

**Results of Proficiency Test  
Hydraulic Fluid (used)  
November 2009**

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

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## **1 INTRODUCTION**

Since 2003, the Institute for Interlaboratory Studies organizes a proficiency test for the analysis of used Hydraulic Fluid every year. It was decided to continue the round robin during the annual program 2009/2010. In this international interlaboratory study, 31 laboratories from 20 different countries have participated. See appendix 2 for a list of number of participants per country in alphabetical order. In this report, the results of the interlaboratory study on used hydraulic fluid test are presented and discussed.

## **2 SET UP**

The Institute for Interlaboratory Studies (i.i.s.) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. It was decided to send two different samples of used oils: one sample of 1 litre used ISO VG 68 Hydraulic Fluid and one sample of 0.1 litre used ISO VG 68 Hydraulic Fluid especially for wear metals. Participants were requested to report rounded and unrounded results. The unrounded 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 on ISO guide 43 and ILAC-G13:2007. This ensures 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 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of November 2008 (i.i.s.-protocol, version 3.1).

### **2.3 CONFIDENTIALITY STATEMENT**

All data present 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 bulk materials for the two samples of used Hydraulic Fluid were obtained from a participating laboratory. The approximately 45 litre of the first bulk material was homogenised in a precleaned drum. After homogenisation, 45 subsamples were transferred to 1 litre amber glass bottles, and labelled #0986. The homogeneity of the subsamples #0986

was checked by determination of Density in accordance with ASTM D4052:02e1, Water in accordance with ASTM D6304:07 and Kinematic Viscosity @ 40°C according to ASTM D445:09 on 4 stratified random selected samples.

	<i>Density @ 15 °C in kg/L</i>	<i>Water in %M/M</i>	<i>Kinematic Viscosity at 40°C in mm<sup>2</sup>/s</i>
Sample #0986-1	0.87891	0.0160	64.04
Sample #0986-2	0.87892	0.0166	64.05
Sample #0986-3	0.87893	0.0154	64.06
Sample #0986-4	0.87893	0.0148	64.05

table 1: homogeneity test of subsamples #0986

From the test results of table 1, the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	<i>Density @15°C in kg/L</i>	<i>Water in %M/M</i>	<i>Kinematic Viscosity at 40°C in mm<sup>2</sup>/s</i>
r (Observed)	0.00003	0.0022	0.023
reference method	D4052:02e1	D6304:07	D445:09
0.3 * R (ref. method)	0.00015	0.0125	0.146

table 2: repeatabilities of subsamples #0986

Each calculated repeatability for Density, Viscosity or Water was equal or less than 0.3 times the corresponding reproducibility of respectively ASTM D4052:02e1, D6304:07 and D445:09. Therefore, homogeneity of the subsamples #0986 was assumed.

The approximately 5 litre of the second bulk material, containing wear metals, was homogenised in a precleaned can. After homogenisation, 44 subsamples were transferred to 100 mL HDPE containers, labelled #0987. The homogeneity of the subsamples #0987 was checked by determination of Phosphorus in accordance with ASTM D5185:09 on 4 stratified random selected samples.

	<i>Phosphorus content in mg/kg</i>
Sample #0987-1	379
Sample #0987-2	388
Sample #0987-3	386
Sample #0987-4	386

table 3: homogeneity test of subsamples #0987

From the test results of table 3, the repeatability was calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	<i>Phosphorous content in mg/kg</i>
r (Observed)	11.1
reference method	D5185:09
0.3* R (ref. method)	25.3

table 4: repeatabilities of subsamples #0987

The calculated repeatability for Phosphorus was equal or less than 0.3 times the corresponding reproducibility of ASTM D5185:09. Therefore, homogeneity of the subsamples #0987 was assumed.

One 1 litre amber glass bottle, labelled #0986 and one 100 mL HDPE container, labelled #0987, were despatched to each of the participating laboratories on October 21, 2009.

## 2.5 STABILITY OF THE SAMPLES

The stability of Hydraulic Fluid, packed in the brown glass bottles or in HDPE containers, was checked. The material was found sufficiently stable for the period of the proficiency test.

## 2.6 ANALYSES

The participants were asked to determine Total Acid Number, Density @ 15°C, Flash Point PMcc, Kinematic Viscosity @ 40°C and @ 100°C, Extractable Organic Halogens, Total Organic Halogens and Water on sample #0986 and 20 elements (17 wear metals and 3 additives) on sample #0987.

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 SDS 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 the appendix 1 of this report. The laboratories are presented by their codenumbers.

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 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 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of November 2008 (i.i.s.-protocol, version 3.1).

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 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 under 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. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3, nr.13-14).

### 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, some major problems were encountered during dispatch and execution. Eight laboratories received the samples too late, and six laboratories reported their results after the final reporting date. One laboratory did not report any results at all.

The 30 reporting participants sent in 520 numerical results. Observed were 35 outlying results, which is 6.7% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal distribution. An abnormal distribution was found for the following determination: Kinematic Viscosity @ 40°C, Aluminium, Chromium, Copper, Iron and Vanadium. In this case the statistical evaluation should be used with due care.

##### 4.1 EVALUATION PER TEST

In this section, the results are discussed per test. The methods that 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.

###### **Sample #0986:**

Acid Number (Total): This determination is not problematic. No statistical outliers were detected. The calculated reproducibility is in good agreement with the requirements of ASTM D664:09. One laboratory reported a false negative result.

Density @ 15°C: This determination is very problematic. Six statistical outliers (25% of the reported density results!) were observed. The calculated reproducibility is, even after rejection of the statistical outliers, not at all in agreement with the requirements of ASTM D4052:02e1.

Flash Point PMcc: This determination is problematic. Only one statistical outlier was detected. However, the calculated reproducibility is, after rejection of the

statistical outlier, not in agreement with the requirements of ASTM D93:08.

Kin.Visco.@ 40°C: This determination is problematic. Three statistical outliers were observed. The calculated reproducibility is, after rejection of the statistical outliers, not at all in agreement with the requirements of ASTM D445:09.

Kin.Visco.@ 100°C: This determination is problematic. Four statistical outliers were detected. The calculated reproducibility is, after rejection of the statistical outliers, not at all in agreement with the requirements of ASTM D445:09.

Extractable Organic Halogens: For this determination, only two numerical results were reported. Therefore no significant conclusions were drawn.

Total Organic Halogens: For this determination, only two numerical results were reported. Therefore no significant conclusions were drawn.

Water: This determination is not problematic. No statistical outliers were detected. The calculated reproducibility is in full agreement with the requirements of ASTM D6304:07. The preferred method to use is ASTM D6304:07 method C. This method is applicable for oils with difficult matrix interferences (presence of additives).

**Sample #0987:**

One participant reported false negative and deviating results for nearly all metals, hence all the reported results for metals of this participant were excluded from calculations.

Aluminium: This determination is not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D5185:09.

Barium: As the average concentration found by the group (79.52 mg/kg) is far above the application range given in ASTM D5185:09 table 3 (0.5 – 4 mg/kg), it was decided to use the estimated reproducibility calculated using the Horwitz equation instead of the reproducibility of ASTM D5185:09. This determination seems not problematic. No statistical outliers were observed, and the calculated reproducibility is in good agreement with the estimated requirements calculated using the Horwitz equation.

Chromium: This determination may be problematic. No statistical outliers were detected. However, the calculated reproducibility is not in agreement with the requirements of ASTM D5185:09.

- Copper: This determination is not problematic. Only one statistical outlier was observed. The calculated reproducibility is, after rejection of the statistical outlier, in full agreement with the requirements of ASTM D5185:09.
- Iron: This determination is not problematic. One statistical outlier was detected. The calculated reproducibility is, after rejection of the statistical outlier, almost in agreement with the requirements of ASTM D5185:09.
- Lead: This determination is not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D5185:09.
- Lithium: This determination is not problematic. Only six results were reported, however, one result may be a false negative. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated requirements calculated using the Horwitz equation.
- Magnesium: This determination is not problematic. One statistical outlier was detected. The calculated reproducibility is, after rejection of the statistical outlier, in full agreement with the requirements of ASTM D5185:09.
- Manganese: This determination is not problematic. One statistical outlier was detected. The calculated reproducibility is, after rejection of the statistical outlier, in agreement with the requirements of ASTM D5185:09.
- Molybdenum This determination is not problematic. One statistical outlier was observed. The calculated reproducibility is, after rejection of the statistical outlier, in full agreement with the requirements of ASTM D5185:09
- Nickel: This determination is not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D5185:09.
- Sodium: This determination is problematic for several laboratories. Three statistical outliers were detected. However, the calculated reproducibility is, after rejection of the statistical outlier, in full agreement with the requirements of ASTM D5185:09.
- Silicon: This determination is not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D5185:09.
- Silver: This determination is not problematic. Two outliers were detected. However, the calculated reproducibility is, after rejection of the statistical outliers, in full agreement with re requirements of ASTM D5185:09.

- Tin: This determination is not problematic. Two statistical outliers were observed. However, the calculated reproducibility is, after rejection of the statistical outliers, in full agreement with the requirements of ASTM D5185:09.
- Titanium: This determination is not problematic. One statistical outlier was detected. The calculated reproducibility was, after rejection of the statistical outlier, in good agreement with the requirements of ASTM D5185:09.
- Vanadium: This determination is not problematic for one laboratory. One statistical outlier was observed. The calculated reproducibility is, after rejection of the statistical outlier, in good agreement with the requirements of ASTM D5185:09.
- Calcium: As the average concentration found by the group (29.54 mg/kg) is below the application range given in ASTM D5185:09 table 3 (40-9000 mg/kg), it was decided to use the estimated reproducibility calculated using the Horwitz equation instead of the reproducibility of ASTM D5185:09. This determination is not problematic. After rejection of the two detected outliers, the calculated reproducibility is in agreement with the strict estimated requirements calculated using the Horwitz equation.
- Phosphorus: This determination is not problematic. Two statistical outliers were observed. The calculated reproducibility is, after rejection of the statistical outliers, in full agreement with the requirements of ASTM D5185:09.
- Zinc: This determination is not problematic. Three statistical outliers were detected. The calculated reproducibility is, after rejection of the statistical outliers, in agreement with the requirements of ASTM D5185:09.

#### 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, IP, ISO and EN standards, see tables in appendix 1).

<i>Parameter</i>	<i>Unit</i>	<i>n</i>	<i>Average</i>	<i>2.8 * sd</i>	<i>R(lit)</i>
Total Acid Number	mg KOH/g	22	0.510	0.187	0.224
Density @ 15 °C	kg/L	18	0.87912	0.00087	0.00050
Flash Point PMcc	°C	22	204.74	14.98	10.00
Kinematic viscosity @ 40 °C	mm <sup>2</sup> /s	27	64.296	0.739	0.489
Kinematic viscosity @ 100 °C	mm <sup>2</sup> /s	25	10.0997	0.1283	0.0768
Organic Halogens Extractable	mg/kg	2	n.a.	n.a.	n.a.
Total Organic Halogens	mg/kg	2	n.a.	n.a.	n.a.
Water	%M/M	21	0.0113	0.0143	0.0288

table 5: reproducibilities of results of sample #0986.

<i>Parameter</i>	<i>Unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R (lit)</i>
Aluminium as Al	mg/kg	19	15.95	5.12	7.81
Barium as Ba	mg/kg	17	79.52	16.34	18.44
Calcium as Ca	mg/kg	17	29.54	6.04	7.95
Chromium as Cr	mg/kg	20	15.14	5.13	4.25
Copper as Cu	mg/kg	20	35.15	7.12	8.44
Iron as Fe	mg/kg	19	17.74	5.61	5.19
Lead as Pb	mg/kg	20	17.82	4.66	8.46
Lithium as Li	mg/kg	5	19.89	4.99	5.68
Magnesium as Mg	mg/kg	16	25.72	3.07	8.75
Manganese as Mn	mg/kg	13	15.46	2.95	3.48
Molybdenum as Mo	mg/kg	16	15.42	2.18	4.46
Nickel as Ni	mg/kg	20	15.12	5.56	5.83
Phosphorus as P	mg/kg	17	386.8	60.9	84.6
Silicon as Si	mg/kg	18	16.10	5.30	8.57
Sodium as Na	mg/kg	13	16.66	4.30	8.10
Silver as Ag	mg/kg	14	15.51	2.78	5.43
Tin as Sn	mg/kg	17	15.64	3.10	11.55
Titanium as Ti	mg/kg	15	15.16	3.80	8.97
Vanadium as V	mg/kg	18	15.17	4.44	5.58
Zinc as Zn	mg/kg	18	360.6	54.7	53.9

table 6: reproducibilities of results of sample #0987 (results in brackets are compared against the estimated reproducibility calculated using the Horwitz equation)

Without further statistical calculations, it could be concluded that for some tests there is 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 OF THE PROFICIENCY TEST OF NOVEMBER 2009 WITH THE PREVIOUS PTs.

	<i>November 2009</i>	<i>November 2008</i>	<i>November 2007</i>	<i>November 2006</i>
Number of reporting labs	30	25	24	18
Number of results reported	520	418	407	338
Statistical outliers	35	32	26	38
Percentage outliers	6.7%	7.7%	6.4%	11.2%

table 7: comparison with previous proficiency tests.

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given the following table:

Determination	<i>November 2009</i>	<i>November 2008</i>	<i>November 2007</i>	<i>November 2006</i>
Total Acid Number	++	++	(--)	+
Density @ 15 °C	--	--	--	--
Flash Point PMcc	--	--	++	--
Kinematic viscosity @ 40 °C	--	--	--	--
Kinematic viscosity @ 100 °C	--	--	--	--
Organic Halogens Extractable	n.e.	n.e.	n.e.	n.e.
Total Organic Halogens	n.e.	n.e.	n.e.	n.e.
Water	++	++	++	++
Aluminium as Al	++	++	++	++
Barium as Ba	(++)	(++)	(++)	++ *)
Calcium as Ca	++	--	(--)	n.e. *)
Chromium as Cr	-	++	++	++
Copper as Cu	++	+/-	++	-
Iron as Fe	+	++	++	++
Lead as Pb	++	++	++	++ *)
Lithium as Li	+	(++)	++	n.e.
Magnesium as Mg	++	++	++	++
Manganese as Mn	++	++	++	++
Molybdenum	++	n.e.	n.e.	n.e.
Nickel as Ni	+	+/-	++	++
Phosphorus as P	++	-	--	-- *)
Silicon as Si	++	++	++	++
Sodium as Na	++	++	++	++
Silver as Ag	++	n.e.	n.e.	n.e.
Tin as Sn	(++)	++	++	++ *)
Titanium as Ti	++	n.e.	n.e.	n.e.
Vanadium as V	++	++	++	++
Zinc as Zn	+	--	(++)	n.e. *)

table 8: comparison determinations against the standard

\*) to be used with care, result was lower (or above) than application range of reference method.

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

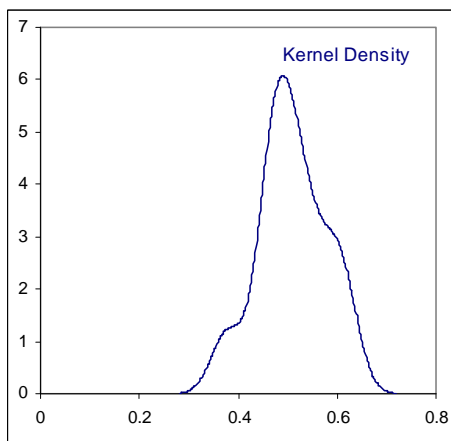
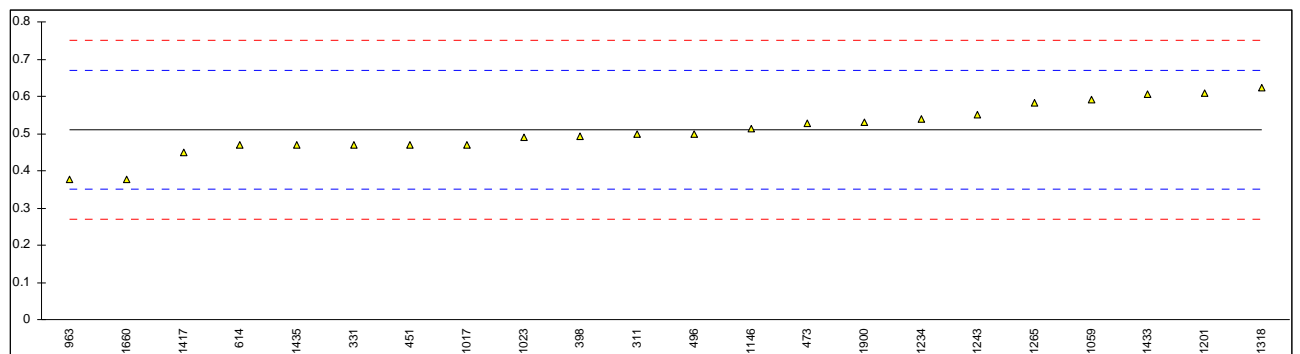
**APPENDIX 1**

Determination of Acid Number (Total) on sample #0986; results in mg KOH/g.

lab	method	value	mark	z(targ)	remarks
255	D664	<0.05	C	<-5.74	False negative? First reported 0.80
311	D664	0.50		-0.12	
317		----		----	
331	D664	0.47		-0.49	
398	D664	0.493		-0.21	
432		----		----	
451	D664	0.47		-0.49	
473	D664mod	0.5281		0.23	
496	D664	0.50		-0.12	
562		----		----	
614	D974	0.47		-0.49	
963	D974	0.3773		-1.65	
1017	D974	0.4703		-0.49	
1023	D664	0.49		-0.24	
1059	ISO6619	0.59		1.00	
1146	D664	0.514		0.06	
1189		----		----	
1201	D664	0.61		1.25	
1234	D664	0.54		0.38	
1243	D664	0.55		0.51	
1258		----		----	
1265	D664	0.5813		0.90	
1318	D664	0.624		1.43	
1417	INHOUSE	0.45		-0.74	
1433	D664	0.6044		1.18	
1435	D664	0.47		-0.49	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D664	0.378		-1.64	
1900	D664	0.53		0.26	

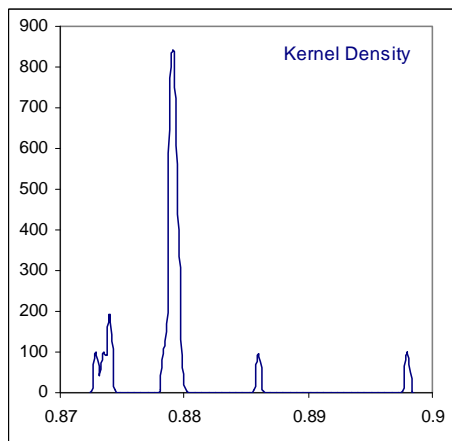
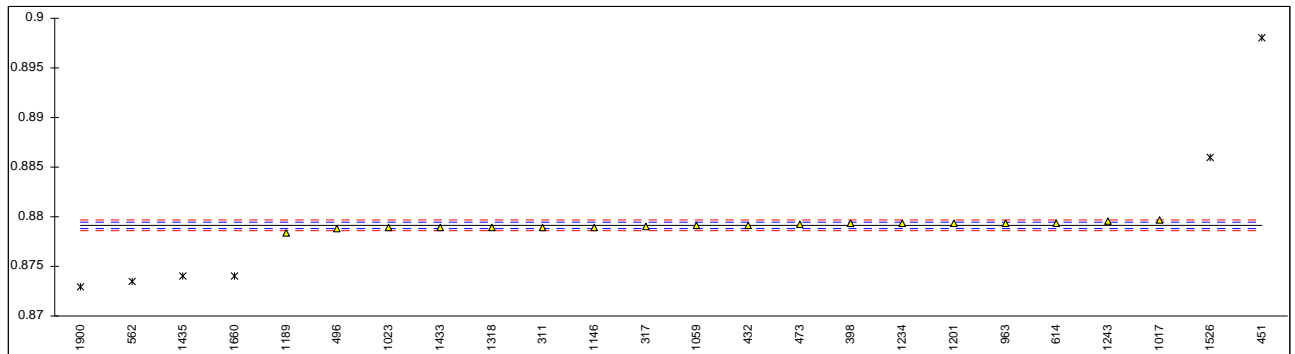
normality OK  
 n 22  
 outliers 0  
 mean (n) 0.510  
 st.dev. (n) 0.0669  
 R(calc.) 0.187  
 R(D664:09) 0.224

Compare to R(D974) = 0.0764



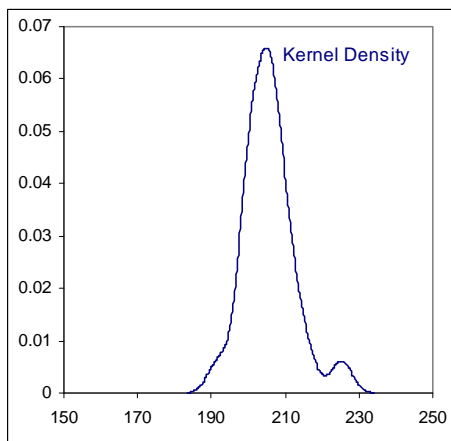
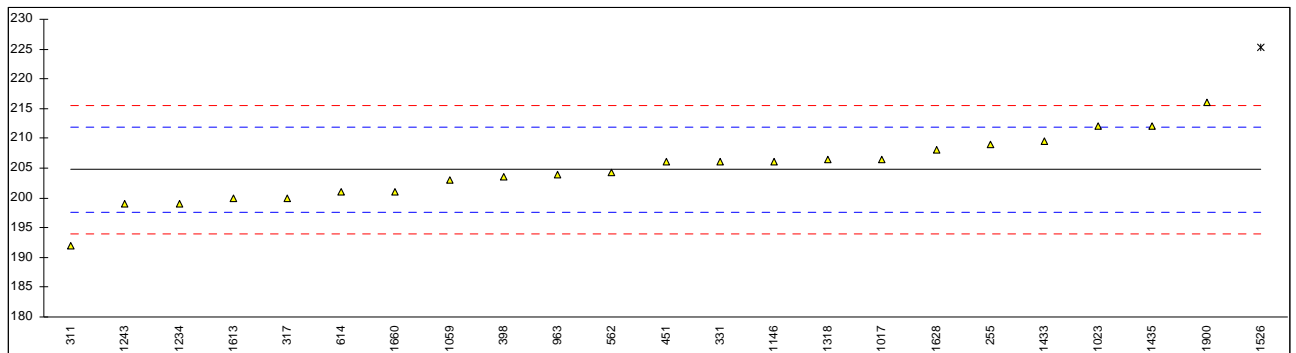
Determination of Density @ 15°C on sample #0986; results in kg/L.

lab	method	value	mark	z(targ)	remarks
255		-----		-----	
311	D4052	0.8789		-1.21	
317	D4052	0.8790		-0.65	
331		-----		-----	
398	D4052	0.8793		1.03	
432	D4052	0.87917		0.30	
451	D4052	0.898	C,G(0.01)	105.75	First reported 0.8761
473	D4052	0.8792		0.47	
496	D4052	0.87880		-1.77	
562	D1298	0.8735	C,G(0.05)	-31.45	First reported 0.8650
614	D4052	0.8794		1.59	
963	D4052	0.8793		1.03	
1017	D4052	0.8797		3.27	
1023	D4052	0.8789		-1.21	
1059	D4052	0.8791		-0.09	
1146	D4052	0.87891		-1.15	
1189	D1298	0.8784		-4.01	
1201	D4052	0.8793		1.03	
1234	D4052	0.8793	U,C	1.03	Reported in deviating unit (879.3 kg/m <sup>3</sup> ), first reported 875.4
1243	ISO12185	0.8796		2.71	
1258		-----		-----	
1265		-----		-----	
1318	D4052	0.87890		-1.21	
1417		-----		-----	
1433	D4052	0.8789	C	-1.21	First reported 0.87585
1435	ISO3675	0.874	G(0.01)	-28.65	
1526	INHOUSE	0.886	G(0.01)	38.55	
1613		-----		-----	
1628		-----		-----	
1660	D4052	0.8740	G(0.05)	-28.65	
1900	INHOUSE	0.8729	G(0.05)	-34.81	
normality		OK			
n		18			
outliers		6			
mean (n)		0.87912			
st.dev. (n)		0.000312			
R(calc.)		0.00087			
R(D4052:02e1)		0.00050			



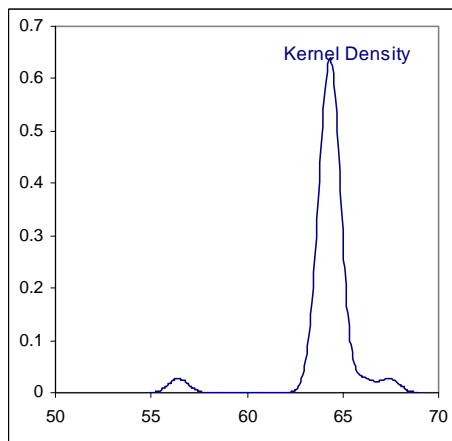
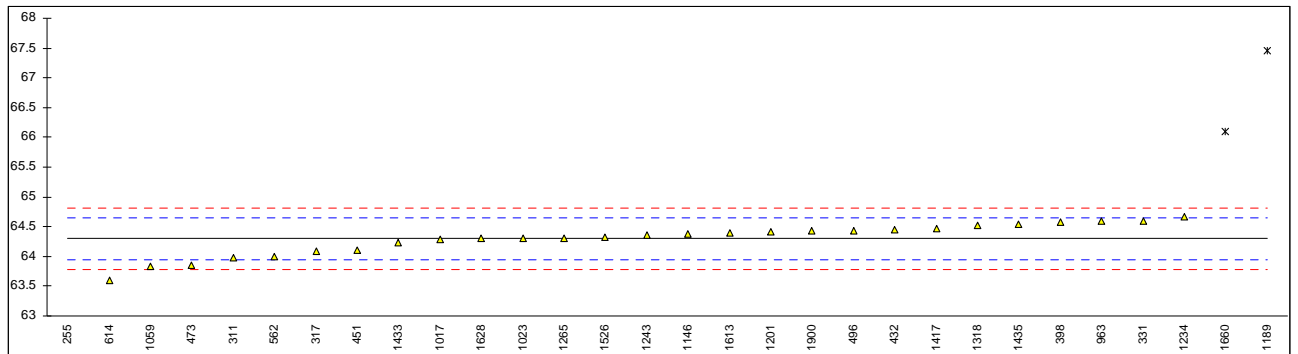
Determination of Flash Point PMcc on sample #0986; results in °C.

lab	method	value	mark	z(targ)	remarks
255	D93	209		1.19	
311	D93	192		-3.57	
317	D93	200.0		-1.33	
331	D93	206.0		0.35	
398	D93	203.5		-0.35	
432		----		----	
451	D93	206		0.35	
473		----		----	
496		----		----	
562	D93	204.3		-0.12	
614	D93	201.0		-1.05	
963	D93	204		-0.21	
1017	D93	206.5		0.49	
1023	D6450	212		2.03	
1059	ISO2719	203.0		-0.49	
1146	INHOUSE	206.0		0.35	
1189		----		----	
1201	D93	>110		----	
1234	D93	199		-1.61	
1243	ISO2719	199		-1.61	
1258		----		----	
1265		----		----	
1318	D93	206.5		0.49	
1417		----		----	
1433	D93	209.5		1.33	
1435	D93	212		2.03	
1526	INHOUSE	225.3	G(0.05)	5.76	
1613	D93	200		-1.33	
1628	D93	208.0		0.91	
1660	D93	201.0		-1.05	
1900	D93	216		3.15	
normality		OK			
n		22			
outliers		1			
mean (n)		204.74			
st.dev. (n)		5.350			
R(calc.)		14.98			
R(D93:08)		10.00			



Determination of Kinematic Viscosity @ 40°C on sample #0986; results in mm<sup>2</sup>.

lab	method	value	mark	z(targ)	remarks
255	D445	56.42	C,G(0.01)	-45.13	First reported 61.94
311	D445	63.98		-1.81	
317	D445	64.08		-1.24	
331	INHOUSE	64.6		1.74	
398	D445	64.572		1.58	
432	D445	64.450		0.88	
451	D7279	64.1		-1.12	
473	D445mod	63.845		-2.59	
496	D445	64.437		0.81	
562	D445	63.989		-1.76	
614	D445	63.600		-3.99	
963	D445	64.595		1.71	
1017	D445	64.2874		-0.05	
1023	D445	64.3		0.02	
1059	ISO3104	63.84		-2.61	
1146	D445	64.385		0.51	
1189	D445	67.45	C,G(0.01)	18.07	First reported 68.15
1201	D445	64.41		0.65	
1234	D445	64.67		2.14	
1243	D7279	64.35		0.31	
1258		-----		-----	
1265	D445	64.30		0.02	
1318	D7042	64.516		1.26	
1417	INHOUSE	64.47		1.00	
1433	D445	64.22976		-0.38	
1435	ISO3104	64.54		1.40	
1526	INHOUSE	64.33		0.19	
1613	D445	64.393		0.55	
1628	ISO3104	64.298		0.01	
1660	D445	66.1	G(0.01)	10.34	
1900	D445	64.43		0.77	
					<u>Only ASTM D445 / ISO3104 data</u>
	normality	not OK			not OK
	n	27			20
	outliers	3			3
	mean (n)	64.296			64.289
	st.dev. (n)	0.2640			0.2708
	R(calc.)	0.739			0.758
	R(D445:09)	0.489			0.489

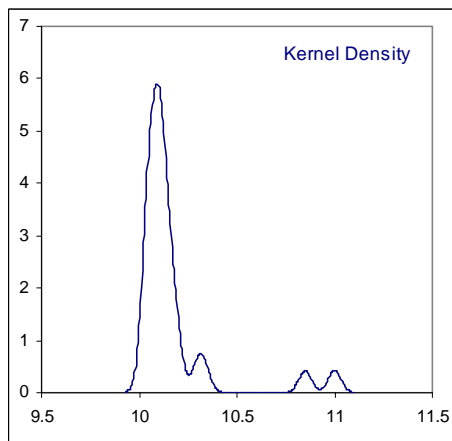
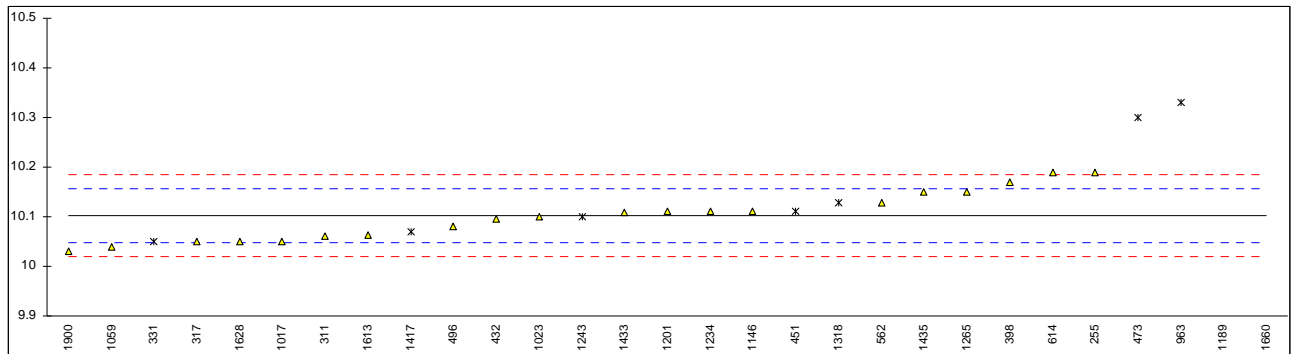


Determination of Kinematic Viscosity @ 100°C on sample #0986; results in mm/s<sup>2</sup>

lab	method	value	mark	z(targ)	remarks
255	D445	10.19	C	3.29	First reported 10.49
311	D445	10.06		-1.45	
317	D445	10.05		-1.81	
331	INHOUSE	10.05		-1.81	
398	D445	10.169		2.53	
432	D445	10.095		-0.17	
451	D7279	10.11		0.38	
473	D445mod	10.2998	G(0.01)	7.30	
496	D445	10.080		-0.72	
562	D445	10.128		1.03	
614	D445	10.189		3.26	
963	D445	10.331	C,G(0.05)	8.44	First reported 9.7157
1017	D445	10.0508		-1.78	
1023	D445	10.1		0.01	
1059	ISO3104	10.04		-2.18	
1146	D445	10.110		0.38	
1189	D445	10.85	C,G(0.01)	27.37	First reported 10.80
1201	D445	10.11		0.38	
1234	D445	10.11		0.38	
1243	D7279	10.10		0.01	
1258		-----		-----	
1265	D445	10.15		1.83	
1318	D7042	10.128		1.03	
1417	INHOUSE	10.07		-1.08	
1433	D445	10.11		0.31	
1435	ISO3104	10.15		1.83	
1526		-----		-----	
1613	D445	10.064		-1.30	
1628	ISO3104	10.0506		-1.79	
1660	D445	11.0	G(0.01)	32.84	
1900	D445	10.03		-2.54	

Only ASTM D445 / ISO3104 data

normality	OK	OK
n	25	20
outliers	4	4
mean (n)	10.0997	10.1017
st.dev. (n)	0.04583	0.04923
R(calc.)	0.1283	0.1379
R(D445:09)	0.0768	0.0768



## Determination of Extractable Organic Chloride on sample #0986; results in mg/kg.

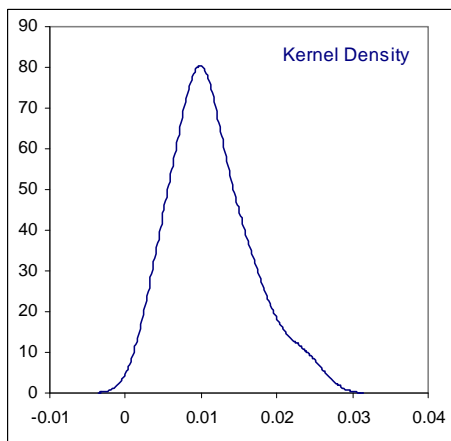
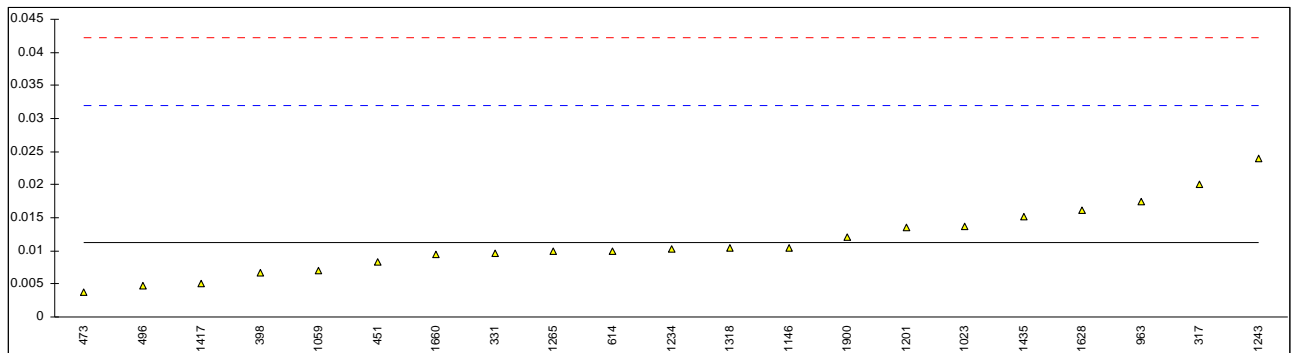
lab	method	value	mark	z(targ)	remarks
255		----		----	
311		----		----	
317		----		----	
331		----		----	
398		----		----	
432		----		----	
451		----		----	
473		----		----	
496		----		----	
562		----		----	
614		----		----	
963		----		----	
1017		----		----	
1023		----		----	
1059		----		----	
1146		----		----	
1189		----		----	
1201		----		----	
1234		----		----	
1243		----		----	
1258		----		----	
1265		----		----	
1318	INHOUSE	3.8		----	
1417		----		----	
1433		----		----	
1435		----		----	
1526	EPA9076	<100		----	
1613		----		----	
1628		----		----	
1660		----		----	
1900	D4929	215		----	
	normality	n.a.			
	n	2			
	outliers	0			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(Horwitz)	n.a.			

## Determination of Total Organic Halogens on sample #0986; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311		----		----	
317	UOP779	2		----	
331		----		----	
398		----		----	
432		----		----	
451		----		----	
473		----		----	
496		----		----	
562		----		----	
614		----		----	
963		----		----	
1017		----		----	
1023		----		----	
1059		----		----	
1146		----		----	
1189		----		----	
1201		----		----	
1234		----		----	
1243	DIN51408	1.2		----	
1258		----		----	
1265		----		----	
1318		----		----	
1417		----		----	
1433		----		----	
1435		----		----	
1526		----		----	
1613		----		----	
1628		----		----	
1660		----		----	
1900		----		----	
	normality	n.a.			
	n	2			
	outliers	0			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(Horwitz)	n.a.			

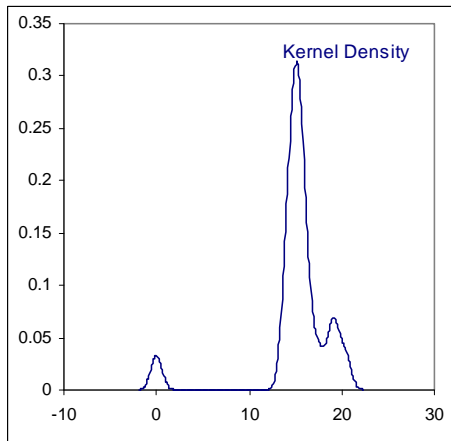
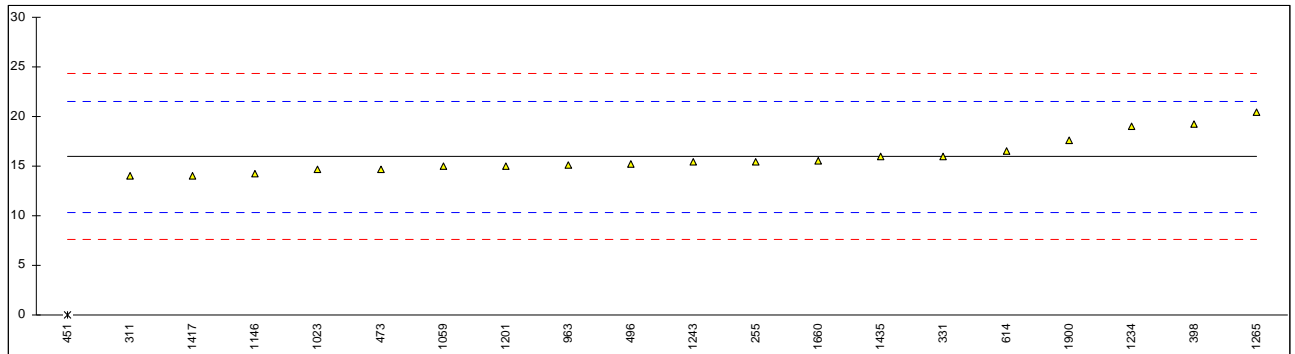
Determination of Water on sample #0986; results in %M/M.

lab	method	value	mark	z(targ)	remarks
255	D95	<0.04		-----	
311	D6304	<0.01		-----	
317	D6304	0.02		0.84	
331	D6304	0.00954		-0.17	
398	D6304	0.00661	C	-0.46	First reported 66.1
432		-----		-----	
451	D6304	0.0083	C	-0.29	First reported 83
473	D6304	0.0038		-0.73	
496	D6304	0.0048		-0.63	
562		-----		-----	
614	D6304	0.01		-0.13	
963	D6304	0.0174		0.59	
1017		-----		-----	
1023	D6304	0.0137		0.23	
1059	D6304mod	0.007		-0.42	
1146	D6304	0.0105		-0.08	
1189		-----		-----	
1201	D6304	0.0135	C	0.21	First reported 135
1234	D6304	0.0102		-0.11	
1243	DIN51777	0.024		1.23	
1258		-----		-----	
1265	D6304	0.01	C	-0.13	First reported 98.83
1318	D6304	0.01047		-0.08	
1417	INHOUSE	0.005		-0.61	
1433		-----		-----	
1435	D1744	0.0151		0.37	
1526	INHOUSE	<0.50		-----	
1613		-----		-----	
1628	KF	0.0162		0.47	
1660	D6304	0.00942	U	-0.18	Reported in deviating unit (94.2 mg/kg)
1900	D6304	0.0120	C	0.07	First reported 0.0094
	normality	OK			
	n	21			
	outliers	0			
	mean (n)	0.0113			
	st.dev. (n)	0.00512			
	R(calc.)	0.0143			
	R(D6304:07)	0.0288			



Determination of Aluminium (Al) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	AAS	15.40		-0.20	
311	D5185	14		-0.70	
317		----		----	
331	D5185	16.0		0.02	
398	D6595	19.2		1.17	
432		----		----	
451	D5185	0	ex	-5.72	False negative? (Zero is not a real value)
473	D5185mod	14.714		-0.44	
496	D5185	15.20		-0.27	
562		----		----	
614	D5185	16.5		0.20	
963	D5185	15.14		-0.29	
1017		----		----	
1023	D5185	14.7		-0.45	
1059	INHOUSE	15		-0.34	
1146	D5185	14.21		-0.62	
1189		----		----	
1201	D5185	15		-0.34	
1234	INHOUSE	19		1.10	
1243	DIN51396	15.4		-0.20	
1258		----		----	
1265	D6595	20.4		1.60	
1318		----		----	
1417	INHOUSE	14.004		-0.70	
1433		----		----	
1435	D5185	16		0.02	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	15.51		-0.16	
1900	D6595	17.58481		0.59	
normality		not OK			
n		19			
outliers		0			
mean (n)		15.95			
st.dev. (n)		1.828			
R(calc.)		5.12			
R(D5185:09)		7.81			

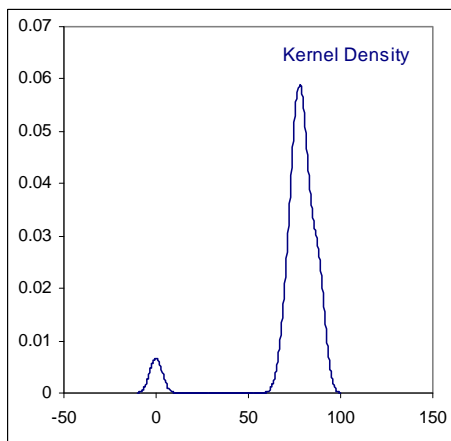
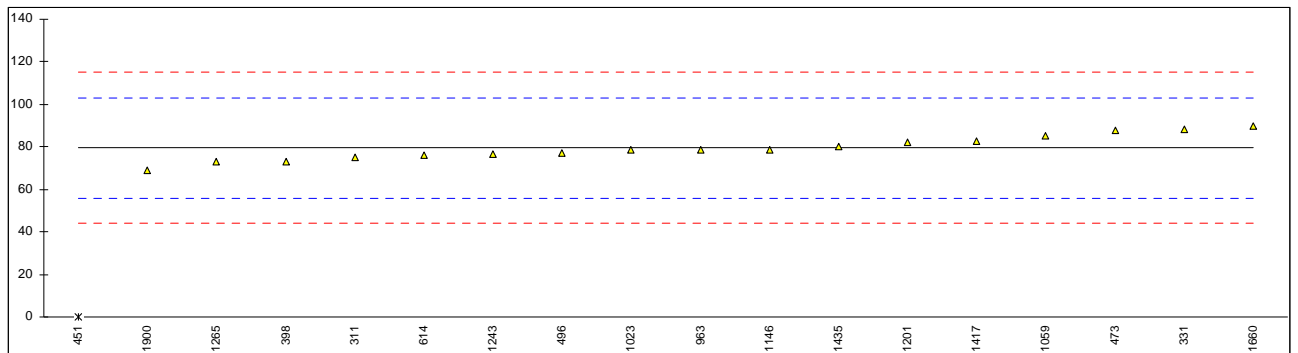


Determination of Barium (Ba) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311	D5185	75		-0.38	
317		----		----	
331	D5185	88.2		0.74	
398	D6595	73.0		-0.55	
432		----		----	
451	D5185	0	ex	-6.74	False negative? (Zero is not a real value)
473	D5185mod	87.831		0.70	
496	D5185	77.32		-0.19	
562		----		----	
614	D5185	76.3		-0.27	
963	D5185	78.61		-0.08	
1017		----		----	
1023	D5185	78.5		-0.09	
1059	INHOUSE	85		0.46	
1146	D5185	78.82		-0.06	
1189		----		----	
1201	D5185	82		0.21	
1234		----		----	
1243	DIN51396	76.5		-0.26	
1258		----		----	
1265	D6595	72.9		-0.56	
1318		----		----	
1417	INHOUSE	82.770		0.28	
1433		----		----	
1435	D5185	80		0.04	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	90.0		0.89	
1900	D6595	69.01953		-0.89	

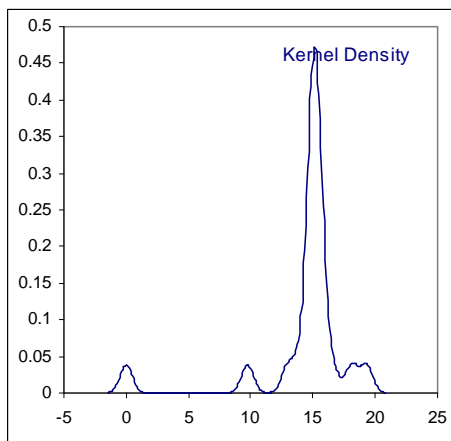
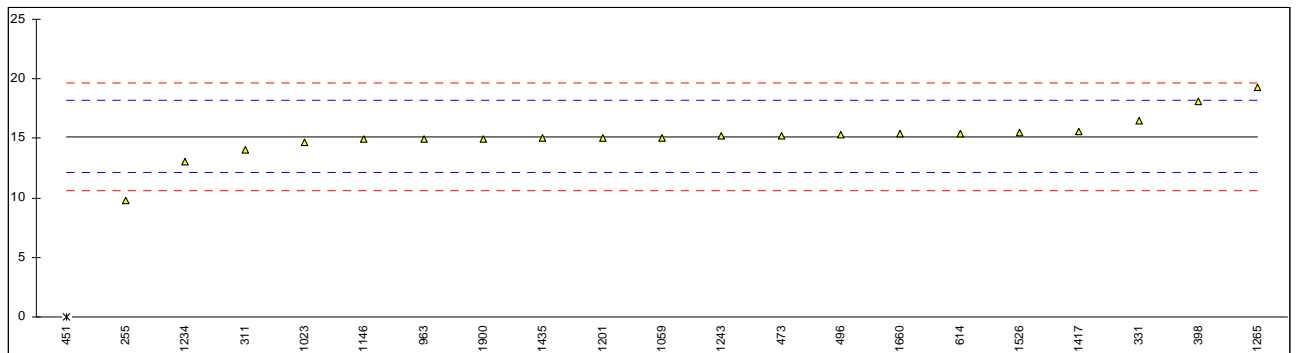
normality OK  
n 17  
outliers 0  
mean (n) 79.52  
st.dev. (n) 5.836  
R(calc.) 16.34  
R(Horwitz) 18.44

Application range = 0.5-4 mg/kg  
Compared with R(D5185) = 33.057 (extrapolated)



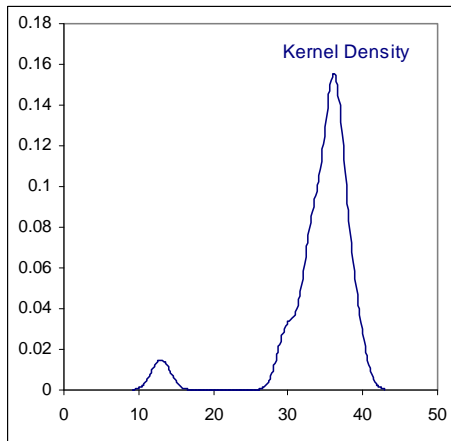
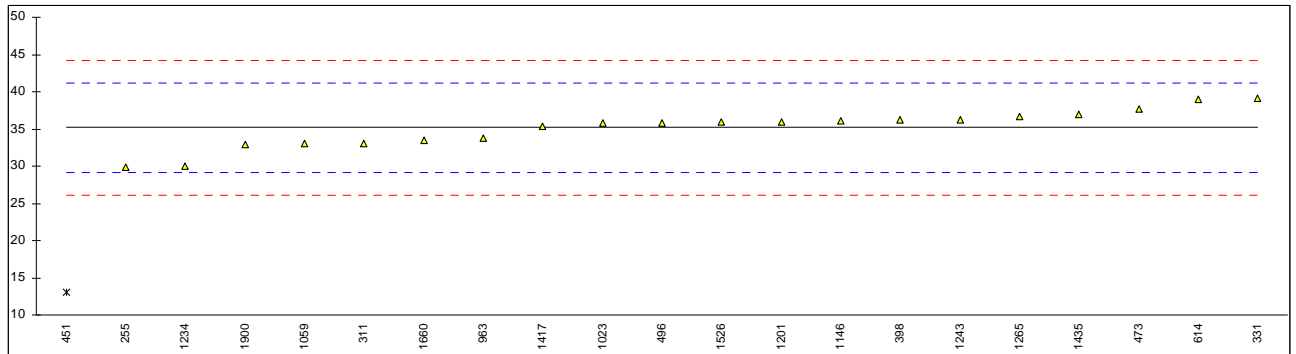
Determination of Chromium (Cr) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	AAS	9.74	C	-3.56	First reported 8.00
311	D5185	14		-0.75	
317		----		----	
331	D5185	16.5		0.90	
398	D6595	18.1		1.95	
432		----		----	
451	D5185	0	ex	-9.98	False negative? (Zero is not a real value)
473	D5185mod	15.251		0.07	
496	D5185	15.35		0.14	
562		----		----	
614	D5185	15.4		0.17	
963	D5185	14.91		-0.15	
1017		----		----	
1023	D5185	14.7		-0.29	
1059	INHOUSE	15		-0.09	
1146	D5185	14.91		-0.15	
1189		----		----	
1201	D5185	15		-0.09	
1234	INHOUSE	13		-1.41	
1243	DIN51396	15.2		0.04	
1258		----		----	
1265	D6595	19.3		2.74	
1318		----		----	
1417	INHOUSE	15.608		0.31	
1433		----		----	
1435	D5185	15		-0.09	
1526	D5185	15.5		0.24	
1613		----		----	
1628		----		----	
1660	D5185	15.39		0.16	
1900	D6595	14.96004		-0.12	
normality		not OK			
n		20			
outliers		0			
mean (n)		15.14			
st.dev. (n)		1.834			
R(calc.)		5.13			
R(D5185:09)		4.25			



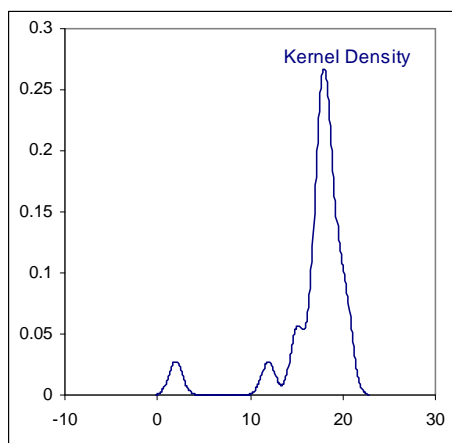
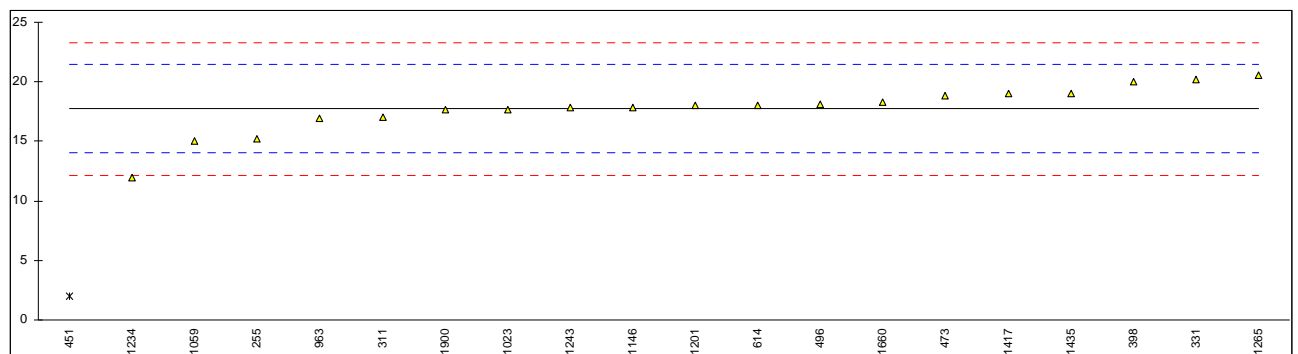
Determination of Copper (Cu) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	AAS	29.80		-1.78	
311	D5185	33		-0.71	
317		----		----	
331	D5185	39.1		1.31	
398	D6595	36.3		0.38	
432		----		----	
451	D5185	13	C,G(0.01)	-7.35	First reported 0
473	D5185mod	37.670		0.84	
496	D5185	35.80		0.22	
562		----		----	
614	D5185	39.0		1.28	
963	D5185	33.74		-0.47	
1017		----		----	
1023	D5185	35.8		0.22	
1059	INHOUSE	33		-0.71	
1146	D5185	36.14		0.33	
1189		----		----	
1201	D5185	36		0.28	
1234	INHOUSE	30		-1.71	
1243	DIN51396	36.3		0.38	
1258		----		----	
1265	D6595	36.7		0.51	
1318		----		----	
1417	INHOUSE	35.312		0.05	
1433		----		----	
1435	D5185	37		0.61	
1526	D5185	36		0.28	
1613		----		----	
1628		----		----	
1660	D5185	33.46		-0.56	
1900	D6595	32.89352		-0.75	
normality		not OK			
n		20			
outliers		1			
mean (n)		35.15			
st.dev. (n)		2.543			
R(calc.)		7.12			
R(D5185:09)		8.44			



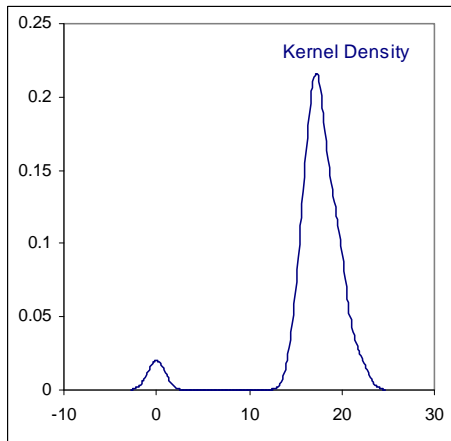
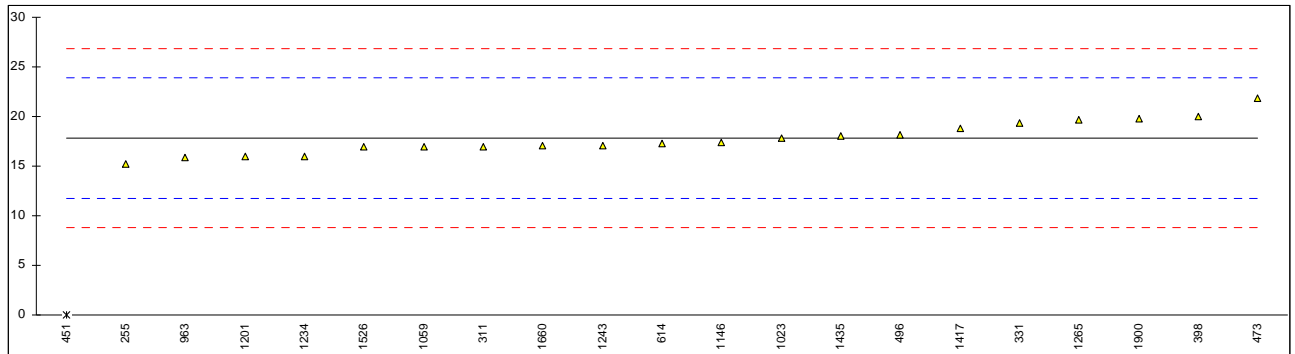
Determination of Iron (Fe) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	AAS	15.20		-1.37	
311	D5185	17		-0.40	
317		----		----	
331	D5185	20.2		1.33	
398	D6595	20.0		1.22	
432		----		----	
451	D5185	2	G(0.01)	-8.49	
473	D5185mod	18.836		0.59	
496	D5185	18.11		0.20	
562		----		----	
614	D5185	18.0		0.14	
963	D5185	16.96		-0.42	
1017		----		----	
1023	D5185	17.7		-0.02	
1059	INHOUSE	15		-1.48	
1146	D5185	17.82		0.04	
1189		----		----	
1201	D5185	18		0.14	
1234	INHOUSE	12		-3.10	
1243	DIN51396	17.8		0.03	
1258		----		----	
1265	D6595	20.6		1.54	
1318		----		----	
1417	INHOUSE	18.983		0.67	
1433		----		----	
1435	D5185	19		0.68	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	18.28		0.29	
1900	D6595	17.63918		-0.06	
normality		not OK			
n		19			
outliers		1			
mean (n)		17.74			
st.dev. (n)		2.004			
R(calc.)		5.61			
R(D5185:09)		5.19			



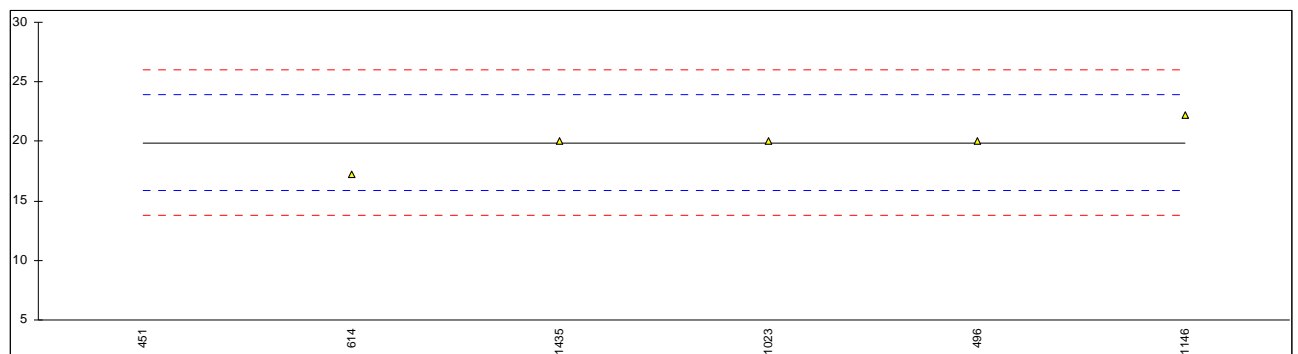
Determination of Lead (Pb) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	AAS	15.20		-0.87	
311	D5185	17		-0.27	
317		----		----	
331	D5185	19.3		0.49	
398	D6595	20.0		0.72	
432		----		----	
451	D5185	0	ex	-5.90	False negative? (Zero is not a real value)
473	D5185mod	21.851		1.33	
496	D5185	18.13		0.10	
562		----		----	
614	D5185	17.3		-0.17	
963	D5185	15.83	C	-0.66	First reported 35.83
1017		----		----	
1023	D5185	17.8		-0.01	
1059	INHOUSE	17		-0.27	
1146	D5185	17.44		-0.12	
1189		----		----	
1201	D5185	16		-0.60	
1234	INHOUSE	16		-0.60	
1243	DIN51396	17.1		-0.24	
1258		----		----	
1265	D6595	19.7		0.62	
1318		----		----	
1417	INHOUSE	18.857		0.34	
1433		----		----	
1435	D5185	18		0.06	
1526	D5185	17		-0.27	
1613		----		----	
1628		----		----	
1660	D5185	17.09		-0.24	
1900	D6595	19.75465		0.64	
normality		OK			
n		20			
outliers		0			
mean (n)		17.82			
st.dev. (n)		1.664			
R(calc.)		4.66			
R(D5185:09)		8.46			



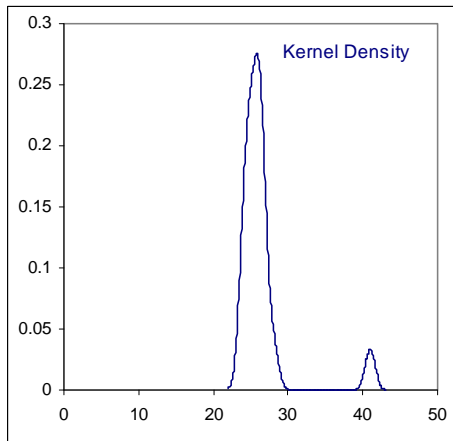
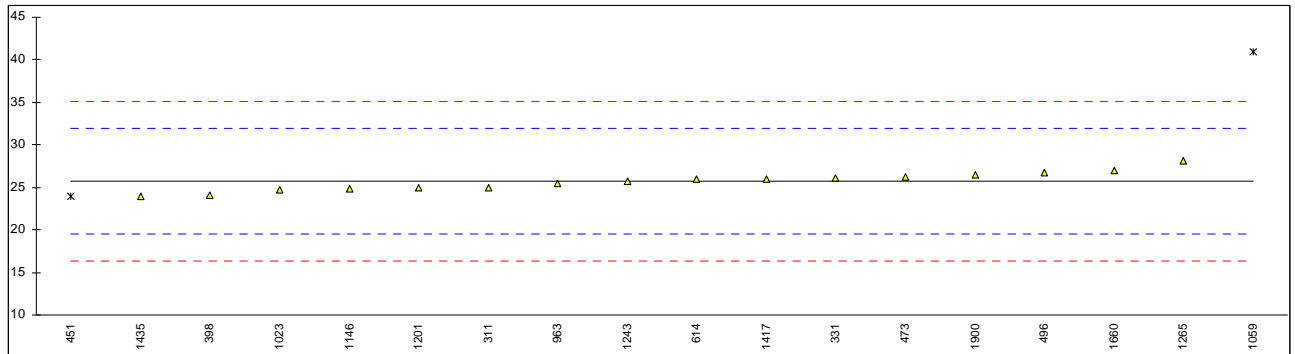
Determination of Lithium (Li) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311		----		----	
317		----		----	
331		----		----	
398		----		----	
432		----		----	
451	D5185	0	ex	-9.80	False negative? (Zero is not a real value)
473		----		----	
496	D5185	20.01		0.06	
562		----		----	
614	D5185	17.2		-1.32	
963		----		----	
1017		----		----	
1023	D5185	20.0		0.06	
1059		----		----	
1146	D5185	22.22		1.15	
1189		----		----	
1201		----		----	
1234		----		----	
1243		----		----	
1258		----		----	
1265		----		----	
1318		----		----	
1417		----		----	
1433		----		----	
1435	D5185	20		0.06	
1526		----		----	
1613		----		----	
1628		----		----	
1660		----		----	
1900		----		----	
normality		OK			
n		5			
outliers		0			
mean (n)		19.89			
st.dev. (n)		1.782			
R(calc.)		4.99			
R(Horwitz)		5.68			



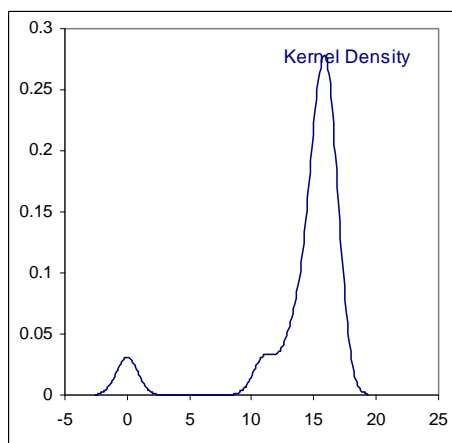
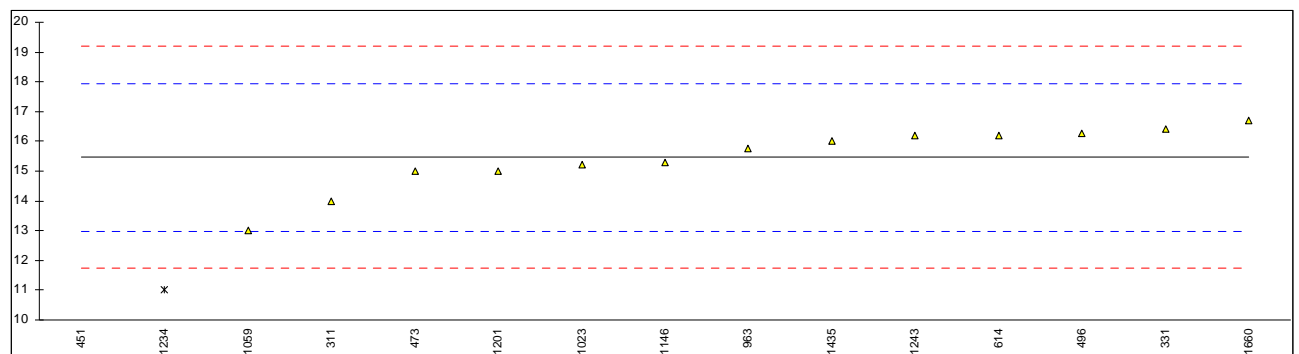
Determination of Magnesium (Mg) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311	D5185	25		-0.23	
317		----		----	
331	D5185	26.1		0.12	
398	D6595	24.1		-0.52	
432		----		----	
451	D5185	24	ex,C	-0.55	See § 4.1, first reported 0
473	D5185mod	26.264		0.17	
496	D5185	26.71		0.32	
562		----		----	
614	D5185	26.0		0.09	
963	D5185	25.51		-0.07	
1017		----		----	
1023	D5185	24.7		-0.33	
1059	INHOUSE	41	G(0.01)	4.89	
1146	D5185	24.81		-0.29	
1189		----		----	
1201	D5185	25		-0.23	
1234		----		----	
1243	DIN51396	25.7		-0.01	
1258		----		----	
1265	D6595	28.1		0.76	
1318		----		----	
1417	INHOUSE	26.005		0.09	
1433		----		----	
1435	D5185	24		-0.55	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	27.0		0.41	
1900	D6595	26.52302		0.26	
	normality	OK			
	n	16			
	outliers	1			
	mean (n)	25.72			
	st.dev. (n)	1.097			
	R(calc.)	3.07			
	R(D5185:09)	8.75			



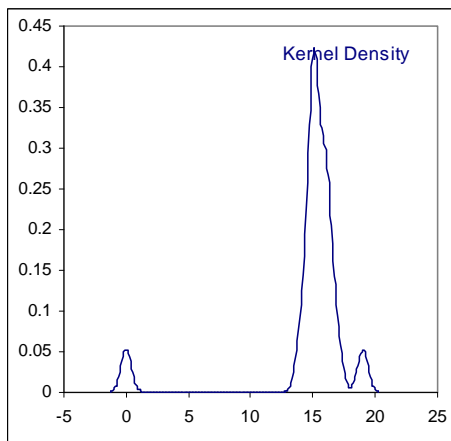
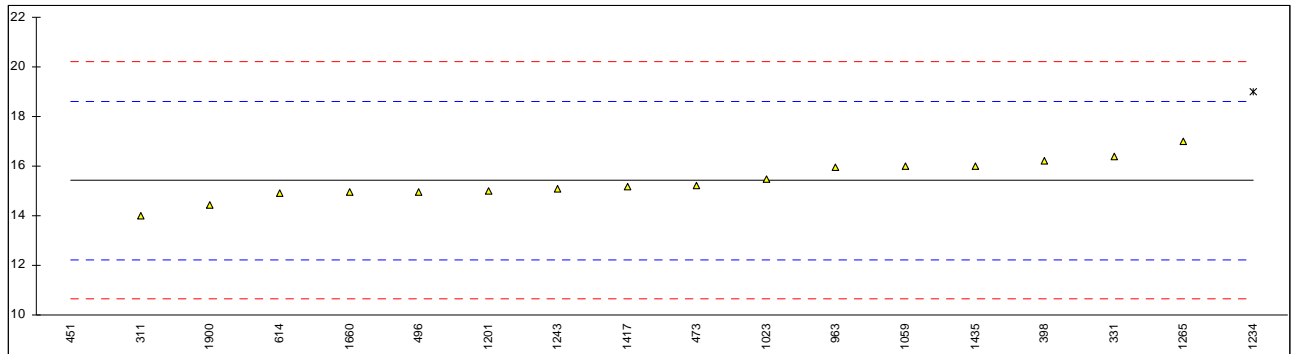
Determination of Manganese (Mn) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311	D5185	14		-1.18	
317		----		----	
331	D5185	16.4		0.76	
398		----		----	
432		----		----	
451	D5185	0	ex	-12.46	False negative? (Zero is not a real value)
473	D5185mod	14.989		-0.38	
496	D5185	16.26		0.64	
562		----		----	
614	D5185	16.2		0.59	
963	D5185	15.75		0.23	
1017		----		----	
1023	D5185	15.2		-0.21	
1059	INHOUSE	13		-1.98	
1146	D5185	15.28		-0.15	
1189		----		----	
1201	D5185	15		-0.37	
1234	INHOUSE	11	G(0.05)	-3.59	
1243	DIN51396	16.2		0.59	
1258		----		----	
1265		----		----	
1318		----		----	
1417		----		----	
1433		----		----	
1435	D5185	16		0.43	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	16.72		1.01	
1900		----		----	
normality		OK			
n		13			
outliers		1			
mean (n)		15.46			
st.dev. (n)		1.053			
R(calc.)		2.95			
R(D5185:09)		3.48			



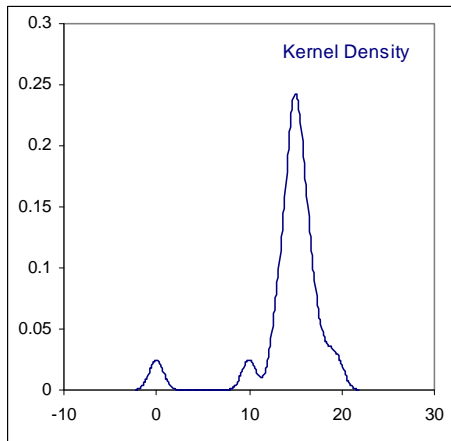
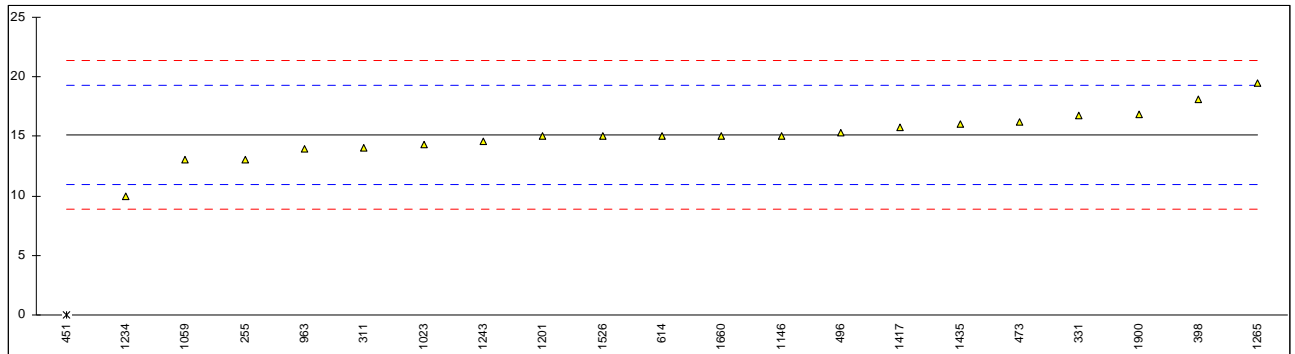
Determination of Molybdenum (Mo) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311	D5185	14		-0.89	
317		----		----	
331	D5185	16.4		0.61	
398	D6595	16.2		0.49	
432		----		----	
451	D5185	0	ex	-9.67	False negative? (Zero is not a real value)
473	D5185mod	15.196		-0.14	
496	D5185	14.96		-0.29	
562		----		----	
614	D5185	14.9		-0.33	
963	D5185	15.95		0.33	
1017		----		----	
1023	D5185	15.5		0.05	
1059	INHOUSE	16		0.36	
1146		----		----	
1189		----		----	
1201	D5185	15		-0.26	
1234	INHOUSE	19	G(0.01)	2.24	
1243	DIN51396	15.1		-0.20	
1258		----		----	
1265	D6595	17.0		0.99	
1318		----		----	
1417	INHOUSE	15.163		-0.16	
1433		----		----	
1435	D5185	16		0.36	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	14.94		-0.30	
1900	D6595	14.4402		-0.62	
normality		OK			
n		16			
outliers		1			
mean (n)		15.42			
st.dev. (n)		0.779			
R(calc.)		2.18			
R(D5185:09)		4.46			



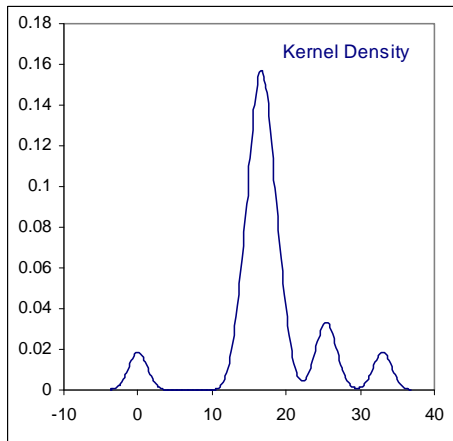
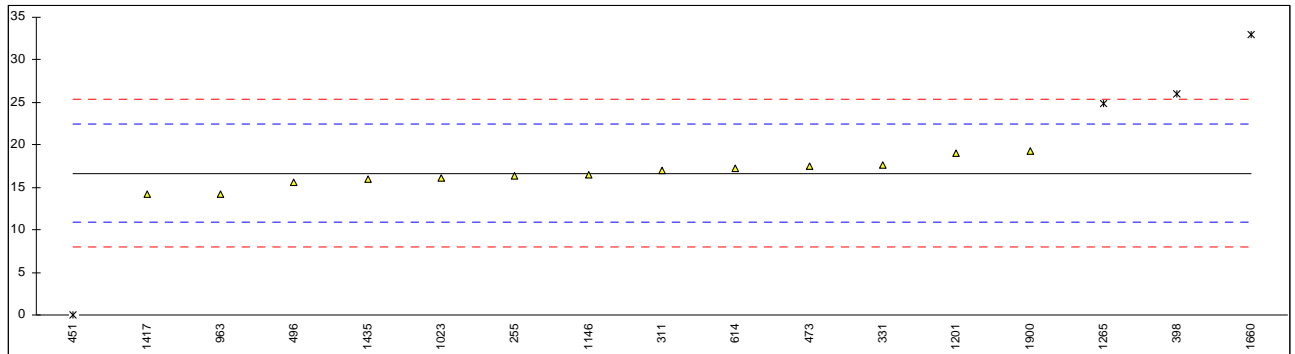
Determination of Nickel (Ni) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	AAS	13.00		-1.02	
311	D5185	14		-0.54	
317		----		----	
331	D5185	16.8		0.81	
398	D6595	18.1		1.43	
432		----		----	
451	D5185	0	ex	-7.26	False negative? (Zero is not a real value)
473	D5185mod	16.188		0.51	
496	D5185	15.35		0.11	
562		----		----	
614	D5185	15.0		-0.06	
963	D5185	13.97		-0.55	
1017		----		----	
1023	D5185	14.3		-0.39	
1059	INHOUSE	13		-1.02	
1146	D5185	15.06		-0.03	
1189		----		----	
1201	D5185	15		-0.06	
1234	INHOUSE	10		-2.46	
1243	DIN51396	14.6		-0.25	
1258		----		----	
1265	D6595	19.5		2.10	
1318		----		----	
1417	INHOUSE	15.725		0.29	
1433		----		----	
1435	D5185	16		0.42	
1526	D5185	15		-0.06	
1613		----		----	
1628		----		----	
1660	D5185	15.01		-0.05	
1900	D6595	16.81495		0.81	
normality		OK			
n		20			
outliers		0			
mean (n)		15.12			
st.dev. (n)		1.985			
R(calc.)		5.56			
R(D5185:09)		5.83			



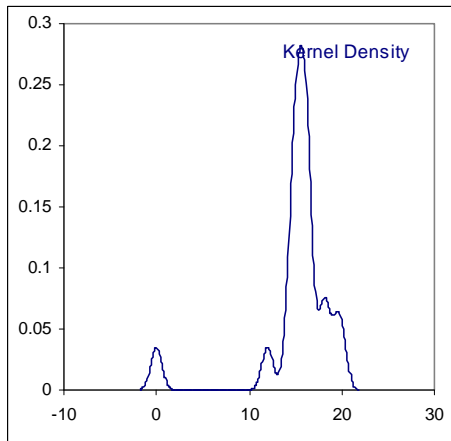
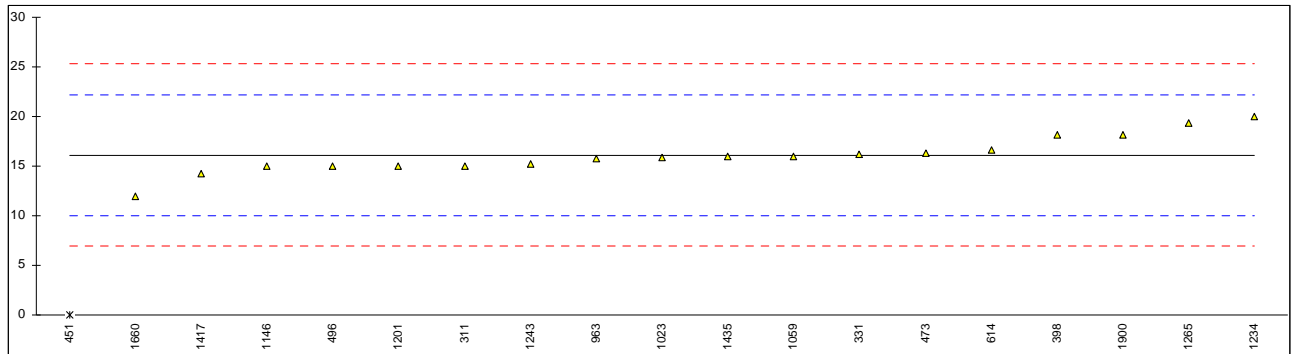
Determination of Sodium (Na) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	AAS	16.40		-0.09	
311	D5185	17		0.12	
317		----		----	
331	D5185	17.6		0.33	
398	D6595	26.0	DG(0.01)	3.23	
432		----		----	
451	D5185	0	ex	-5.75	False negative? (Zero is not a real value)
473	D5185mod	17.511		0.29	
496	D5185	15.61		-0.36	
562		----		----	
614	D5185	17.3		0.22	
963	D5185	14.21		-0.85	
1017		----		----	
1023	D5185	16.1		-0.19	
1059		----		----	
1146	D5185	16.44		-0.08	
1189		----		----	
1201	D5185	19		0.81	
1234		----		----	
1243		----		----	
1258		----		----	
1265	D6595	24.8	DG(0.01)	2.81	
1318		----		----	
1417	INHOUSE	14.176		-0.86	
1433		----		----	
1435	D5185	16		-0.23	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	33.0	G(0.05)	5.65	
1900	D6595	19.21248		0.88	
normality		OK			
n		13			
outliers		3			
mean (n)		16.66			
st.dev. (n)		1.535			
R(calc.)		4.30			
R(D5185:09)		8.10			



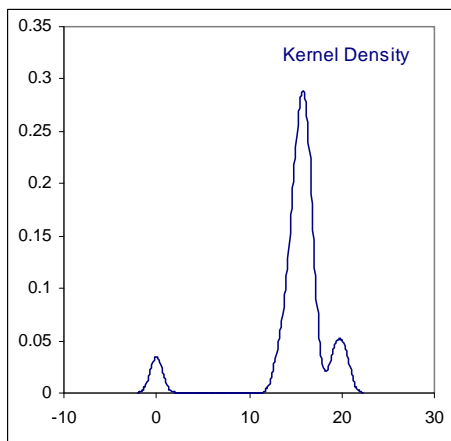
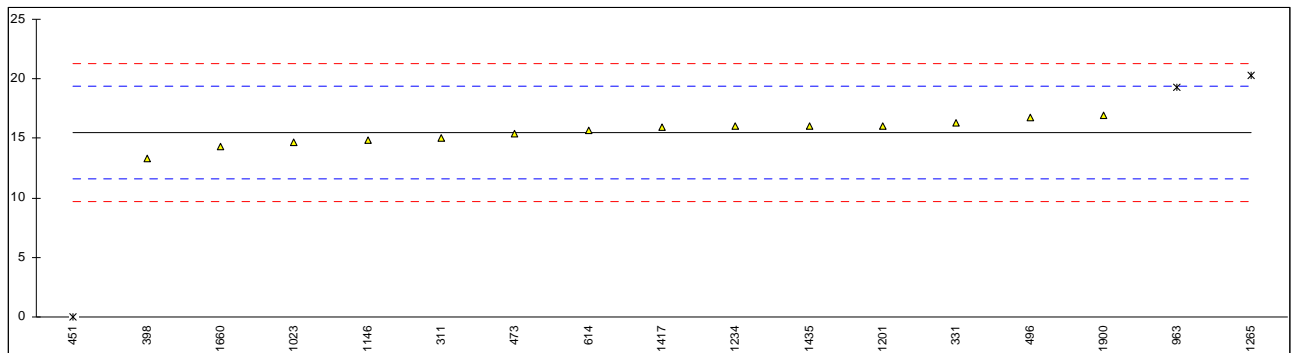
Determination of Silicon (Si) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311	D5185	15		-0.36	
317		----		----	
331	D5185	16.2		0.03	
398	D6595	18.1		0.65	
432		----		----	
451	D5185	0	ex	-5.26	False negative? (Zero is not a real value)
473	D5185mod	16.336		0.08	
496	D5185	14.98		-0.37	
562		----		----	
614	D5185	16.6		0.16	
963	D5185	15.71		-0.13	
1017		----		----	
1023	D5185	15.9		-0.07	
1059	INHOUSE	16		-0.03	
1146	D5185	14.98		-0.37	
1189		----		----	
1201	D5185	15		-0.36	
1234	INHOUSE	20		1.27	
1243	DIN51396	15.2		-0.29	
1258		----		----	
1265	D6595	19.4		1.08	
1318		----		----	
1417	INHOUSE	14.257		-0.60	
1433		----		----	
1435	D5185	16		-0.03	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	12.0		-1.34	
1900	D6595	18.16234		0.67	
	normality	OK			
	n	18			
	outliers	0			
	mean (n)	16.10			
	st.dev. (n)	1.892			
	R(calc.)	5.30			
	R(D5185:09)	8.57			



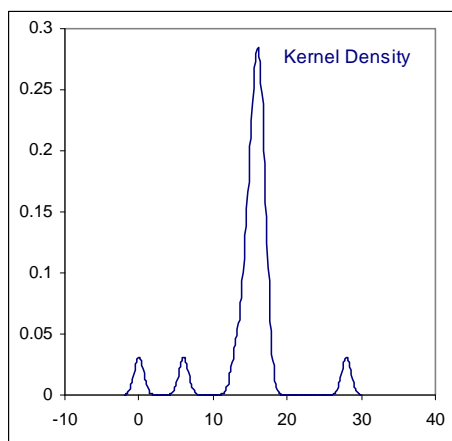
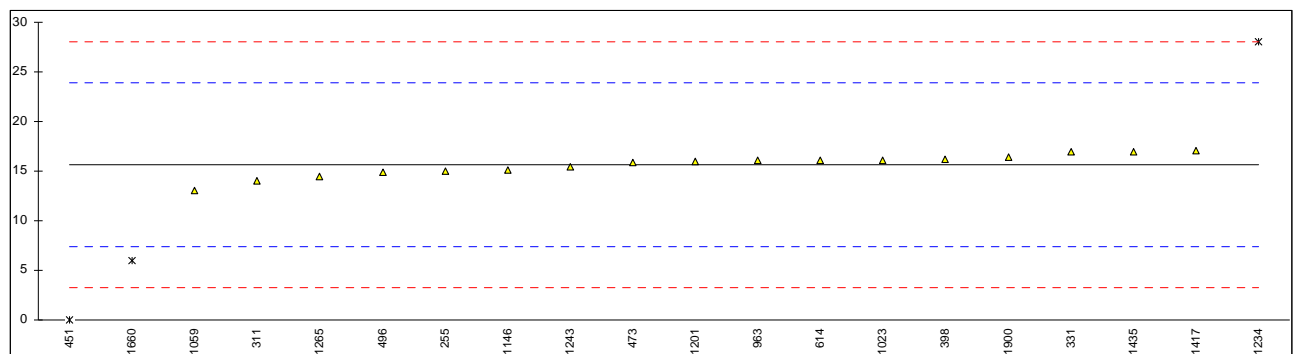
Determination of Silver (Ag) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311	D5185	15		-0.26	
317		----		----	
331	D5185	16.3		0.41	
398	D6595	13.3		-1.14	
432		----		----	
451	D5185	0	ex	-8.00	False negative? (Zero is not a real value)
473	D5185mod	15.372		-0.07	
496	D5185	16.78		0.66	
562		----		----	
614	D5185	15.7		0.10	
963	D5185	19.26	DG(0.05)	1.93	
1017		----		----	
1023	D5185	14.7		-0.42	
1059		----		----	
1146	D5185	14.87		-0.33	
1189		----		----	
1201	D5185	16		0.25	
1234	INHOUSE	16		0.25	
1243		----		----	
1258		----		----	
1265	D6595	20.3	DG(0.05)	2.47	
1318		----		----	
1417	INHOUSE	15.911		0.21	
1433		----		----	
1435	D5185	16		0.25	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	14.3		-0.62	
1900	D6595	16.9		0.72	
	normality	OK			
	n	14			
	outliers	2			
	mean (n)	15.51			
	st.dev. (n)	0.991			
	R(calc.)	2.78			
	R(D5185:09)	5.43			



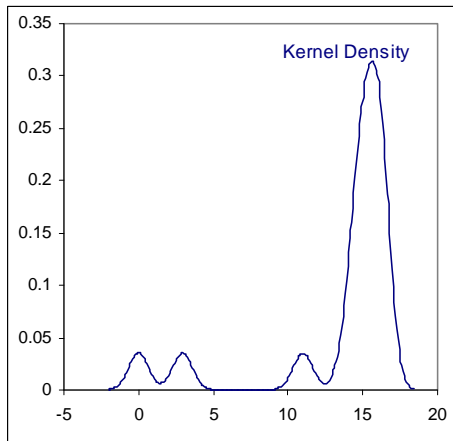
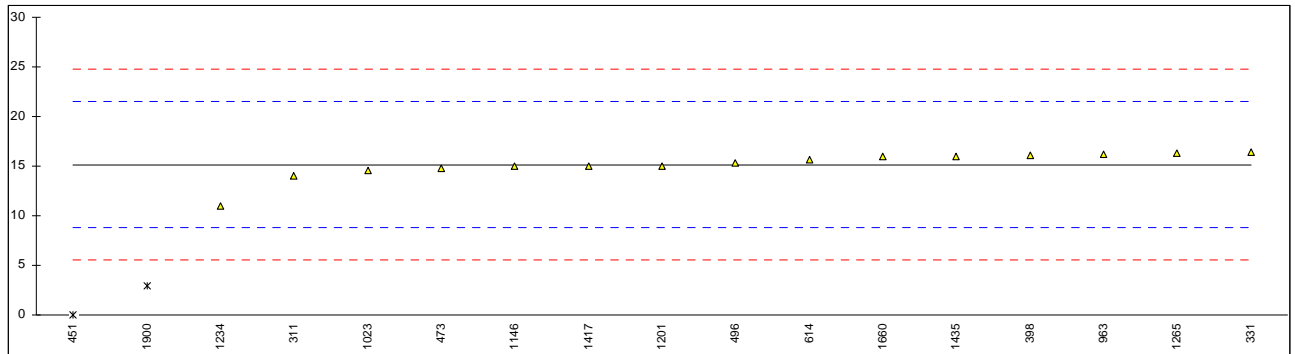
Determination of Tin (Sn) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	AAS	15.00		-0.15	
311	D5185	14		-0.40	
317		----		----	
331	D5185	17.0		0.33	
398	D6595	16.2		0.14	
432		----		----	
451	D5185	0	ex	-3.79	False negative? (Zero is not a real value)
473	D5185mod	15.852		0.05	
496	D5185	14.94		-0.17	
562		----		----	
614	D5185	16.1		0.11	
963	D5185	16.04		0.10	
1017		----		----	
1023	D5185	16.1		0.11	
1059	INHOUSE	13		-0.64	
1146	D5185	15.16		-0.12	
1189		----		----	
1201	D5185	16		0.09	
1234	INHOUSE	28	G(0.01)	3.00	
1243	DIN51396	15.4		-0.06	
1258		----		----	
1265	D6595	14.5		-0.28	
1318		----		----	
1417	INHOUSE	17.072		0.35	
1433		----		----	
1435	D5185	17		0.33	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	6.03	G(0.01)	-2.33	
1900	D6595	16.44711		0.20	
	normality	OK			
	n	17			
	outliers	2			
	mean (n)	15.64			
	st.dev. (n)	1.107			
	R(calc.)	3.10			
	R(D5185:09)	11.55			



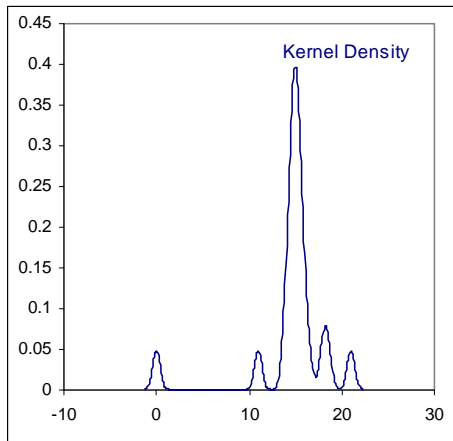
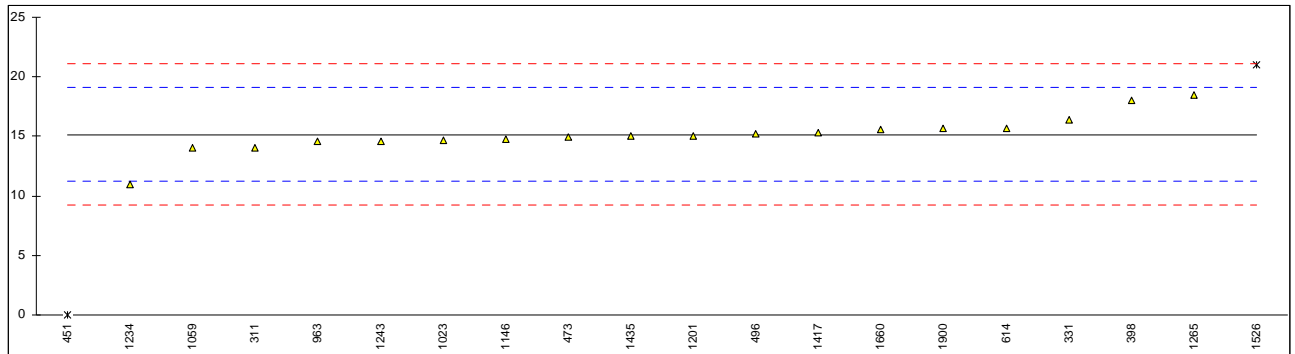
Determination of Titanium (Ti) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311	D5185	14		-0.36	
317		----		----	
331	D5185	16.4		0.39	
398	D6595	16.1		0.29	
432		----		----	
451	D5185	0	ex	-4.73	False negative? (Zero is not a real value)
473	D5185mod	14.805		-0.11	
496	D5185	15.36		0.06	
562		----		----	
614	D5185	15.7		0.17	
963	D5185	16.24		0.34	
1017		----		----	
1023	D5185	14.6		-0.18	
1059		----		----	
1146	D5185	14.99		-0.05	
1189		----		----	
1201	D5185	15		-0.05	
1234	INHOUSE	11		-1.30	
1243		----		----	
1258		----		----	
1265	D6595	16.3		0.35	
1318		----		----	
1417	INHOUSE	14.990		-0.05	
1433		----		----	
1435	D5185	16		0.26	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	15.96		0.25	
1900	D6595	2.93	C,G(0.01)	-3.82	First reported 8.47
normality		OK			
n		15			
outliers		1			
mean (n)		15.16			
st.dev. (n)		1.356			
R(calc.)		3.80			
R(D5185:09)		8.97			



Determination of Vanadium (V) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311	D5185	14		-0.59	
317		----		----	
331	D5185	16.4		0.62	
398	D6595	18.0		1.42	
432		----		----	
451	D5185	0	ex	-7.62	False negative? (Zero is not a real value)
473	D5185mod	14.935		-0.12	
496	D5185	15.26		0.04	
562		----		----	
614	D5185	15.7		0.27	
963	D5185	14.54		-0.32	
1017		----		----	
1023	D5185	14.7		-0.24	
1059	INHOUSE	14		-0.59	
1146	D5185	14.80		-0.19	
1189		----		----	
1201	D5185	15		-0.09	
1234	INHOUSE	11		-2.09	
1243	DIN51396	14.6		-0.29	
1258		----		----	
1265	D6595	18.5		1.67	
1318		----		----	
1417	INHOUSE	15.352		0.09	
1433		----		----	
1435	D5185	15		-0.09	
1526	D5185	21	G(0.05)	2.93	
1613		----		----	
1628		----		----	
1660	D5185	15.61		0.22	
1900	D6595	15.68399		0.26	
normality		not OK			
n		18			
outliers		1			
mean (n)		15.17			
st.dev. (n)		1.585			
R(calc.)		4.44			
R(D5185:09)		5.58			

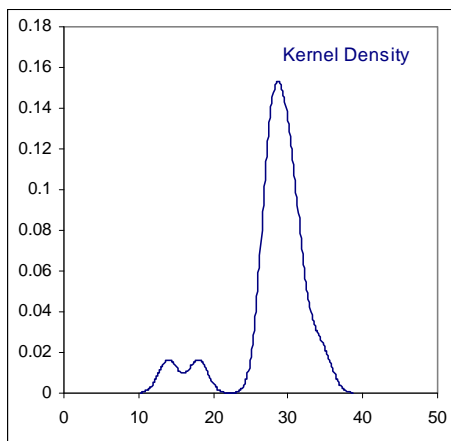
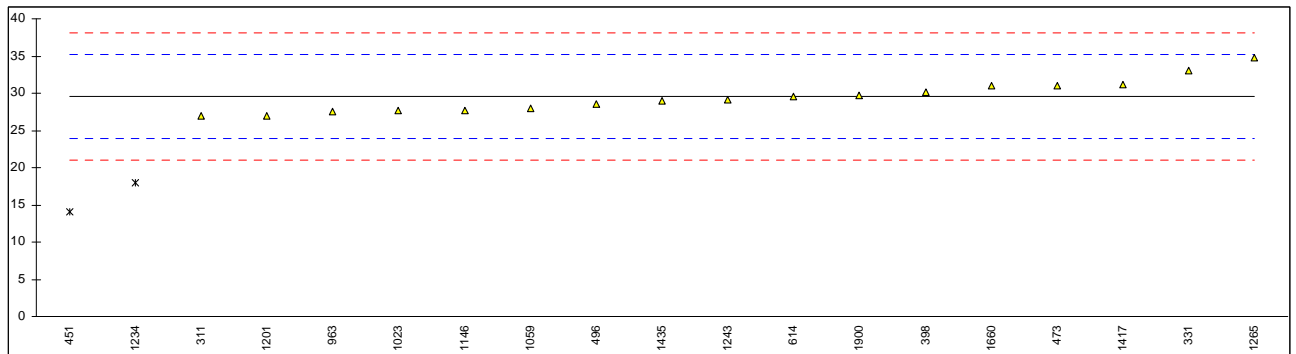


Determination of Calcium (Ca) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311	D5185	27		-0.89	
317		----		----	
331	D5185	33.0		1.22	
398	D6595	30.1		0.20	
432		----		----	
451	D5185	14	C,G(0.01)	-5.47	First reported 11
473	D5185mod	31.043		0.53	
496	D5185	28.59		-0.33	
562		----		----	
614	D5185	29.6		0.02	
963	D5185	27.56		-0.70	
1017		----		----	
1023	D5185	27.7		-0.65	
1059	INHOUSE	28		-0.54	
1146	D5185	27.74		-0.63	
1189		----		----	
1201	D5185	27		-0.89	
1234	INHOUSE	18	G(0.01)	-4.06	
1243	DIN51396	29.1		-0.16	
1258		----		----	
1265	D6595	34.8		1.85	
1318		----		----	
1417	INHOUSE	31.194		0.58	
1433		----		----	
1435	D5185	29		-0.19	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	31.0		0.51	
1900	D6595	29.77004		0.08	

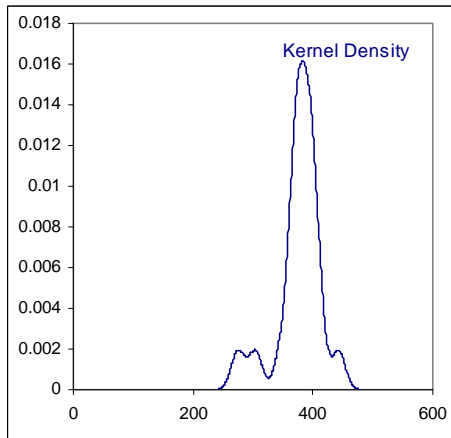
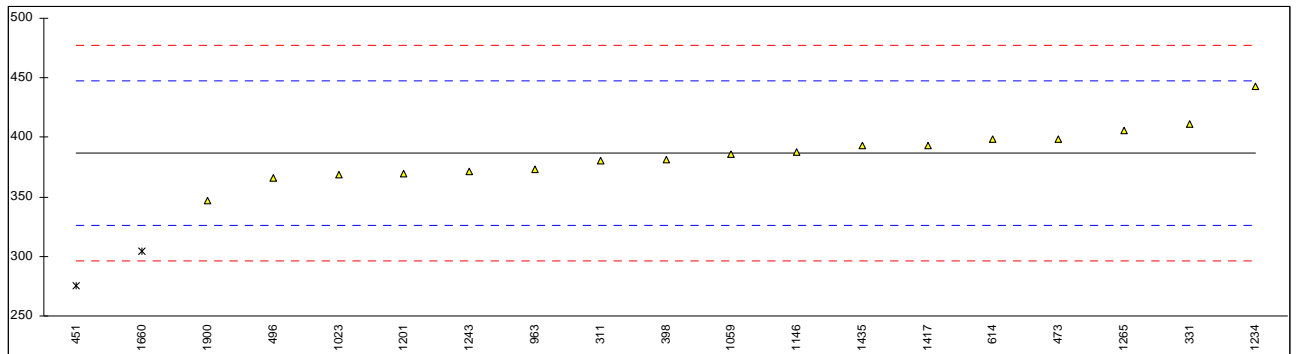
normality OK  
n 17  
outliers 2  
mean (n) 29.54  
st.dev. (n) 2.156  
R(calc.) 6.04  
R(Horwitz) 7.95

Application range D5185 = 40 – 9000 mg/kg  
Compare with R(D5185:09) = 1.22 mg/kg (extrapolated)



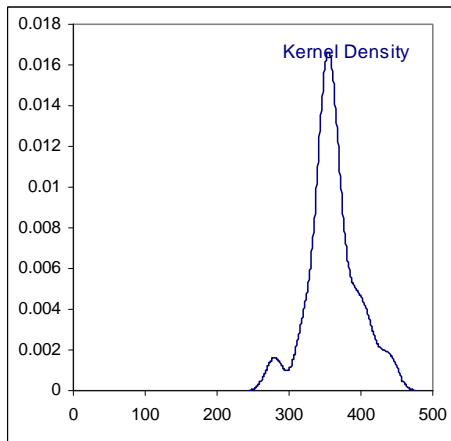
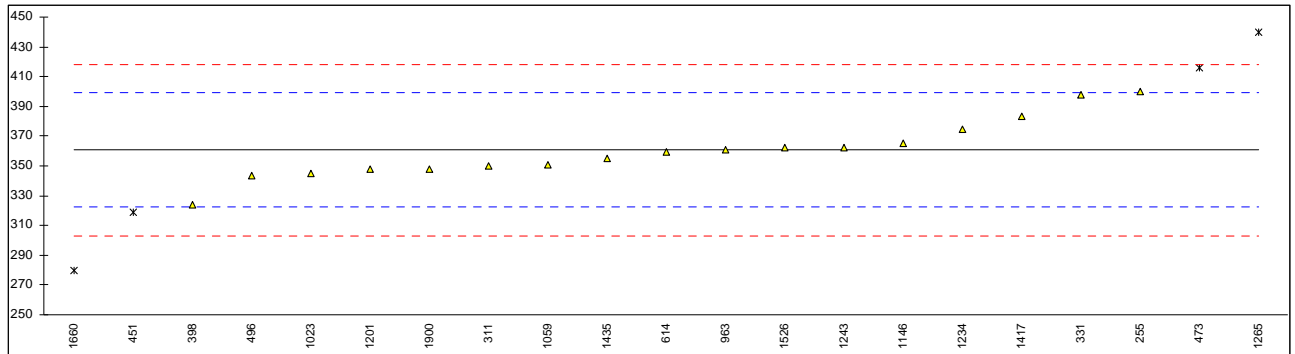
Determination of Phosphorus (P) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311	D5185	380		-0.23	
317		----		----	
331	D5185	411.0		0.80	
398	D6595	381.4		-0.18	
432		----		----	
451	D5185	275	C,G(0.05)	-3.70	First reported 304
473	D5185mod	398.874		0.40	
496	D5185	366.1		-0.69	
562		----		----	
614	D5185	398.8		0.40	
963	D5185	372.8		-0.46	
1017		----		----	
1023	D5185	369		-0.59	
1059	INHOUSE	386		-0.03	
1146	D5185	388.10		0.04	
1189		----		----	
1201	D5185	370		-0.56	
1234	INHOUSE	443		1.86	Reported as 0.0443%
1243	DIN51396	371.6		-0.50	
1258		----		----	
1265	D6595	406		0.63	
1318		----		----	
1417	INHOUSE	393.188		0.21	
1433		----		----	
1435	D5185	393		0.20	
1526		----		----	
1613		----		----	
1628		----		----	
1660	D5185	304.0	G(0.05)	-2.74	
1900	D6595	347.2833		-1.31	
	normality	OK			
	n	17			
	outliers	2			
	mean (n)	386.8			
	st.dev. (n)	21.76			
	R(calc.)	60.9			
	R(D5185:09)	84.6			



Determination of Zinc (Zn) on sample #0987; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	AAS	400.00		2.04	
311	D5185	350		-0.55	
317		-----		-----	
331	D5185	398.0		1.94	
398	D6595	324.2		-1.89	
432		-----		-----	
451	D5185	319	ex,C	-2.16	See §4.1, first reported 323
473	D5185mod	415.884	DG(0.05)	2.87	
496	D5185	343.6		-0.88	
562		-----		-----	
614	D5185	359.4		-0.06	
963	D5185	360.8		0.01	
1017		-----		-----	
1023	D5185	345		-0.81	
1059	INHOUSE	351		-0.50	
1146	D5185	365.10		0.23	
1189		-----		-----	
1201	D5185	348		-0.66	
1234	INHOUSE	375		0.75	Reported as 0.0375%
1243	DIN51396	362.6		0.10	
1258		-----		-----	
1265	D6595	440	G(0.05)	4.12	
1318		-----		-----	
1417	INHOUSE	383.067		1.16	
1433		-----		-----	
1435	D5185	355		-0.29	
1526	D5185	362		0.07	
1613		-----		-----	
1628		-----		-----	
1660	D5185	280.0	G(0.05)	-4.19	
1900	D6595	348.1014		-0.65	
normality		OK			
n		17			
outliers		4			
mean (n)		360.6			
st.dev. (n)		19.52			
R(calc.)		54.7			
R(D5185:09)		53.9			



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

<b>Name of laboratories</b>	<b>Country</b>
1 laboratory in	AUSTRALIA
1 laboratory in	AUSTRIA
2 laboratories in	BELGIUM
1 laboratory in	CHILE
2 laboratories in	ESTONIA
1 laboratory in	FRANCE
2 laboratories in	GERMANY
2 laboratories in	ITALY
1 laboratory in	JORDAN
4 laboratories in	NORWAY
1 laboratory in	POLAND
1 laboratory in	SAUDI ARABIA
1 laboratory in	SERBIA
1 laboratory in	SINGAPORE
1 laboratory in	SLOVENIA
1 laboratory in	SPAIN
1 laboratory in	TANZANIA
4 laboratories in	THE NETHERLANDS
1 laboratory in	TURKEY
2 laboratories in	UNITED KINGDOM

## 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
W	= withdrawn
Mod.	= modified
SDS	= Material Safety Data Sheet

### Literature:

- 1 i.i.s. Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, November 2008
- 2 ASTM E178-89
- 3 ASTM E1301-89
- 4 ISO 5725-86
- 5 ISO 5725, parts 1-6, 1994
- 6 ISO 13528-05
- 7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367/84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 Analytical Methods Committee Technical Brief, No4 January 2001
- 14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see <http://www.rsc.org/suppdata/an/b2/b205600n/>)