Results of Proficiency Test
AdBlue, 32% Urea Solution
May 2020

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 in accordance with the latest version of the ISO22241 part 1 every year. During the annual proficiency testing program 2019/2020 it was decided to continue the round robin for the analysis of AdBlue 32% Urea Solution.
In this interlaboratory study 22 laboratories in 14 different countries registered for participation. See appendix 2 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 #20072.
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

Approximately 30 liters of AdBlue 32% Urea solution was obtained from a local supplier. After homogenization 30 wide-neck HDPE bottles of 1L were filled and labelled #20072. The homogeneity of the subsamples #20072 was checked by determination of Density at 20°C in accordance with ISO12185 on 5 stratified randomly selected subsamples.

	Density at 20°C in kg/m^3
sample #20072-1	1089.2
sample #20072-2	1089.2
sample #20072-3	1089.2
sample #20072-4	1089.2
sample #20072-5	1089.2

Table 1: homogeneity test results of subsamples #20072

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)	0.00
reference test method	ISO12185:96
0.3 x R (reference test method)	0.15

Table 2: evaluation of repeatability of the subsamples #20072

The calculated repeatability was 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 bottle of 1L AdBlue 32% Urea solution labelled #20072 was sent on April 29, 2020. 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 that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. 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	good
1 <	|z|	< 2	satisfactory
2 <	|z|	< 3	questionable
3 <	|z|		unsatisfactory

4 EVALUATION

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another seven weeks. Finally, four participants did not report any test results. Not all laboratories were able to perform all analyzes requested. In total 18 participants reported 161 numerical test results. Observed were 8 outlying test results, which is 5.0%. 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 test methods are also in the tables together with the original data. The abbreviations, used in these tables, are explained 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 full agreement with the requirements of ISO22241-2:19.
The consensus value (0.24 mg/kg) is below the application range of Aldehyde as Formaldehyde in ISO22241-2:19 (0.5 - 10 mg/kg). Most of the participants reported a numerical test result below this application range without difficulties, therefore it was decided to calculate z-scores.

Alkalinity as NH_3: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO22241-2:19.

Biuret: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO22241-2:19.

Density at 20°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96.

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

Phosphate as PO_4: This determination may not be problematic. Almost all participants agreed that the Phosphate as PO_4 is <0.2 mg/kg. It was decided not to calculate z-scores due to the low Phosphate content.

Refractive Index at 20°C: 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 ISO22241-2:19.

Urea content (by total Nitrogen): Only three participants reported a test result. 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 not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO22241-2:19.

Trace elements: The majority of the participants agreed on a concentration near or below the limit of detection for all trace elements mentioned in paragraph 2.6. Therefore, no z-scores were calculated.

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. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ISO standards) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Aldehyde as Formaldehyde	mg/kg	11	0.24	0.45	0.46
Alkalinity as NH_3	%M/M	13	0.051	0.054	0.124
Biuret	%M/M	16	0.16	0.02	0.04
Density at 20°C	kg/m^3	16	1089.3	0.3	0.5
Insoluble Matter	mg/kg	14	3.46	7.96	8.22
Phosphate as PO_4	mg/kg	14	<0.2	n.a.	n.a.
Refractive index at 20°C		16	1.3825	0.0002	0.0003
Urea content (by total Nitrogen)	%M/M	3	32.67	0.86	1.05
Urea content (by refractive index)	%M/M	13	32.29	0.15	0.21

Table 3: reproducibilities of tests on sample #20072

Without further statistical calculations, it can be concluded that for all tests there is a good compliance of the group of participants with the relevant reference test methods.

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

	May 2020	June 2019	June 2018	June 2017
Number of reporting laboratories	18	18	14	14
Number of test results	161	192	146	196
Number of statistical outliers	8	7	2	4
Percentage of statistical outliers	5.0%	3.6%	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 reference test methods. The conclusions are given in the following table.

	May 2020	June 2019	June 2018	June 2017
Aldehyde as Formaldehyde	+/-	+/-	+/-	--
Alkalinity as NH_3	++	++	-	-
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)	+	-	++	++

Table 5: comparison determinations against the reference test methods
Result between brackets should be used with due care

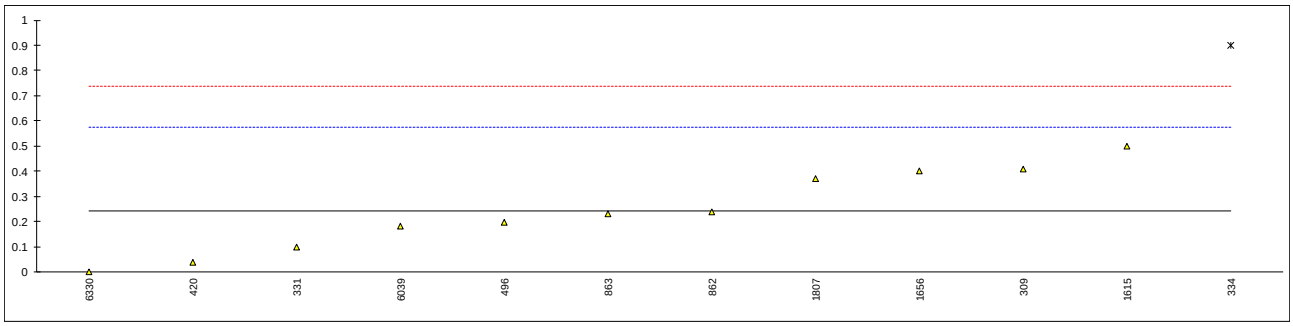
The following performance categories were used:

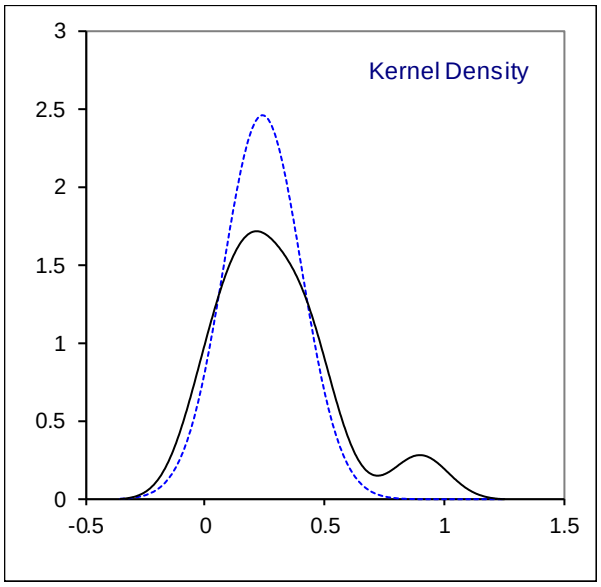
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

APPENDIX 1

Determination of Aldehyde as Formaldehyde on sample #20072; result in mg/kg

lab	method	value	mark	z(targ)	remarks
309	ISO22241-2 Annex F	0.409		1.01	
331	ISO22241-2 Annex F	0.1		-0.86	
334	ISO22241-2 Annex F	0.90	C,G(0.05)	3.97	first reported 0.98
343	ISO22241-2 Annex F	<0,5		-----	
398	ISO22241-2 Annex F	<0,5		-----	
420	ISO22241-2 Annex F	0.0367		-1.24	
445		-----		-----	
496	ISO22241-2 Annex F	0.196		-0.28	
541	ISO22241-2 Annex F	<0.50		-----	
551		-----		-----	
862	ISO22241-2 Annex F	0.24		-0.01	
863	ISO22241-2 Annex F	0.23		-0.07	
864	ISO22241-2 Annex F	<0.5		-----	
1299		-----		-----	
1367		-----		-----	
1615	ISO22241-2 Annex F	0.50		1.56	
1656	ISO22241-2 Annex F	0.40		0.95	
1807	ISO22241-2 Annex F	0.37		0.77	
6039	ISO22241-2 Annex F	0.182		-0.36	
6256		-----		-----	
6262		-----		-----	
6330	ISO22241-2 Annex F	0		-1.46	
	normality	OK			
	n	11			
	outliers	1			
	mean (n)	0.242			
	st.dev. (n)	0.1620			
	R(calc.)	0.454			
	st.dev.(ISO22241-2:19)	0.1657			
	R(ISO22241-2:19)	0.464			application range: 0.5 – 10mg/kg

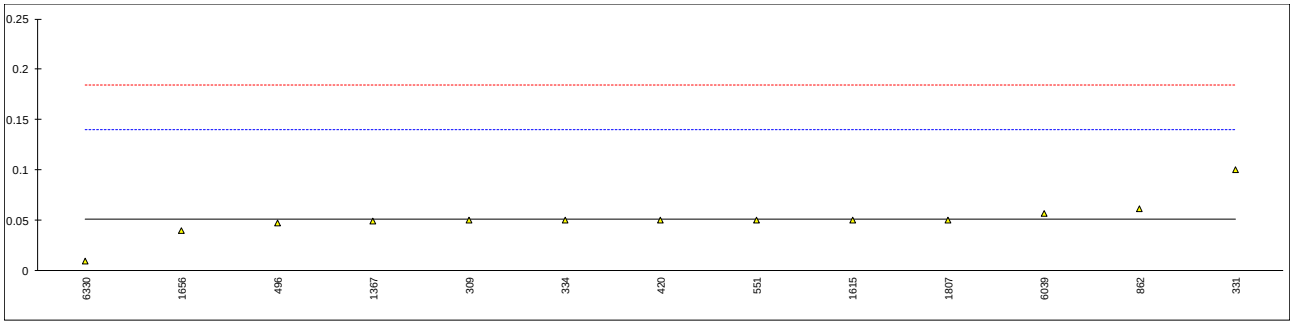


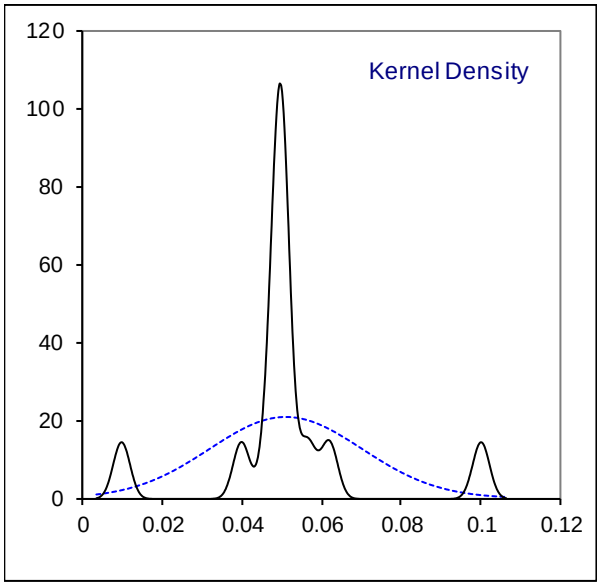


Determination of Alkalinity as NH_3 on sample #20072; result in %M/M

lab	method	value	mark	z(targ)	remarks
309	ISO22241-2 Annex D	0.05		-0.03	
331	ISO22241-2 Annex D	0.1		1.10	
334	ISO22241-2 Annex D	0.05		-0.03	
343	ISO22241-2 Annex D	<0,1		-----	
398	ISO22241-2 Annex D	<0,1		-----	
420	ISO22241-2 Annex D	0.05		-0.03	
445		-----		-----	
496	ISO22241-2 Annex D	0.047		-0.09	
541	ISO22241-2 Annex D	<0.10		-----	
551	ISO22241-2 Annex D	0.05		-0.03	
862	ISO22241-2 Annex D	0.062		0.25	
863	ISO22241-2 Annex D	<0.10		-----	
864	ISO22241-2 Annex D	<0.1		-----	
1299		-----		-----	
1367	ISO22241-2 Annex D	0.049		-0.05	
1615	ISO22241-2 Annex D	0.05		-0.03	
1656	ISO22241-2 Annex D	0.04		-0.25	
1807	ISO22241-2 Annex D	0.05		-0.03	
6039	ISO22241-2 Annex D	0.0565		0.12	
6256		-----		-----	
6262		-----		-----	
6330	ISO22241-2 Annex D	0.01		-0.93	

normality	not OK
n	13
outliers	0
mean (n)	0.0511
st.dev. (n)	0.01911
R(calc.)	0.0535
st.dev.(ISO22241-2:19)	0.04429
R(ISO22241-2:19)	0.124

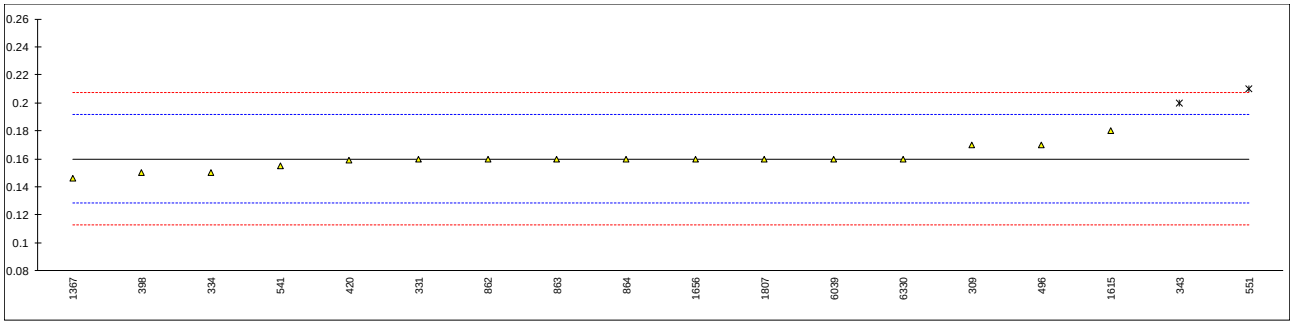


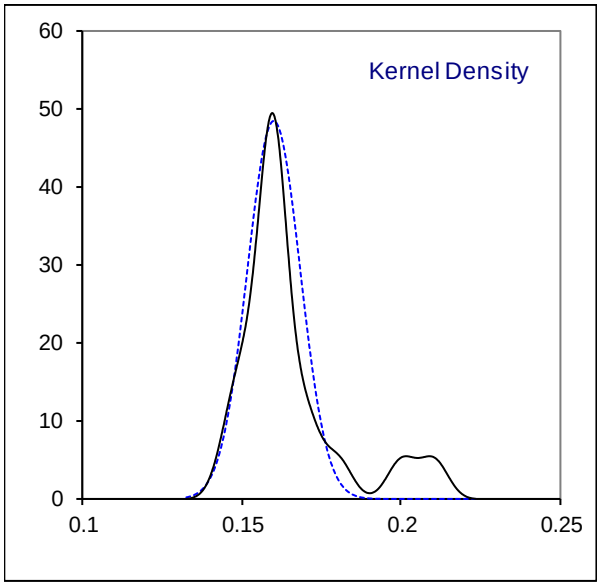


Determination of Biuret on sample #20072; result in %M/M

lab	method	value	mark	z(targ)	remarks
309	ISO22241-2 Annex E	0.17		0.64	
331	ISO22241-2 Annex E	0.16		0.00	
334	ISO22241-2 Annex E	0.15		-0.64	
343	ISO22241-2 Annex E	0.20	G(0.05)	2.54	
398	ISO22241-2 Annex E	0.15		-0.64	
420	ISO22241-2 Annex E	0.1593		-0.05	
445		-----		-----	
496	ISO22241-2 Annex E	0.170		0.64	
541	ISO22241-2 Annex E	0.155		-0.32	
551	ISO22241-2 Annex E	0.21	G(0.05)	3.18	
862	ISO22241-2 Annex E	0.16		0.00	
863	ISO22241-2 Annex E	0.16		0.00	
864	ISO22241-2 Annex E	0.16		0.00	
1299		-----		-----	
1367	ISO22241-2 Annex E	0.146		-0.89	
1615	ISO22241-2 Annex E	0.18		1.27	
1656	ISO22241-2 Annex E	0.16		0.00	
1807	ISO22241-2 Annex E	0.16		0.00	
6039	ISO22241-2 Annex E	0.16		0.00	
6256		-----		-----	
6262		-----		-----	
6330	ISO22241-2 Annex E	0.16		0.00	

normality	suspect
n	16
outliers	2
mean (n)	0.1600
st.dev. (n)	0.00825
R(calc.)	0.0231
st.dev.(ISO22241-2:19)	0.01571
R(ISO22241-2:19)	0.044

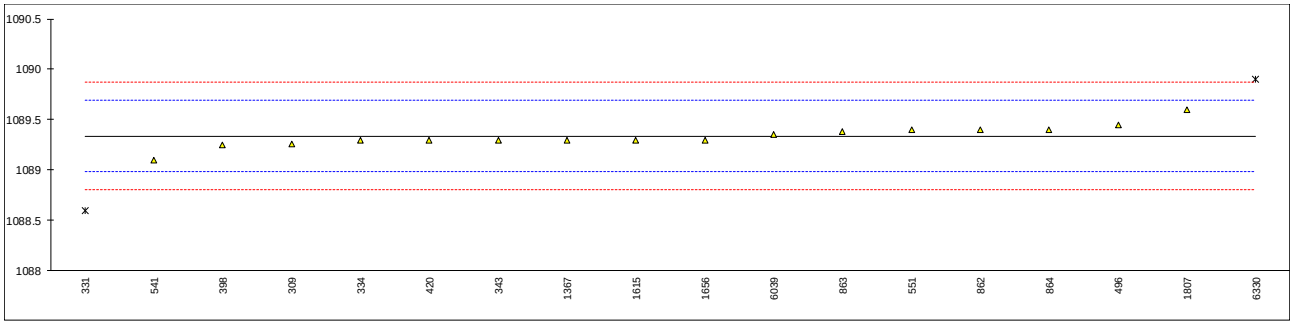


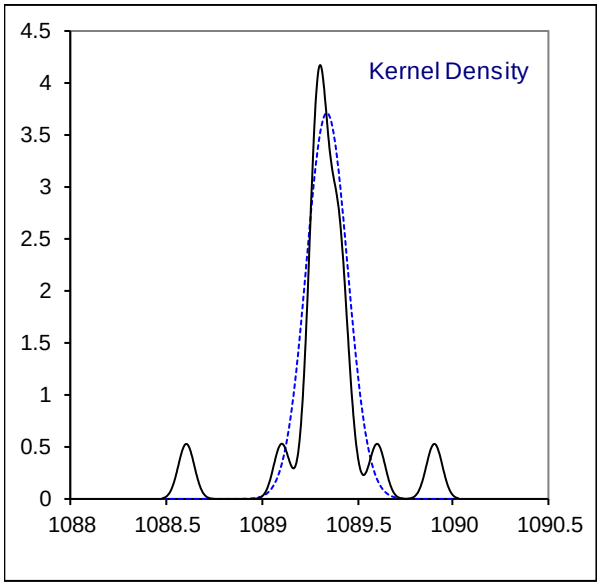


Determination of Density at 20°C on sample #20072; result in kg/m^3

lab	method	value	mark	z(targ)	remarks
309	D4052	1089.255		-0.46	
331	ISO12185	1088.6	G(0.01)	-4.12	
334	ISO12185	1089.3		-0.20	
343	ISO12185	1089.3		-0.20	
398	ISO12185	1089.25		-0.48	
420	ISO12185	1089.3		-0.20	
445		-----		-----	
496	ISO12185	1089.45		0.64	
541	ISO12185	1089.1	C	-1.32	first reported 1087.91
551	D4052	1089.4		0.36	
862	ISO12185	1089.4		0.36	
863	ISO12185	1089.38		0.24	
864	ISO12185	1089.4		0.36	
1299		-----		-----	
1367	ISO12185	1089.3		-0.20	
1615	ISO12185	1089.3		-0.20	
1656	D4052	1089.3		-0.20	
1807	D4052	1089.6		1.48	
6039	ISO12185	1089.35		0.08	
6256		-----		-----	
6262		-----		-----	
6330	ISO12185	1089.9	G(0.05)	3.16	

normality	not OK
n	16
outliers	2
mean (n)	1089.3365
st.dev. (n)	0.10786
R(calc.)	0.3020
st.dev.(ISO12185:96)	0.17857
R(ISO12185:96)	0.5

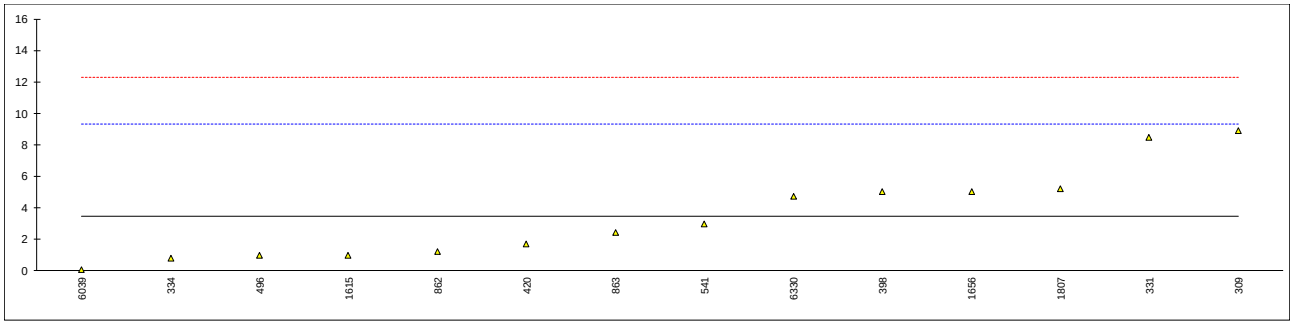


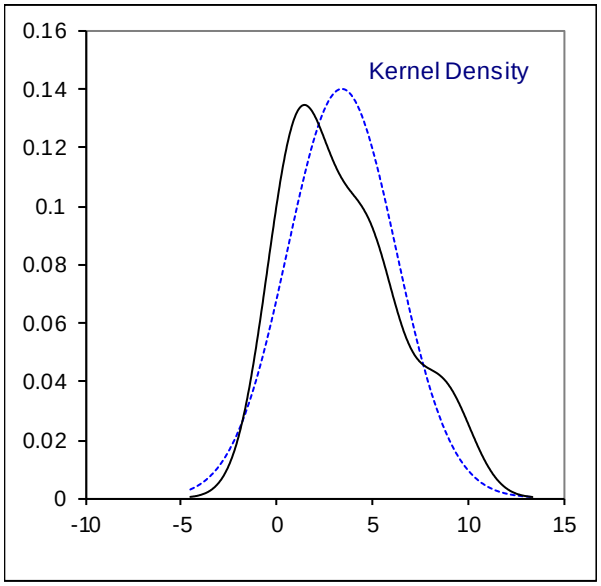


Determination of Insoluble Matter on sample #20072; result in mg/kg

lab	method	value	mark	z(targ)	remarks
309	ISO22241-2 Annex G	8.9		1.85	
331	ISO22241-2 Annex G	8.5		1.72	
334	ISO22241-2 Annex G	0.77		-0.92	
343		-----		-----	
398	ISO22241-2 Annex G	5.0		0.52	
420	ISO22241-2 Annex G	1.72		-0.59	
445		-----		-----	
496	ISO22241-2 Annex G	0.955		-0.85	
541	ISO22241-2 Annex G	3.00		-0.16	
551		-----		-----	
862	ISO22241-2 Annex G	1.2		-0.77	
863	ISO22241-2 Annex G	2.4		-0.36	
864	ISO22241-2 Annex G	<1		-----	
1299		-----		-----	
1367	ISO22241-2 Annex G	<1		-----	
1615	ISO22241-2 Annex G	1.0		-0.84	
1656	ISO22241-2 Annex G	5		0.52	
1807	ISO22241-2 Annex G	5.2		0.59	
6039	ISO22241-2 Annex G	0.062		-1.16	
6256		-----		-----	
6262		-----		-----	
6330	ISO22241-2 Annex G	4.746		0.44	

normality	OK
n	14
outliers	0
mean (n)	3.4609
st.dev. (n)	2.84198
R(calc.)	7.9576
st.dev.(ISO22241-2:19)	2.93571
R(ISO22241-2:19)	8.220





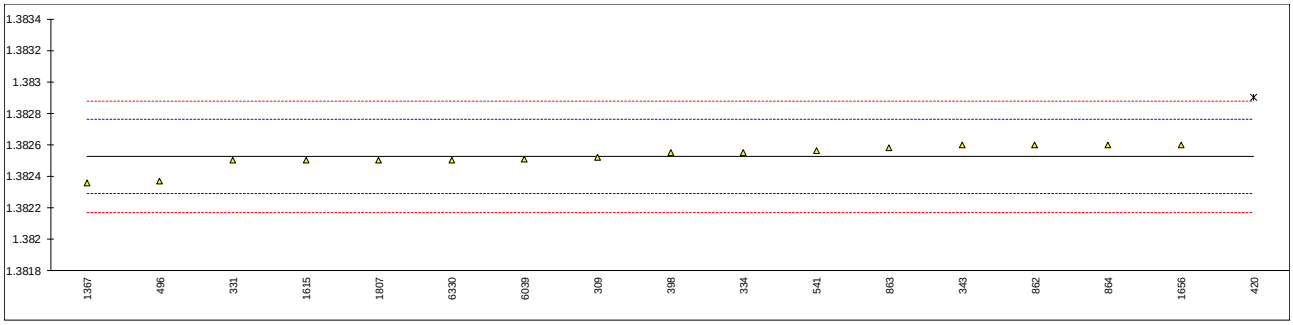
Determination of Phosphate as PO_4 on sample #20072; result in mg/kg

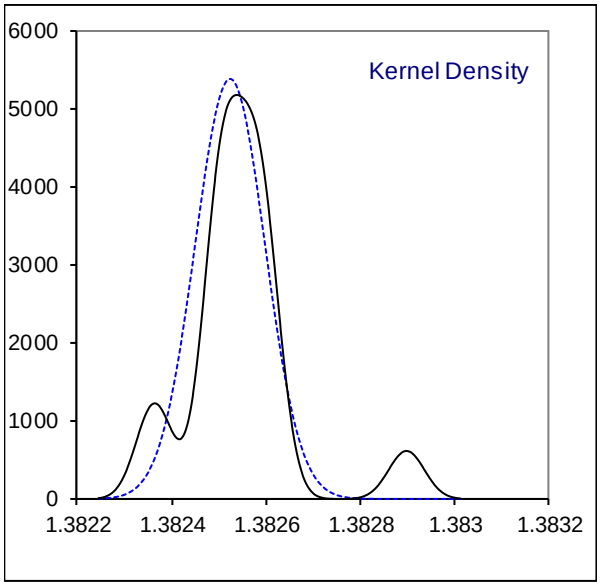
lab	method	value	mark	z(targ)	remarks
309	ISO22241-2 Annex H	0.03		-----	
331	ISO22241-2 Annex H	<0.05		-----	
334	ISO22241-2 Annex H	0.38	C	-----	first reported 0.49
343	ISO22241-2 Annex H	0.06		-----	
398	ISO22241-2 Annex H	<0,05		-----	
420	ISO22241-2 Annex H	0.3067		-----	
445		-----		-----	
496	ISO22241-2 Annex H	0.002		-----	
541	ISO22241-2 Annex H	0.074		-----	
551	ISO22241-2 Annex H	<0,05		-----	
862	ISO22241-2 Annex H	<0.05		-----	
863	ISO22241-2 Annex H	<0.05		-----	
864	ISO22241-2 Annex H	<0.05		-----	
1299		-----		-----	
1367		-----		-----	
1615	ISO22241-2 Annex I	<0.10		-----	
1656		-----		-----	
1807	ISO22241-2 Annex H	<0.2		-----	
6039	ISO22241-2 Annex H	0.036		-----	
6256		-----		-----	
6262		-----		-----	
6330	ISO22241-2 Annex H	0.01		-----	
	n	14			
	mean (n)	<0.2			

Determination of Refractive index at 20°C on sample #20072;

lab	method	value	mark	z(targ)	remarks
309	ISO22241-2 Annex C	1.38252		-0.05	
331	ISO22241-2 Annex C	1.3825		-0.22	
334	ISO22241-2 Annex C	1.38255		0.21	
343	ISO22241-2 Annex C	1.3826		0.63	
398	ISO22241-2 Annex C	1.38255		0.21	
420	ISO22241-2 Annex C	1.3829	G(0.01)	3.18	
445		-----		-----	
496	ISO22241-2 Annex C	1.38237		-1.32	
541	ISO22241-2 Annex C	1.382563		0.32	
551		-----		-----	
862	ISO22241-2 Annex C	1.3826		0.63	
863	ISO22241-2 Annex C	1.38258		0.46	
864	ISO22241-2 Annex C	1.3826		0.63	
1299		-----		-----	
1367	ISO22241-2 Annex C	1.38236	C	-1.40	first reported 1.382
1615	ISO22241-2 Annex C	1.38250		-0.22	
1656	ISO22241-2 Annex C	1.3826		0.63	
1807	ISO22241-2 Annex C	1.3825		-0.22	
6039	ISO22241-2 Annex C	1.38251		-0.13	
6256		-----		-----	
6262		-----		-----	
6330	ISO22241-2 Annex C	1.3825		-0.22	

normality	suspect
n	16
outliers	1
mean (n)	1.382525
st.dev. (n)	0.0000740
R(calc.)	0.000207
st.dev.(ISO22241-2:19)	0.0001179
R(ISO22241-2:19)	0.00033





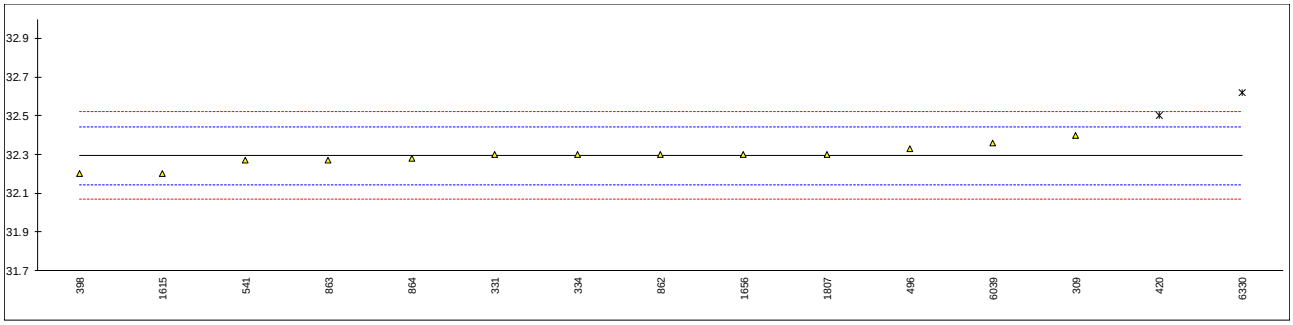
Determination of Urea content (by total Nitrogen) on sample #20072; result in %M/M

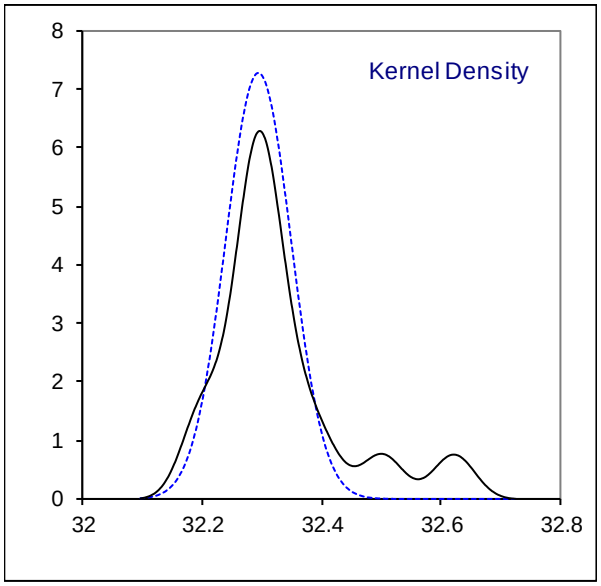
lab	method	value	mark	z(targ)	remarks
309		-----		-----	
331		-----		-----	
334		-----		-----	
343	ISO22241-2 Annex B	33.0		0.88	
398		-----		-----	
420		-----		-----	
445		-----		-----	
496		-----		-----	
541		-----		-----	
551		-----		-----	
862	ISO22241-2 Annex B	32.4		-0.71	
863		-----		-----	
864		-----		-----	
1299		-----		-----	
1367		-----		-----	
1615		-----		-----	
1656		-----		-----	
1807		-----		-----	
6039		-----		-----	
6256		-----		-----	
6262		-----		-----	
6330	ISO22241-2 Annex B	32.602		-0.17	
	normality	unknown			
	n	3			
	outliers	0			
	mean (n)	32.667			
	st.dev. (n)	0.3053			
	R(calc.)	0.855			
	st.dev.(ISO22241-2:19)	0.3761			
	R(ISO22241-2:19)	1.053			

Determination of Urea content (by refractive index) on sample #20072; result in %M/M

lab	method	value	mark	z(targ)	remarks
309	ISO22241-2 Annex C	32.4	C	1.42	first reported 32.1
331	ISO22241-2 Annex C	32.3		0.09	
334	ISO22241-2 Annex C	32.3	C	0.09	first reported 32.5
343		-----		-----	
398	ISO22241-2 Annex C	32.2		-1.24	
420	ISO22241-2 Annex C	32.5	G(0.05)	2.75	
445		-----		-----	
496	ISO22241-2 Annex C	32.331		0.50	
541	ISO22241-2 Annex C	32.27		-0.31	
551		-----		-----	
862	ISO22241-2 Annex C	32.3		0.09	
863	ISO22241-2 Annex C	32.27		-0.31	
864	ISO22241-2 Annex C	32.28		-0.17	
1299		-----		-----	
1367		-----		-----	
1615	ISO22241-2 Annex C	32.20		-1.24	
1656	ISO22241-2 Annex C	32.3		0.09	
1807	ISO22241-2 Annex C	32.3		0.09	
6039	ISO22241-2 Annex C	32.359		0.87	
6256		-----		-----	
6262		-----		-----	
6330	ISO22241-2 Annex C	32.620	C,G(0.05)	4.34	first reported 32.626

normality	OK
n	13
outliers	2
mean (n)	32.293
st.dev. (n)	0.0548
R(calc.)	0.153
st.dev.(ISO22241-2:19)	0.0754
R(ISO22241-2:19)	0.211





Determination of Trace Elements on sample #20072; result in mg/kg

lab	method	Al	Ca	Cr	Cu	Fe	Mg	Ni	K	Na	Zn
309		<0.01	<0.01	0.08	<0.01	<0.01	<0.01	0.03	<0.2	<0.2	0.02
331	*)	<0.5	<0.5	<0.2	<0.2	<0.5	<0.5	<0.2	<0.5	<0.5	<0.2
334	*)	0.01	0.04	0.10	<0.01	0.01	0.02	0.03	0.05	0.04	0.02
343	*)	<0,5	<0,5	<0,2	<0,2	<0,5	<0,5	<0,2	<0,5	<0,5	<0,2
398		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
420	*)	<0,03	<0,03	0.03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03
445	*)	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
496		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
541	*)	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
551		-----	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
862	*)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
863	*)	<0.10	<0.20	<0.10	<0.10	<0.10	<0.10	<0.10	<0.20	<0.20	<0.10
864	*)	<0.5	<0.5	<0.2	<0.2	<0.5	<0.5	<0.2	<0.5	<0.5	<0.2
1299		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1367	*)	0.05	0.11	0.07	0	0.02	0	0.01	0.2	0	0
1615	*)	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
1656	*)	<0.5	<0.5	<0.2	<0.2	<0.5	<0.5	<0.2	<0.5	<0.5	<0.2
1807	*)	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15
6039	*)	0.029	0.010	0.099	0.010	0.005	0.002	0.020	0.041	0.024	0.040
6256		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
6262		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
6330	*)	0	0	0.075	0	0	0	0.084	0.006	0	0

*) ISO22241-2 Annex I

APPENDIX 2

Number of participants per country

1 lab in	ARGENTINA
1 lab in	BELGIUM
1 lab in	BRAZIL
3 labs in	CHINA, People's Republic
1 lab in	CZECH REPUBLIC
2 labs in	FRANCE
2 labs in	GERMANY
1 lab in	INDIA
1 lab in	ITALY
1 lab in	MALAYSIA
1 lab in	NETHERLANDS
3 labs 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
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
E = possibly an error in calculations
W = test result withdrawn on request of participant
ex = test result excluded from statistical evaluation
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported
SDS = Safety Data Sheet

Literature

1. iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2. Horwitz, R. Albert, J. AOAC Int, 79, 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, 76, 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, 331, 513, (1988)
12. J.N. Miller, Analyst, 118, 455, (1993)
13. Analytical Methods Committee Technical Brief, No 4, January 2001
14. P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364 (2002)
15. Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)