

**Results of Proficiency Test
Naphtha
April 2021**

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink

Correctors: ing. A.S. Noordman-de Neef & ing. G.A. Oosterlaken-Buijs

Report: iis21N01

July 2021

CONTENTS

1 INTRODUCTION 3

2 SET UP 3

2.1 ACCREDITATION 3

2.2 PROTOCOL 4

2.3 CONFIDENTIALITY STATEMENT 4

2.4 SAMPLES 4

2.5 STABILITY OF THE SAMPLES 8

2.6 ANALYZES 9

3 RESULTS 9

3.1 STATISTICS 9

3.2 GRAPHICS 10

3.3 Z-SCORES 11

4 EVALUATION 11

4.1 EVALUATION PER SAMPLE AND PER TEST 12

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES 13

4.3 COMPARISON OF THE PROFICIENCY TEST OF APRIL 2021 WITH PREVIOUS PTS 14

Appendices:

1. Data, statistical and graphic results 16

2. Number of participants per country 40

3. Abbreviations and literature 41

1 INTRODUCTION

Since 1994 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Naphtha every year. The interlaboratory study on Naphtha was extended with separate PTs for the determination for Mercury, Arsenic/Lead and Vapor Pressure. In the annual proficiency testing program of 2020/2021, it was decided to continue the 4 PTs on Naphtha. It was decided to prepare a separate report for the proficiency test PIONA/PNA on Naphtha (iis21N01PIONA).

In this interlaboratory study registered for participation:

- 90 laboratories in 37 countries on Naphtha (iis21N01),
- 50 laboratories in 23 countries for Mercury in Naphtha (iis21N01Hg),
- 33 laboratories in 18 countries for Arsenic and Lead in Naphtha (iis21N01AsPb),
- 54 laboratories in 22 countries for Vapor Pressure (iis21N01DVPE).

In total 98 laboratories in 38 countries registered for participation in one or more PTS. See appendix 2 for the number of participants per country. In this report the results of the Naphtha proficiency tests are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.

In this proficiency test the participants received, depending on the registration, from one up to six different samples of Naphtha, see table below.

Samples	Type of bottle	Purpose	Matrix
#21035	0.5L	For regular analyzes	Real Naphtha
#21037	0.5L	For Mercury	Artificial Naphtha
#21038	0.5L	For Mercury	Real Naphtha
#21039	0.5L	For Arsenic and Lead	Artificial Naphtha
#21040	0.5L	For Arsenic and Lead	Real Naphtha
#21041	0.25L	For DVPE	Real Naphtha

Table 1: Six different Naphtha samples used in iis21N01

Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website site www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

For the preparation of the sample for the regular PT Naphtha a batch of approximately 70L of Naphtha was obtained from a local refinery and spiked with Chloroform. After homogenization 130 amber glass bottles of 0.5L were filled and labelled #21035. The homogeneity of the subsamples was checked by the determination of Density at 15°C in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/L
sample #21035-1	0.71901
sample #21035-2	0.71903
sample #21035-3	0.71905
sample #21035-4	0.71902
sample #21035-5	0.71903
sample #21035-6	0.71901
sample #21035-7	0.71905
sample #21035-8	0.71904

Table 2: homogeneity test results of subsamples #21035

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 15°C in kg/L
r (observed)	0.00004
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 3: evaluation of the repeatability of subsamples #21035

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

For the preparation of the sample Mercury in Naphtha a batch of approximately 45L of Naphtha was obtained from a local refinery and spiked with Mercury Chloride and a Mercury Conostan standard. After homogenization 80 amber glass bottles of 0.5L were filled and labelled #21038.

The homogeneity of the subsamples was checked by the determination of Mercury in accordance with UOP938 on 8 stratified randomly selected subsamples.

	Mercury in µg/kg
sample #21038-1	239.6
sample #21038-2	245.6
sample #21038-3	245.7
sample #21038-4	244.9
sample #21038-5	231.2
sample #21038-6	245.1
sample #21038-7	252.5
sample #21038-8	247.2

Table 4: homogeneity test results of subsamples #21038

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility calculated with the Horwitz equation in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Mercury in µg/kg
r (observed)	17.5
reference method	Horwitz
0.3 x R (reference method)	40.5

Table 5: evaluation of the repeatability of subsamples #21038

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

For the preparation of the sample Arsenic and Lead in Naphtha a batch of approximately 30L of Naphtha was obtained from a local refinery and spiked with an Arsenic Conostan standard and Aviation Gasoline (for Lead). After homogenization 60 amber glass bottles of 0.5L were filled and labelled #21040.

The homogeneity of the subsamples was checked by the determination of Lead in accordance with UOP952 on 8 stratified randomly selected subsamples.

	Lead in $\mu\text{g}/\text{kg}$
sample #21040-1	31
sample #21040-2	32
sample #21040-3	34
sample #21040-4	33
sample #21040-5	33
sample #21040-6	32
sample #21040-7	35
sample #21040-8	36

Table 6: homogeneity test results of subsamples #21040

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility calculated with the Horwitz equation in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Lead in $\mu\text{g}/\text{kg}$
r (observed)	5
reference method	Horwitz
0.3 x R (reference method)	7

Table 7: evaluation of the repeatability of subsamples #21040

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

For the preparation of the sample for Vapor Pressure on Naphtha a batch of approximately 25L of Naphtha was obtained from a local refinery. After homogenization 90 amber glass bottles of 0.25L were filled and labelled #21041. The homogeneity of the subsamples was checked by the determination of DVPE in accordance with ASTM D5191 on 8 stratified randomly selected subsamples. One Dixon outlier was found in this data set.

	DVPE in psi
sample #21041-1	6.28
sample #21041-2	6.25
sample #21041-3	6.26
sample #21041-4	6.22
sample #21041-5	6.25
sample #21041-6	6.03 D(0.01)
sample #21041-7	6.25
sample #21041-8	6.24

Table 8: homogeneity test results of subsamples #21041

Subsample 6 is a Dixon outlier and therefore excluded for statistical evaluation of the homogeneity.

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	DVPE in psi
r (observed)	0.05
reference test method	ASTM D5191:20
0.3 x R (reference test method)	0.07

Table 9: evaluation of the repeatability of subsamples #21041

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

Furthermore, a batch of approximately 45L artificial Naphtha was prepared and spiked with Mercury Chloride and Mercury Conostan especially for Mercury determination. After homogenization 80 amber glass bottles of 0.5L were filled and labelled #21037. The homogeneity of the subsamples was checked by determination of Mercury in accordance with UOP938 on 8 stratified randomly selected subsamples.

	Mercury in µg/kg
sample #21037-1	104.4
sample #21037-2	102.7
sample #21037-3	96.7
sample #21037-4	93.8
sample #21037-5	96.7
sample #21037-6	98.5
sample #21037-7	101.5
sample #21037-8	93.2

Table 10: homogeneity test results of subsamples #21037

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility calculated with the Horwitz equation in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Mercury in µg/kg
r (observed)	11.5
reference method	Horwitz
0.3 x R (reference method)	18.8

Table 11: evaluation of the repeatability of subsamples #21037

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

For the preparation of the sample Arsenic and Lead in Naphtha a batch of approximately 30L of artificial Naphtha was obtained from a local refinery and spiked with an Arsenic Conostan standard and Aviation Gasoline (for Lead). After homogenization 60 amber glass bottles of 0.5L were filled and labelled #21039.

The homogeneity of the subsamples was checked by the determination of Lead in accordance with UOP952 on 8 stratified randomly selected subsamples.

	Lead in µg/kg
sample #21039-1	35
sample #21039-2	37
sample #21039-3	39
sample #21039-4	34
sample #21039-5	32
sample #21039-6	38
sample #21039-7	36
sample #21039-8	37

Table 12: homogeneity test results of subsamples #21039

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility calculated with the Horwitz equation in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Lead in µg/kg
r (observed)	6
reference method	Horwitz
0.3 x R (reference method)	8

Table 13: evaluation of the repeatability of subsamples #21039

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories, depending on the registration, the appropriate set of samples were sent on March 10, 2021. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Naphtha packed in amber glass bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were asked to determine on sample #21035: Organic Chlorides, Color Saybolt (Manual and/or Automated), Copper Corrosion 3hrs at 50°C, Density at 15°C, Distillation at 760 mmHg (IBP, 50% recovered and FBP), Mercaptan Sulfur as S and Sulfur. On samples #21037 and #21038: Mercury only. On samples #21039 and #21040: Arsenic and Lead only. On sample #21041: TVP / DVPE only.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the appropriate reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

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

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

3.1 STATISTICS

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

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

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

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

The $Z_{(\text{target})}$ scores are listed in the test result tables in appendix 1.

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare. Therefore, the usual interpretation of z-scores is as follows:

$ z < 1$	good
$1 < z < 2$	satisfactory
$2 < z < 3$	questionable
$3 < z $	unsatisfactory

4 EVALUATION

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week.

For the regular Naphtha PT ten participants reported test results after the final reporting date and seven other participants did not report any test results.

For the Mercury in Naphtha PT six participants reported test results after the final reporting date and fourteen other participants did not report any test results.

For the Arsenic and Lead in Naphtha PT two participants reported test results after the final reporting date and thirteen other participants did not report any test results.

For the Vapor Pressure in Naphtha PT seven participants reported test results after the final reporting date and six other participants did not report any test results.

In total 90 participants reported 753 numerical test results. Observed were 17 outlying test results, which is 2.3%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER SAMPLE AND PER TEST

In this section the reported test results are discussed per sample and per test. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data. The abbreviations, used in these tables, are explained in appendix 3.

Unfortunately, a suitable reference test method providing the precision data is not available for all determinations. For the tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

Sample #21035

Organic Chlorides: This determination was problematic. One statistical outlier was observed.

The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirement of UOP779:08, nor in agreement with the requirements of ASTM D5808:20 but is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Color Saybolt: This determination was problematic for the automated and manual mode. One statistical outlier was observed. The calculated reproducibilities for the automated and the manual modes are both not in agreement with the respective requirements of ASTM D6045:20 and ASTM D156:15.

Copper Corrosion: This determination was not problematic. All reporting laboratories agreed on a result of 1 (1a/1b).

Density at 15°C: This determination was problematic for a number of laboratories. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirement of ISO12185:96.

Distillation: This determination was not problematic. In total three statistical outliers were observed. All calculated reproducibilities after rejection of the statistical outliers are in agreement with the requirements of ASTM D86:20b for the automated and manual mode.

Mercaptan Sulfur: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirement of ASTM D3227:16.

Sulfur: This determination was problematic dependent on the test method used. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirement of ASTM D4294:16e1 but not with the strict ASTM D2622:16 or ASTM D5453:20.

Samples #21037 and #21038

Mercury: This determination was not problematic for both samples. No statistical outliers were observed. The calculated reproducibilities are in agreement with the estimated reproducibilities calculated with the Horwitz equation.

Samples #21039 and #21040

Arsenic: This determination for sample #21039 and #21040 was very problematic. Both samples were spiked with Arsenic, but the laboratories did not find Arsenic in the samples. All participants agreed on a test result less than 50 µg/kg. Therefore, no z-scores were calculated. It is assumed that Arsenic had adsorbed to the wall of the glass bottle. It is recommended to rinse with strong acid for this determination.

Lead: This determination may be problematic for both samples. In total one statistical outlier was observed. Both calculated reproducibilities after rejection of the statistical outlier are not in agreement with the estimated reproducibilities calculated with the Horwitz equation.

Sample #21041

TVP: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D5191:20.

DVPE: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D5191:20.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method or as declared by the estimated target reproducibility calculated with the Horwitz equation and the reproducibility as found for the group of the participating laboratories. The number of significant test results, the average, the calculated reproducibility ($2.8 \times$ standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM, UOP and ISO reference test methods) or estimated calculated with the Horwitz equation are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R(lit)
Organic Chlorides	mg/kg	41	6.1	2.2	1.4
Color Saybolt (automated)		40	28.4	2.6	1.2
Color Saybolt (manual)		29	28.3	3.2	2
Copper Corrosion		62	1(1a/1b)	n.a.	n.a.
Density at 15°C	kg/L	78	0.7191	0.0004	0.0005
Initial Boiling Point	°C	71	37.4	4.7	4.7
50% recovered	°C	72	108.0	1.7	4.1
Final Boiling Point	°C	73	166.0	7.4	7.1

Parameter	unit	n	average	2.8 * sd	R(lit)
Mercaptan Sulfur as S	mg/kg	57	76.4	7.5	6.3
Sulfur	mg/kg	77	255.9	47.0	68.4

Table 14: reproducibilities of tests on sample #21035

Parameter	unit	n	average	2.8 * sd	R(lit)
Mercury as Hg #21037	µg/kg	36	83.7	48.7	54.5
Mercury as Hg #21038	µg/kg	36	213	115	120

Table 15: reproducibilities of tests on sample #21037 and #21038

Parameter	unit	n	average	2.8 * sd	R(lit)
Arsenic as As #21039	µg/kg	11	<50	n.e.	n.e.
Arsenic as As #21040	µg/kg	10	<50	n.e.	n.e.
Lead as Pb #21039	µg/kg	18	46.6	42.3	33.1
Lead as Pb #21040	µg/kg	17	51.8	46.1	36.2

Table 16: reproducibilities of tests on sample #21039 and #21040

Parameter	unit	n	average	2.8 * sd	R(lit)
TVP	psi	38	7.06	0.31	0.23
DVPE	psi	46	6.27	0.35	0.23

Table 17: reproducibilities of tests on sample #21041

Without further statistical calculations, it can be concluded that for a number of tests there is a good compliance of the group of participating laboratories with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF APRIL 2021 WITH PREVIOUS PTS

	April 2021	April 2020	April 2019	April 2018	April 2017
Number of reporting laboratories	90	74	93	104	100
Number of test results	753	1446	1635	1831	1723
Number of statistical outliers	17	130	73	88	84
Percentage of statistical outliers	2.3%	9.0%	4.5%	4.8%	4.9%

Table 18: 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 reference test methods. The conclusions are given in the following table.

Parameter	April 2021	April 2020	April 2019	April 2018	April 2017
Organic Chlorides	-	--	--	n.e.	+/-
Color Saybolt	--	--	+	--	++
Density at 15°C	+	+/-	+	+	+/-
Distillation	+	+/-	+/-	+/-	+/-
Mercaptan Sulfur as S	-	-	-	--	-
Sulfur	+	+	+	+	+/-
Mercury	+	+	+	+	++
Arsenic	--	--	+/-	+/-	+/-
Lead	-	-	+/-	+/-	-
Total Vapor Pressure	-	-	-	+	++
DVPE acc. to D5191	-	-	-	+	+

Table 19: comparison determinations against the reference test methods

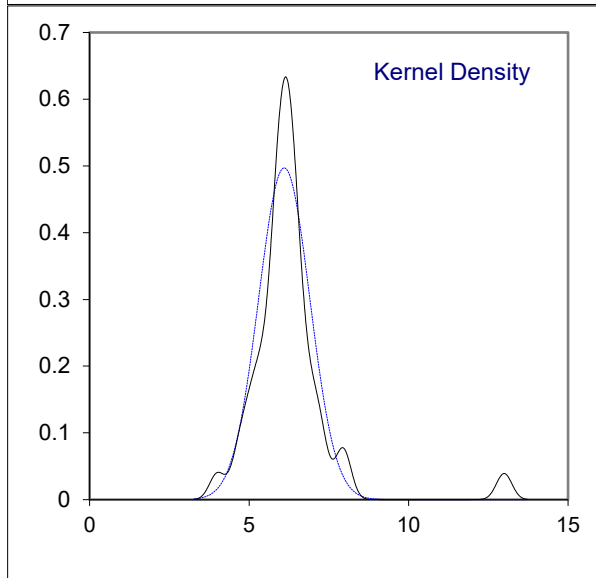
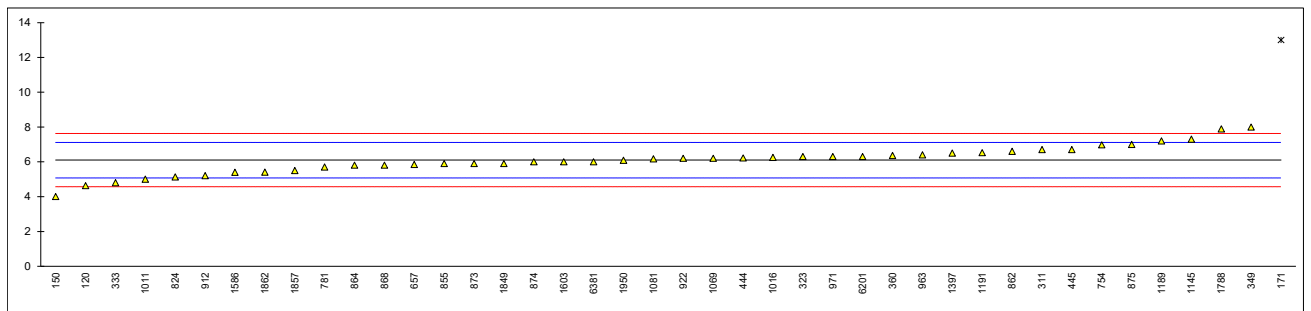
The following performance categories were used:

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

APPENDIX 1**Determination of Organic Chlorides on sample #21035; results in mg/kg**

lab	method	value	mark	z(targ)	remarks
120	D5808	4.63		-2.88	
140		----		----	
150	D7359	4.0		-4.12	
158		----		----	
171	UOP779	13	R(0.01)	13.54	
225		----		----	
237		----		----	
238		----		----	
311	D5808	6.7		1.18	
323	UOP779	6.3		0.39	
328		----		----	
333	D5808	4.8		-2.55	
334		----		----	
337		----		----	
349	UOP588	8		3.73	
360	UOP779	6.36		0.51	
399		----		----	
444	IP510	6.22		0.24	
445	IP510	6.7		1.18	
492		----		----	
495		----		----	
541		----		----	
608		----		----	
657	UOP779	5.849		-0.49	
663		----		----	
750		----		----	
753		----		----	
754	UOP779	6.97		1.71	
779		----		----	
781	UOP779	5.7		-0.78	
785		----		----	
798		----		----	
824	UOP779	5.13		-1.90	
855	UOP779	5.9		-0.39	
862	D5808	6.6		0.98	
864	D5808	5.8		-0.59	
868	D5808	5.8		-0.59	
872		----		----	
873	UOP779	5.9		-0.39	
874	UOP779	6.0		-0.20	
875	UOP779	7.0		1.77	
912	D4929B	5.2		-1.77	
914		----		----	
922	D4929-A	6.2		0.20	
962		----		----	
963	UOP779	6.4		0.59	
971	UOP779	6.3		0.39	
974		----		----	
994		----		----	
1011	D5808	5		-2.16	
1012		----		----	
1016	In house	6.25		0.30	
1026		----		----	
1062		----		----	
1065		----		----	
1069	D7359	6.2		0.20	
1081	D5808	6.17		0.14	
1097		----		----	
1145	D5808	7.30		2.36	
1189	UOP779	7.2		2.16	
1191	UOP779	6.52		0.83	
1254		----		----	
1320		----		----	
1381		----		----	
1397	D4929	6.5		0.79	
1457		----		----	
1556		----		----	
1586	UOP779	5.4		-1.37	
1603	In house	6		-0.20	
1656		----		----	
1737		----		----	
1776		----		----	
1788	D5808	7.89		3.51	
1796		----		----	

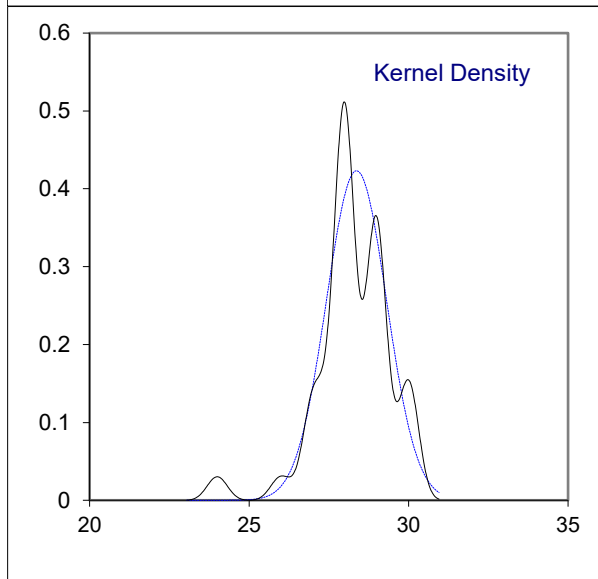
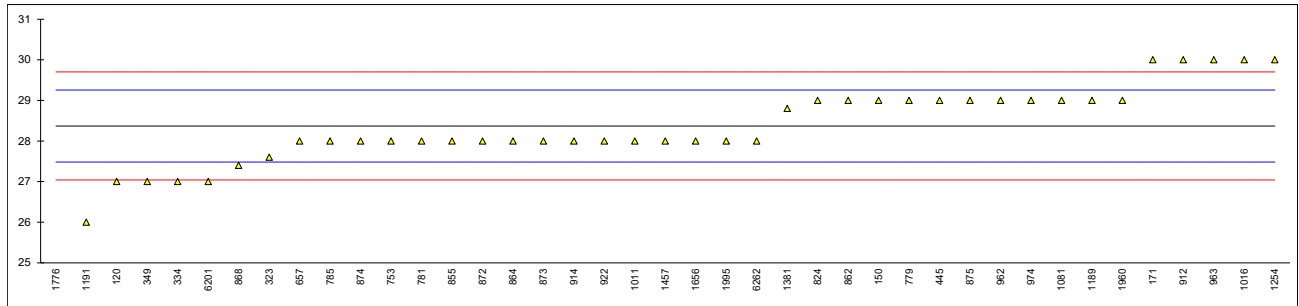
lab	method	value	mark	z(targ)	remarks
1849	D7359	5.9		-0.39	
1857	UOP779	5.50		-1.18	
1862	UOP779	5.41		-1.35	
1949		-----		-----	
1950	UOP779	6.08		-0.04	
1960		-----		-----	
1995		-----		-----	
6198		-----		-----	
6200		-----		-----	
6201	UOP779	6.3		0.39	
6262		-----		-----	
6299		-----		-----	
6381	D4929	6		-0.20	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
normality		OK			
n		41			
outliers		1			
mean (n)		6.099			
st.dev. (n)		0.8028			
R(calc.)		2.248			
st.dev.(UOP779:08)		0.5096			
R(UOP779:08)		1.427			Application range: 0.3 – 1000 mg/kg
Compare					
R(D5808:20)		1.3			Application range: 1 – 25 mg/kg
R(Horwitz)		2.081			



Determination of Color Saybolt (automated) on sample #21035

lab	method	value	mark	z(targ)	cuvette	remarks
120	D6045	27		-3.09	50 mm	
140		----		----		
150	D6045	29		1.42	100 mm	
158		----		----		
171	D6045	30		3.68		
225		----		----		
237		----		----		
238		----		----		
311		----		----		
323	D6045	27.6		-1.74	50 mm	
328		----		----		
333		----		----		
334	D6045	27		-3.09	50 mm	
337		----		----		
349	D6045	27		-3.09		
360		----		----		
399		----		----		
444	D6045	>30		>3.68	50 mm	possibly a false positive test result?
445	D6045	29		1.42	50 mm	
492		----		----		
495		----		----		
541	D6045	>30	C	>3.68	100 mm	possibly a false positive test result? First reported -9
608		----		----		
657	D6045	28		-0.84	100 mm	
663		----		----		
750		----		----		
753	D6045	28		-0.84	100 mm	
754		----		----		
779	D6045	29		1.42	50 mm	
781	D6045	28		-0.84	100 mm	
785	D6045	28		-0.84	50 mm	
798		----		----		
824	D6045	29		1.42	50 mm	
855	D6045	28		-0.84	50 mm	
862	D6045	29		1.42	50 mm	
864	D6045	28		-0.84	100 mm	
868	D6045	27.4		-2.19		
872	D6045	28		-0.84		
873	D6045	28		-0.84	100 mm	
874	D6045	28		-0.84	33 mm	
875	D6045	29		1.42	50 mm	
912	D6045	30		3.68		
914	D6045	28		-0.84		
922	D6045	28		-0.84	100 mm	
962	D6045	29		1.42	50 mm	
963	D6045	30		3.68		
971		----		----		
974	D6045	29		1.42	100 mm	
994		----		----		
1011	D6045	28		-0.84	100 mm	
1012	D6045	>+30		>3.68	50 mm	possibly a false positive test result?
1016	D156	30		3.68	100 mm	
1026		----		----		
1062		----		----		
1065		----		----		
1069		----		----		
1081	D6045	29		1.42	100 mm	
1097		----		----		
1145		----		----		
1189	D6045	29		1.42	100 mm	
1191	D6045	26		-5.35	100 mm	
1254	D6045	30		3.68	100 mm	
1320		----		----		
1381	D6045	28.8		0.97	50 mm	
1397		----		----		
1457	D6045	28		-0.84	50 mm	
1556		----		----		
1586	D6045	>+30		>3.68	50 mm	possibly a false positive test result?
1603		----		----		
1656	D5386	28		-0.84	50 mm	
1737		----		----		
1776	D6045	24	R(0.01)	-9.87		
1788		----		----		
1796		----		----		
1849		----		----		

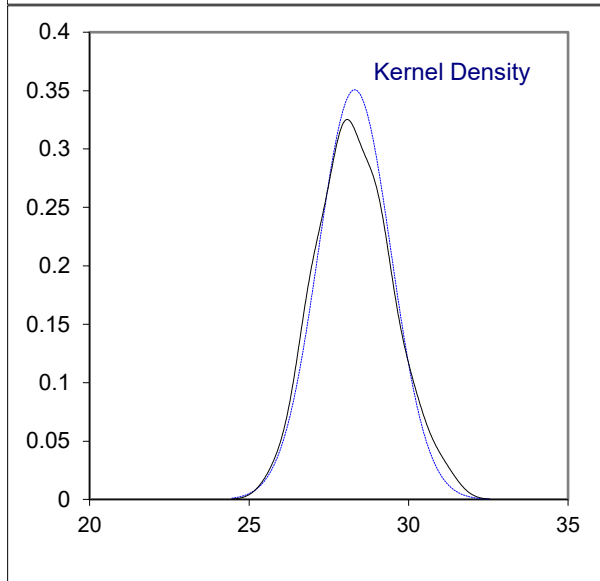
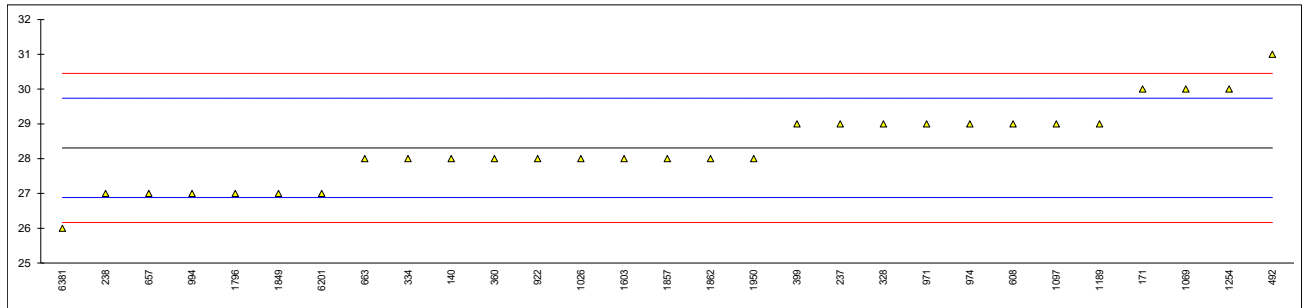
lab	method	value	mark	z(targ)	cuvette	remarks
1857		----		----		
1862		----		----		
1949		----		----		
1950		----		----		
1960	D6045	29		1.42	100 mm	
1995	D6045	28		-0.84	33 mm	
6198		----		----		
6200		----		----		
6201	D6045	27		-3.09		
6262	D6045	28.0		-0.84	50 mm	
6299		----		----		
6381		----		----		
9057		----		----		
9058		----		----		
9061		----		----		
normality		OK				
n		40				
outliers		1				
mean (n)		28.370				
st.dev. (n)		0.9430				
R(calc.)		2.641				
st.dev.(D6045:20)		0.4429				
R(D6045:20)		1.24				



Determination of Color Saybolt (manual) on sample #21035

lab	method	value	mark	z(targ)	filter	remarks
120		----		----		
140	D156	28		-0.43		
150		----		----		
158		----		----		
171	D156	30		2.37		
225		----		----		
237	D156	29		0.97		
238	D156	27		-1.83		
311		----		----		
323		----		----		
328	D156	29		0.97		
333		----		----		
334	D156	28		-0.43	0.5	
337		----		----		
349		----		----		
360	D156	28		-0.43	0.5	
399	D156	29		0.97		
444		----		----		
445		----		----		
492	D156	31		3.77		
495		----		----		
541		----		----		
608	D156	29		0.97		
657	D156	27		-1.83	0.5	
663	D156	28		-0.43		
750		----		----		
753		----		----		
754		----		----		
779		----		----		
781		----		----		
785		----		----		
798		----		----		
824		----		----		
855		----		----		
862		----		----		
864		----		----		
868		----		----		
872		----		----		
873		----		----		
874		----		----		
875		----		----		
912		----		----		
914		----		----		
922	D156	28		-0.43	0.5	
962		----		----		
963		----		----		
971	D156	29		0.97	0.5	
974	D156	29		0.97	0.5	
994	D156	27		-1.83	0.5	
1011		----		----		
1012		----		----		
1016		----		----		
1026	D156	28		-0.43		
1062		----		----		
1065		----		----		
1069	D156	30		2.37		
1081		----		----		
1097	NF M07-203	29		0.97	0.5	
1145		----		----		
1189	D156	29		0.97		
1191		----		----		
1254	D156	30		2.37	0.5	
1320		----		----		
1381		----		----		
1397		----		----		
1457		----		----		
1556		----		----		
1586		----		----		
1603	In house	28		-0.43		
1656		----		----		
1737		----		----		
1776		----		----		
1788		----		----		
1796	D156	27		-1.83		
1849	TS2991	27		-1.83	0.5	

lab	method	value	mark	z(targ)	filter	remarks
1857	D156	28		-0.43	0.5	
1862	D156	28		-0.43		
1949		----		----		
1950	D156	28		-0.43		
1960		----		----		
1995		----		----		
6198		----		----		
6200		----		----		
6201	D156	27		-1.83		
6262		----		----		
6299		----		----		
6381	D156	26		-3.23		
9057		----		----		
9058		----		----		
9061		----		----		
normality		OK				
n		29				
outliers		0				
mean (n)		28.310				
st.dev. (n)		1.1371				
R(calc.)		3.184				
st.dev.(D156:15)		0.7143				
R(D156:15)		2				



Determination of Copper Corrosion 3 hrs at 50°C on sample #21035

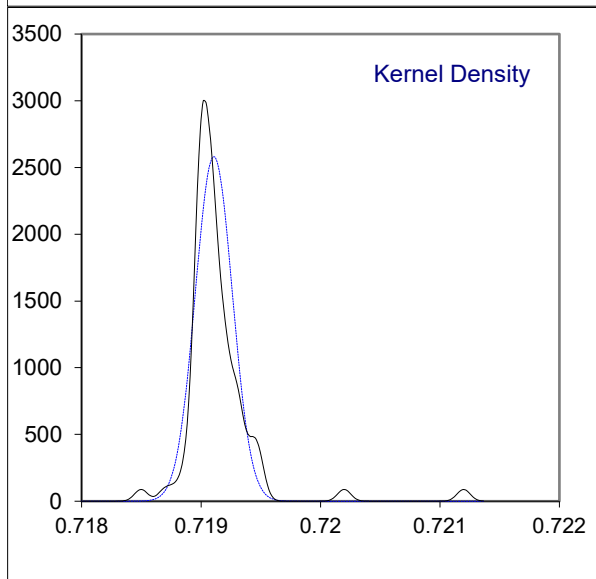
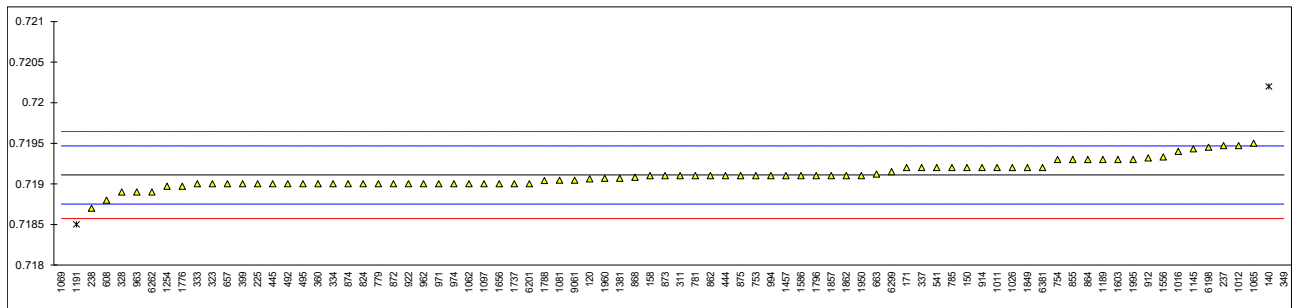
lab	method	value	mark	z(targ)	remarks
120	D130	1A		----	
140	D130	1a		----	
150	D130	1A		----	
158	D130	1A		----	
171	D130	1a		----	
225	D130	1a		----	
237	D130	1A		----	
238	D130	1a		----	
311	D130	1A		----	
323		----		----	
328	D130	1		----	
333		----		----	
334	D130	1A		----	
337		----		----	
349		----		----	
360	D130	1A		----	
399	D130	1A		----	
444		----		----	
445	D130	1a		----	
492		----		----	
495		----		----	
541	D130	1a		----	
608	D130	1a		----	
657	D130	1A		----	
663	D130	1a		----	
750		----		----	
753	D130	1A		----	
754	D130	1a		----	
779		----		----	
781	D130	1a		----	
785	D130	1a		----	
798		----		----	
824	D130	1a		----	
855	D130	1a		----	
862	D130	1a		----	
864	D130	1a		----	
868	D130	1a		----	
872		----		----	
873	D130	1a		----	
874	D130	1a		----	
875	D130	1a		----	
912	D130	1A		----	
914	D130	1a		----	
922	D130	1a		----	
962	D130	1a		----	
963	D130	1a		----	
971	D130	1a		----	
974	D130	1a		----	
994	D130	1a		----	
1011	D130	1a		----	
1012	D130	1a		----	
1016	D130	1A		----	
1026	ISO2160	1A		----	
1062		----		----	
1065		----		----	
1069		----		----	
1081		----		----	
1097	ISO2160	1a		----	
1145		----		----	
1189		----		----	
1191	ISO2160	1a		----	
1254	D130	1A		----	
1320		----		----	
1381	ISO2160	1A		----	
1397		----		----	
1457	D130	1a		----	
1556	ISO2160	Class 1a		----	
1586	D130	1A		----	
1603	In house	1A		----	
1656	IP154	1a		----	
1737		----		----	
1776		----		----	
1788	D130	1a		----	
1796	D130	1a		----	
1849	ISO2160	1a		----	

lab	method	value	mark	z(targ)	remarks
1857	D130	1a		----	
1862	D130	1A		----	
1949		----		----	
1950	D130	1a		----	
1960		----		----	
1995	D130	1a		----	
6198		----		----	
6200		----		----	
6201	D130	1a		----	
6262	D130	1B		----	
6299	ISO2160	1a		----	
6381	ISO2160	1a		----	
9057		----		----	
9058		----		----	
9061		----		----	
	n	62			
	mean (n)	1 (1a/1b)			

Determination of Density at 15°C on sample #21035; results in kg/L

lab	method	value	mark	z(targ)	remarks
120	D4052	0.71906		-0.27	
140	D4052	0.7202	R(0.01)	6.11	
150	D4052	0.7192		0.51	
158	D4052	0.7191	C	-0.05	first reported 0.1786
171	D4052	0.7192		0.51	
225	D4052	0.7190		-0.61	
237	D4052	0.71947		2.02	
238	D4052	0.7187		-2.29	
311	ISO12185	0.7191		-0.05	
323	D4052	0.7190		-0.61	
328	ISO12185	0.7189		-1.17	
333	ISO12185	0.7190		-0.61	
334	ISO12185	0.7190		-0.61	
337	ISO12185	0.7192		0.51	
349	D4052	0.7212	R(0.01)	11.71	
360	D4052	0.7190		-0.61	
399	D4052	0.7190		-0.61	
444	D4052	0.7191		-0.05	
445	IP365	0.7190		-0.61	
492	D4052	0.719		-0.61	
495	ISO12185	0.7190		-0.61	
541	ISO12185	0.7192	C	0.51	first reported 0.74657
608	D4052	0.7188		-1.73	
657	ISO12185	0.7190		-0.61	
663	D4052	0.71912		0.06	
750		----		----	
753	D4052	0.7191		-0.05	
754	D4052	0.7193		1.07	
779	D4052	0.7190		-0.61	
781	ISO12185	0.7191		-0.05	
785	D4052	0.7192		0.51	
798		----		----	
824	ISO12185	0.7190		-0.61	
855	D4052	0.7193		1.07	
862	D4052	0.7191		-0.05	
864	D4052	0.7193		1.07	
868	D4052	0.71908		-0.16	
872	ISO12185	0.7190		-0.61	
873	ISO12185	0.7191		-0.05	
874	ISO12185	0.7190		-0.61	
875	D4052	0.7191		-0.05	
912	D4052	0.71932		1.18	
914	D4052	0.7192		0.51	
922	D4052	0.7190		-0.61	
962	D4052	0.7190		-0.61	
963	ISO12185	0.7189		-1.17	
971	D4052	0.7190		-0.61	
974	D4052	0.7190		-0.61	
994	D4052	0.7191		-0.05	
1011	ISO12185	0.7192		0.51	
1012	D4052	0.71947		2.02	
1016	D4052	0.7194		1.63	
1026	D4052	0.7192		0.51	
1062	D4052	0.7190		-0.61	
1065	D4052	0.7195		2.19	
1069	D4052	0.7153	R(0.01)	-21.33	Reported Density at 20°C
1081	D4052	0.719045		-0.36	
1097	ISO12185	0.7190		-0.61	
1145	D4052	0.71943		1.80	
1189	D4052	0.7193		1.07	
1191	ISO12185	0.7185	R(0.05)	-3.41	
1254	D4052	0.71897		-0.78	
1320		----		----	
1381	ISO12185	0.71907		-0.22	
1397		----		----	
1457	D4052	0.7191		-0.05	
1556	ISO12185	0.71933		1.24	
1586	D4052	0.7191		-0.05	
1603	In house	0.7193		1.07	
1656	D4052	0.7190		-0.61	
1737	D4052	0.7190		-0.61	
1776	ISO12185	0.71897		-0.78	
1788	D4052	0.71904		-0.39	
1796	D4052	0.7191		-0.05	
1849	ISO12185	0.7192		0.51	

lab	method	value	mark	z(target)	remarks
1857	ISO12185	0.7191		-0.05	
1862	D4052	0.7191		-0.05	
1949		-----		-----	
1950	D4052	0.7191		-0.05	
1960	D4052	0.719068		-0.23	
1995	D4052	0.7193		1.07	
6198	D4052	0.71945		1.91	
6200		-----		-----	
6201	D4052	0.7190		-0.61	
6262	D4052	0.7189		-1.17	
6299	ISO12185	0.71915		0.23	
6381	ISO12185	0.7192		0.51	
9057		-----		-----	
9058		-----		-----	
9061	D5002	0.719045		-0.36	
normality		OK			
n		78			
outliers		4			
mean (n)		0.71911			
st.dev. (n)		0.000155			
R(calc.)		0.00043			
st.dev.(ISO12185:96)		0.000179			
R(ISO12185:96)		0.0005			

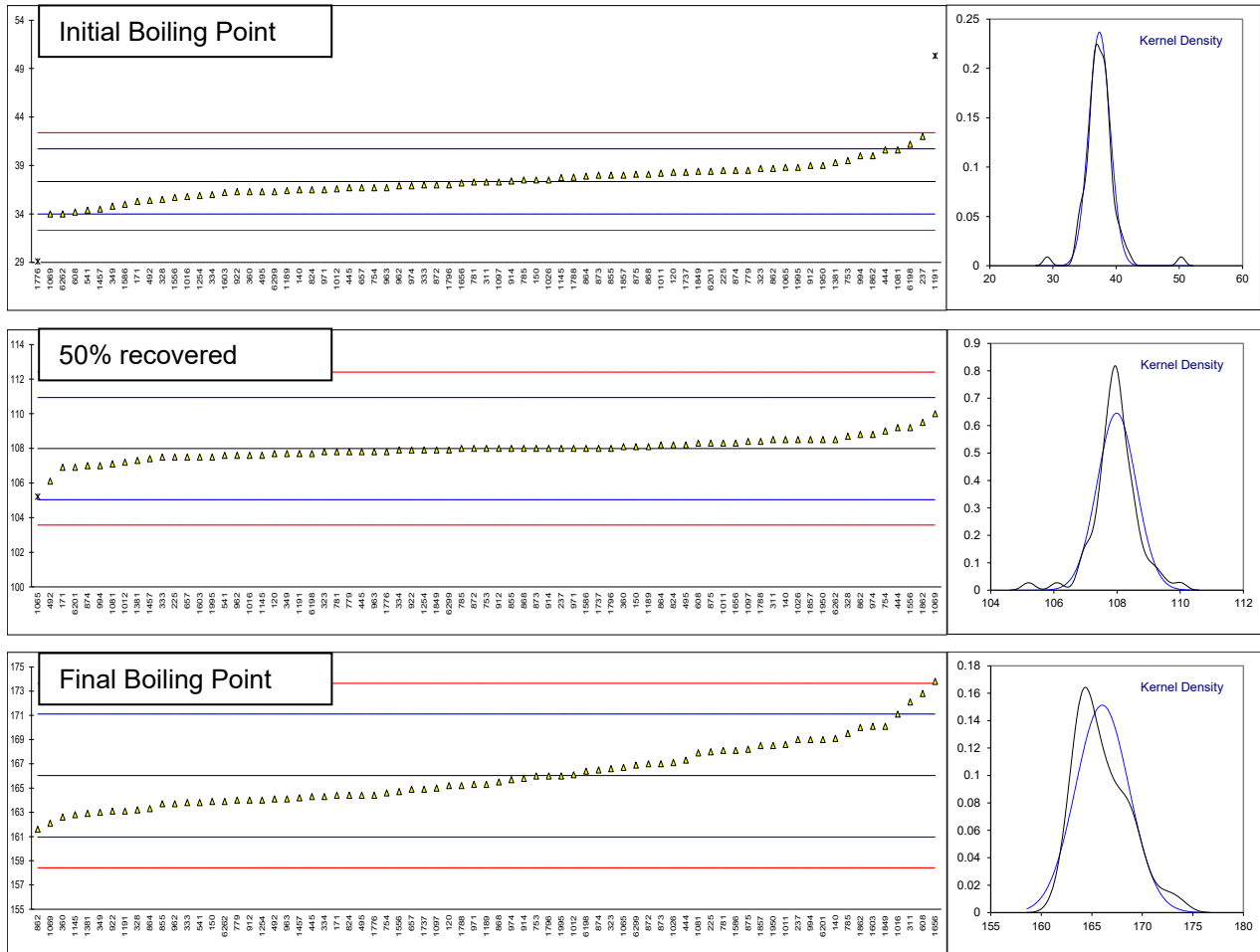


Determination of Distillation at 760 mmHg on sample #21035; results in °C

lab	method	IBP	mark	z(targ)	50%rec	mark	z(targ)	FBP	mark	z(targ)
120	D86-automated	38.3		0.57	107.7		-0.19	165.2		-0.33
140	D86-automated	36.5		-0.51	108.5		0.35	169.1		1.21
150	D86-automated	37.5		0.09	108.1		0.08	163.9		-0.84
158		----		----	----		----	----		----
171	D86-automated	35.3		-1.22	106.9		-0.74	164.4		-0.64
225	D86-manual	38.5		0.68	107.5		-0.33	168.0		0.78
237	D86-manual	42.0		2.77	108.0		0.01	169.0		1.17
238		----		----	----		----	----		----
311	D86-automated	37.3		-0.03	108.5		0.35	172.1		2.39
323	D86-automated	38.7		0.80	107.8		-0.13	166.6		0.22
328	D86	35.5		-1.10	108.7		0.49	163.2		-1.12
333	D86-automated	37.0		-0.21	107.5		-0.33	163.8		-0.88
334	D86-automated	36.0		-0.81	107.9		-0.06	164.3		-0.68
337		----		----	----		----	----		----
349	D86-automated	34.8		-1.52	107.7		-0.19	163.0		-1.20
360	D86-automated	36.3		-0.63	108.1		0.08	162.6		-1.35
399		----		----	----		----	----		----
444	D86-automated	40.6		1.94	109.2		0.82	167.3		0.50
445	D86-automated	36.7		-0.39	107.8		-0.13	164.3		-0.68
492	D86	35.4		-1.16	106.1		-1.28	164.1		-0.76
495	D86-automated	36.3		-0.63	108.2		0.15	164.4		-0.64
541	D86-automated	34.4	C	-1.76	107.6	C	-0.26	163.8	C	-0.88
608	D86-automated	34.2		-1.88	108.3		0.21	172.8		2.67
657	D86-automated	36.7		-0.39	107.5		-0.33	164.9		-0.45
663		----		----	----		----	----		----
750		----		----	----		----	----		----
753	D86-manual	39.5		1.28	108.0		0.01	166.0		-0.01
754	D86-automated	36.7		-0.39	109.0		0.69	164.6		-0.56
779	D86-manual	38.5		0.68	107.8		-0.13	164.0		-0.80
781	D86-automated	37.3		-0.03	107.8		-0.13	168.1		0.82
785	D86-manual	37.5		0.09	108.0		0.01	169.5		1.37
798		----		----	----		----	----		----
824	D86-automated	36.5		-0.51	108.2		0.15	164.4		-0.64
855	D86-automated	38.0		0.39	108.0		0.01	163.7		-0.92
862	D86-automated	38.7		0.80	108.8	C	0.55	161.6		-1.75
864	D86-automated	37.9		0.33	108.2		0.15	163.3		-1.08
868	D86-automated	38.1		0.45	108.0		0.01	165.5		-0.21
872	D86-manual	37		-0.21	108		0.01	167		0.38
873	D86-manual	38.0		0.39	108.0		0.01	167.0		0.38
874	D86-manual	38.5		0.68	107.0		-0.67	166.5		0.18
875	D86-automated	38.1		0.45	108.3		0.21	168.2		0.86
912	D86	39.0		0.98	108.0		0.01	164.0		-0.80
914	D86	37.4		0.03	108.0		0.01	165.8		-0.09
922	D86-automated	36.3		-0.63	107.9		-0.06	163.1		-1.16
962	D86-automated	36.9		-0.27	107.6		-0.26	163.7		-0.92
963	D86-automated	36.7		-0.39	107.8		-0.13	164.1		-0.76
971	D86-automated	36.5		-0.51	108.0		0.01	165.3		-0.29
974	D86-automated	36.9		-0.27	108.8		0.55	165.7		-0.13
994	D86-manual	40.0		1.58	107.0		-0.67	169.0		1.17
1011	D86-automated	38.2		0.51	108.3		0.21	168.6		1.01
1012	D86-automated	36.6		-0.45	107.2		-0.53	166.1		0.03
1016	D86-automated	35.8		-0.92	107.6		-0.26	171.1		2.00
1026	ISO3405-automated	37.5		0.09	108.5		0.35	167.1		0.42
1062		----		----	----		----	----		----
1065		38.8		0.86	105.2	R(0.01)	-1.89	166.7		0.26
1069	D86-automated	34.0		-2.00	110.0		1.37	162.1		-1.55
1081		40.6		1.94	107.1		-0.60	167.9		0.74
1097	ISO3405-automated	37.3		-0.03	108.4		0.28	165.0		-0.41
1145		37.75		0.24	107.60		-0.26	162.80		-1.27
1189	D86-automated	36.4		-0.57	108.1		0.08	165.3		-0.29
1191	ISO3405-automated	50.3	R(0.01)	7.71	107.7		-0.19	163.1		-1.16
1254	D86-automated	35.9		-0.86	107.9		-0.06	164.0		-0.80
1320		----		----	----		----	----		----
1381	ISO3405-automated	39.30		1.16	107.30		-0.47	162.90		-1.23
1397		----		----	----		----	----		----
1457	D86-automated	34.5		-1.70	107.4		-0.40	164.2		-0.72
1556	Other (mention	35.7		-0.98	109.2		0.82	164.7		-0.53
1586	D86-automated	35.0		-1.40	108.0		0.01	168.1		0.82
1603	D86-automated	36.2		-0.69	107.5		-0.33	170.1		1.60
1656	IP123-automated	37.2		-0.09	108.3		0.21	173.8	C	3.06
1737		38.3		0.57	108.0		0.01	164.9		-0.45
1776	ISO3405-automated	29.1	R(0.01)	-4.92	107.8		-0.13	164.4		-0.64
1788	D86-automated	37.8		0.27	108.4		0.28	165.2		-0.33
1796	D86-automated	37		-0.21	108.0		0.01	166.0		-0.01
1849	ISO3405-automated	38.4		0.62	107.9		-0.06	170.1		1.60
1857	D86-manual	38.0		0.39	108.5		0.35	168.5		0.97

lab	method	IBP	mark	z(targ)	50%rec	mark	z(targ)	FBP	mark	z(targ)
1862	D86-manual	40.0		1.58	109.5		1.03	170.0		1.57
1949		----		----	----		----	----		----
1950	D86-manual	39.0		0.98	108.5		0.35	168.5		0.97
1960		----		----	----		----	----		----
1995	D86-automated	38.8		0.86	107.5		-0.33	166.0		-0.01
6198	D86-automated	41.2		2.29	107.7		-0.19	166.4		0.15
6200		----		----	----		----	----		----
6201	D86-automated	38.4		0.62	106.9		-0.74	169.0		1.17
6262	D86-automated	34.0		-2.00	108.5		0.35	163.9		-0.84
6299	ISO3405-automated	36.3		-0.63	107.9		-0.06	166.9		0.34
6381		----		----	----		----	----		----
9057		----		----	----		----	----		----
9058		----		----	----		----	----		----
9061		----		----	----		----	----		----
	normality	OK			suspect			OK		
	n	71			72			73		
	outliers	2			1			0		
	mean (n)	37.35			107.99			166.03		
	st.dev. (n)	1.685			0.618			2.636		
	R(calc.)	4.72			1.73			7.38		
	st.dev.(D86-A:20b)	1.679			1.472			2.536		
	R(D86-A:20b)	4.7			4.12			7.1		
Compare										
	R(D86-M:20b)	5.6			4.24			7.2		

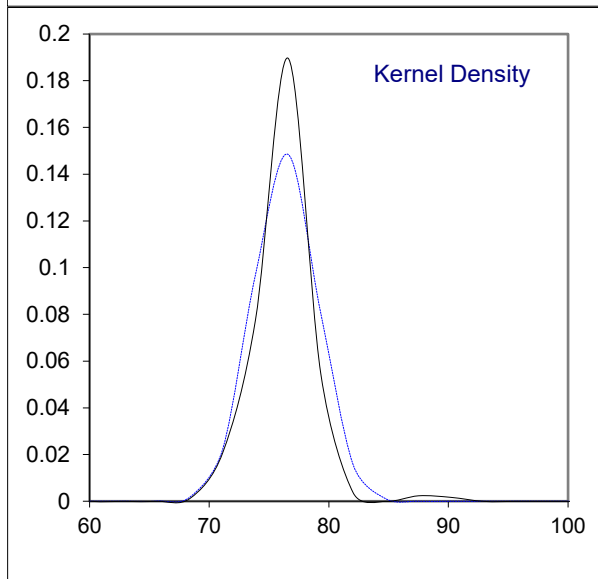
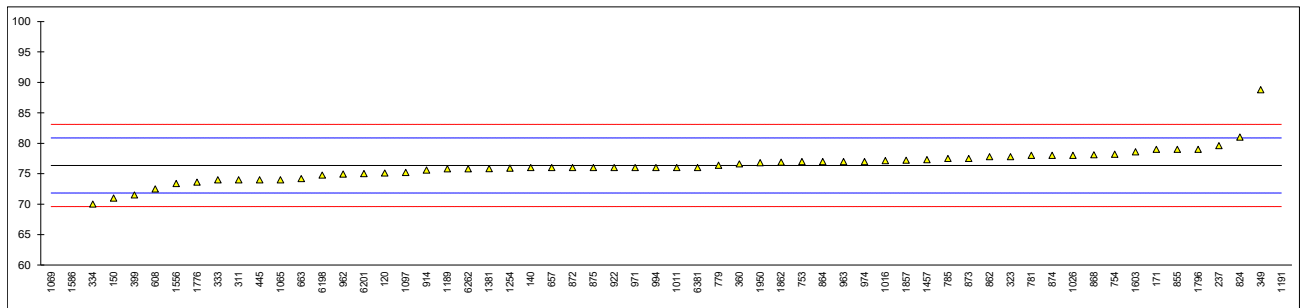
Lab 541: first reported 34.70, 94.90, 175.80 respectively
 Lab 862: first reported 180.8
 Lab 1656: first reported 179.2



Determination of Mercaptan Sulfur as S on sample #21035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	D3227	75.1		-0.56	
140	UOP163	76.0		-0.16	
150	D3227	71		-2.38	
158		----		----	
171	D3227	79		1.17	
225		----		----	
237	D3227	79.6		1.44	
238		----		----	
311	UOP163	74		-1.05	
323	UOP163	77.8		0.64	
328		----		----	
333	D3227	74		-1.05	
334	D3227	70		-2.82	
337		----		----	
349	UOP163	88.8		5.52	
360	D3227	76.6	C	0.11	first reported 59.6
399	D3227	71.5		-2.16	
444		----		----	
445	D3227	74		-1.05	
492		----		----	
495		----		----	
541		----		----	
608	UOP163	72.5	C	-1.71	first reported 68
657	D3227	76		-0.16	
663	D3227	74.2		-0.96	
750		----		----	
753	UOP163	77		0.29	
754	UOP163	78.2		0.82	
779	UOP163	76.4		0.02	
781	D3227	78		0.73	
785	UOP163	77.497		0.51	
798		----		----	
824	D3227	81		2.06	
855	D3227	79		1.17	
862	D3227	77.8		0.64	
864	D3227	77		0.29	
868	D3227	78.1		0.77	
872	D3227	76		-0.16	
873	D3227	77.5		0.51	
874	UOP163	78		0.73	
875	UOP163	76		-0.16	
912		----		----	
914	D3227	75.6		-0.34	
922	D3227	76		-0.16	
962	D3227	74.95		-0.62	
963	D3227	77		0.29	
971	D3227	76		-0.16	
974	D3227	77		0.29	
994	D3227	76		-0.16	
1011	D3227	76		-0.16	
1012		----		----	
1016	D3227	77.14		0.35	
1026	D3227	78		0.73	
1062		----		----	
1065	D3227	74		-1.05	
1069	D5623	16.3	R(0.01)	-26.66	
1081		----		----	
1097	ISO3012	75.2		-0.51	
1145		----		----	
1189	D3227	75.8		-0.25	
1191	UOP163	707.15	R(0.01)	280.04	
1254	D3227	75.9		-0.20	
1320		----		----	
1381	UOP163	75.84		-0.23	
1397		----		----	
1457	UOP163	77.3		0.42	
1556	UOP163	73.4		-1.31	
1586	D3227	56	C,R(0.01)	-9.04	first reported 65
1603	In house	78.6	C	1.00	first reported 65
1656		----		----	
1737		----		----	
1776	UOP163	73.6		-1.22	
1788		----		----	
1796	UOP163	79		1.17	
1849		----		----	

lab	method	value	mark	z(targ)	remarks
1857	UOP163	77.2		0.37	
1862	UOP163	76.9		0.24	
1949		----		----	
1950	UOP163	76.8		0.20	
1960		----		----	
1995		----		----	
6198	UOP163	74.765		-0.71	
6200		----		----	
6201	D3227	75		-0.60	
6262	D3227	75.8	C	-0.25	first reported 65.9
6299		----		----	
6381	UOP163	76		-0.16	
9057		----		----	
9058		----		----	
9061		----		----	
normality		not OK			
n		57			
outliers		3			
mean (n)		76.358			
st.dev. (n)		2.6776			
R(calc.)		7.497			
st.dev.(D3227:16)		2.2525			
R(D3227:16)		6.307			

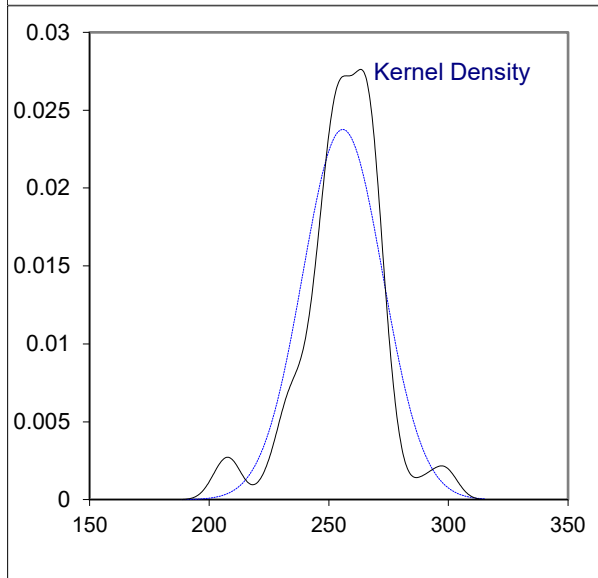
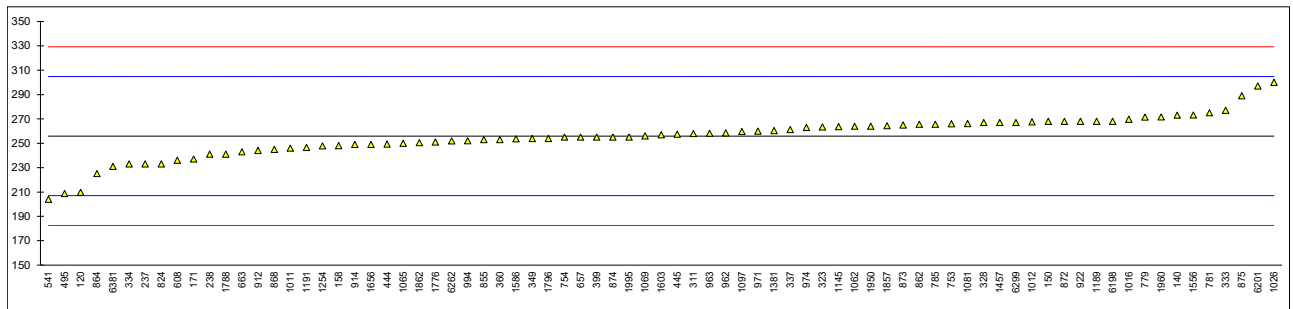


Determination of Sulfur on sample #21035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	D4294	209.7		-1.89	
140	D2622	273		0.70	
150	D2622	268		0.50	
158	D2622	248		-0.32	
171	D4294	237		-0.77	
225		----		----	
237	D5453	233		-0.94	
238	D4294	241.0		-0.61	
311	D2622	258		0.09	
323	D5453	263.4		0.31	
328	ISO20847	267		0.46	
333	D4294	277		0.87	
334	D4294	233		-0.94	
337	D5453	261.2		0.22	
349	D7039	254		-0.08	
360	D4294	253		-0.12	
399	D4294	255		-0.04	
444	D5453	249.3		-0.27	
445	D4294	257.4		0.06	
492		----		----	
495	ISO8754	208.7		-1.93	
541	D4294	204	C	-2.12	first reported <100
608	D5453	236		-0.81	
657	D4294	255		-0.04	
663	D5453	243		-0.53	
750		----		----	
753	D4294	266		0.42	
754	D4294	255		-0.04	
779	D4294	271.5		0.64	
781	D4294	275		0.78	
785	D2622	265.6		0.40	
798		----		----	
824	D5453	233		-0.94	
855	D5453	253		-0.12	
862	D5453	265.6		0.40	
864	D4294	225		-1.26	
868	D5453	245		-0.44	
872	D4294	268		0.50	
873	D4294	265		0.37	
874	D4294	255		-0.04	
875	D4294	289		1.36	
912	D5453	244		-0.49	
914	D4294	249		-0.28	
922	D4294	268		0.50	
962	D4294	258.6		0.11	
963	D4294	258.2		0.10	
971	D4294	260		0.17	
974	D4294	263		0.29	
994	D4294	252		-0.16	
1011	D4294	246		-0.40	
1012	D5453	267.565		0.48	
1016	D2622	269.7		0.57	
1026	D2622	300		1.81	
1062	D4294	264		0.33	
1065	D4294	250		-0.24	
1069	D5623	256.0		0.01	
1081	D2622	266.13		0.42	
1097	D5453	259.69		0.16	
1145	D5453	263.6		0.32	
1189	D4294	268		0.50	
1191	ISO8754	246.6		-0.38	
1254	D5453	247.8		-0.33	
1320		----		----	
1381	ISO8754	260.3		0.18	
1397		----		----	
1457	D4294	267		0.46	
1556	ISO20884	273.2		0.71	
1586	D5453	253.6		-0.09	
1603	In house	257		0.05	
1656	IP336	249		-0.28	
1737		----		----	
1776	ISO8754	251.0		-0.20	
1788	D5453	241.00		-0.61	
1796	D4294	254		-0.08	
1849		----		----	

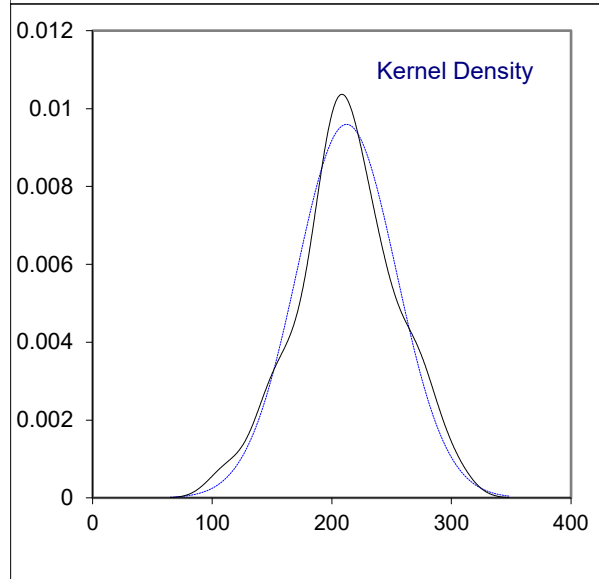
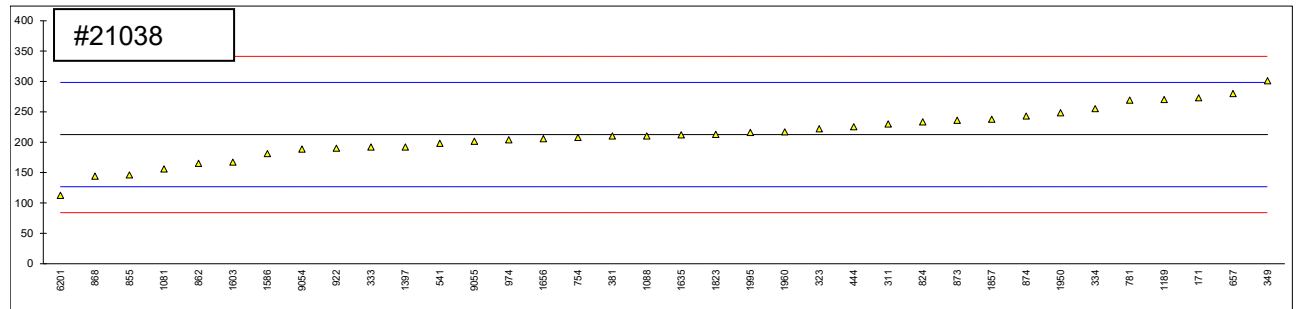
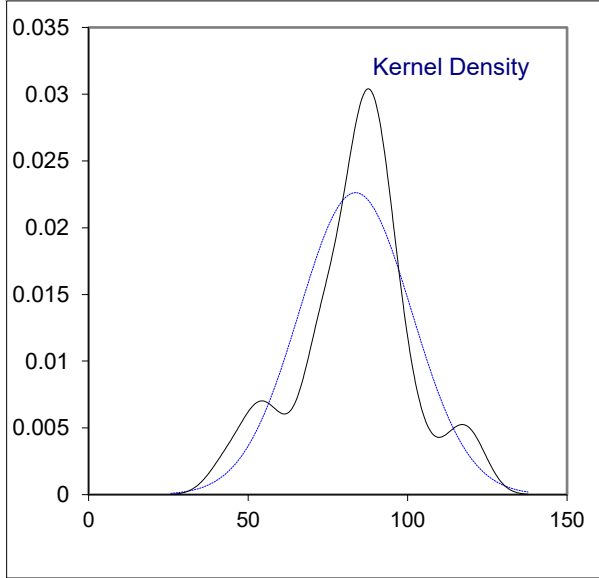
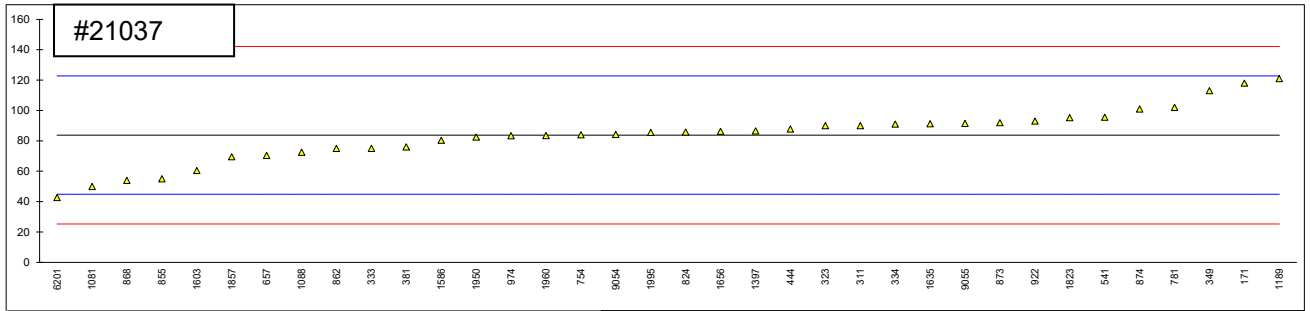
lab	method	value	mark	z(targ)	remarks
1857	D4294	264.4		0.35	
1862	D4294	250.5		-0.22	
1949		-----		-----	
1950	D4294	264		0.33	
1960	D5453	271.660		0.65	
1995	D4294	255		-0.04	
6198	D5453	268		0.50	
6200		-----		-----	
6201	D4294	297		1.68	
6262	D5453	251.9		-0.16	
6299	D5453	267		0.46	
6381	D2622	231		-1.02	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	

normality suspect
 n 77
 outliers 0
 mean (n) 255.855
 st.dev. (n) 16.7917
 R(calc.) 47.017
 st.dev.(D4294:16e1) 24.4303
 R(D4294:16e1) 68.405
 Compare
 R(D2622:16) 36.370
 R(D5453:20) 32.417



Determination of Mercury as Hg on sample #21037 and #21038; results in µg/kg

lab	method	#21037	mark	z(targ)	#21038	mark	z(targ)	remarks
140		----		----	----		----	
171	UOP938	118		1.76	273		1.41	
311	UOP938	90		0.32	230		0.41	
323	UOP938	90		0.32	222		0.22	
333	EPA7423	75		-0.45	192		-0.48	
334	INH-09003	91		0.37	255		0.99	
349	UOP938	113		1.50	301		2.06	
381	INH-118	76	C	-0.40	210		-0.06	first reported 233
444	UOP938	87.78		0.21	225.2		0.29	
541	INH-244	95.5		0.60	198.0		-0.34	
657	UOP938	70.4		-0.69	280		1.57	
663		----		----	----		----	
750		----		----	----		----	
754	UOP938	84.055		0.02	207.721		-0.11	
781	D7622	102		0.94	269		1.31	
798		----		----	----		----	
824	UOP938	85.797		0.11	233.326		0.48	
855	UOP938	55		-1.48	146		-1.55	
862	UOP938	75		-0.45	165		-1.11	
868	UOP938	54		-1.53	144		-1.60	
873	UOP938	92		0.42	236		0.55	
874	UOP938	101		0.89	243		0.71	
912		----		----	----		----	
922	INH-001	93		0.48	190		-0.53	
963		----		----	----		----	
974	UOP938	83.40		-0.02	203.8		-0.20	
1081	In house	50		-1.73	156		-1.32	
1088	D6350	72.49		-0.58	210.0		-0.06	
1135		----		----	----		----	
1145		----		----	----		----	
1189	UOP938	121		1.91	270		1.34	
1397	In house	86.5		0.14	192		-0.48	
1586	UOP938	80.3420		-0.17	181.1257		-0.73	
1603	In house	60.4	C	-1.20	167.1		-1.06	first reported 227.9
1635	In house	91.3		0.39	212.0		-0.01	
1656	UOP938	86.3		0.13	205.8		-0.16	
1823	D7623	95.23		0.59	212.7		0.00	
1857	UOP938	69.5		-0.73	237.7		0.59	
1949		----		----	----		----	
1950	UOP938	82.5		-0.06	248.4		0.83	
1960	UOP938	83.4043		-0.02	216.872		0.10	
1995	UOP938	85.6		0.10	215.8		0.08	
6200		----		----	----		----	
6201	UOP938	42.6		-2.11	112.4		-2.33	
6262		----		----	----		----	
9054	UOP938	84.2		0.02	188.55		-0.56	
9055	UOP938	91.5		0.40	201.4		-0.26	
9057		----		----	----		----	
9058		----		----	----		----	
9061		----		----	----		----	
	normality	OK			OK			
	n	36			36			
	outliers	0			0			
	mean (n)	83.744			212.553			
	st.dev. (n)	17.3865			40.9947			
	R(calc.)	48.682			114.785			
	st.dev.(Horwitz)	19.4620			42.9351			
	R(Horwitz)	54.494			120.218			
Compare								
	R(UOP938:20)	10.937			27.760			

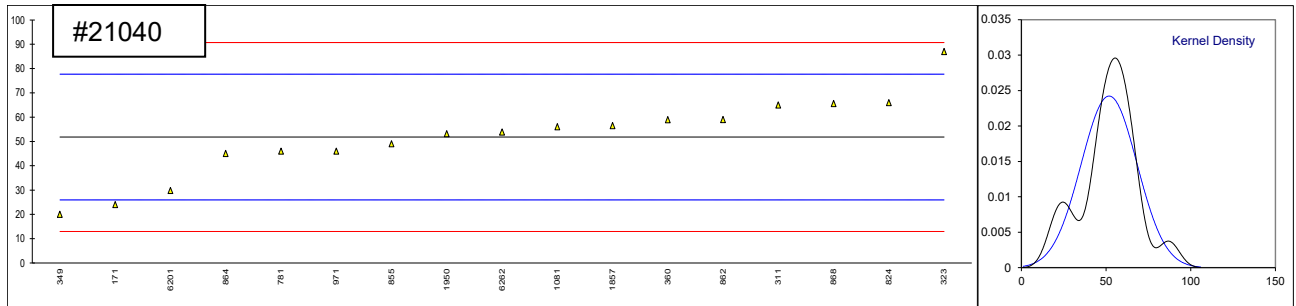
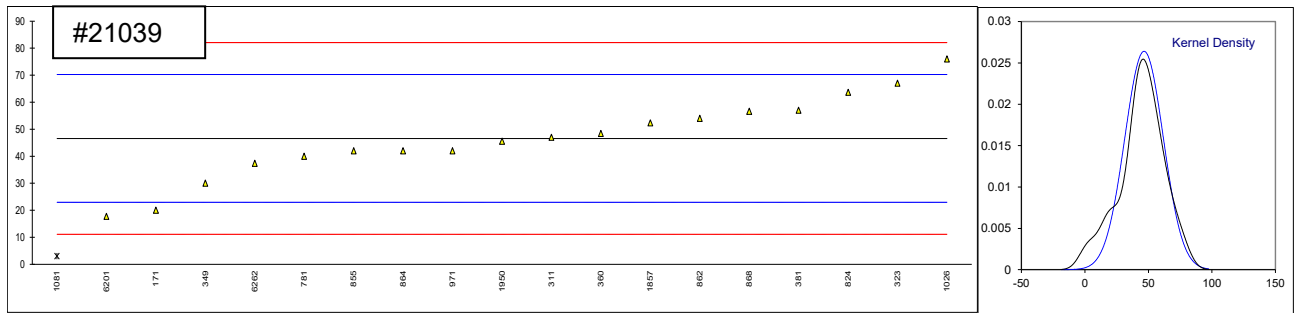


Determination of Arsenic as As on sample #21039 and #21040; results in µg/kg

lab	method	#21039	mark	z(targ)	#21040	mark	z(targ)	remarks
140		----		----	----		----	
150		----		----	----		----	
171	INH-014	<5		----	<5		----	
237		----		----	----		----	
311		----		----	----		----	
323	INH-018	< 10		----	< 10		----	
349	IFP9312	<5		----	<5		----	
360		----		----	----		----	
381	INH-118	<5		----	----		----	
444		----		----	----		----	
445		----		----	----		----	
657		----		----	----		----	
750		----		----	----		----	
781	UOP946	<5		----	<5		----	
824		----		----	----		----	
855		----		----	----		----	
862	SH/T0629	<10		----	<10		----	
864		----		----	----		----	
868		----		----	----		----	
874		----		----	----		----	
912		----		----	----		----	
963		----		----	----		----	
971	UOP946	----		----	6		----	
1026	In house	<6		----	----		----	
1081	In house	38		----	1		----	
1320		----		----	----		----	
1603		----		----	----		----	
1788		----		----	----		----	
1857	UOP946	4.8		----	3.2		----	
1949		----		----	----		----	
1950		----		----	----		----	
6201	In house	2.23		----	1.18		----	
6262	In house	1.9		----	0		----	
	n	11			10			
	mean (n)	<50			<50			

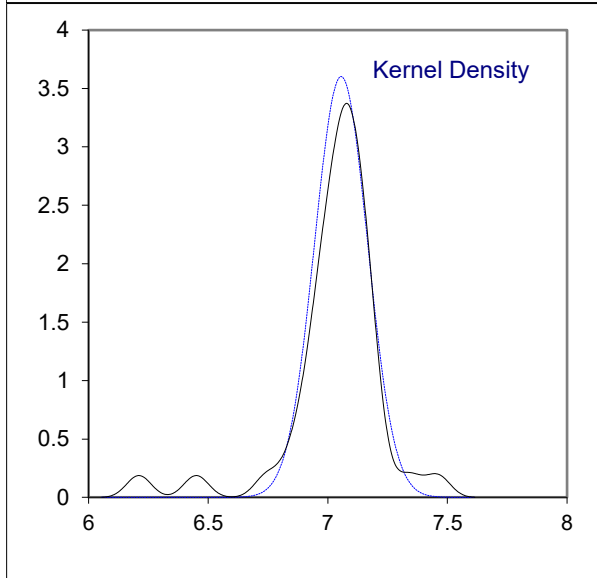
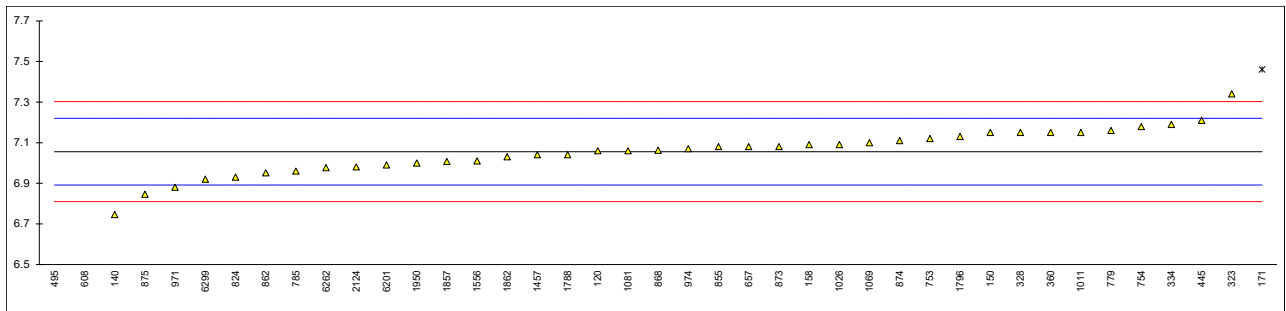
Determination of Lead as Pb on sample #21039 and #21040; results in µg/kg

lab	method	#21039	mark	z(targ)	#21040	mark	z(targ)	remarks
140		----		----	----		----	
150	UOP952	<10		<-3.09	<10		<-3.23	Possibly false neg test results?
171	INH-014	20		-2.25	24		-2.15	
237		----		----	----		----	
311	UOP952	47		0.04	65		1.02	
323	INH-018	67		1.73	87		2.72	
349	UOP952	30		-1.40	20		-2.46	
360	INH-06/15	48.40		0.15	58.93		0.55	
381	INH-118	57		0.88	----		----	
444		----		----	----		----	
445		----		----	----		----	
657		----		----	----		----	
750		----		----	----		----	
781	UOP952	40		-0.56	46		-0.45	
824	UOP952	63.64		1.44	65.94		1.09	
855	SH/T0242	42		-0.39	49		-0.22	
862	UOP952	54		0.63	59		0.56	
864	UOP952	42		-0.39	45		-0.53	
868	UOP952	56.6		0.85	65.6		1.07	
874		----		----	----		----	
912		----		----	----		----	
963		----		----	----		----	
971	UOP952	42		-0.39	46		-0.45	
1026	In house	76		2.49	----		----	
1081	In house	3	G(0.05)	-3.69	56		0.32	
1320		----		----	----		----	
1603		----		----	----		----	
1788		----		----	----		----	
1857	UOP952	52.3		0.48	56.5		0.36	
1949		----		----	----		----	
1950	UOP952	45.5		-0.09	53.2		0.11	
6201	In house	17.7	C	-2.44	29.8	C	-1.70	first reported 4.34, 7.77
6262	In house	37.3425		-0.78	53.8788		0.16	
normality		OK			OK			
n		18			17			
outliers		1			0			
mean (n)		46.582			51.815			
st.dev. (n)		15.1113			16.4731			
R(calc.)		42.312			46.125			
st.dev.(Horwitz)		11.8248			12.9439			
R(Horwitz)		33.109			36.243			



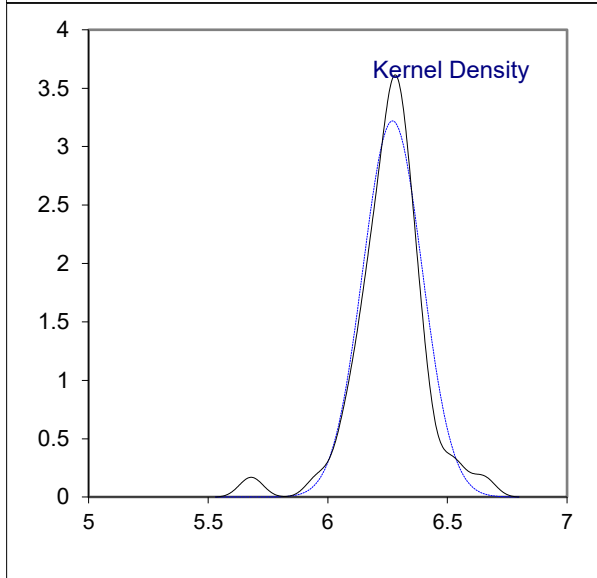
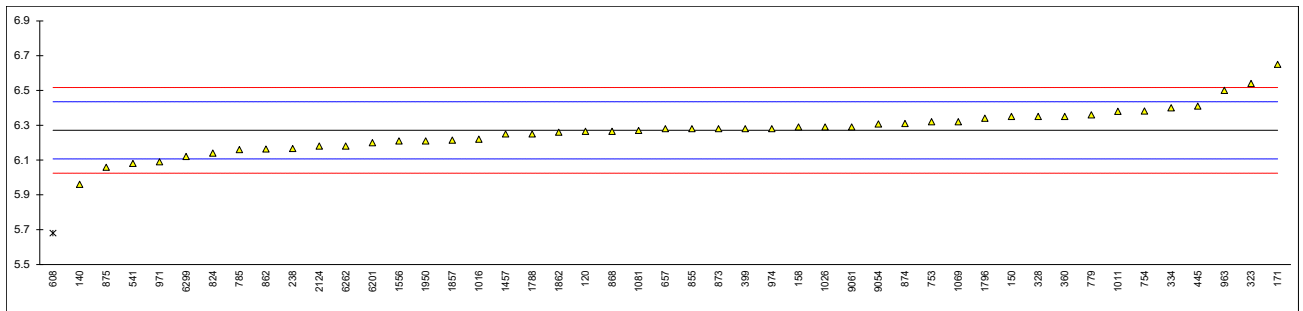
Determination of Total Vapor Pressure on sample #21041; results in psi

lab	method	value	mark	z(targ)	remarks
120	D5191	7.06		0.05	
140	D5191	6.746		-3.77	
150	D5191	7.15		1.15	
158	D5191	7.09		0.42	
171	D5191	7.46	R(0.05)	4.92	
237		----		----	
238		----		----	
323	D5191	7.34		3.46	
328	D5191	7.15		1.15	
334	EN13016-1	7.19		1.63	
360	D5191	7.15		1.15	
399		----		----	
445	D5191	7.21		1.88	
495	EN13016-1	6.21	R(0.01)	-10.30	
541		----		----	
608	D5191	6.45	R(0.01)	-7.37	
657	D5191	7.08		0.30	
750		----		----	
753	D5191	7.12		0.78	
754	D5191	7.179		1.50	
779	D5191	7.16		1.27	
785	D5191	6.96		-1.17	
798		----		----	
824	D5191	6.93		-1.53	
855	D5191	7.08		0.30	
862	D5191	6.951		-1.28	
864		----		----	
868	D5191	7.063		0.09	
873	D5191	7.08		0.30	
874	D5191	7.11		0.66	
875	D5191	6.846		-2.55	
963		----		----	
971	D5191	6.88		-2.14	
974	D5191	7.07		0.17	
1011	EN13016-1	7.15	C	1.15	first reported 43.8 kPa (=6.38 psi)
1016		----		----	
1026	D5191	7.09		0.42	
1069	D5191	7.10		0.54	
1081	D5191	7.06		0.05	
1457	D5191	7.04		-0.19	
1556	EN13016-1	7.01		-0.56	
1788	D5191	7.04		-0.19	
1796	D5191	7.13		0.90	
1857	D5191	7.007		-0.59	
1862	D5191	7.03		-0.31	
1950	D5191	7.00		-0.68	
2124	D5191	6.98		-0.92	
6200		----		----	
6201	D5191	6.99		-0.80	
6262	D5191	6.98		-0.97	
6299	EN13016-1	6.92		-1.65	
9054		----		----	
9058		----		----	
9061		----		----	
	normality	suspect			
	n	38			
	outliers	3			
	mean (n)	7.056			
	st.dev. (n)	0.1107			
	R(calc.)	0.310			
	st.dev.(D5191:20)	0.0821			
	R(D5191:20)	0.23			



Determination of DVPE acc. to D5191 on sample #21041; results in psi

lab	method	value	mark	z(targ)	remarks
120	D5191	6.265		-0.07	
140	D5191	5.96		-3.78	
150	D5191	6.35		0.97	
158	D5191	6.29		0.24	
171	D5191	6.65		4.62	
237		----		----	
238	D5191	6.166		-1.27	
323	D5191	6.54		3.28	
328	D5191	6.35		0.97	
334	EN13016-1	6.40		1.57	
360	D5191	6.35		0.97	
399	D5191	6.28		0.11	
445	D5191	6.41		1.70	
495		----		----	
541	D6378	6.08		-2.32	
608	D5191	5.68	C,R(0.01)	-7.19	first reported 7.25
657	D5191	6.28		0.11	
750		----		----	
753	D5191	6.32		0.60	
754	D5191	6.382		1.36	
779	D5191	6.36		1.09	
785	D5191	6.16		-1.35	
798		----		----	
824	D5191	6.14		-1.59	
855	D5191	6.28		0.11	
862	D5191	6.163		-1.31	
864		----		----	
868	D5191	6.265		-0.07	
873	D5191	6.28		0.11	
874	D5191	6.31		0.48	
875	D5191	6.058	C	-2.59	first reported 5.976
963	D5191	6.5		2.79	
971	D5191	6.09		-2.20	
974	D5191	6.28		0.11	
1011	EN13016-1	6.38		1.33	
1016	D5191	6.22		-0.62	
1026	D5191	6.29		0.24	
1069	D5191	6.32		0.60	
1081	D5191	6.27		-0.01	
1457	D5191	6.25		-0.25	
1556	EN13016-1	6.21		-0.74	
1788	D5191	6.25		-0.25	
1796	D5191	6.34		0.84	
1857	D5191	6.214		-0.69	
1862	D5191	6.26		-0.13	
1950	D5191	6.21		-0.74	
2124	D5191	6.18		-1.10	
6200		----		----	
6201	D5191	6.20		-0.86	
6262	D5191	6.18		-1.10	
6299	EN13016-1	6.12		-1.83	
9054	D5191	6.3075		0.45	
9058		----		----	
9061	D5191	6.29		0.24	
	normality	suspect			
	n	46			
	outliers	1			
	mean (n)	6.271			
	st.dev. (n)	0.1240			
	R(calc.)	0.347			
	st.dev.(D5191:20)	0.0821			
	R(D5191:20)	0.23			



APPENDIX 2**Number of participants per country**

1 lab in ARGENTINA
3 labs in AUSTRALIA
1 lab in AZERBAIJAN
4 labs in BELGIUM
1 lab in BULGARIA
6 labs in CHINA, People's Republic
1 lab in COTE D'IVOIRE
2 labs in CROATIA
1 lab in EGYPT
1 lab in ESTONIA
2 labs in FINLAND
7 labs in FRANCE
3 labs in GERMANY
2 labs in INDIA
1 lab in ISRAEL
1 lab in ITALY
1 lab in LATVIA
1 lab in MALAYSIA
1 lab in MALTA
9 labs in NETHERLANDS
3 labs in NIGERIA
2 labs in NORWAY
1 lab in PAKISTAN
1 lab in POLAND
2 labs in PORTUGAL
16 labs in RUSSIAN FEDERATION
2 labs in SAUDI ARABIA
1 lab in SERBIA
1 lab in SINGAPORE
1 lab in SLOVAKIA
1 lab in SOUTH KOREA
1 lab in SPAIN
2 labs in SWEDEN
2 labs in THAILAND
1 lab in TURKEY
2 labs in UNITED ARAB EMIRATES
5 labs in UNITED KINGDOM
5 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature

1. iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2. ISO5725:86
3. ISO5725 parts 1-6:94
4. ISO13528:05
5. M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
6. W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7. P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
8. J.N. Miller, Analyst, 118, 455, (1993)
9. Analytical Methods Committee, Technical Brief, No 4, January 2001
10. P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11. W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12. Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)