Results of Proficiency Test
Ad Blue, 32% Urea Solution
June 2017

Organised by: **Institute for Interlaboratory Studies (iis)**
Spijkenisse, the Netherlands

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CONTENTS

Appendices:

1   INTRODUCTION

On request of several participants, the Institute for Interlaboratory Studies decided to organize a new proficiency test for the analysis of AdBlue (32% Urea Solution) in accordance with the latest applicable version of ISO22241 part 1, during the annual proficiency testing program 2016/2017.

In this interlaboratory study 14 laboratories in 9 different countries registered for participation. See appendix 2 for the number of participants per country.

In this report, the results of the 2017 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 organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send an 1 litre HDPE bottle filled with AdBlue (labelled #17092).

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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3   CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The necessary sample material of approximately 30 litres of AdBlue (32%Urea solution) was purchased from at local supplier. After homogenisation in a pre-cleaned drum, 28 wide-neck HDPE bottles of 1 litre (labelled #17092) were filled. The homogeneity of the subsamples #17092 was checked by determination of the Density at 20°C in accordance with ASTM D4052 on 4 stratified randomly selected samples.

	Density at 20°C in kg/m^3
sample #17092-1	1.08900
sample #17092-2	1.08900
sample #17092-3	1.08901
sample #17092-4	1.08901

Table 1: homogeneity test results of the subsamples #17092

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

	Density at 20°C in kg/m^3
r (observed)	0.00001
reference test method	ISO12185:96
0.3 x R(reference test method)	0.00015

Table 2: evaluation of repeatability of the subsamples #17092

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the target method. Therefore, homogeneity of the subsamples #17092 was assumed.

To each of the participating laboratories 1 * 1 litre wide-neck HDPE bottle, labelled #17092 was sent on May 17, 2017. 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 ANALYSES

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 (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 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 calculations.

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 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 reanalysis). 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4).
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 ISO 5725 the original test results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying these with a factor of 2.8.

3.2   GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.
Furthermore, Kernel Density Graphs were made. The Kernel Density Graph 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. ISO reproducibilities, 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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

The z-scores were calculated according to:

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

The $z_{(target)}$ scores are listed in the 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:

$$|z| < 1 \quad \text{good}$$
$$1 < |z| < 2 \quad \text{satisfactory}$$
$$2 < |z| < 3 \quad \text{questionable}$$
$$3 < |z| \quad \text{unsatisfactory}$$

4   EVALUATION

In this proficiency test, no problems were encountered. One laboratory reported the test results after the final reporting date. Not all laboratories were able to perform all analyses requested. Finally, 14 laboratories did report 196 numerical test results. Observed were 4 outlying test results, which is 2.0%. In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1   EVALUATION PER 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 methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

Aldehyde as Formaldehyde: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ISO22241-2 Annex K:06. One false negative test result was reported.

Alkalinity as NH_3: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ISO22241-2 Annex K:06.

Biuret: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ISO22241-2 Annex K:06.

Density at 20°C: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ISO22241-2 Annex K:06.

Insoluble Matter: This determination was problematic. Two statistical outliers were observed. The reported test results vary over a wide range, from 0.4 mg/kg up to 16.6 mg/kg. The reported 0.0014 mg/kg contains probably a unit error. As all reported test results are within the specification limit of 20 mg/kg (ISO22241-1:06, table 1), it is decided not to calculate any z-scores.

Phosphate as PO_4: 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 Annex K:06.

Refractive Index at 20°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ISO22241-2 Annex K:06.

Urea (total Nitrogen): Regretfully, only two laboratories reported a test result. To evaluate both test results the mean of the urea content (by refractive index) was used to calculate z-scores. Laboratory 541 reported a test result which would be classified as 'off spec'- in accordance with ISO22241-1:06 table 1 (limits 31.8 – 33.2 %M/M).

Urea (refractive Index): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ISO22241-2 Annex K:06.

Trace elements: Requested was to determine Aluminium, Calcium, Chromium, Copper, Iron, Magnesium, Nickel, Potassium, Sodium and Zinc. None of the requested elements was found positive. Therefore, no significant conclusions were drawn.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The assigned values, calculated reproducibilities and target reproducibilities, derived from literature reference test methods (in casu ISO, EN reference test methods) are compared in the next table.

Parameters	Unit	n	average	2.8 * sd	R (lit)
Aldehyde as Formaldehyde	mg/kg	13	1.7	1.5	0.9
Alkalinity as NH_3	%M/M	12	0.036	0.011	0.007
Biuret	%M/M	14	0.17	0.05	0.04
Density at 20°C	kg/L	13	1.0891	0.0002	0.0005
Insoluble Matter	mg/kg	11	1.72	3.15	(0.65)
Phosphate as PO_4	mg/kg	6	0.014	0.043	0.03
Refractive index at 20°C	--	14	1.3824	0.0006	0.0010
Urea content (total Nitrogen)	%M/M	2	n.a.	n.a.	n.a.
Urea content (by refractive index)	%M/M	13	32.25	0.52	1.0
Trace elements	mg/kg	n.a.	n.e.	n.e.	n.e.

Table 3: summary of test results samples #17092
Result between brackets has to be evaluate with due care

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

4.3   EVALUATION OF THE INTERLABORATORY STUDY OF JUNE 2017

	June 2017
Number of reporting labs	14
Number of results reported	196
Statistical outliers	4
Percentage outliers	2.0%

Table 4:summary of statistics

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 standards. The conclusions are given the following table:

	June 2017
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 (total Nitrogen)	n.e.
Urea content (by refractive	++
Trace elements	n.e.

Table 5: comparison determinations against the standard
Result between brackets has to be evaluate with due care

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

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

APPENDIX 1

Determination of Aldehyde as Formaldehyde on sample #17092; result in mg/kg

lab	method	value	mark	z(targ)	remarks
311	ISO22241-2 Annex F	1.8		0.20	
420	ISO22241-2 Annex F	2.48		2.39	
444	ISO22241-2 Annex F	1.24		-1.60	
496	ISO22241-2 Annex F	1.9		0.52	
541	ISO22241-2 Annex F	1.100		-2.05	
551	ISO22241-2 Annex F	<0.50		<3.99	False negative test result?
1276	ISO22241-2 Annex F	1.23		-1.64	
1300	ISO22241-2 Annex F	2.19		1.46	
1367	In house	2.5	C	2.46	First reported 4.8
1656	ISO22241-2 Annex F	1.47		-0.86	
1807	ISO22241-2 Annex F	0.90		-2.70	
1938	ISO22241-2 Annex F	1.47		-0.86	
6039	ISO22241-2 Annex F	2.067		1.06	
6079	ISO22241-2 Annex F	2.24		1.62	

normality	OK	
n	13	
outliers	0	
mean (n)	1.737	
st.dev. (n)	0.5389	
R(calc.)	1.509	
R(ISO22241-2 annex K:06)	0.869	

Kernel Density
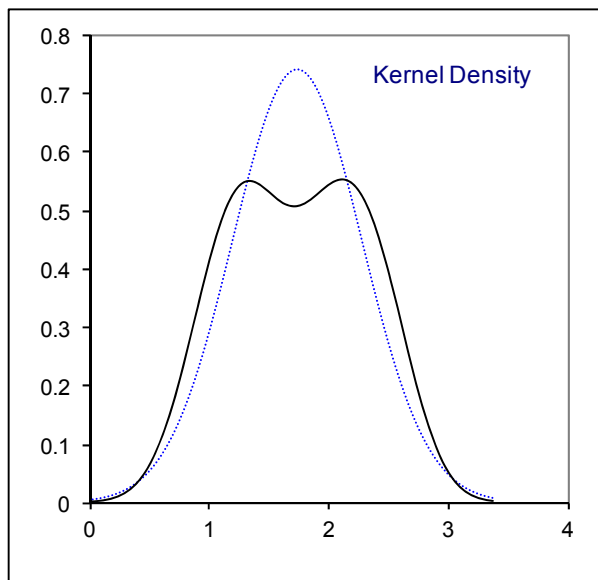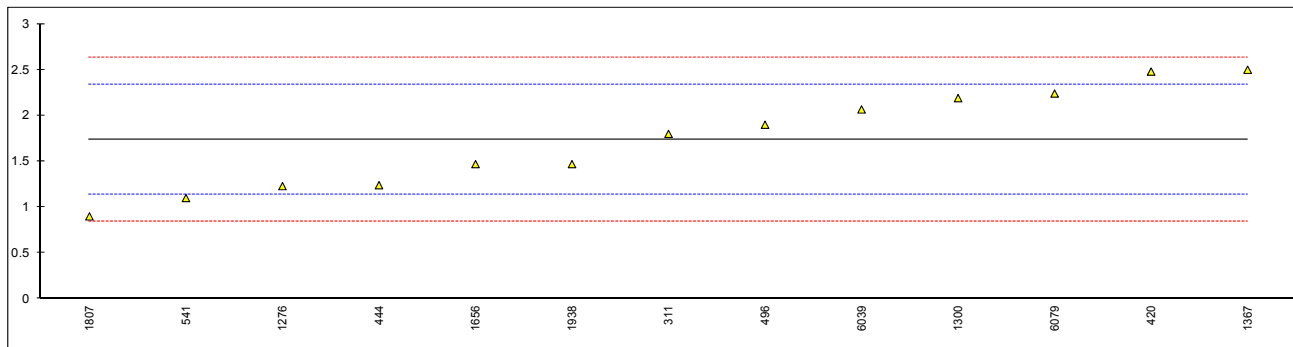
Determination of Alkalinity as NH_3 on sample #17092; result in %M/M

lab	method	value	mark	z(targ)	remarks
311	ISO22241-2 Annex D	0.04		1.65	
420	ISO22241-2 Annex D	0.036		0.08	
444	ISO22241-2 Annex D	0.036		0.08	
496	ISO22241-2 Annex D	0.033		-1.09	
541	ISO22241-2 Annex D	<0.10		-----	
551	ISO22241-2 Annex D	<0.10		-----	
1276	ISO22241-2 Annex D	0.039		1.26	
1300	ISO22241-2 Annex D	0.0385		1.06	
1367	ISO22241-2 Annex D	0.03		-1.68	
1656	ISO22241-2 Annex D	0.03		-2.26	
1807	ISO22241-2 Annex D	0.03		-2.26	
1938	ISO22241-2 Annex D	0.038		0.87	
6039	ISO22241-2 Annex D	0.0374		0.63	
6079	ISO22241-2 Annex D	0.04		1.65	

normality	OK	
n	12	
outliers	0	
mean (n)	0.0358	
st.dev. (n)	0.00374	
R(calc.)	0.0105	
R(ISO22241-2 annex K:06)	0.0072	

Kernel Density
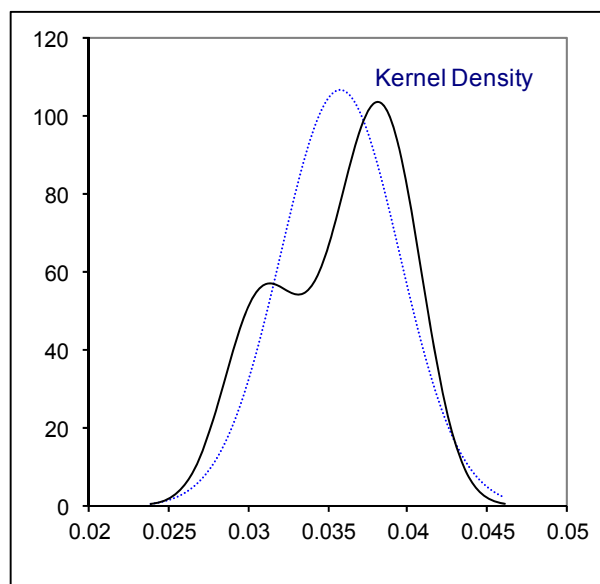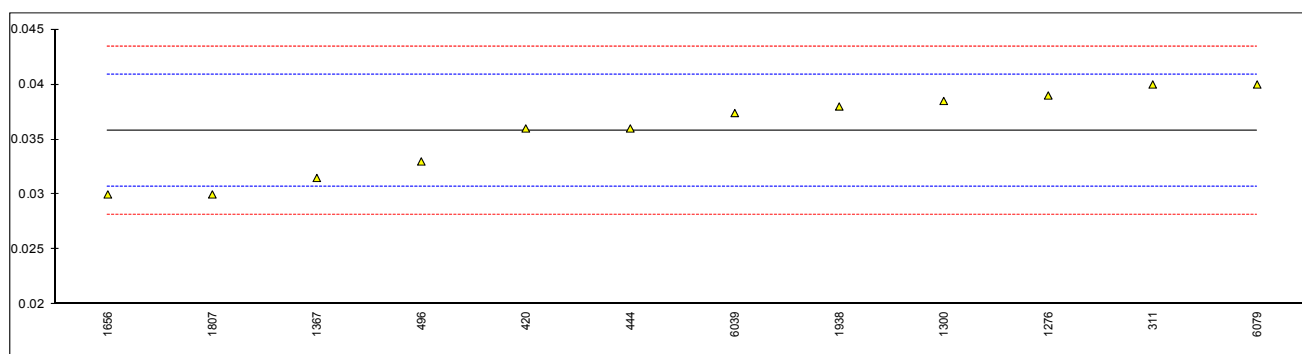
Determination of Biuret on sample #17092; result in %M/M

lab	method	value	mark	z(targ)	remarks
311	ISO22241-2 Annex E	0.18		0.60	
420	ISO22241-2 Annex E	0.16		-0.80	
444	ISO22241-2 Annex E	0.179		0.53	
496	ISO22241-2 Annex E	0.15		-1.50	
541	ISO22241-2 Annex E	0.17		-0.10	
551	ISO22241-2 Annex E	0.2		2.00	
1276	ISO22241-2 Annex E	0.17		-0.10	
1300	ISO22241-2 Annex E	0.1879		1.15	
1367	ISO22241-2 Annex E	0.2		2.00	
1656	ISO22241-2 Annex E	0.14		-2.20	
1807	ISO22241-2 Annex E	0.17		-0.10	
1938	ISO22241-2 Annex E	0.15		-1.50	
6039	ISO22241-2 Annex E	0.16		-0.80	
6079	ISO22241-2 Annex E	0.183		0.81	

normality	OK
n	14
outliers	0
mean (n)	0.1714
st.dev. (n)	0.01828
R(calc.)	0.0512
R(ISO22241-2 annex K:06)	0.04
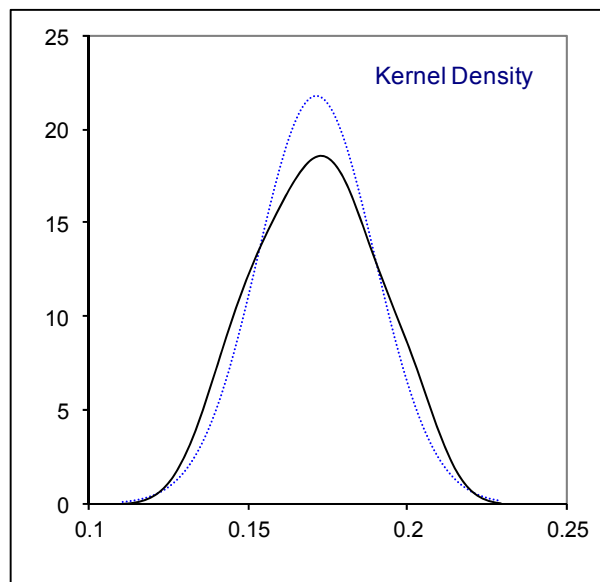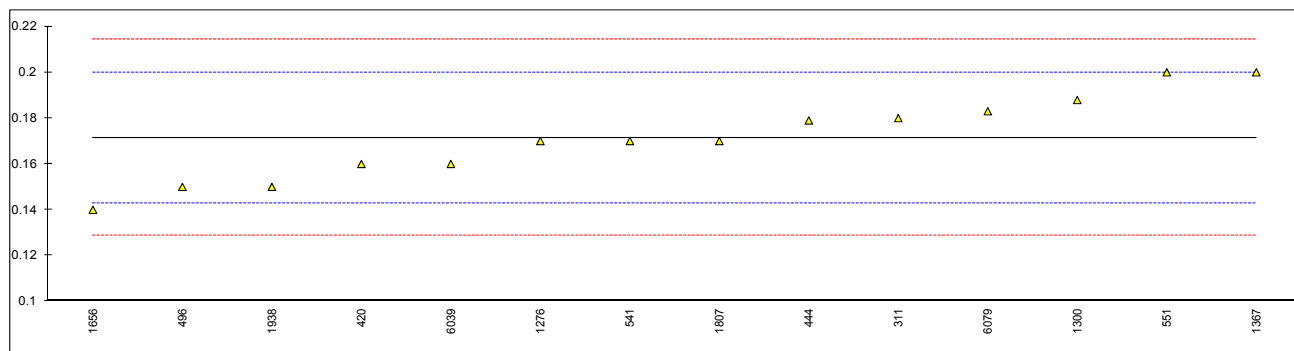
## Determination of Density at 20°C on sample #17092; result in kg/L

lab	method	value	mark	z(targ)	remarks
311	ISO12185	1.0890		-0.34	
420	ISO12185	1.0890		-0.34	
444	D4052	1.0890		-0.34	
496	ISO12185	1.08909	C	0.16	Reported 1089.09 kg/L
541	ISO12185	1.0891		0.22	
551	ISO12185	1.0891		0.22	
1276	ISO12185	1.08903	C	-0.17	Reported 1089.03 kg/L
1300	ISO12185	1.08897		-0.51	
1367	ISO12185	1.0891	C	0.22	First reported 1089.1
1656	D4052	1.0891		0.22	
1807	ISO12185	1.0894	G(0.05)	1.90	
1938	ISO12185	1.0891	C	0.22	First reported 1089.1
6039	ISO12185	1.08924		1.00	
6079	ISO12185	1.08896		-0.56	

normality	suspect
n	13
outliers	1
mean (n)	1.08906
st.dev. (n)	0.000077
R(calc.)	0.00022
R(ISO22241-2 annex K:06)	0.0005

Kernel Density
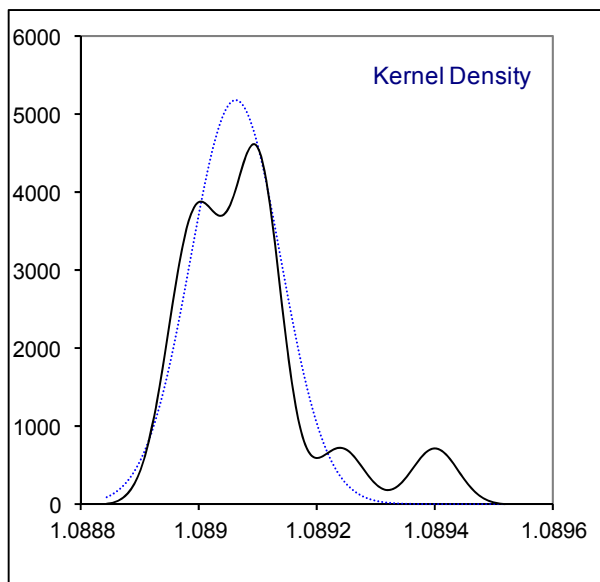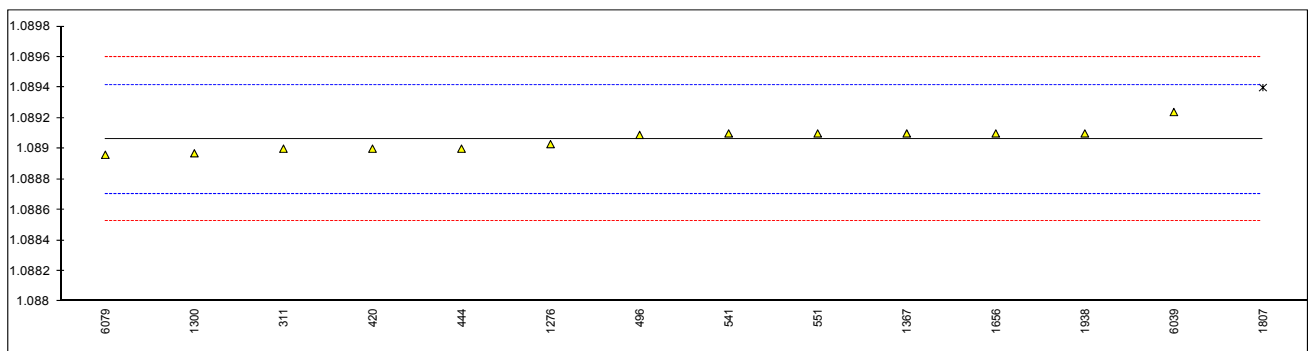
Determination of Insoluble Matter on sample #17092; result in mg/kg

lab	method	value	mark	z(targ)	remarks
311	ISO22241-2 Annex G	16.6	C,G(0.01)	-----	First reported 8.4
420	ISO22241-2 Annex G	0.0014	G(0.05)	-----	
444	ISO22241-2 Annex G	1.5		-----	
496	ISO22241-2 Annex G	1.954		-----	
541	ISO22241-2 Annex G	<1.0		-----	
551	ISO22241-2 Annex G	1.0		-----	
1276	ISO22241-2 Annex G	2.73		-----	
1300	ISO22241-2 Annex G	2.973		-----	
1367	ISO22241-2 Annex G	0.870		-----	
1656	ISO22241-2 Annex G	4.0		-----	
1807	ISO22241-2 Annex G	1.8		-----	
1938	ISO22241-2 Annex G	0.5	C	-----	First reported 7.8
6039	ISO22241-2 Annex G	1.2019		-----	
6079	ISO22241-2 Annex G	0.4		-----	

normality	OK
n	11
outliers	2
mean (n)	1.7208
st.dev. (n)	1.12420
R(calc.)	3.1478
R(ISO22241-2 annex K:06)	(0.6539)
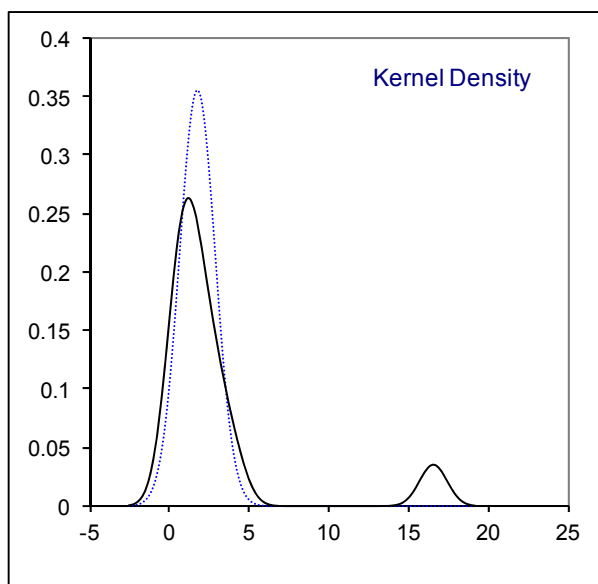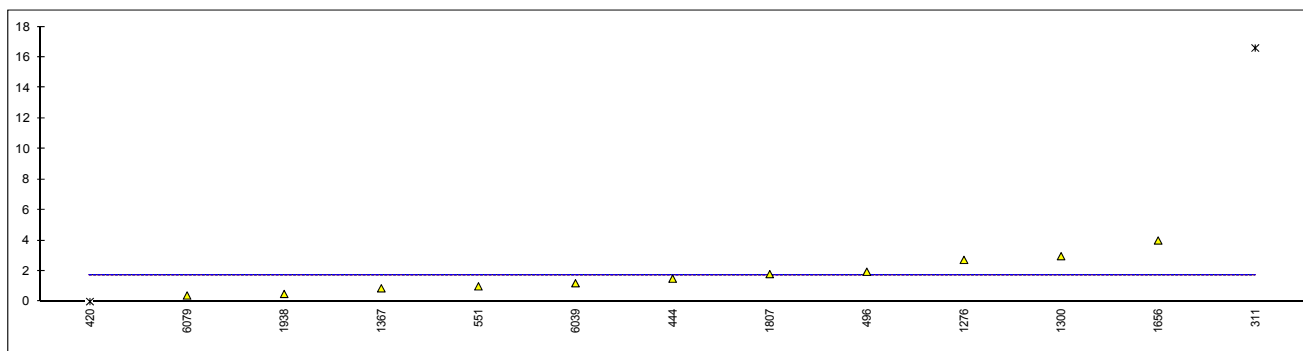
## Determination of Phosphate as PO_4 on sample #17092; result in mg/kg

lab	method	value	mark	z(targ)	remarks
311	ISO22241-2 Annex H	<0.05		-----	
420		-----		-----	
444		-----		-----	
496	ISO22241-2 Annex H	0.02		0.58	
541	ISO22241-2 Annex H	<0.05		-----	
551	ISO22241-2 Annex H	<0.05		-----	
1276	ISO22241-2 Annex H	0.0036		-0.96	
1300	ISO22241-2 Annex H	0.2869	C,G(0.01)	25.49	First reported 0.1819
1367		-----		-----	
1656	ISO22241-2 Annex H	0.04	C	2.44	First reported 0.12
1807		-----		-----	
1938	ISO22241-2 Annex H	0.0014		-1.16	
6039	ISO22241-2 Annex H	0.018		0.39	
6079	ISO22241-2 Annex H	0.000		-1.29	

normality	unknown	
n	6	
outliers	1	
mean (n)	0.0138	
st.dev. (n)	0.01543	
R(calc.)	0.0432	
R(ISO22241-2 annex K:06)	0.03	
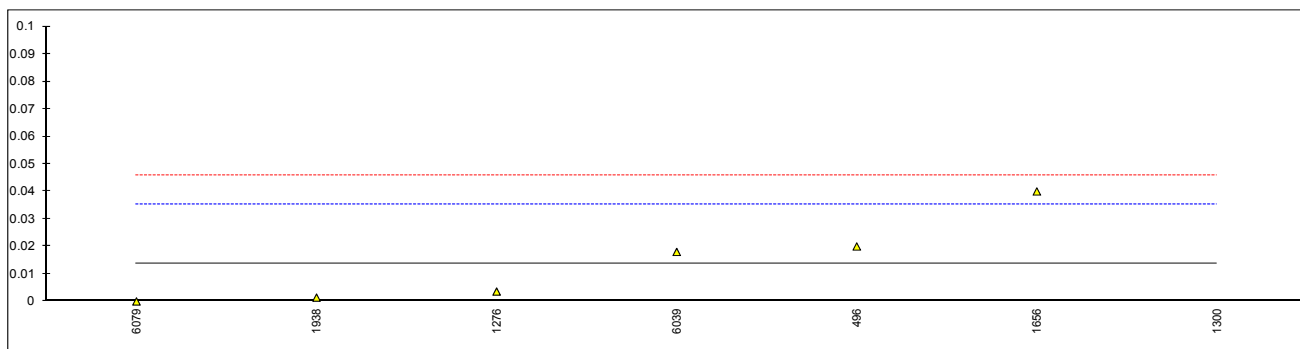
Determination of Refractive index at 20°C on sample #17092;

lab	method	value	mark	z(targ)	remarks
311	ISO22241-2 Annex C	1.38243		0.03	
420	ISO22241-2 Annex C	1.38246		0.12	
444	ISO22241-2 Annex C	1.3824		-0.05	
496	ISO22241-2 Annex C	1.38229		-0.36	
541	ISO22241-2 Annex C	1.38243		0.03	
551	ISO22241-2 Annex C	1.3824		-0.05	
1276	ISO22241-2 Annex C	1.3830		1.63	
1300	ISO22241-2 Annex C	1.3820		-1.17	
1367	ISO22241-2 Annex C	1.382465		0.13	
1656	ISO22241-2 Annex C	1.3825		0.23	
1807	ISO22241-2 Annex C	1.3821		-0.89	
1938	ISO22241-2 Annex C	1.3825		0.23	
6039	ISO22241-2 Annex C	1.38246		0.12	
6079	ISO22241-2 Annex C	1.382414		-0.01	

|	
normality	not OK
n	14
outliers	0
mean (n)	1.382418
st.dev. (n)	0.0002238
R(calc.)	0.000627
R(ISO22241-2 annex K:06)	0.0010
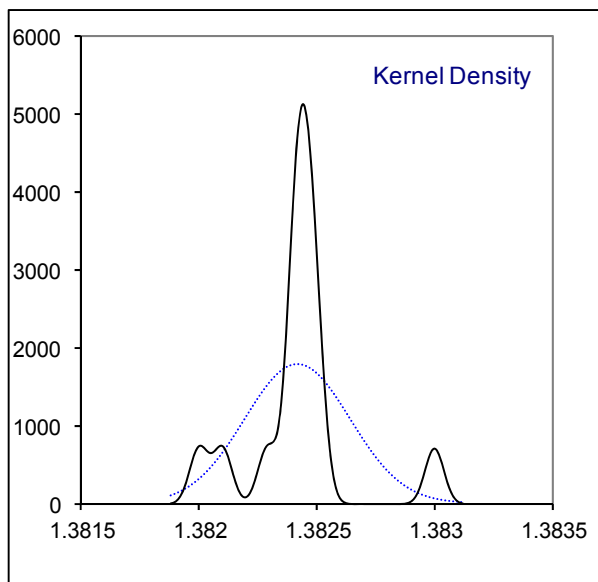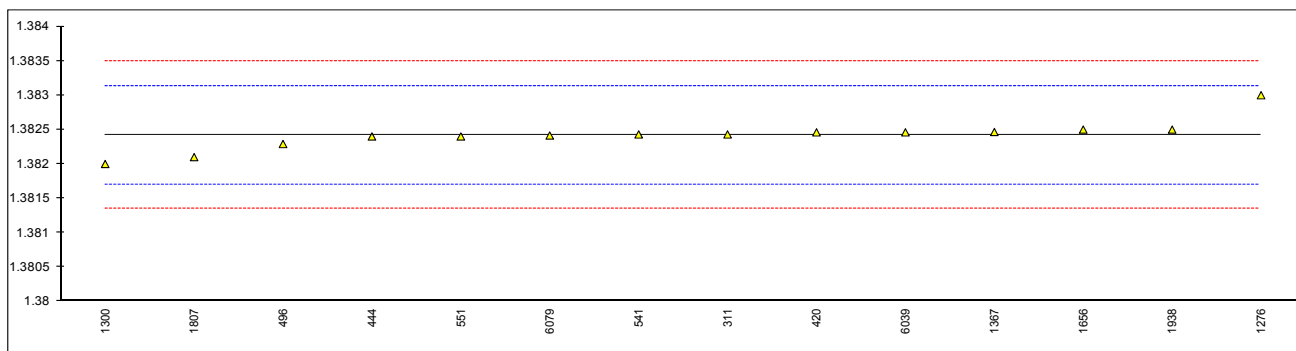
Determination of Urea content (total Nitrogen) on sample #17092; result in %M/M

lab	method	value	mark	z(targ)	remarks
311		-----		-----	
420		-----		-----	
444		-----		-----	
496		-----		-----	
541	ISO22241-2 Annex B	31.41		-2.35	
551		-----		-----	
1276		-----		-----	
1300	ISO22241-2 Annex B	32.47		0.61	
1367		-----		-----	
1656		-----		-----	
1807		-----		-----	
1938		-----		-----	
6039		-----		-----	
6079		-----		-----	

normality	unknown	
n	2	
outliers	n.a.	
mean (n)	32.251	Consensus value used from Urea by Refractive index
st.dev. (n)	n.a.	
R(calc.)	n.a.	
R(ISO22241-2 annex K:06)	1.0	

Determination of Urea content (by refractive index) on sample #17092; result in %M/M

lab	method	value	mark	z(targ)	remarks
311	ISO22241-2 Annex C	32.1		-0.42	
420	ISO22241-2 Annex C	32.2		-0.14	
444	ISO22241-2 Annex C	32.3		0.14	
496	ISO22241-2 Annex C	32.21		-0.11	
541		-----		-----	
551	ISO22241-2 Annex C	32.5		0.70	
1276	ISO22241-2 Annex C	32.52		0.75	
1300	ISO22241-2 Annex C	31.92		-0.93	
1367	ISO22241-2 Annex C	32.5		0.70	
1656	ISO22241-2 Annex C	32.3		0.14	
1807	ISO22241-2 Annex C	32.0		-0.70	
1938	ISO22241-2 Annex C	32.25		0.00	
6039	ISO22241-2 Annex C	32.32		0.19	
6079	ISO22241-2 Annex C	32.14		-0.31	

normality	OK	
n	13	
outliers	0	
mean (n)	32.251	
st.dev. (n)	0.1866	
R(calc.)	0.523	
R(ISO22241-2 annex K:06)	1.0	
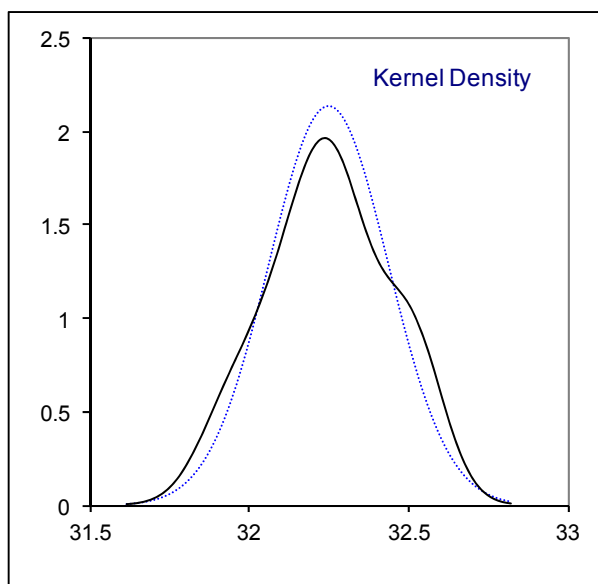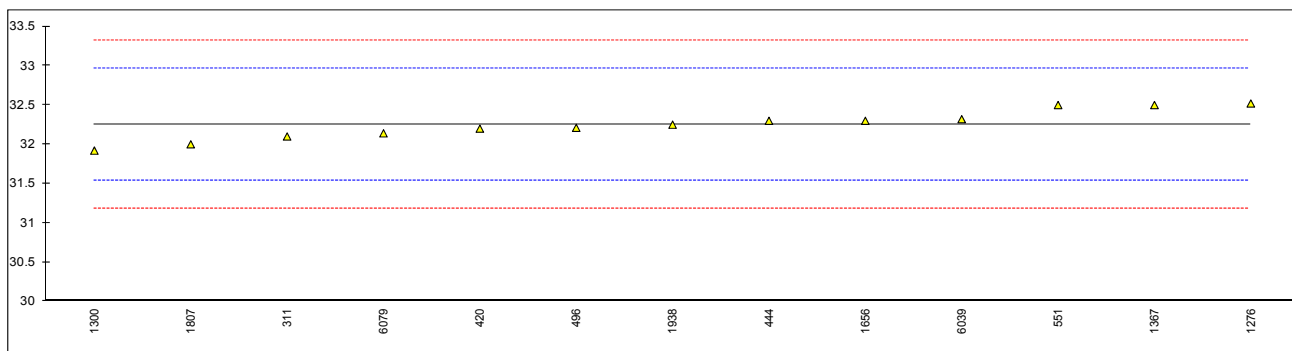
Determination of Trace Elements on sample #17092; result in mg/kg

lab	method	Al	Ca	Cr	Cu	Fe	Mg	Ni	K	Na	Zn
311	*)	<0.5	<0.5	<0.2	<0.2	<0.5	<0.5	<0.2	<0.5	<0.5	<0.2
420	*)	<0,05	<0,05	0.06	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
444		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
496	*)	0.000	0.071	0.001	0.000	0.001	0.005	0.000	0.000	0.034	0.031
541	*)	<0.5	<0.5	<0.2	<0.2	<0.5	<0.5	<0.2	<0.5	<0.5	<0.2
551	*)	0.00	0.24	0.08	0.00	0.18	0.06	0.00	0.00	0.00	0.31
1276	*)	0.056	0.002	0.014	0.093	0.067	0.041	0.013	0.117	0.150	0.002
1300	*)	0.0024	0.2461	0.0018	0.0132	0.0113	0.0442	0.0044	0.124	0.1643	0.071
1367	*)	0.01	0.4	0.07	0.02	0.03	0.03	0.01	0.02	0.0	0.17
1656	*)	<0.01	<0.01	0.05	<0.01	<0.01	<0.01	0.02	<0.01	<0.01	0.05
1807	*)	0.00	0.00	0.015	0.00	0.00	0.015	0.01	0.09	0.28	0.005
1938	*)	0.002	0.02	0.011	0.013	0.04	0.01	0.023	0.01	0.038	0.13
6039		0.025	0.031	0.086	0.035	0.003	0.020	0.027	0.010	0.100	0.113
6079		0.000	0.000	0.059	0.034	0.000	0.003	0.020	0.000	0.058	0.131

*) ISO22241-2 Annex I

APPENDIX 2

Number of participants per country

    1 lab in ARGENTINA
    1 lab in BRAZIL
    1 lab in CZECH REPUBLIC
    1 lab in ESTONIA
  3 labs in GERMANY
    1 lab in NETHERLANDS
    1 lab in SPAIN
  2 labs in TURKEY
  3 labs in UNITED KINGDOM

APPENDIX 3

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluations
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported test result
SDS	= Safety Data Sheet

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