO22241-2	0.05		-----	
6439	ISO22241-2 Annex H	0.125	C	-----	first reported 1.25
6441	ISO22241-2 Annex H	0.1037		-----	
6450	ISO22241-2 Annex H	ND		-----	
	normality	not OK			
	n	10			
	outliers	2			
	mean (n)	0.077			
	st.dev. (n)	0.0806			
	R(calc.)	0.226			
	st.dev.(ISO22241-2:19)	(0.0268)			
	R(ISO22241-2:19)	(0.075)			Application range: 0.22 – 1.007 mg/kg

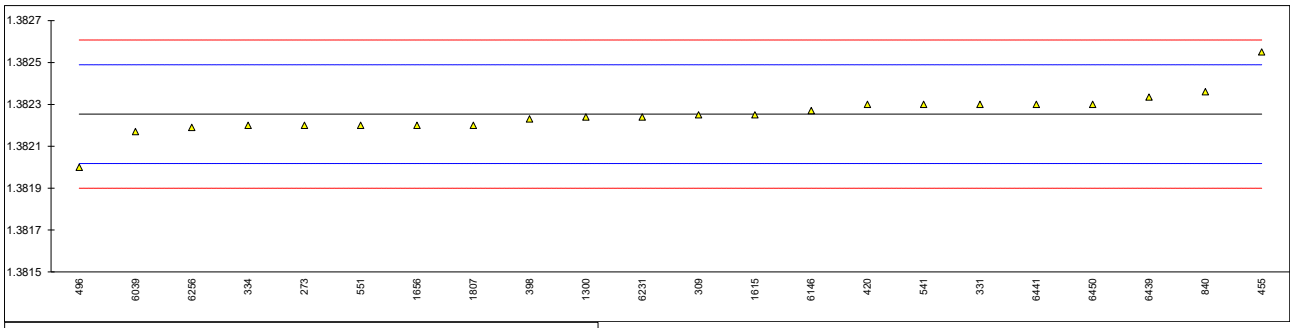


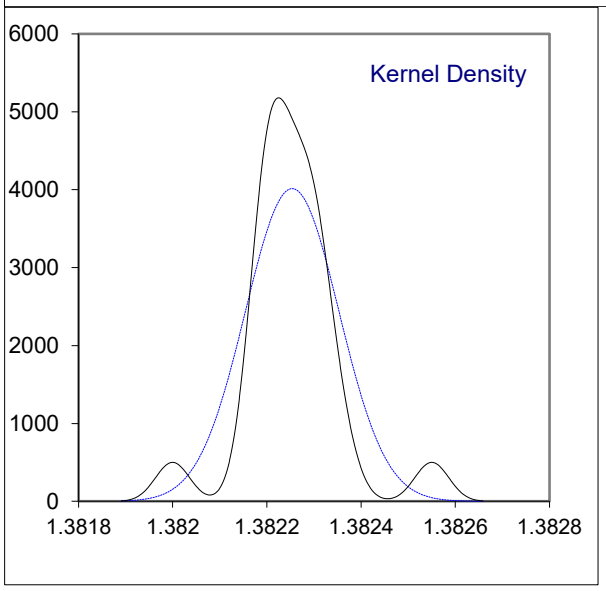


Determination of Refractive index at 20 °C on sample #22082;

lab	method	value	mark	z(targ)	remarks
273	ISO22241-2 Annex C	1.3822	C	-0.46	first reported 1.3816
309	ISO22241-2 Annex C	1.38225		-0.03	
331	ISO22241-2 Annex C	1.3823		0.39	
334	ISO22241-2 Annex C	1.3822		-0.46	
398	ISO22241-2 Annex C	1.38223		-0.20	
405		-----		-----	
420	ISO22241-2 Annex C	1.3823	C	0.39	first reported 1.3825
455	ISO22241-2 Annex C	1.38255		2.51	
496	ISO22241-2 Annex C	1.38200		-2.15	
541	ISO22241-2 Annex C	1.3823		0.39	
551	ISO22241-2 Annex C	1.3822		-0.46	
840	ISO22241-2 Annex C	1.38236		0.90	
862		-----		-----	
863		-----		-----	
864		-----		-----	
1300	ISO22241-2 Annex C	1.38224		-0.12	
1615	ISO22241-2 Annex C	1.38225		-0.03	
1656	ISO22241-2 Annex C	1.3822		-0.46	
1743		-----		-----	
1807	ISO22241-2 Annex C	1.38220		-0.46	
6039	ISO22241-2 Annex C	1.38217		-0.71	
6139		-----		-----	
6146		1.38227		0.14	
6231	ISO22241-2 Annex C	1.38224		-0.12	
6256	ISO22241-2	1.38219		-0.54	
6439	ISO22241-2 Annex C	1.382335		0.69	
6441	ISO22241-2 Annex C	1.3823		0.39	
6450	ISO22241-2 Annex C	1.3823		0.39	

normality	not OK
n	22
outliers	0
mean (n)	1.382254
st.dev. (n)	0.0000994
R(calc.)	0.000278
st.dev.(ISO22241-2:19)	0.0001179
R(ISO22241-2:19)	0.00033

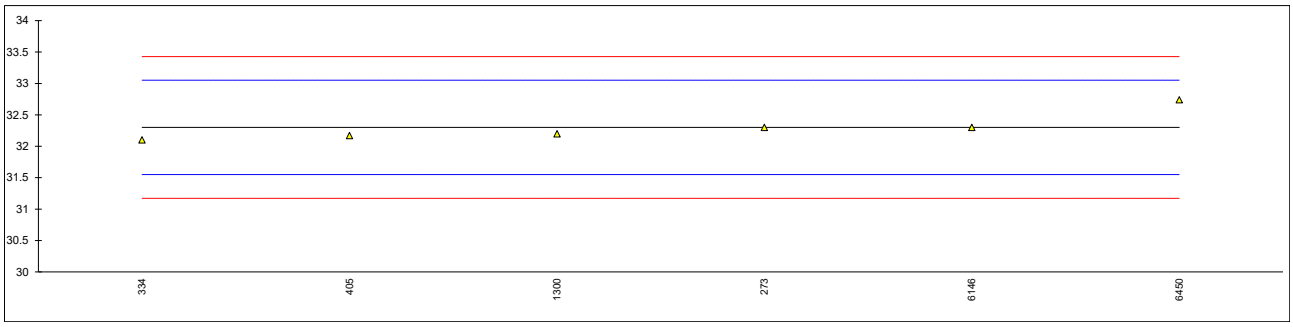




Determination of Urea content (by total Nitrogen) on sample #22082; result in %M/M

lab	method	value	mark	z(targ)	remarks
273	ISO22241-2 Annex B	32.3		0.00	
309		-----		-----	
331		-----		-----	
334	ISO22241-2 Annex B	32.1		-0.53	
398		-----		-----	
405	ISO22241-2 Annex B	32.17		-0.35	
420		-----		-----	
455		-----		-----	
496		-----		-----	
541		-----		-----	
551		-----		-----	
840		-----		-----	
862		-----		-----	
863		-----		-----	
864		-----		-----	
1300	ISO22241-2 Annex B	32.197		-0.28	
1615		-----		-----	
1656		-----		-----	
1743		-----		-----	
1807		-----		-----	
6039		-----		-----	
6139		-----		-----	
6146		32.3		0.00	
6231		-----		-----	
6256		-----		-----	
6439		-----		-----	
6441		-----		-----	
6450	ISO22241-2 Annex B	32.74		1.17	

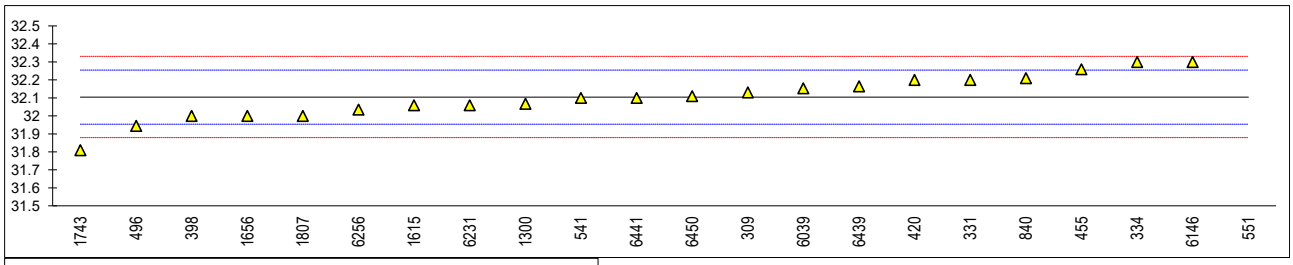
normality	unknown
n	6
outliers	0
mean (n)	32.301
st.dev. (n)	0.2285
R(calc.)	0.640
st.dev.(ISO22241-2:19)	0.3761
R(ISO22241-2:19)	1.053

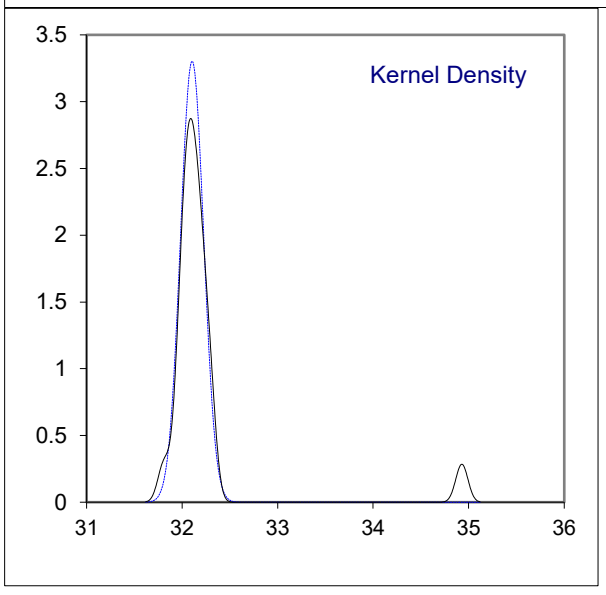


Determination of Urea content (by refractive index) on sample #22082; result in %M/M

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
309	ISO22241-2 Annex C	32.132		0.35	
331	ISO22241-2 Annex C	32.2		1.26	
334	ISO22241-2 Annex C	32.3		2.58	
398	ISO22241-2 Annex C	32.0		-1.40	
405		-----		-----	
420	ISO22241-2 Annex C	32.2		1.26	
455	ISO22241-2 Annex C	32.26000		2.05	
496	ISO22241-2 Annex C	31.946		-2.11	
541	ISO22241-2 Annex C	32.1		-0.07	
551	ISO22241-2 Annex C	34.93	G(0.01)	37.48	
840	ISO22241-2 Annex C	32.21		1.39	
862		-----		-----	
863		-----		-----	
864		-----		-----	
1300	ISO22241-2 Annex C	32.068		-0.49	
1615	ISO22241-2 Annex C	32.06		-0.60	
1656	ISO22241-2 Annex C	32.0		-1.40	
1743	ISO22241-2 Annex C	31.81		-3.92	
1807	ISO22241-2 Annex C	32.0		-1.40	
6039	ISO22241-2 Annex C	32.154		0.65	
6139		-----		-----	
6146		32.3		2.58	
6231	ISO22241-2 Annex C	32.06		-0.60	
6256	ISO22241-2	32.0353		-0.93	
6439	ISO22241-2 Annex C	32.165		0.79	
6441	ISO22241-2 Annex C	32.1		-0.07	
6450	ISO22241-2 Annex C	32.11		0.06	

normality	OK
n	21
outliers	1
mean (n)	32.105
st.dev. (n)	0.1207
R(calc.)	0.338
st.dev.(ISO22241-2:19)	0.0754
R(ISO22241-2:19)	0.211

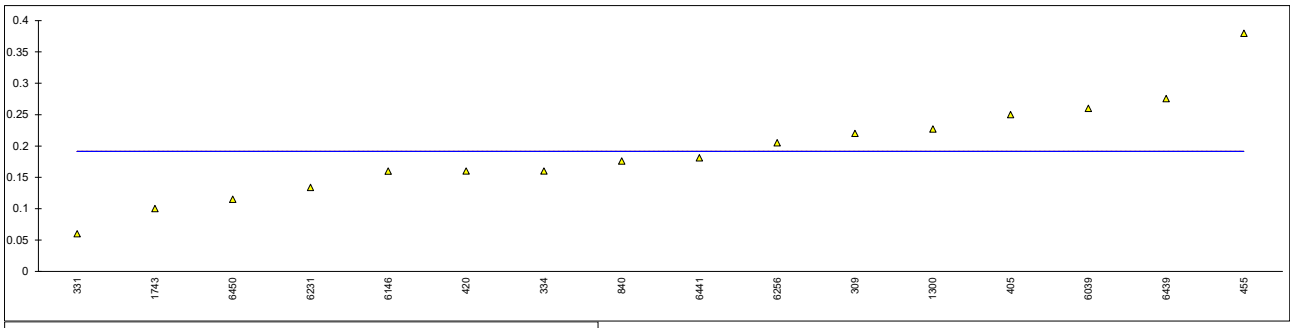


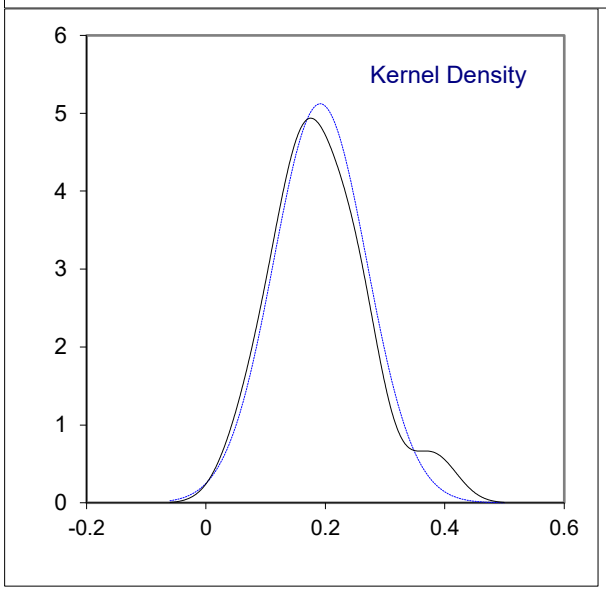


Determination of Sodium as Na on sample #22082; result in mg/kg

lab	method	value	mark	z(targ)	remarks
273	ISO22241-2 Annex I	<0.1		-----	
309	ISO22241-2 Annex I	0.22		-----	
331	ISO22241-2 Annex I	0.06		-----	
334	ISO22241-2 Annex I	0.16		-----	
398		-----		-----	
405	ISO22241-2 Annex I	0.25	C	-----	first reported 0.42
420	ISO22241-2 Annex I	0.16		-----	
455	ISO22241-2 Annex I	0.37968		-----	
496		-----		-----	
541		-----		-----	
551	ISO22241-2 Annex I	<0.10		-----	
840	ISO22241-2 Annex I	0.176		-----	
862		-----		-----	
863		-----		-----	
864		-----		-----	
1300	ISO22241-2 Annex I	0.227		-----	
1615	ISO22241-2 Annex I	<0.05		-----	
1656	ISO22241-2 Annex I	<0.5		-----	
1743	ISO22241-2 Annex I	0.1		-----	
1807		-----		-----	
6039	ISO22241-2 Annex I	0.260		-----	
6139		-----		-----	
6146		0.1597		-----	
6231	ISO22241-2 Annex I	0.134		-----	
6256	ISO22241-2	0.205		-----	
6439	ISO22241-2 Annex I	0.27555		-----	
6441	ISO22241-2 Annex I	0.181		-----	
6450	ISO22241-2 Annex I	0.115		-----	

normality	suspect
n	16
outliers	0
mean (n)	0.191
st.dev. (n)	0.0779
R(calc.)	0.218
st.dev.(ISO22241-2:19)	(0.0107)
R(ISO22241-2:19)	(0.03)





APPENDIX 2

Summary of other reported elements on sample #22082; result in mg/kg

lab	method	Al	Ca	Cr	Cu	Fe	Mg	Ni	K	Zn
273	ISO22241-2 Annex I	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
309	ISO22241-2 Annex I	<0.2	0.12	0.02	<0.2	<0.5	<0.5	<0.2	<0.5	0.03
331	ISO22241-2 Annex I	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00
334	ISO22241-2 Annex I	0.02	0.06	0.01	<0.01	<0.01	***0.04***	0.02	0.11	0.02
398		-----	-----	-----	-----	-----	-----	-----	-----	-----
405	ISO22241-2 Annex I	0.018	0.20	0.018	0.010	***0.19***	0.030	0.0088	0.059	0.041
420	ISO22241-2 Annex I	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03
455	ISO22241-2 Annex I	0	0.90512	0	0	0	0.03774	0	0.11243	0
496		-----	-----	-----	-----	-----	-----	-----	-----	-----
541		-----	-----	-----	-----	-----	-----	-----	-----	-----
551	ISO22241-2 Annex I	<0.10	0.14	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
840	ISO22241-2 Annex I	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
862		-----	-----	-----	-----	-----	-----	-----	-----	-----
863		-----	-----	-----	-----	-----	-----	-----	-----	-----
864		-----	-----	-----	-----	-----	-----	-----	-----	-----
1300	ISO22241-2 Annex I	<0.1	<0.2	<0.2	<0.2	<0.1	<0.4	<0.2	<0.2	<0.2
1615	ISO22241-2 Annex I	0.07	0.16	***<0.05***	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
1656	ISO22241-2 Annex I	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
1743	ISO22241-2 Annex I	0	0	0	0	0	0	0	0	0
1807		-----	-----	-----	-----	-----	-----	-----	-----	-----
6039	ISO22241-2 Annex I	0.028	0.150	0.017	0.003	0.004	0.014	0.010	0.150	0.033
6139		-----	-----	-----	-----	-----	-----	-----	-----	-----
6146		0	0.06774	0.01598	0	0	0	0.00556	0.04804	0.00785
6231	ISO22241-2 Annex I	<0,05	0.075	0.011	<0,05	<0,05	<0,05	<0,05	0.034	0.0295
6256	ISO22241-2	0.066	0.120	0.018	0.002	0.009	0.063	0.016	-0.0137	0.031
6439	ISO22241-2 Annex I	0.0014	0.2561	***0.02603***	0.00915	0.04055	0.0376	0.009	0.1258	0.037
6441	ISO22241-2 Annex I	0.035	0.116	0.016	0.017	0.003	0.011	0.006	0.054	0.019
6450	ISO22241-2 Annex I	ND	0.011	0.0117	ND	ND	ND	ND	ND	0.0280

Results bold, Italic and underline in the table first reported as test result:
lab 334 first reported Mg 0.33
lab 405 first reported Fe 0.15
lab 1615 first reported Cr 0.15
lab 6439 first reported Cr 0.0412

APPENDIX 3

Number of participants per country

1 lab in ARGENTINA
1 lab in BRAZIL
1 lab in BULGARIA
4 labs in CHINA, People's Republic
1 lab in CZECH REPUBLIC
2 labs in ESTONIA
3 labs in FRANCE
3 labs in GERMANY
1 lab in HUNGARY
1 lab in INDIA
2 labs in ITALY
1 lab in MALAYSIA
1 lab in NETHERLANDS
1 lab in ROMANIA
1 lab in SOUTH AFRICA
1 lab in SPAIN
2 labs in UNITED KINGDOM
1 lab in VIETNAM

APPENDIX 4

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
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)