

**Results of Proficiency Test
Biodiesel
May 2003**

**Organised by: Institute for Interlaboratory Studies
Dordrecht, the Netherlands**

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1 INTRODUCTION

On request of several participants, the Institute for Interlaboratory Studies decided to organise again a proficiency test for the analysis of Biodiesel, as part of the annual proficiency test program of 2002/2003. In this international interlaboratory study of the annual program, 20 laboratories from 14 different countries have participated. See appendix 3 for a list of participants in alphabetical country order.

In this report the results of the Biodiesel proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Dordrecht, The Netherlands, was the organizer of this proficiency test. To get maximum information from this study it was decided to send one sample Biodiesel (labelled 0336). Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Dordrecht, the Netherlands, is accredited in agreement with ISO guide 43 and ILAC-G13:2000, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie).

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test is described in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of August 1998 (iis98protocol, version 2.0).

2.3 SAMPLES

In this proficiency test one sample was used. The necessary 45 litre bulk of Pure Biodiesel was purchased from a German manufacturer. After homogenisation in a precleaned metal drum, the Biodiesel was transferred to 30 brown glass bottles of 1000 mL and to 30 brown glass bottles of 500 mL and both labelled 0336. The homogeneity of the subsamples was checked by determination of the Density @ 15°C in accordance with ASTM D4052:02 on 4 stratified random selected samples:

	<i>Density @15°C</i>
sample 0336-1	0.8848
sample 0336-2	0.8848
sample 0336-3	0.8848
sample 0336-4	0.8847

table 1: homogeneity test of subsamples 0336

	<i>Density @15°C</i>
r (sample 0336)	0.0001
Reference test	ASTM D4052:02
r (reference test)	0.0001

table 2: repeatability of subsample 0336

The repeatability of the results was in agreement with the repeatability as required by the reference test. Therefore, homogeneity of the samples was assumed.

One 1000 mL bottle and one 500 mL bottle Biodiesel, labelled 0336, were despatched to each of the participating laboratories on April 23, 2003.

2.4 STABILITY OF THE SAMPLES

In order to be sure that the material, which is used in this proficiency test, is stable for the valid period, the stability of the material packed in the brown glass bottles was checked prior to use.

2.5 ANALYSES

The participants were asked to determine Density @15°C, Viscosity @40°C, Flash Point PMcc, Cold Filter Plugging Point (CFPP), Water, Sulphur, Sulphated Ash, Carbon Residue, Total Contamination, Copper Corrosion, Methanol, Monoglycerides, Diglyceride, Triglyceride, Free Glycerol, Total Glycerol, Total Ester content, Linolein acid-methylester, Oxidation Stability, Iodine number, Sodium, Potassium, Phosphorus, Calcium and Magnesium.

To get comparable results a detailed report form, on which the units were prescribed, was sent together with each set of samples. Also a letter of instructions and a MSDS were added to the package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code number.

Directly after the deadline the available results were screened for suspect data. A 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 results. Additional or corrected data are put under 'Remarks' in the result tables in appendix 1.

Results that came in after deadline were not taken into account in the screening for suspect data and thus these participants were not requested to check the raw data for obvious errors.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test is described in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of August 1998 (iis98protocol, version 2.0).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. After removal of outliers this check was repeated. In case a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by G(0.01) for the Dixon test and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by G(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

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

3.2 GRAPHICS

In order to visualise 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 results from a sample are plotted. The corresponding laboratory numbers are under the X-axis. A straight line presents the average of the reported data. The reproducibility limits of the relevant standard, calculated as mean \pm target reproducibility, are presented by two dotted lines parallel to the average line. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

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. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated in accordance with:

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

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare. 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 during the execution. Two participants did not report any results and four participants reported after the deadline. Not all laboratories were able to perform all analyses requested. So finally, 18 participants reported 220 numerical results were reported. Observed were 18 statistical outlying results, which is 8.2% of the numerical results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal. Not all data sets proved to have a normal distribution. An abnormal distribution was found for the following determinations: Acid number, Density, Iodine number, Water, Linolein acid-methylester and Sodium.

4.1 EVALUATION PER TEST

In this section the results are discussed per test. The methods, which are used by the various laboratories, are 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.

Acid number: This determination is not problematic. No results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outlier, in good agreement with the requirements of ASTM D664:01

Carbon residue: This determination is not problematic. Only one result was outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outlier, in good agreement with the requirements of ISO10370:95.

CFPP: This determination is somewhat problematic. Three results were outside the reproducibility limits and the calculated reproducibility limit is, after rejection of the statistical outliers, not in agreement with the requirements of EN116:97. When the EN116 data are evaluated

separately the calculated reproducibility is smaller, but still not in agreement.

Copper Corrosion: No problems have been observed, although the reporting for the rounded results can be improved as in accordance with ISO2160:98 and ASTM D130:00 as one should report only the classification (e.g. 1,2,3,4). One participant reported a false positive result.

Total Contamin.: This determination is very problematic. Five results were found outside the reproducibility limits. The calculated reproducibility is not at all in agreement with the requirements of EN12662:98.

Density @15°C: This determination is not problematic. Only one result was found outside the reproducibility limits and the calculated reproducibility limits is, after rejection of the statistical outlier, in agreement with the requirements of D4052:02.

Flash Point PMcc: This determination is very problematic. Ten results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outlier, not at all in agreement with the requirements of EN22719:93. When the ASTM D93 data are evaluated separately, the calculated reproducibility is somewhat improved, but still not in agreement with the requirements of ASTM D93:02. None of the participants used the test method ISO 3679 as prescribed in En 14214:01.

Iodine Number: This determination is problematic. Two results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outlier, not in agreement with the requirements of EN14111:01 or ISO3961:96 (methods are identical)

Kin.Visco. @ 40°C: This determination is problematic. Four results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outliers, not in agreement with the requirements of ISO3104:94. When the ISO3104 and ASTM D445 data are evaluated separate, both calculated reproducibilities were not in agreement with the requirements of the respective standards. Thus the large spread may not be explained by the variety of methods used.

Oxidation Stability: Unfortunately, no reference method exists for the determination of Oxidation Stability of Biodiesel. Therefore no concrete conclusions could be made.

- Sulphated Ash: This determination is very problematic. Six results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outlier, not at all in agreement with the requirements of ASTM D874:00.
- Sulphur: This determination is somewhat problematic. Two results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outliers, almost in agreement with the requirements of ASTM D2622:98.
- Water: This determination is not problematic. Only one result was outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outlier, in good agreement with the requirements of ISO12937:00. When the ISO12937 data are evaluated separately the calculated reproducibility is even much smaller.
- Glycerides: This determination is somewhat problematic. For the three determinations, in total only one result was outside the reproducibility limits. However, for all three determinations the calculated reproducibilities were not in agreement with the requirements of EN14105:01. Perhaps the low number of numeric reporting results may explain this.
- Glycerol (Tot./Free): This determination seems somewhat problematic. For the two determinations, in total only one result was outside the reproducibility limits. However, for both determinations the calculated reproducibilities were not in agreement with the requirements of EN14105:01. Perhaps the low number of numeric reporting results may explain this.
- Methanol: This determination is problematic. Two results were outside the reproducibility limits. The calculated reproducibility is, even after rejection of the statistical outlier, not at all in agreement with the requirements of EN14110:01.
- Linolein acid-ME: This determination is not problematic. No results were outside the reproducibility limits and the calculated reproducibility is in good agreement with the requirements of EN14103:01.
- Total Ester: This determination is problematic. Only one result was outside the reproducibility limits. The calculated reproducibility is not at all in good agreement with the requirements of EN14103:01.

<u>Calcium:</u>	Due the lack of a suitable method for the determination of Calcium in Biodiesel, no conclusions can be drawn.
<u>Magnesium:</u>	No conclusions could be drawn due the low number of numeric results reported and the lack of a suitable method for Magnesium in Biodiesel.
<u>Phosphorous:</u>	The concentration found, was below the application range of EN14107:01, therefore no concrete conclusions could be made.
<u>Potassium:</u>	Unfortunately no reference method for the determination of Potassium in Biodiesel exists. However, one statistical outlier was noticed and the calculated reproducibility is not at all in agreement with the estimated reproducibility, calculated by the Horwitz Equation
<u>Sodium:</u>	The concentration found, was below the application range of EN14108:01, therefore no concrete conclusions could be made.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and these parameters as found for the group of participating laboratories. The average results and the calculated reproducibilities are compared in the next tables with the reproducibilities, derived from literature standards (in casu the ASTM and ISO standards, see tables in appendix 1).

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>R (Calc.)</i>	<i>R (lit)</i>
Acid number	Mg KOH/g	13	0.613	0.110	0.270
Carbon residue	%M/M	12	0.0563	0.0254	0.0360
Cold Filter Plugging Point	°C	13	-11.3	5.6	3.7
Total Contamination	mg/kg	9	16.26	21.30	4.88
Density @15°C	kg/m ³	16	0.88477	0.00037	0.00050
Flash Point PMcc	°C	12	89.70	21.96	3.50
Iodine number	g I/100g	8	116.58	7.49	5.00
Kin. Viscosity @ 40 °C	mm ² /S	15	4.5547	0.0728	0.0456
Oxidation Stability	hours	5	3.45	4.34	unknown
Sulphated Ash	%M/M	12	0.00702	0.01407	0.00279
Sulphur	mg/kg	10	3.10	5.60	5.6
Water	mg/kg	14	824.4	134.4	197.5
Monoglyceride	%M/M	5	0.7110	0.3014	0.2212
Diglyceride	%M/M	4	0.4043	0.1491	0.1026
Triglyceride	%M/M	4	1.2018	0.9343	0.3164
Free Glycerol	%M/M	4	0.0308	0.0312	0.0214
Total Glycerol	%M/M	5	0.3606	0.2814	0.1513
Methanol	%M/M	7	0.323	0.127	0.074
Linolein acid-ME	%M/M	6	7.783	0.579	2.441
Total Ester	%M/M	6	95.498	5.139	2.960
Calcium	mg/kg	6	0.617	1.269	unknown
Magnesium	mg/kg	3	0.255	0.763	unknown
Phosphorous	mg/kg	9	0.790	1.587	0.177
Potassium	mg/kg	8	16.26	28.58	unknown
Sodium	mg/kg	8	0.291	0.726	1.432

table 3: reproducibilities of results of sample 0336

Without further statistical calculations it can be concluded that for many tests there is not a good compliance of the group of participating laboratories with the relevant standards. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON WITH RESULTS OF PREVIOUS PROFICIENCY TEST OF MAY 2002

Since 2002 the number of reporting participants has increased from 9 to 18. The absolute number of statistical outliers increased from 3 to 18, which means that the relative percentage of outliers increased from 2.4% to 8.2% of the reported results.

Comparison of the group performances of this year to last year (2002):

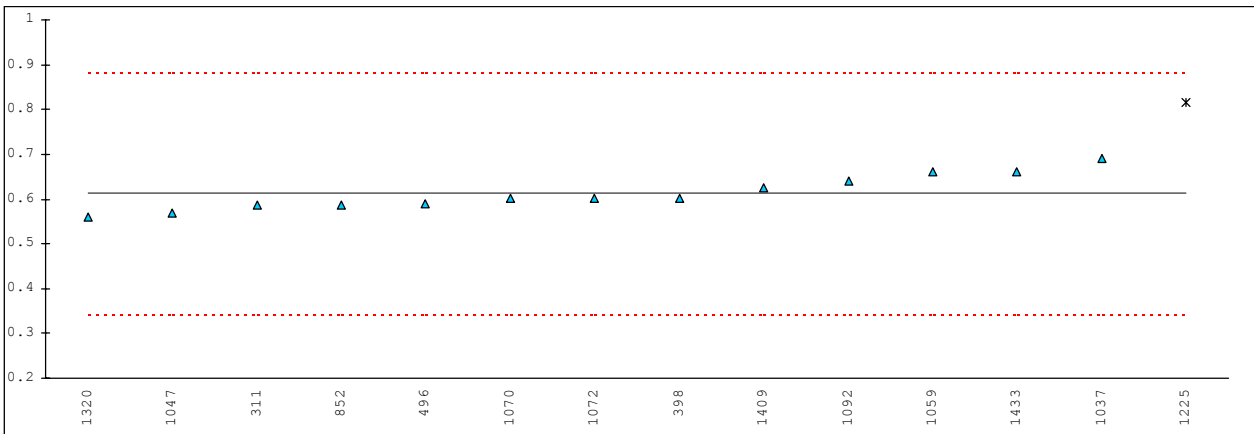
2003 better than 2002	2003 the same as 2002	2003 worse than 2002
Carbon Residue	Oxidation Stability	Cold Filter Plugging Point
Total Contamination	Sulphated Ash	Flash Point PMcc
Density	Glycerol (Free+Total)	Iodine number
Linolein acid-ME	Calcium	Kinematic Viscosity
	Magnesium	Sulphur
	Phosphorous	Glycerides (mono+di+tri)
	Potassium	Total Ester content
	Methanol	Sodium
	Water	

table 7: comparison of the results in 2002 and in 2003

APPENDIX 1

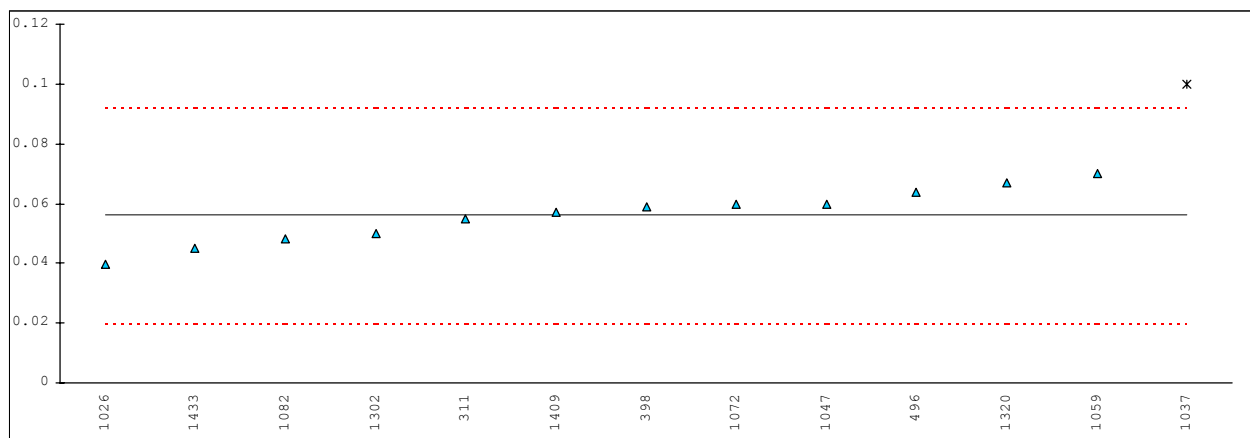
Determination of Acid number on sample 0336; results in mg KOH/gram

lab	method	value	mark	Z(targ)	Remarks
311	D664	0.586		-0.28	
398	EN14104	0.601		-0.13	
496	EN14104	0.59		-0.24	
592		----		----	
852	D974	0.588		-0.26	
1026		----		----	
1035		----		----	
1037	D474	0.691		0.81	
1047	D664	0.57		-0.45	
1059	ISO6619	0.66		0.49	
1070	DIN51558-2	0.60		-0.14	
1072	D664	0.6		-0.14	
1082		----		----	
1092	D664	0.64		0.28	
1225	In house	0.816	G(0.01)	2.10	
1302		----		----	
1320	D664	0.56		-0.55	
1409	D664	0.62416		0.11	
1433	D664	0.6613		0.50	
1435		----		----	
					<u>Only ASTM D664 data:</u>
normality	not OK			OK	
n	13			7	
outliers	1			0	
mean (n)	0.613			0.606	
st.dev. (n)	0.0393			0.0374	
R(calc.)	0.110			0.105	
R(D664:01)	0.270			0.267	



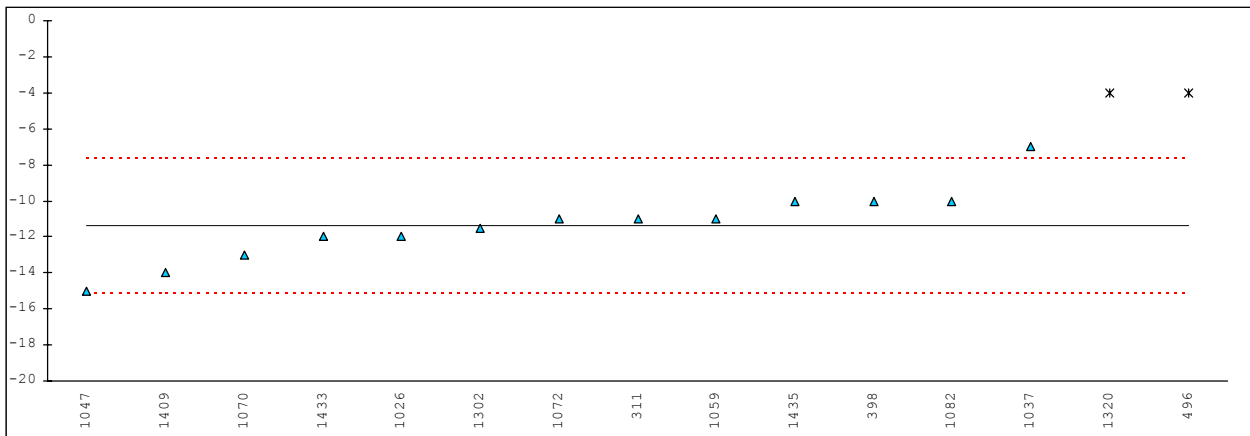
Determination of Carbon Residue on sample 0336; results in %M/M

lab	method	value	mark	Z(targ)	Remarks
311	D4530	0.055		-0.10	
398	D189	0.059		0.21	
496	ISO10370	0.064		0.60	
592		----		----	
852		----		----	
1026	D4530	0.0398		-1.28	
1035		----		----	
1037	PN-C-04075	0.10	G(0.01)	3.40	
1047	ISO10370	0.06		0.29	
1059	ISO10370	0.07		1.07	Measured on 10% distillation residue
1070		----		----	
1072	D4530	0.06		0.29	
1082	ISO10370	0.048		-0.64	
1092		----		----	
1225		----		----	
1302	ISO10370	0.050		-0.49	
1320	D189	0.067		0.84	
1409	ISO10370	0.057		0.06	
1433	D4530	0.0452		-0.86	
1435		----		----	
					<u>Only ISO10370 data:</u>
normality	OK			OK	
n	12			6	
outliers	1			0	
mean (n)	0.0563			0.0582	
st.dev. (n)	0.00906			0.00835	
R(calc.)	0.0254			0.0234	
R(ISO10370:93)	0.0360			0.0368	



Determination of Cold Filter Plugging Point on sample 0336; results in °C

lab	method	value	mark	Z(targ)	Remarks
311	EN116	-11		0.26	
398	EN116	-10		1.01	
496	EN116	-4	DG(0.05)	5.49	
592		----		----	
852		----		----	
1026	EN116	-12		-0.49	
1035		----		----	
1037	PN-C-04114	-7		3.25	
1047	EN116	-15		-2.73	
1059	EN116	-11		0.26	
1070	EN116	-13		-1.24	
1072	IP309	-11		0.26	
1082	EN116	-10		1.01	
1092		----		----	
1225		----		----	
1302	EN116	-11.5		-0.12	
1320	EN116	-4	DG(0.05)	5.49	
1409	EN116	-14		-1.98	
1433	EN116	-12		-0.49	
1435	IP309	-10		1.01	
					<u>Only EN116 data:</u>
normality	OK			OK	
n	13			10	
outliers	2			2	
mean (n)	-11.3			-12.0	
st.dev. (n)	2.01			1.64	
R(calc.)	5.6			4.6	
R(EN116:97)	3.7			3.8	

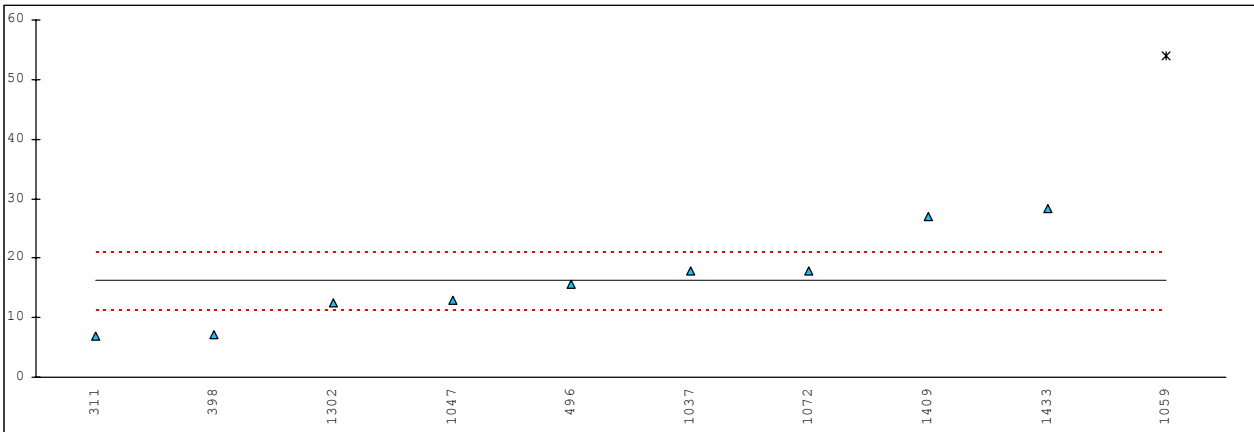


Determination of Copper Corrosion on sample 0336

lab	method	value	mark	Z(targ)	Remarks
311	D130	1A		----	
398	EN2160	1A		----	
496	ISO2160	1A		----	
592		----		----	
852		----		----	
1026	D130	1A		----	
1035		----		----	
1037	D130	1		----	
1047	ISO2160	1A		----	
1059	ISO2160	1A		----	
1070		----		----	
1072	D130	1A		----	
1082	ISO2160	3A		----	False positive?
1092		----		----	
1225		----		----	
1302	ISO2160	1		----	
1320	ISO2160	1A		----	
1409	ISO2160	1A		----	
1433	D130	1A		----	
1435		----		----	
	normality	n.a.			
	n	13			
	outliers	0			
	mean (n)	1A			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D130:00)	Unknown			

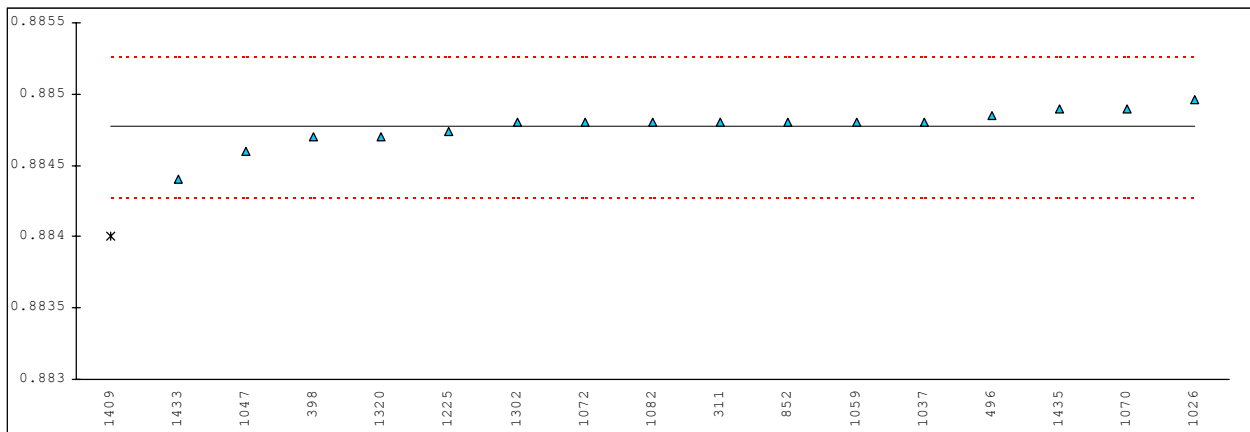
Determination of Total Contamination on sample 0336; results in mg/kg

lab	method	value	mark	Z(targ)	Filter type	Remarks
311	EN12662	7.0		-5.32	S&S me27 Membrane 0.8 um	
398	EN12662	7.09		-5.26	Cellulose nitrate 0.8 um	
496	EN12662	15.6		-0.38	Cellulose nitrate 0.8 um Sartorius	
592		----		----		
852		----		----		
1026		----		----		
1035		----		----		
1037	EN12662	17.888		0.93	Millipore 0.8 um	
1047	EN12662	13.0		-1.87	Cellulose nitrate 0.8 um Whatman	
1059	EN12662	54	G(0.05)	21.65	Millipore	
1070		----		----		
1072	DIN51419	17.9		0.94	0.8 um White AAWP04700	
1082		----		----		
1092		----		----		
1225		----		----		
1302	EN12662	12.5		-2.16	Sartorius	
1320		----		----		
1409	EN12662	27.0		6.16	0.8 um	
1433	In house	28.40		6.96		
1435		----		----		
normality	OK					
n	9					
outliers	1					
mean (n)	16.26					
st.dev. (n)	7.608					
R(calc.)	21.30					
R(EN12662:98)	4.88					



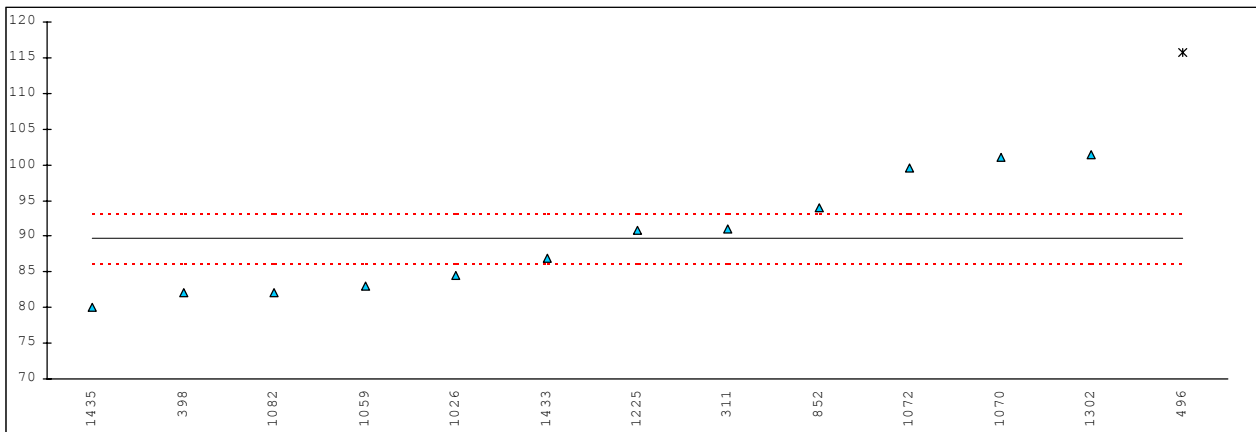
Determination of Density @ 15 °C on sample 0336; results in kg/L

lab	method	value	mark	Z(targ)	Remarks
311	D4052	0.8848		0.16	
398	EN3675	0.8847		-0.40	
496	ISO12185	0.88485		0.44	
592		-----		-----	
852	D4052	0.8848		0.16	
1026	D4052	0.88496		1.05	
1035		-----		-----	
1037	D4052	0.8848		0.16	
1047	ISO12185	0.8846		-0.96	
1059	ISO12185	0.8848		0.16	
1070	DIN51757	0.8849		0.72	
1072	D4052	0.8848		0.16	
1082	ISO12185	0.8848		0.16	
1092		-----		-----	
1225	D4052	0.88474		-0.18	
1302	ISO12185	0.8848		0.16	
1320	ISO12185	0.8847		-0.40	
1409	ISO3675	0.884	G(0.01)	-4.32	
1433	D4052	0.8844		-2.08	
1435	D4052	0.8849		0.72	
					<u>Only ASTM D4052/ ISO12185 data:</u>
normality		not OK		not OK	
n		16		14	
outliers		1		0	
mean (n)		0.88477		0.88477	
st.dev. (n)		0.000131		0.000135	
R(calc.)		0.00037		0.00038	
R(D4052:02)		0.00050		0.00050	



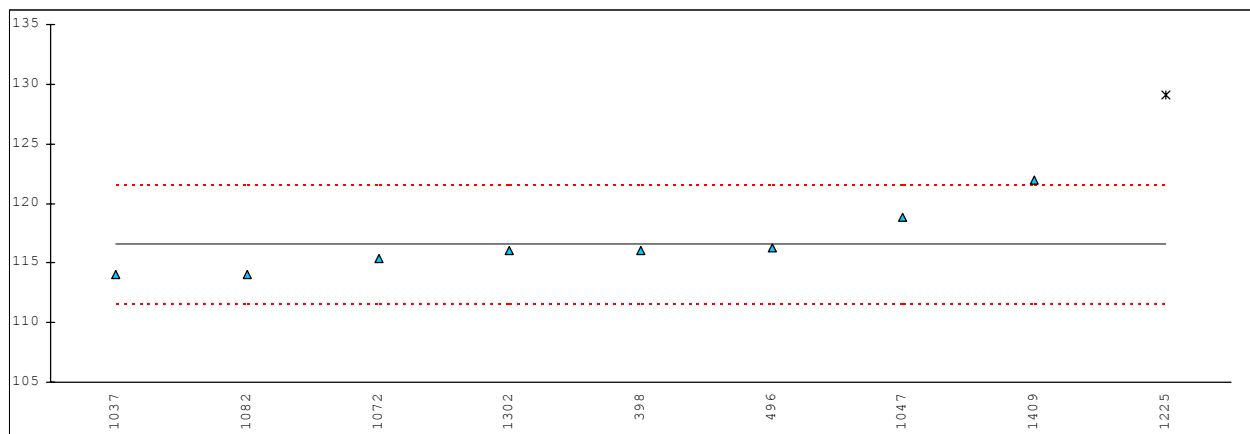
Determination of Flash Point PMcc on sample 0336; results in °C

lab	method	value	mark	Z(targ)	Remarks
311	D93	91.0		1.04	
398	D93	82.0		-6.16	
496	EN22719	115.8	G(0.05)	20.88	
592		----		----	
852	D93	94		3.44	
1026	D93	84.5		-4.16	
1035		----		----	
1037		----		----	
1047		----		----	
1059	EN22719	83		-5.36	Reported results varies between 68-83
1070	D93	101		9.04	
1072	D93	99.5		7.84	
1082	EN22719	82.0		-6.16	
1092		----		----	
1225	D93	90.9		0.96	
1302	EN22719	101.5		9.44	
1320		----		----	
1409		----		----	Reported 186.0 via method ASTM D92 (= COC)
1433	D93	87		-2.16	
1435	D93	80		-7.76	
					<u>Only ASTM D93 data:</u>
normality	OK			OK	
n	12			9	
outliers	1			0	
mean (n)	89.70			89.99	
st.dev. (n)	7.842			7.351	
R(calc.)	21.96			20.58	
R(EN22179:93)	3.50			6.00	



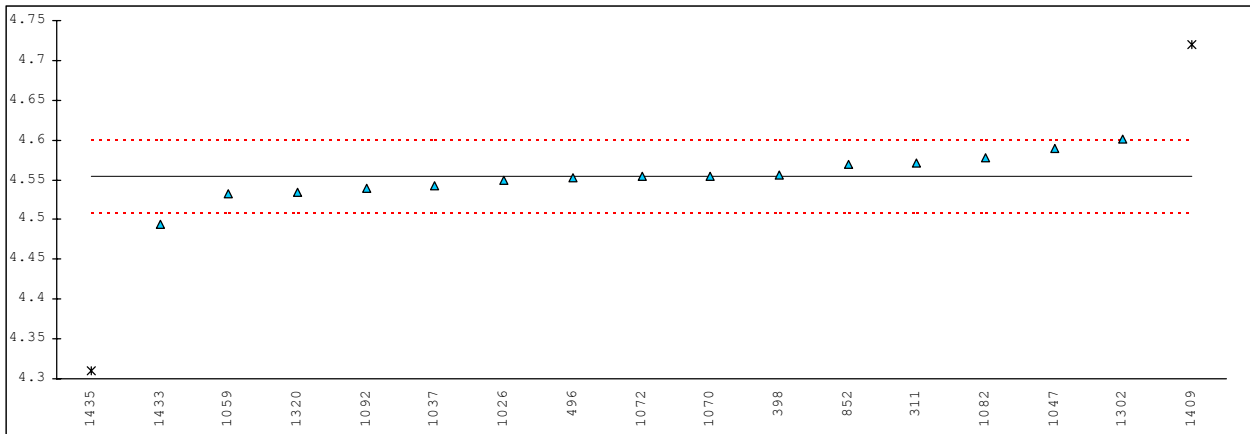
Determination of Iodine Number on sample 0336; results in g I/100g

lab	method	value	mark	Z(targ)	Remarks
311		----		----	
398	EN14111	116.03		-0.31	
496	EN14111	116.3		-0.15	
592		----		----	
852		----		----	
1026		----		----	
1035		----		----	
1037	ISO3961	114.0		-1.44	
1047	EN14111	118.88		1.29	
1059		----		----	
1070		----		----	
1072	D5554	115.4		-0.66	
1082	D5554	114		-1.44	
1092		----		----	
1225	In house	129.1	G(0.05)	7.01	
1302	EN14111	116		-0.32	
1320		----		----	
1409	D1959	122		3.04	
1433		----		----	
1435		----		----	
normality		not OK			
n		8			
outliers		1			
mean (n)		116.58			
st.dev. (n)		2.675			
R(calc.)		7.49			
R(EN14111:01)		5.00			



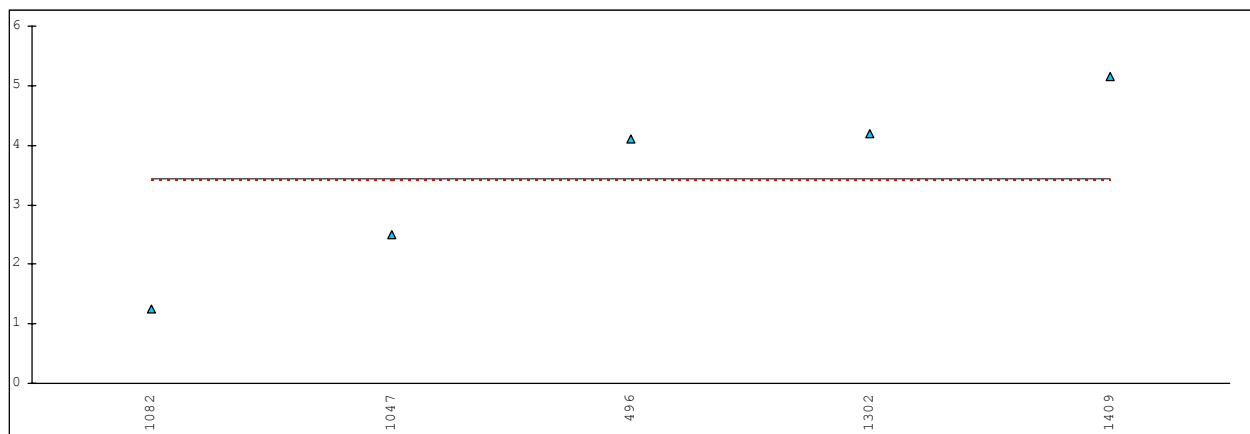
Determination of Kinematic Viscosity @40°C on sample 0336; results in mm²/S

lab	method	value	mark	Z(targ)	Remarks
311	D445	4.5708		0.99	
398	EN3104	4.5562		0.09	
496	EN1304	4.553		-0.11	
592		----		----	
852	D445	4.570		0.94	
1026	D445	4.55		-0.29	
1035		----		----	
1037	D445	4.542		-0.78	
1047	ISO3104	4.59		2.17	
1059	ISO3104	4.532		-1.40	
1070	ISO3104	4.555		0.02	
1072	D445	4.555		0.02	
1082	ISO3104	4.5781		1.44	
1092	D445	4.54		-0.91	
1225		----		----	
1302	DIN51562-1	4.601		2.84	
1320	ISO3104	4.534		-1.27	
1409	ISO3104	4.72	C,G(0.01)	10.16	First reported 4.51
1433	D445	4.494		-3.73	
1435	D445	4.31	G(0.01)	-15.04	
					<u>Only ISO3104 data</u>
normality	OK				OK
n	15				7
outliers	2				1
mean (n)	4.5547				4.5460
st.dev. (n)	0.02600				0.02596
R(calc.)	0.0728				0.0727
R(ISO3104:94)	0.0456				0.0295
					<u>Only ASTM D445 data:</u>
					OK
					7
					1
					4.5460
					0.02596
					0.0727
					0.0295



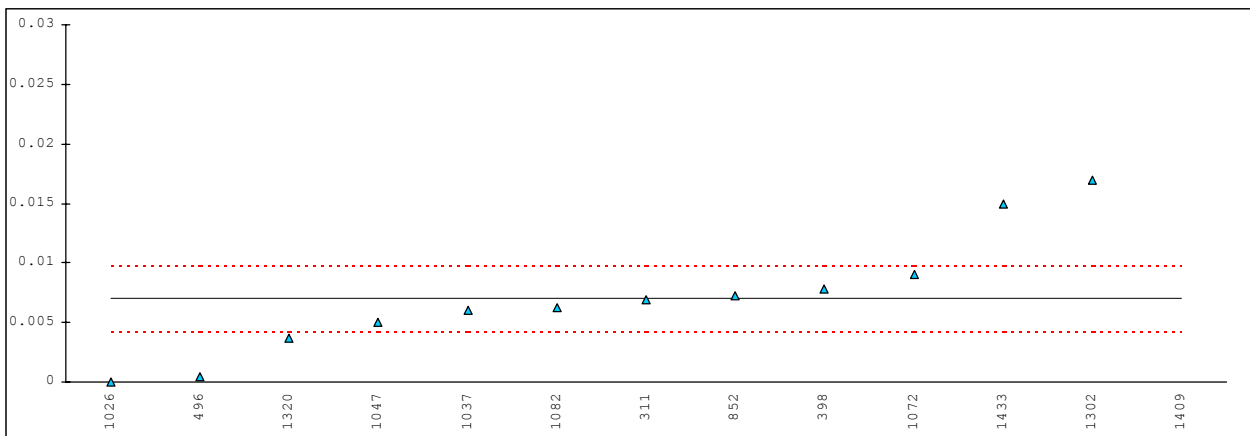
Determination of Oxidation Stability on sample 0336; results in hours

lab	method	value	mark	Z(targ)	Remarks
311		----		----	
398		----		----	
496	EN14112	4.11		----	
592		----		----	
852		----		----	
1026		----		----	
1035		----		----	
1037		----		----	
1047	EN14112	2.5		----	
1059		----		----	
1070		----		----	
1072		----		----	
1082	EN14112	1.26		----	
1092		----		----	
1225		----		----	
1302	EN14112	4.2		----	
1320		----		----	
1409	ISO6886	5.16	C	----	First reported 68
1433		----		----	
1435		----		----	
normality		OK			
n		5			
outliers		0			
mean (n)		3.45			
st.dev. (n)		1.551			
R(calc.)		4.34			
R(EN14112)		unknown			



Determination of Sulphated Ash on sample 0336; results in %M/M

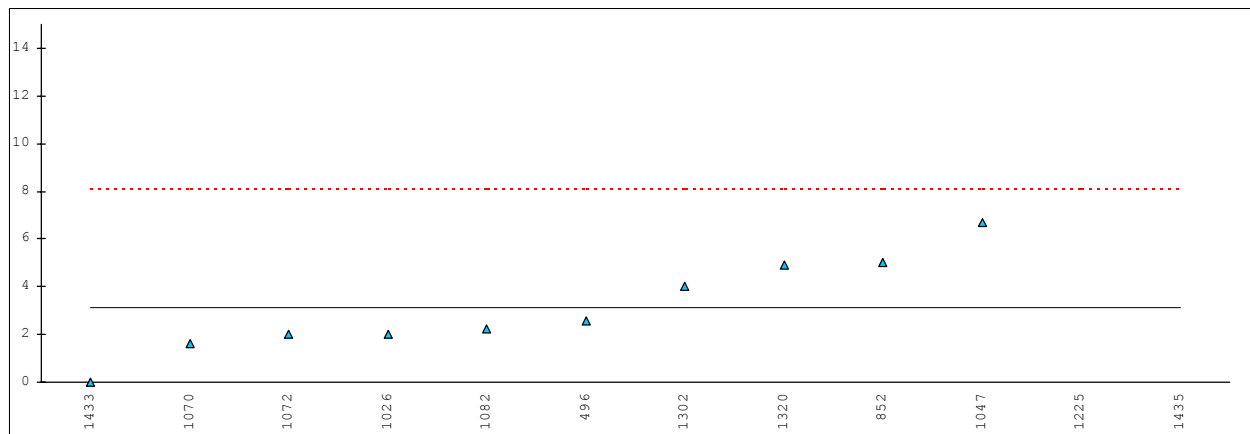
lab	method	value	mark	Z(targ)	Remarks
311	D874	0.0069		-0.13	
398	EN3987	0.0078		0.78	
496	ISO3987	0.0004		-6.64	
592		----		----	
852	D874	0.0072		0.18	
1026	D874	0.000		-7.04	
1035		----		----	
1037	PN-C-04077	0.006		-1.03	
1047	ISO3987	0.005		-2.03	
1059		----		----	
1070		----		----	
1072	D482	0.009		1.98	
1082	D874	0.0063		-0.73	
1092		----		----	
1225		----		----	
1302	ISO3987	0.017		10.00	
1320	D874	0.0037		-3.33	
1409	D874	0.0773	C,G(0.01)	70.44	First reported 0.0991
1433	D874	0.015		7.99	
1435		----		----	
					<u>Only ASTM D874 data:</u>
normality	OK			OK	
n	12			6	
outliers	1			1	
mean (n)	0.00702			0.00652	
st.dev. (n)	0.005024			0.004960	
R(calc.)	0.01407			0.01389	
R(D874:00)	0.00279			0.00262	



Determination of Sulphur on sample 0336; results in mg/kg

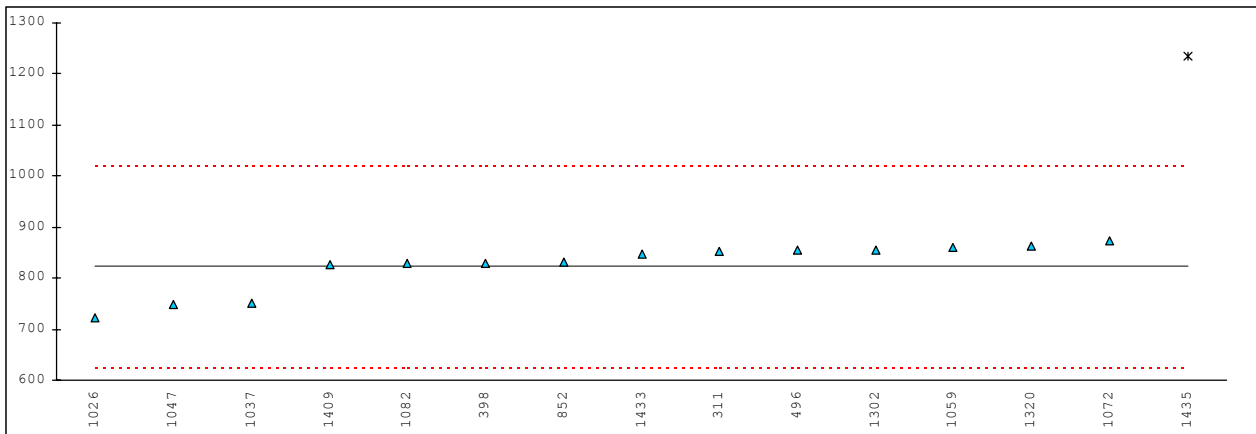
lab	method	value	mark	Z(targ)	Remarks
311	D2622	<3		----	
398				----	
496	ISO20846	2.56		-0.30	
592				----	
852	D2622	5		1.05	
1026	D2622	2		-0.61	
1035				----	
1037				----	
1047	D5453	6.7		1.99	
1059	ISO14596	<10		----	
1070	D2622	1.6		-0.83	
1072	D2622	2		-0.61	
1082	D5453	2.23		-0.48	
1092	X-RAY	<3		----	
1225	D4294	29.75	G(0.01)	14.75	
1302	ISO20846	4		0.50	
1320	D3120	4.9		1.00	
1409	ISO14596	<10		----	
1433	D2622	0		-1.71	
1435	IP336	76	G(0.01)	40.34	
normality		OK			
n		10			
outliers		2			
mean (n)		3.10			
st.dev. (n)		1.999			
R(calc.)		5.60			
R(D2622:98)		5.06			

Compare R(D5453:00) = 1.35



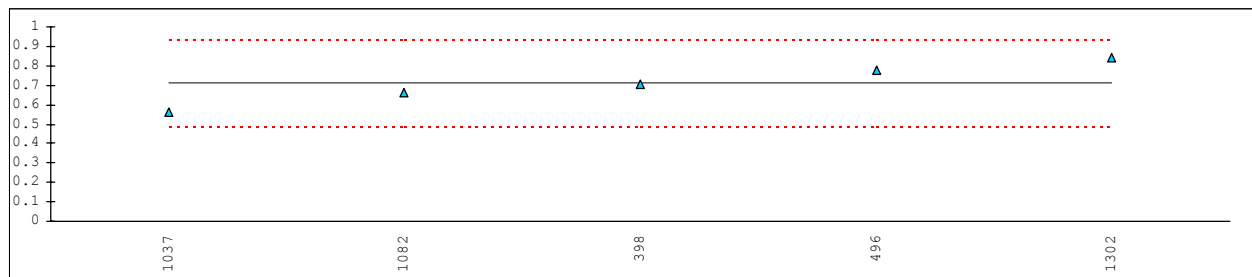
Determination of Water on sample 0336; results in mg/kg

lab	method	value	mark	Z(targ)	Remarks
311	D6304	852		0.39	
398	EN12937	829.6		0.07	
496	ISO12937	854		0.42	
592		----		----	
852	D1744	831.02		0.09	
1026	D6304	722		-1.45	
1035		----		----	
1037	IN HOUSE	750		-1.05	
1047	D1744	749	C	-1.07	First reported 1260
1059	ISO12937	860		0.51	
1070		----		----	
1072	UOP481	872		0.68	
1082	ISO12937	828		0.05	
1092		----		----	
1225		----		----	
1302	ISO12937	856		0.45	
1320	ISO60814	862.4		0.54	
1409	D1744	827		0.04	
1433	D4377	848		0.34	
1435	D1744	1235	G(0.01)	5.82	
					<u>Only ISO12937 data:</u>
normality	not OK			OK	
n	14			5	
outliers	1			0	
mean (n)	824.4			845.5	
st.dev. (n)	48.01			15.43	
R(calc.)	134.4			43.2	
R(ISO12937:00)	197.5			200.0	

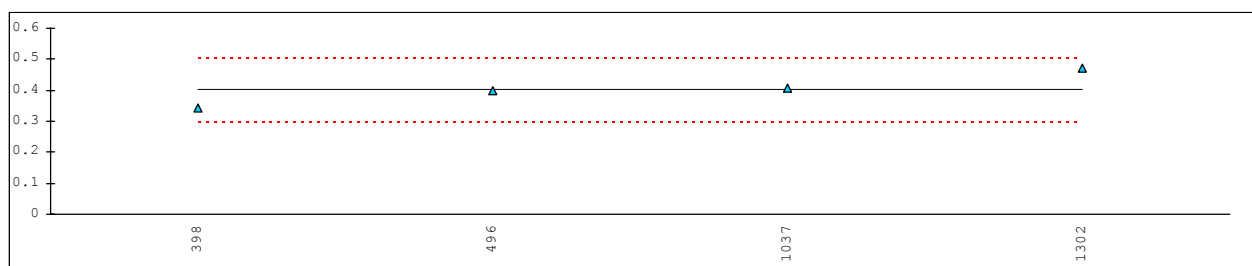


Determination of Glycerides on sample 0336; results in %M/M

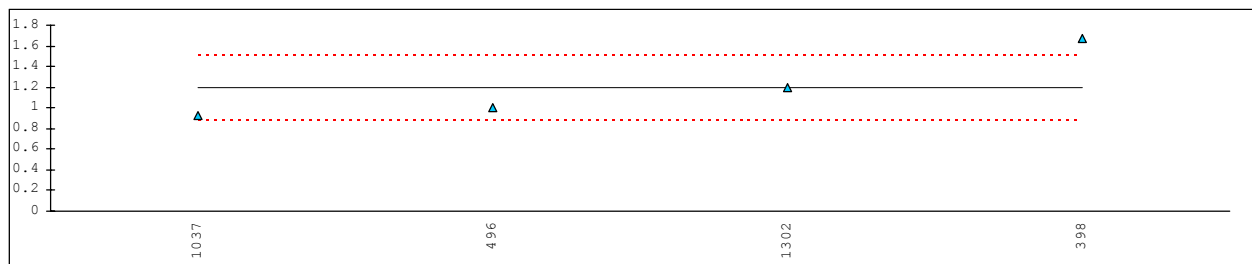
lab	method	Mono glyceride	mark	Z(targ)	Di glyceride	mark	Z(targ)	Tri glyceride	mark	Z(targ)	remarks
311		----		----	----		----	----		----	
398	EN14105	0.707		-0.05	0.342		-1.70	1.672		4.16	
496	EN14105	0.777		0.84	0.398		-0.17	1.007		-1.72	
592		----		----	----		----	----		----	
852		----		----	----		----	----		----	
1026		----		----	----		----	----		----	
1035		----		----	----		----	----		----	
1037	In house	0.564		-1.86	0.405		0.02	0.928		-2.42	
1047		----		----	----		----	----		----	
1059		----		----	----		----	----		----	
1070		----		----	----		----	----		----	
1072		----		----	----		----	----		----	
1082	EN14105	0.662		-0.62	----		----	----		----	
1092		----		----	----		----	----		----	
1225		----		----	----		----	----		----	
1302	EN14105	0.845		1.70	0.472		1.85	1.20		-0.02	
1320		----		----	----		----	----		----	
1409		----		----	----		----	----		----	
1433		----		----	----		----	----		----	
1435		----		----	----		----	----		----	
	normality	OK			OK			OK			
	n	5			4			4			
	outliers	0			0			0			
	mean (n)	0.7110			0.4043			1.2018			
	st.dev. (n)	0.10763			0.05324			0.33367			
	R(calc.)	0.3014			0.1491			0.9343			
	R(EN14105:01)	0.2212			0.1026			0.3164			



Monoglycerides



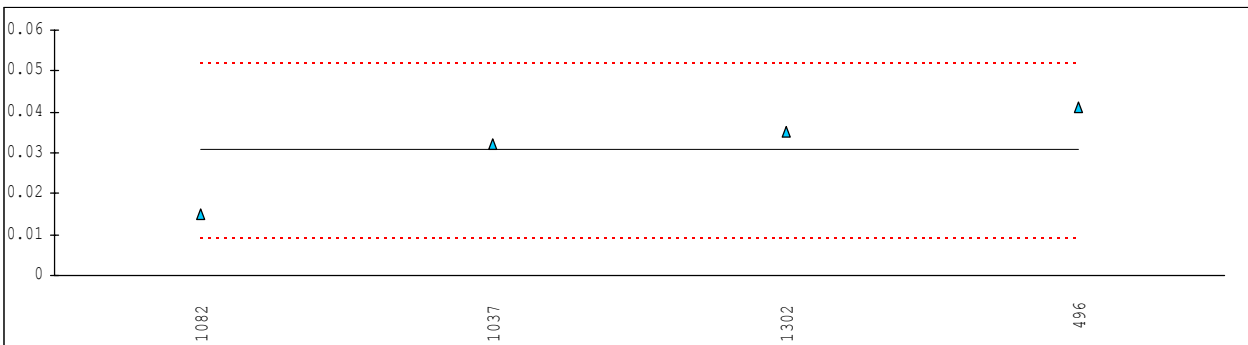
Diglycerides



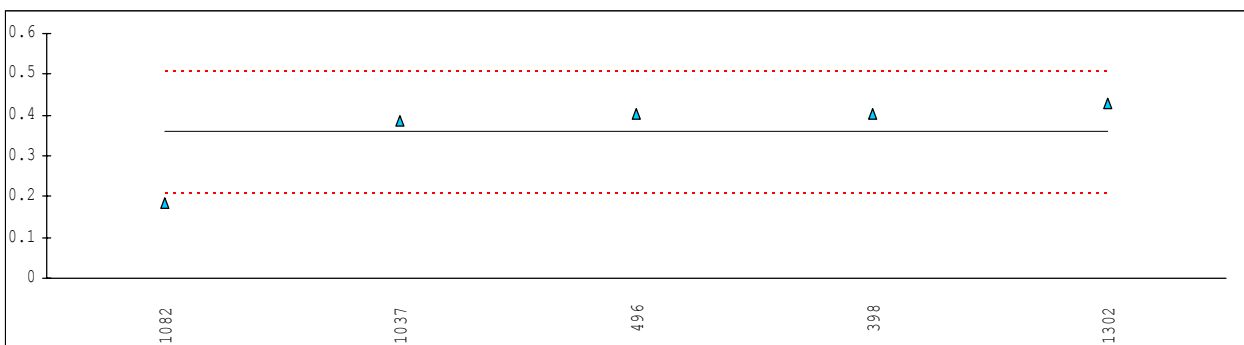
Triglycerides

Determination of Glycerol (Free + Total) on sample 0336; results in %M/M

lab	method	Free glycerol	mark	Z(targ)	Total glycerol	Mark	Z(targ)	remarks
311		----		----	----		----	
398	EN14106	<0.01		----	0.402		0.77	
496	EN14106	0.041		1.34	0.401		0.75	
592		----		----	----		----	
852		----		----	----		----	
1026		----		----	----		----	
1035		----		----	----		----	
1037	In house	0.032		0.16	0.387		0.49	
1047		----		----	----		----	
1059		----		----	----		----	
1070		----		----	----		----	
1072		----		----	----		----	
1082	EN14105	0.015		-2.06	0.183		-3.29	
1092		----		----	----		----	
1225		----		----	----		----	
1302	EN14105	0.035		0.56	0.43		1.28	
1320		----		----	----		----	
1409		----		----	----		----	
1433		----		----	----		----	
1435		----		----	----		----	
normality		OK			not OK			
n		4			5			
outliers		0			0			
mean (n)		0.0308			0.3606			
st.dev. (n)		0.01115			0.10050			
R(calc.)		0.0312			0.2814			
R(EN14105:01)		0.0214			0.1513			



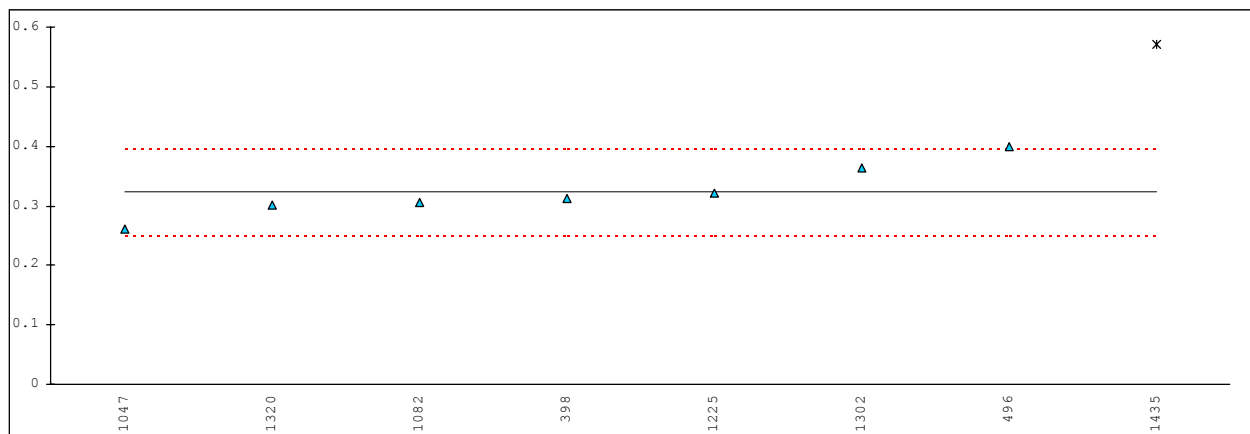
Free glycerol



Total glycerol

Determination of Methanol on sample 0336; results in %M/M

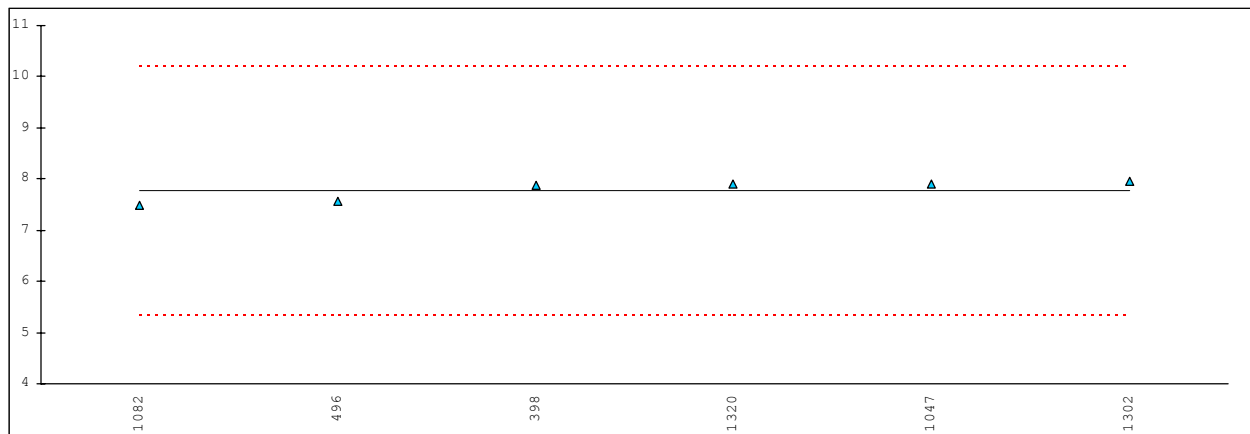
lab	method	value	mark	Z(targ)	Remarks
311		----		----	
398	EN14110	0.3122		-0.42	
496	EN14110	0.40		2.88	
592		----		----	
852		----		----	
1026		----		----	
1035		----		----	
1037		----		----	
1047	EN14110	0.26		-2.38	
1059		----		----	
1070		----		----	
1072		----		----	
1082	EN14110	0.306		-0.65	
1092		----		----	
1225	GC	0.322		-0.05	
1302	EN14110	0.363		1.49	
1320	DIN51413	0.3		-0.88	
1409		----		----	
1433		----		----	
1435	GC	0.57	G(0.05)	9.28	
normality	OK				
n	7				
outliers	1				
mean (n)	0.323				
st.dev. (n)	0.0455				
R(calc.)	0.127				
R(EN14110:01)	0.074				



Determination of Linolein acid-Methylester content on sample 0336; results in %M/M

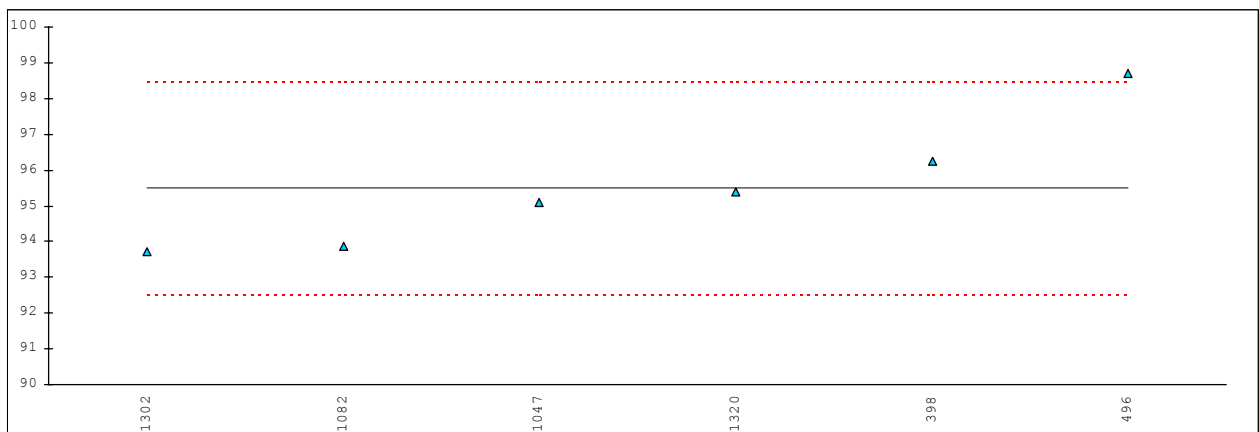
lab	method	value	mark	Z(targ)	Remarks
311		----		----	
398	EN14103	7.89		0.12	
496	EN14103	7.56		-0.26	
592		----		----	
852		----		----	
1026		----		----	
1035		----		----	
1037		----		----	
1047	EN14103	7.91		0.15	
1059		----		----	
1070		----		----	
1072		----		----	
1082	EN14103	7.48		-0.35	
1092		----		----	
1225		----		----	
1302	EN14103	7.96		0.20	
1320	In house	7.9		0.13	
1409		----		----	
1433		----		----	
1435		----		----	

normality not OK
n 6
outliers 0
mean (n) 7.783
st.dev. (n) 0.2070
R(calc.) 0.579
R(EN14103:01) 2.441



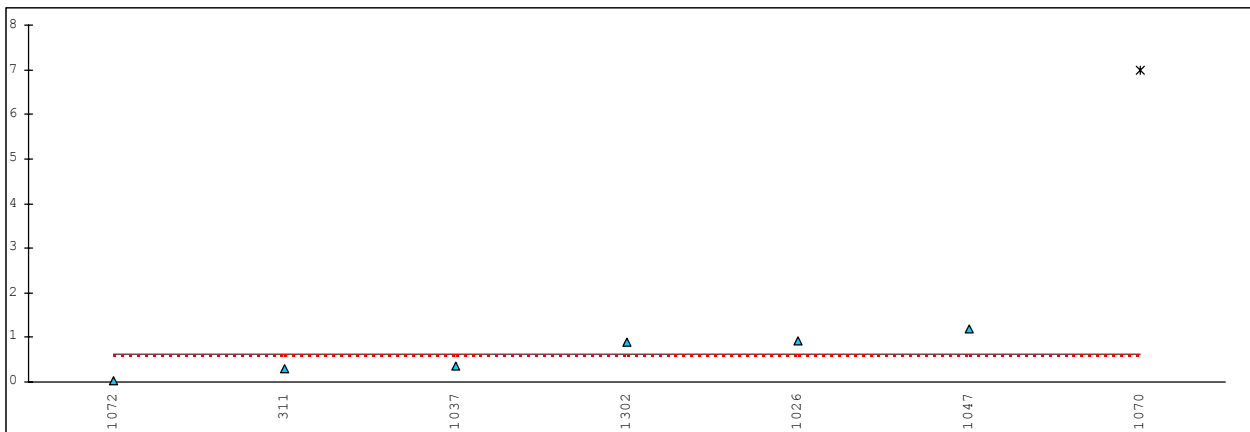
Determination of Total Ester content on sample 0336; results in %M/M

lab	method	value	mark	Z(targ)	Remarks
311		----		----	
398	EN14103	96.229		0.69	
496	EN14103	98.70		3.03	
592		----		----	
852		----		----	
1026		----		----	
1035		----		----	
1037		----		----	
1047	EN14103	95.09		-0.39	
1059		----		----	
1070		----		----	
1072		----		----	
1082	EN14103	93.85		-1.56	
1092		----		----	
1225		----		----	
1302	EN14103	93.72		-1.68	
1320	In house	95.4		-0.09	
1409		----		----	
1433		----		----	
1435		----		----	
normality	OK				
n	6				
outliers	0				
mean (n)	95.498				
st.dev. (n)	1.8352				
R(calc.)	5.139				
R(EN14103:01)	2.960				



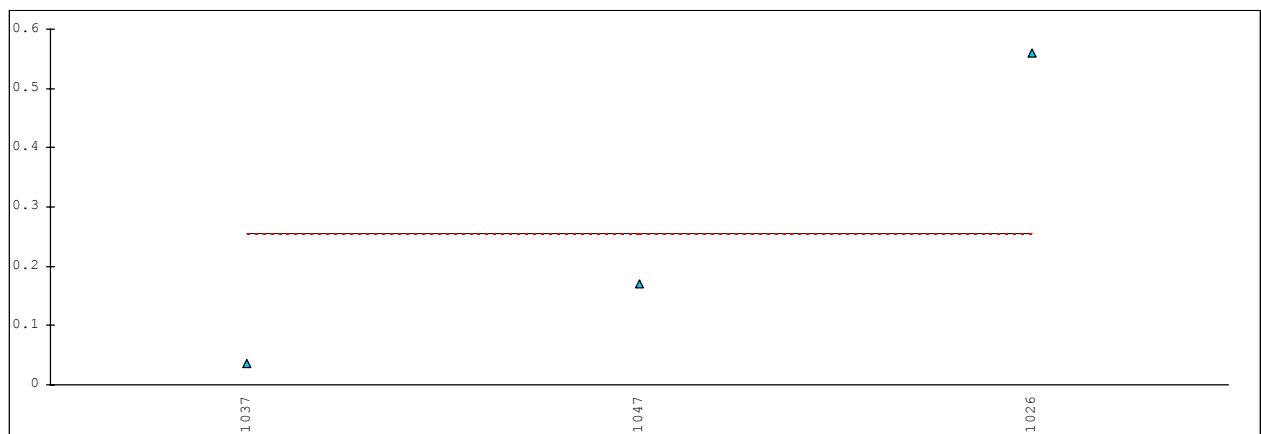
Determination of Calcium on sample 0336; results in mg/kg

lab	Method	value	mark	Z(targ)	Remarks
311	In house	0.3		----	
398	EN14108	<0.1		----	
496	EN14538	<1.0		----	
592		----		----	
852		----		----	
1026	EN14538	0.92		----	
1035		----		----	
1037	PN-V-04030	0.352		----	
1047	D5185	1.2		----	
1059		----		----	
1070	DIN51790	7	G(0.01)	----	
1072	In house	0.03		----	
1082	In house	<1		----	
1092	X-ray	<3		----	
1225		----		----	
1302	EN14538	0.9		----	
1320	D5185	<0.01		----	
1409		----		----	
1433		----		----	
1435		----		----	
normality		OK			
n		6			
outliers		1			
mean (n)		0.617			
st.dev. (n)		0.4532			
R(calc.)		1.269			
R(lit)		unknown			Compare R(iis2002) = 0.700



Determination of Magnesium on sample 0336; results in mg/kg

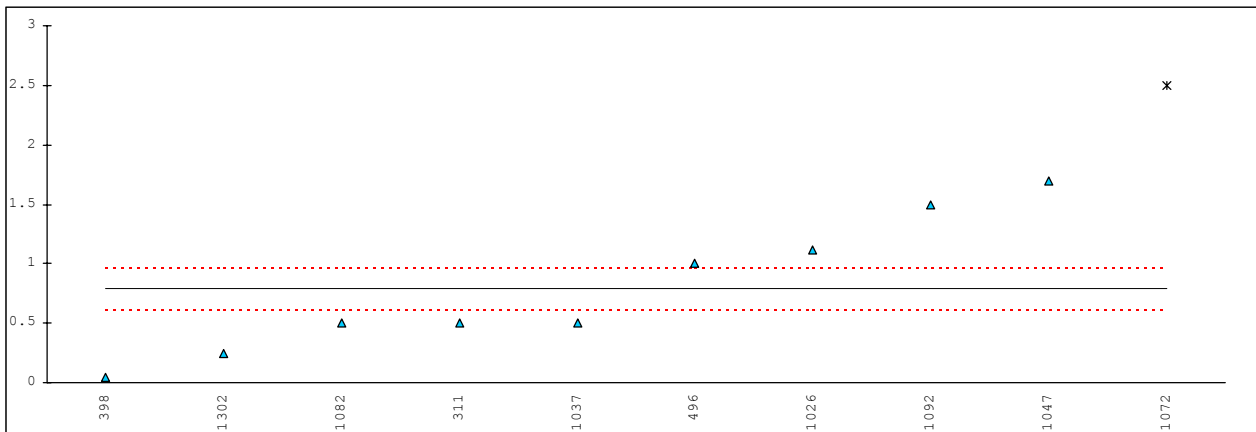
lab	Method	value	mark	Z(targ)	Remarks
311	In house	<1		----	
398	EN14108	<0.1		----	
496	EN14108	<1.0		----	
592		----		----	
852		----		----	
1026	EN14538	0.56		----	
1035		----		----	
1037	PN-V-04030	0.035		----	
1047	D5185	0.17		----	
1059		----		----	
1070		----		----	
1072	In house	<0.01		----	
1082	In house	<1		----	
1092	X-ray	<30		----	
1225		----		----	
1302	EN14538	<0.5		----	
1320	D5185	<0.01		----	
1409		----		----	
1433		----		----	
1435		----		----	
	normality	not OK			
	n	3			
	outliers	0			
	mean (n)	0.255			
	st.dev. (n)	0.2726			
	R(calc.)	0.763			
	R(lit)	unknown			
					Compare R(iis2002) = 0.139



Determination of Phosphorous on sample 0336; results in mg/kg

lab	Method	value	mark	Z(targ)	Remarks
311	In house	<1		-4.59	
398	EN14107	<0.1		-11.71	
496	EN14107	<2.0		3.32	
592		----		----	
852		----		----	
1026	EN14107	1.11		5.06	
1035		----		----	
1037	PN-V-04030	0.503		-4.55	
1047	EN14107	1.7		14.39	
1059		----		----	
1070		----		----	
1072	D4927	<5	G(0.01)	27.05	
1082	In house	<1		-4.59	
1092	X-ray	<3		11.23	
1225		----		----	
1302	EN14107	<0.5		-8.55	
1320		----		----	
1409		----		----	
1433		----		----	
1435		----		----	
normality	OK				
n	9				
outliers	1				
mean (n)	0.790				
st.dev. (n)	0.5666				
R(calc.)	1.587				
R(EN14107:01)	0.177				

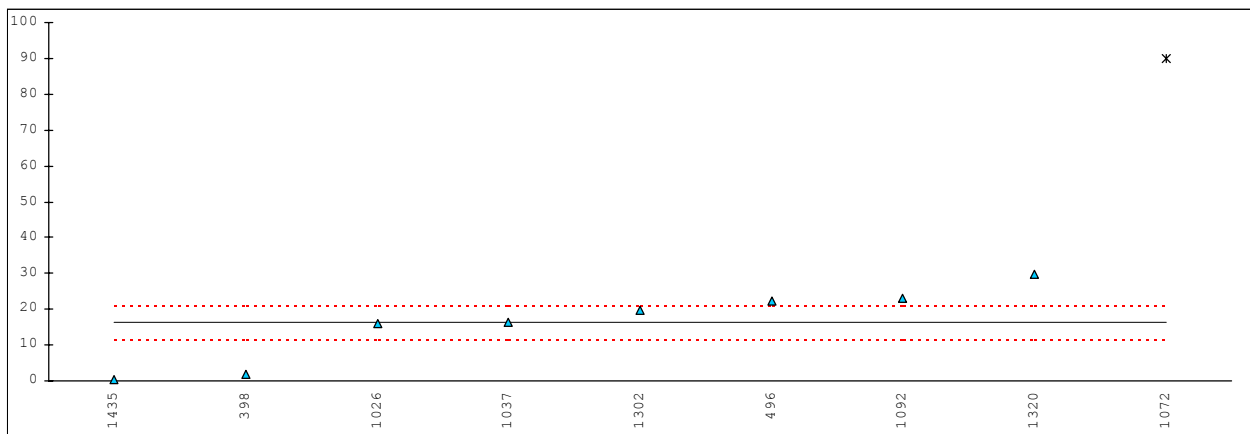
Note: In the calculation of the mean, standard deviation, the reproducibility and the in below graph a reported value of '<X' is changed into 'X/2' (for example <1 into 0.5)



Determination of Potassium on sample 0336; results in mg/kg

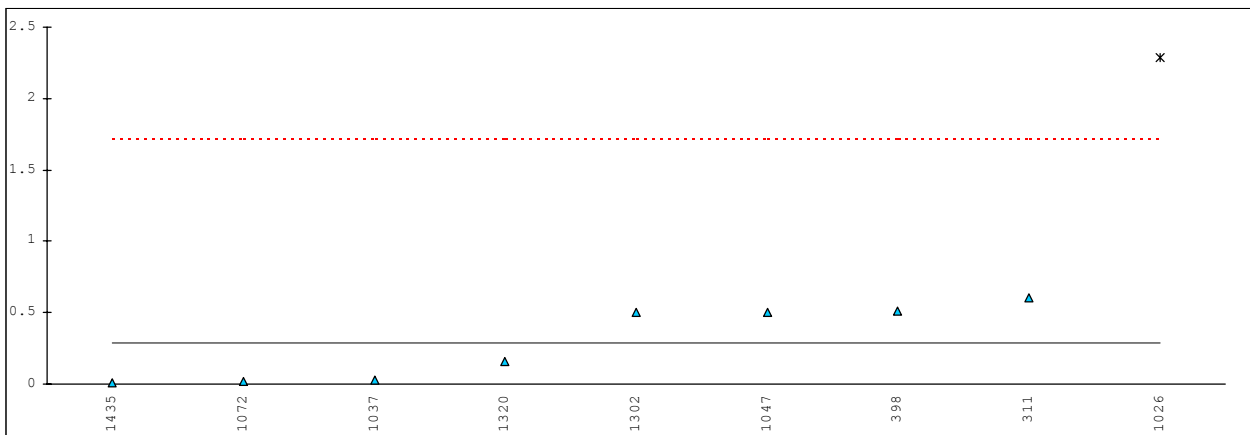
lab	Method	value	mark	Z(targ)	Remarks
311	In house	<1		----	
398	EN14108	2.01		-8.33	
496	EN14538	22.41		3.60	
592		----		----	
852		----		----	
1026	ICP	16.16		-0.06	
1035		----		----	
1037	PN-V-04030	16.32		0.04	
1047	D5185	<0.5	C	----	
1059		----		----	
1070		----		----	
1072	In house	90	G(0.01)	43.11	
1082		----		----	
1092	X-ray	23		3.94	
1225		----		----	
1302	EN14538	19.8		2.07	
1320	D5185	29.8		7.92	
1409		----		----	
1433		----		----	
1435	D5863	0.5432		-9.18	
	normality	OK			
	n	8			
	outliers	1			
	mean (n)	16.26			
	st.dev. (n)	10.207			
	R(calc.)	28.58			
	R(lit)	4.79			

Compare R(D5185:02) = 16.678



Determination of Sodium on sample 0336; results in mg/kg

lab	Method	value	mark	Z(targ)	Remarks
311	In house	0.6		0.60	
398	EN14108	0.511		0.43	
496	EN14538	<1.0		----	
592		----		----	
852		----		----	
1026	ICP	2.29	G(0.01)	3.91	
1035		----		----	
1037	PN-V-04030	0.03		-0.51	
1047	D5185	0.5		0.41	
1059		----		----	
1070		----		----	
1072	In house	0.02		-0.53	
1082		----		----	
1092	X-ray	<100		----	
1225		----		----	
1302	EN14538	0.5		0.41	
1320	D5185	0.16		-0.26	
1409		----		----	
1433		----		----	
1435	D5863	0.0065		-0.56	
	normality	not OK			
	n	8			
	outliers	1			
	mean (n)	0.291			
	st.dev. (n)	0.2594			
	R(calc.)	0.726			
	R(EN14108:01)	1.432			



APPENDIX 2**List of participants on (alphabetical) country order**

Number of labs	Country
2	Austria
2	Belgium
1	Estonia
1	Finland
2	Germany
1	Hong Kong
1	Italy
1	Latvia
1	Mexico
3	Poland
1	Slovakia
1	Slovenia
1	Spain
2	The Netherlands

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect 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
ex	= excluded from calculations
S	= scope of the reported method is not applicable
n.a.	= not applicable
MSDS	= Material Safety Data Sheet

Literature:

- 1 i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation, August 1998
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- 6 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 7 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
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- 10 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
- 11 J.N. Miller, Analyst, 118, 455, (1993)