Results of Proficiency Test AdBlue, 32% Urea Solution June 2018

Organised by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

Author: A. Lewinska, MSc.

Correctors: ing. A.S. Noordman-de Neef & ing. C.M. Nijssen-Wester

Report: iis18G04

CONTENTS

1	INTRODUCTION	3
2	SET UP	3
2.1	QUALITY SYSTEM	3
2.2	PROTOCOL	3
2.3	CONFIDENTIALITY STATEMENT	3
2.4	SAMPLES	4
2.5	STABILITY OF THE SAMPLES	4
2.6	ANALYSES	4
3	RESULTS	5
3.1	STATISTICS	5
3.2	GRAPHICS	6
3.3	Z-SCORES	6
4	EVALUATION	7
4.1	EVALUATION PER TEST	7
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	9
4.3	COMPARISON OF PROFICIENCY TEST OF JUNE 2018 WITH PREVIOUS PT	9

Appendices:

1.	Data, statistical results and graphic results	11
2.	Number of participants per country	21
2	Abbreviations and literature	20

1 Introduction

Last year the Institute for Interlaboratory Studies (iis) organizes a proficiency test (PT) for AdBlue (32% Urea Solution) in accordance with the latest applicable version of ISO22241 part 1. During the annual proficiency testing program 2017/2018 it was decided to continue the round robin for the analysis of AdBlue (32% Urea Solution).

In this interlaboratory study 14 laboratories in 10 different countries registered for participation. See appendix 2 for the number of participants per country.

In this report, the results of the 2018 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one HDPE bottle of 1L filled with AdBlue and labelled #18092.

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 a local supplier. After homogenisation in a pre-cleaned drum, 24 wide-neck HDPE bottles of 1 litre (labelled #18092) were filled. The homogeneity of the subsamples #18092 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/L
sample #18092-1	1.09115
sample #18092-2	1.09116
sample #18092-3	1.09118
sample #18092-4	1.09116

Table 1: homogeneity test results of subsamples #18092

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/L
r (observed)	0.00004
reference test method	ISO22241-2 Annex K:06
0.3 x R(reference test method)	0.00015

Table 2: evaluation of repeatability of the subsamples #18092

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the target method. Therefore, homogeneity of the subsamples #18092 was assumed.

To each of the participating laboratories one sample of 1L AdBlue (32% Urea solution), labelled #18092 was sent on May 16, 2018. 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 NH₃, Biuret, Density at 20°C, Insoluble matter, Phosphate as PO₄, Refractive Index at 20°C, Urea content (total Nitrogen and by Refractive Index) and Trace elements (AI, 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 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. 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 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_{\text{(target)}} = \text{(test result - average of PT)} / \text{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, no problems were encountered. Not all laboratories were able to perform all analyses requested. Finally, all 14 laboratories did report 146 numerical test results. Observed were 2 outlying test results, which is 1.4%. 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 not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO22241-2 Annex K:06.

Alkalinity as NH₃: 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.

<u>Biuret:</u> 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.

- <u>Density at 20°C</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ISO22241-2 Annex K:06.
- Insoluble Matter: This determination was problematic. One statistical outlier was observed. The reported test results vary over a wide range, from 0.41 mg/kg up to 2.7 mg/kg. As all reported test results except one (45.9 mg/kg) 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 PO₄: No problems have been observed, all except one participant agreed that the Phosphate as PO₄ is <0.052 mg/kg.
- 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 Annex K:06.
- <u>Urea (total Nitrogen)</u>: 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.
- <u>Urea (refractive Index)</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO22241-2 Annex K:06.
- <u>Trace elements</u>: Requested was to determine Aluminium, Calcium, Chromium, Copper, Iron, Magnesium, Nickel, Potassium, Sodium and Zinc. None of the requested elements was found positive. All participants had agreed on the absence of trace elements. 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 reference test method and the reproducibility as found for the group of participating laboratories that participated. The average results, calculated reproducibilities and reproducibilities derived from 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	11	1.0	0.5	0.5
Alkalinity as NH₃	%M/M	10	0.045	0.014	0.009
Biuret	%M/M	13	0.18	0.05	0.04
Density at 20°C	kg/L	14	1.0891	0.0005	0.0005
Insoluble Matter	mg/kg	9	1.90	2.25	(0.72)
Phosphate as PO ₄	mg/kg	9	<0.05	n.a.	n.a.
Refractive index at 20°C		13	1.3824	0.0003	0.0010
Urea content (total Nitrogen)	%M/M	2	32.2	n.a.	n.a.
Urea content (by refractive index)	%M/M	12	32.23	0.28	1.0
Trace elements	mg/kg	n.a.	n.e.	n.e.	n.e.

Table 3: reproducibilities of tests on sample #18092

Result between brackets has to be evaluate with due care.

Without further statistical calculations, it can be concluded that for some 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 Comparison of the Proficiency Test of June 2018 with previous PT

	June 2018	June 2017
Number of reporting labs	14	14
Number of results reported	146	196
Statistical outliers	2	4
Percentage outliers	1.4%	2.0%

Table 4: comparison with previous proficiency test

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 respective reference test methods. The conclusions are given in the following table:

	June 2018	June 2017
Aldehyde as Formaldehyde	+/-	
Alkalinity as NH₃	-	-
Biuret	-	-
Density at 20°C	+/-	++
Insoluble Matter	()	()
Phosphate as PO ₄	n.e.	-
Refractive index at 20°C	++	++
Urea content (total Nitrogen)	n.e.	n.e.
Urea content (by refractive index)	++	++
Trace elements	n.e.	n.e.

Table 5: comparison determinations against the respective reference test methods Result between brackets has to be evaluate with due care

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

++: group performed much better than the reference test method

+ : group performed better than the reference test method

+/-: group performance equals the reference test method

- : group performed worse than the reference test method

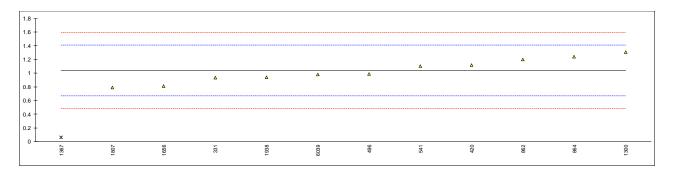
-- : group performed much worse than the reference test method

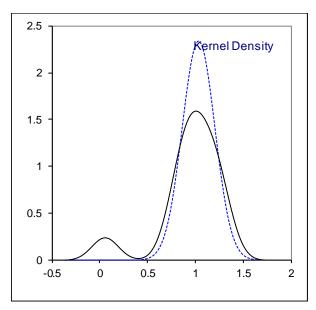
n.e.: not evaluated

APPENDIX 1

Determination of Aldehyde as Formaldehyde on sample #18092; result in mg/kg

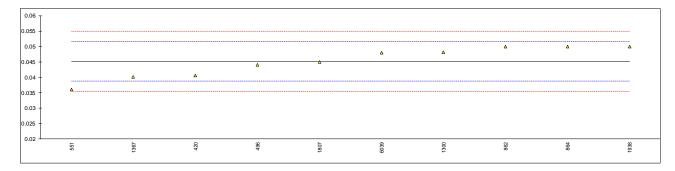
lab	method	value	mark	z(targ)	remarks
331	ISO22241-2 Annex F	0.93		-0.58	
420	ISO22241-2 Annex F	1.117		0.43	
444					
496	ISO22241-2 Annex F	0.99		-0.25	
541	ISO22241-2 Annex F	1.100		0.34	
551					
862	ISO22241-2 Annex F	1.20		0.88	
864	ISO22241-2 Annex F	1.24		1.10	
1300	ISO22241-2 Annex F	1.309		1.47	
1367	ISO22241-2 Annex F	0.06	C,G(0.01)	-5.28	First reported 0.09
1656	ISO22241-2 Annex F	0.81		-1.22	
1807	ISO22241-2 Annex F	0.79		-1.33	
1938	ISO22241-2 Annex F	0.94		-0.52	
6039	ISO22241-2 Annex F	0.978		-0.32	
	normality	OK			
	n	11			
	outliers	1			
	mean (n)	1.037			
	st.dev. (n)	0.1707			
	R(calc.)	0.478			
	st.dev.(ISO22241-2 annex K:06)	0.1851			
	R(ISO22241-2 annex K:06)	0.518			

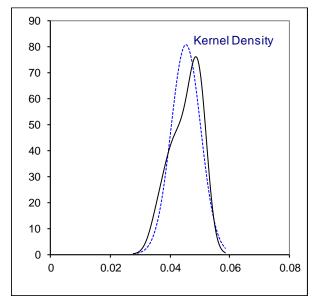




Determination of Alkalinity as NH₃ on sample #18092; result in %M/M

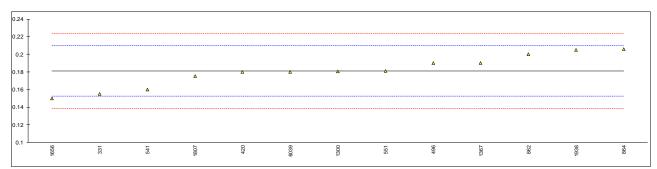
lab	method	value	mark	z(targ)	remarks
331	ISO22241-2 Annex D	<0.1			
420	ISO22241-2 Annex D	0.04055		-1.44	
444					
496	ISO22241-2 Annex D	0.044		-0.37	
541	ISO22241-2 Annex D	<0.10			
551	ISO22241-2 Annex D	0.036		-2.85	
862	ISO22241-2 Annex D	0.05		1.49	
864	ISO22241-2 Annex D	0.05		1.49	
1300	ISO22241-2 Annex D	0.0482		0.93	
1367	ISO22241-2 Annex D	0.0401		-1.58	
1656	ISO22241-2 Annex D	<0.01		<-10.90	Possibly a false negative test result?
1807	ISO22241-2 Annex D	0.045		-0.06	
1938	ISO22241-2 Annex D	0.05		1.49	
6039	ISO22241-2 Annex D	0.048		0.87	
	normality	OK			
	n	10			
	outliers	0			
	mean (n)	0.0452			
	st.dev. (n)	0.00494			
	R(calc.)	0.0138			
	st.dev.(ISO22241-2 annex K:06)	0.00323			
	R(ISO22241-2 annex K:06)	0.0090			

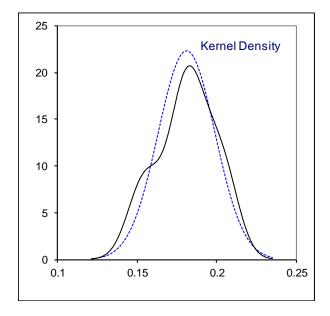




Determination of Biuret on sample #18092; result in %M/M

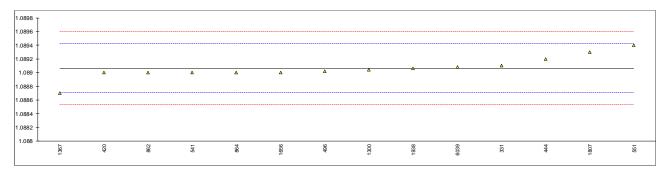
lab	method	value	mark z(targ)	remarks
331	ISO22241-2 Annex E	0.155	-1.82	
420	ISO22241-2 Annex E	0.18	-0.07	
444				
496	ISO22241-2 Annex E	0.190	0.63	
541	ISO22241-2 Annex E	0.160	-1.47	
551	ISO22241-2 Annex E	0.181	0.00	
862	ISO22241-2 Annex E	0.20	1.33	
864	ISO22241-2 Annex E	0.206	1.75	
1300	ISO22241-2 Annex E	0.1805	-0.03	
1367	ISO22241-2 Annex E	0.19	0.63	
1656	ISO22241-2 Annex E	0.15	-2.17	
1807	ISO22241-2 Annex E	0.175	-0.42	
1938	ISO22241-2 Annex E	0.205	1.68	
6039	ISO22241-2 Annex E	0.180	-0.07	
	normality	OK		
	n	13		
	outliers	0		
	mean (n)	0.1810		
	st.dev. (n)	0.01789		
	R(calc.)	0.0501		
	st.dev.(ISO22241-2 annex K:06)	0.01429		
	R(ISO22241-2 annex K:06)	0.04		

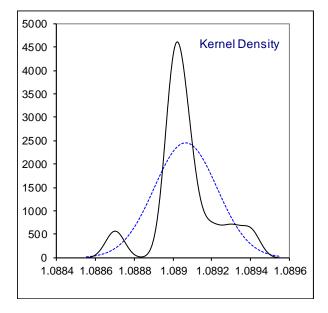




Determination of Density at 20°C on sample #18092; result in kg/L

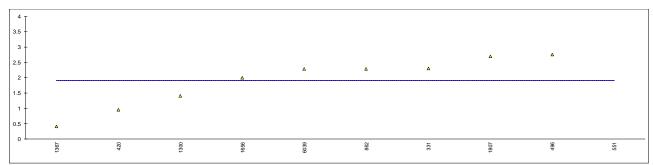
lab	method	value	mark	z(targ)	remarks
331	ISO12185	1.0891	С	0.20	First reported 1089.1 kg/L
420	ISO12185	1.0890		-0.36	
444	D4052	1.0892		0.76	
496	ISO12185	1.08902		-0.25	
541	ISO12185	1.08900		-0.36	
551	ISO12185	1.0894		1.88	
862	D4052	1.0890		-0.36	
864	D4052	1.0890		-0.36	
1300	ISO12185	1.08904		-0.14	
1367	ISO12185	1.0887	С	-2.04	First reported 1088.7 kg/L
1656	D4052	1.0890		-0.36	
1807	D4052	1.0893		1.32	
1938	ISO12185	1.08906		-0.02	
6039	ISO12185	1.08908		0.09	
	normality	not OK			
	n	14			
	outliers	0			
	mean (n)	1.08906			
	st.dev. (n)	0.000163			
	R(calc.)	0.00046			
	st.dev.(ISO22241-2 annex K:06)	0.000179			
	R(ISO22241-2 annex K:06)	0.0005			
	,				

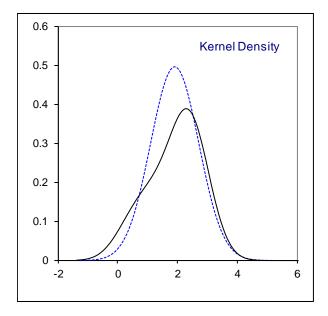




Determination of Insoluble Matter on sample #18092; result in mg/kg

lab	method	value	mark	z(targ)	remarks
331	ISO22241-2 Annex G	2.3	•		
420	ISO22241-2 Annex G	0.962			
444					
496	ISO22241-2 Annex G	2.76			
541	ISO22241-2 Annex G	<1.0			
551	ISO22241-2 Annex G	45.9	C,G(0.01)		First reported 43.94
862	ISO22241-2 Annex G	2.29			
864					
1300	ISO22241-2 Annex G	1.41			
1367	ISO22241-2 Annex G	0.41			
1656	ISO22241-2 Annex G	2			
1807	ISO22241-2 Annex G	2.7			
1938					
6039	ISO22241-2 Annex G	2.279			
	normality	OK			
	n	9			
	outliers	1			
	mean (n)	1.9012			
	st.dev. (n)	0.80509			
	R(calc.)	2.2543			
	st.dev.(ISO22241-2 annex K:06)	(0.25802)			
	R(ISO22241-2 annex K:06)	(0.7225)			



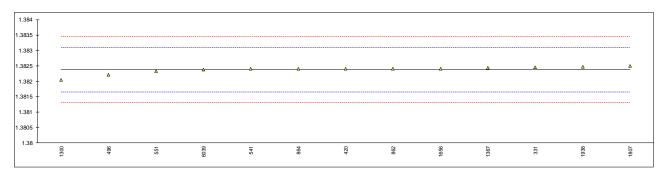


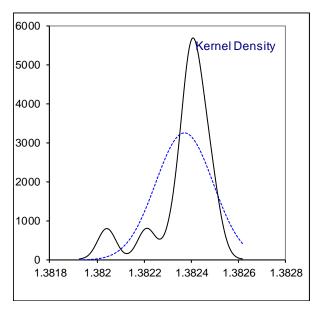
Determination of Phosphate as PO₄ on sample #18092; result in mg/kg

lab	method	value	mark	z(targ)	remarks
331	ISO22241-2 Annex H	< 0.05			
420	ISO22241-2 Annex H	<0,05			
444					
496	ISO22241-2 Annex H	0.015			
541	ISO22241-2 Annex H	< 0.05			
551					
862	ISO22241-2 Annex H	< 0.05			
864					
1300	ISO22241-2 Annex H	0.002			
1367	ISO22241-2 Annex H	< 0.01			
1656			W		First reported 0.32
1807	ISO22241-2 Annex H	0.25			Possibly a false positive test result?
1938	ISO22241-2 Annex H	0.004			
6039	ISO22241-2 Annex H	0.052			
	n	9			
	mean (n)	< 0.05			

Determination of Refractive index at 20°C on sample #18092;

lab	method	value	mark	z(targ)	remarks
331	ISO22241-2 Annex C	1.3824535		0.23	
420	ISO22241-2 Annex C	1.3824		0.08	
444					
496	ISO22241-2 Annex C	1.38221		-0.45	
541	ISO22241-2 Annex C	1.38240		0.08	
551	ISO22241-2 Annex C	1.38233		-0.11	
862	ISO22241-2 Annex C	1.3824		0.08	
864	ISO22241-2 Annex C	1.3824		0.08	
1300	ISO22241-2 Annex C	1.38204		-0.92	
1367	ISO22241-2 Annex C	1.38244		0.20	
1656	ISO22241-2 Annex C	1.3824		0.08	
1807	ISO22241-2 Annex C	1.3825		0.36	
1938	ISO22241-2 Annex C	1.38247		0.28	
6039	ISO22241-2 Annex C	1.38237		0.00	
	normality	not OK			
	n	13			
	outliers	0			
	mean (n)	1.382370			
	st.dev. (n)	0.0001223			
	R(calc.)	0.000342			
	st.dev.(ISO22241-2 annex K:06)	0.0003571			
	R(ISO22241-2 annex K:06)	0.0010			



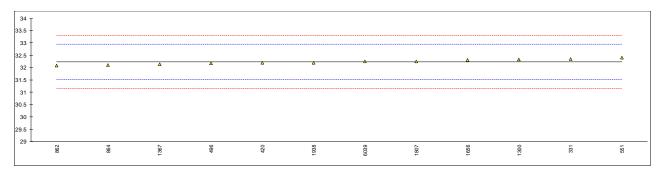


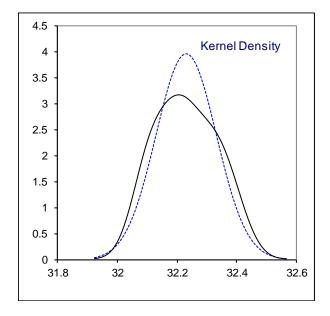
Determination of Urea content (total Nitrogen) on sample #18092; result in %M/M

lab	method	value	mark	z(targ)	remarks
331					
420					
444					
496					
541					
551					
862	ISO22241-2 Annex B	32.2		-0.09	
864					
1300	ISO22241-2 Annex B	32.171		-0.17	
1367					
1656					
1807					
1938					
6039					
	n o rea ality	unlenguen			
	normality	unknown			
	n outliers	2 0			
		32.231		Concon	aug value used from Urea by Defrective index
	mean (n) st.dev. (n)	n.a.		Consens	sus value used from Urea by Refractive index
	R(calc.)	n.a.			
	st.dev.(ISO22241-2 annex K:06)	0.3571			
	R(ISO22241-2 annex K:06)	1			
	K(13022241-2 affilex K:00)	ı			

Determination of Urea content (by refractive index) on sample #18092; result in %M/M

lab	method	value	mark z(targ)	remarks
331	ISO22241-2 Annex C	32.35	0.33	
420	ISO22241-2 Annex C	32.2	-0.09	
444				
496	ISO22241-2 Annex C	32.17	-0.17	
541				
551	ISO22241-2 Annex C	32.4	0.47	
862	ISO22241-2 Annex C	32.09	-0.39	
864	ISO22241-2 Annex C	32.1	-0.37	
1300	ISO22241-2 Annex C	32.333	0.29	
1367	ISO22241-2 Annex C	32.13	-0.28	
1656	ISO22241-2 Annex C	32.3	0.19	
1807	ISO22241-2 Annex C	32.25	0.05	
1938	ISO22241-2 Annex C	32.20	-0.09	
6039	ISO22241-2 Annex C	32.249	0.05	
	normality	OK		
	n	12		
	outliers	0		
	mean (n)	32.231		
	st.dev. (n)	0.1007		
	R(calc.)	0.282		
	st.dev.(ISO22241-2 annex K:06)	0.3571		
	R(ISO22241-2 annex K:06)	1.0		





Determination of Trace Elements on sample #18092; result in mg/kg

lab	method	Al	Ca	Cr	Cu	Fe	Mg	Ni	K	Na	Zn
331	*)	<0.5	<0.5	<0.2	<0.2	<0.2	<0.5	<0.2	<0.5	<0.5	<0.2
420	*)	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
444											
496	*)	0.0065	0.0485	0.0117	0.00	0.016	0.00175	0.00225	0.0222	0.0585	0.00675
541	*)	< 0.5	< 0.5	< 0.2	< 0.2	< 0.5	< 0.5	< 0.2	< 0.5	< 0.5	<0.2
551	*)	0.0	0.0	0.1	0.0	0.0	0.0	0.00 C	0.0	0.0	0.0
862	*)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
864											
1300	*)	0.036	0.025	0.032	0.022	0.018	0.020	0.027	0.075	0.068	0.023
1367	*)	< 0.01	0.02	0.08	< 0.01	< 0.01	< 0.01	0.01	< 0.01	< 0.01	0.04
1656	*)	< 0.01	< 0.01	<0.1	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	<0.1
1807	*)	< 0.15	< 0.15	0.11	< 0.15	< 0.15	< 0.25	< 0.15	< 0.25	< 0.25	<0.15
1938	*)	0.007	0.008	0.051	0.00	0.012	0.003	0.016	0.001	0.007	0.040
6039	*)	0.036	0.019	0.099	0.014	0.007	0.009	0.028	0.012	0.040	0.0001

^{*)} ISO22241-2 Annex I

Lab 551 first reported 0.59

APPENDIX 2

Number of participants per country

- 1 lab in ARGENTINA
- 1 lab in BRAZIL
- 2 labs in CHINA, People's Republic
- 1 lab in CZECH REPUBLIC
- 1 lab in ESTONIA
- 1 lab in FRANCE
- 2 labs in GERMANY
- 1 lab in SPAIN
- 1 lab in TURKEY
- 3 labs in UNITED KINGDOM

APPENDIX 3

Abbreviations

C = final test result after checking of first reported suspect test result

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \\ DG(0.05) &= \text{straggler in Double Grubbs' outlier test} \\ \end{array}$

R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
R(1) = outlier in Rosner's outlier test
R(5) = straggler in Rosner's outlier test
E = probably an error in calculations

U = test result reported probably in a different unit
W = test result withdrawn on request of participant
ex = test result excluded from the statistical evaluation

n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported

SDS = Material Safety Data Sheet

Literature

- 1. iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
- 2. Horwitz, R. Albert, J. AOAC Int, <u>79</u>, 3, 589, (1996)
- 3. ASTM E178:08
- 4. ASTM E1301:03
- 5. ISO 5725:86
- 6. ISO 5725, parts 1-6:94
- 7. ISO13528:05
- 8. M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 9. W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 10. DIN 38402 T41/42
- 11. P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
- 12. J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 13. Analytical Methods Committee Technical Brief, No. 4 January 2001
- 14. P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, Analyst 2002, <u>127</u>, 1359-1364
- 15. Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)