Results of Proficiency Test Trace Metals in Body Cream & Foundation February 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Corrector: ing. A.S. Noordman-de Neef
Report: iis19H01

March 2019

CONTENTS

1 INTRODUCTION

Heavy metals like Lead, Arsenic, Mercury, Aluminum, Zinc, Chromium and Iron are found in a wide variety of cosmetics or personal care products like lipstick, whitening toothpaste, eyeliner, body cream and foundation. Some metals are intentionally added as ingredients, while others are contaminants. Exposure to metals has been linked to health concerns including reproductive, immune and nervous system toxicity.
No reference materials (RMs) for Trace Metals in cosmetics are available to optimise the determination of the metals. As an alternative participation in a proficiency test may enable the laboratories to check their performance and thus to increase this comparability.

On request of a number of laboratories, the Institute for Interlaboratory Studies (iis) decided to set up a new proficiency test of the determination of Trace Metals in Body Cream and in Foundation during the annual testing program 2018/2019.

In this interlaboratory study 19 laboratories from 14 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2019 proficiency test are presented and discussed. This report is also electronically available through the iis website ww.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send in this proficiency test one sample of Body Cream (labelled #19505) and one sample of Foundation (labelled #19506), both were made positive (artificially fortified) with a number of heavy metals.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

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

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

For the first batch, a regular Body Cream was purchased from a supermarket and was artificially fortified with the metals: Cadmium as Cd (20 mg/kg), Chromium as Cr (20 mg/kg), Lead as Pb (5 mg/kg), Mercury as Hg (1.0 mg/kg) and Nickel as Ni (20 mg/kg).
From this batch 34 cups of 15 ml were filled with approximately 5 grams Body Cream and labelled #19505. The homogeneity of the subsamples #19505 was checked by determination of Cadmium and Nickel by using ICP-MS on five stratified randomly selected subsamples. See the following table for the test results.

	Cadmium as Cd in mg/kg	Nickel as Ni in mg/kg
sample #19505-1	19.0	17.7
sample #19505-2	19.5	17.7
sample #19505-3	18.7	17.7
sample #19505-4	18.9	17.4
sample #19505-5	18.6	17.7

Table 1: homogeneity test results of subsamples #19505

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

	Cadmium as Cd in mg/kg	Nickel as Ni in mg/kg
r (observed)	1.0	0.4
reference method	Horwitz	Horwitz
0.3 * R (ref. method)	1.6	1.5

Table 2: evaluation of the repeatability of subsamples #19505

The calculated repeatability of sample #19505 is in agreement with 0.3 times the reproducibility of the reference method. Therefore, homogeneity of the subsamples #19505 was assumed.

For the second batch, a regular Foundation was purchased from a supermarket and was artificially fortified with the metals: Cadmium as Cd (22 mg/kg), Chromium as Cr (22 mg/kg), Lead as Pb (5 mg/kg), Mercury as Hg (1.0 mg/kg) and Nickel as Ni (22 mg/kg).

From this batch 34 cups of 15 ml were filled with approximately 5 grams Foundation and labelled #19506. The homogeneity of the subsamples #19506 was checked by determination of Cadmium and Nickel by using ICP-MS on five stratified randomly selected subsamples. See the following table for the test results.

	Cadmium as Cd in mg/kg	Nickel as Ni in mg/kg
sample #19506-1	21.7	18.0
sample #19506-2	22.0	17.7
sample #19506-3	20.8	17.7
sample #19506-4	20.6	17.3
sample #19506-5	21.3	17.5

Table 3: homogeneity test results of subsamples #19506

From the above test results the repeatabilities were 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.

	Cadmium as Cd in mg/kg	Nickel as Ni in mg/kg
r (observed)	1.7	0.7
reference method	Horwitz	Horwitz
0.3 * R (ref. method)	1.8	1.5

Table 4: evaluation of the repeatability of subsamples #19506

The calculated repeatability of sample #19506 is in agreement with 0.3 times the reproducibility of the reference method. Therefore, homogeneity of the subsamples #19506 was assumed.

To each of the participating laboratories 1 sample labelled #19505 (Body Cream) and 1 sample labelled #19506 (Foundation) was sent on January 16, 2019.

2.5 ANALYSES

The participants were requested to determine on both samples the concentrations of the metals: Arsenic as As, Cadmium as Cd, Chromium as Cr, Lead as Pb, Mercury as Hg and Nickel as Ni, applying the analytical procedure that is routinely used in the laboratory. Also, some analytical details were asked

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 results, but report as much significant figures as possible. It was also requested not to report ‘less than’ results, which are above the detection limit, because such results cannot be used for meaningful statistical evaluations.

To get comparable 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 method 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-cts. 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-cts/. The reported test results are tabulated per determination in appendix 1 and 2 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 reanalyses). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the 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 dataset does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon’s, Grubbs’ and/or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.
Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

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

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

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study.
The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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

The z-scores were calculated according to:

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

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

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

During the execution of this proficiency test some problems occurred. Two participants reported to have received a sample Body Cream which had leaked. A new sample was sent to these participants.
One participant did not report any test results. The 18 participants reported 155 numerical test results. Observed were 6 outlying test results, which is 3.9% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section, the results are discussed per sample and per component. The evaluation of the test results reported on the samples are summarised in appendix 1. The abbreviations, used in these tables, are listed in appendix 4.

Unfortunately, a suitable reference test method, providing the precision data, is not available for the determinations of heavy metals in personal care products, therefore the calculated reproducibilities were compared against the reproducibility estimated from the Horwitz equation.

Sample #19505, Body Cream

Arsenic as As: Almost all reporting participants reported a less then test result, which is close to or below the quantification limit of Arsenic. Therefore, no z-scores were calculated.

Cadmium as Cd: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of statistical outlier is in agreement with the estimated reproducibility using the Horwitz equation.
The average recovery of Cadmium (theoretical increment of 20.0 mg Cadmium/kg) may be good (<104%), the actual Cadmium content is not known.

Chromium as Cr: This determination may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of statistical outliers is not in agreement with the estimated reproducibility using the Horwitz equation.
The average recovery of Chromium (theoretical increment of 20.0 mg Chromium/kg) may be good (<106%), the actual Chromium content is not known.

Lead as Pb: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the estimated reproducibility using the Horwitz equation.
The average recovery of Lead (theoretical increment of 5.0 mg Lead/kg) may be good (<108%), the actual Lead content is not known.

Mercury as Hg: This determination was problematic. The group seems to be bimodal divided. Therefore, no z-scores were calculated.

Nickel as Ni: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the estimated reproducibility using the Horwitz equation.
The average recovery of Nickel (theoretical increment of 20.0 mg Nickel/kg) may be good (<106%), the actual Nickel content is not known.

Sample #19506, Foundation

Arsenic as As: Almost all reporting participants reported a less then test result, which is close to or below the quantification limit of Arsenic. Therefore, no z-scores were calculated.

Cadmium as Cd: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of statistical outlier is in agreement with the estimated reproducibility using the Horwitz equation.
The average recovery of Cadmium (theoretical increment of 21.8 mg Cadmium/kg) may be good (<97%), the actual Cadmium content is not known.

Chromium as Cr: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of statistical outlier is in agreement with the estimated reproducibility using the Horwitz equation.
The average recovery of Chromium (theoretical increment of 21.8 mg Chromium/kg) may be good (<103%), the actual Chromium content is not known.

Lead as Pb: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the estimated reproducibility using the Horwitz equation.
The average recovery of Lead (theoretical increment of 5.4 mg Lead/kg) may be good (<97%), the actual Lead content is not known.

Mercury as Hg: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the estimated reproducibility using the Horwitz equation.
The average recovery of Mercury (theoretical increment of 1.099 mg Mercury/kg) is unsatisfactory (<9%), the actual Mercury content is not known.

Nickel as Ni: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the estimated reproducibility using the Horwitz equation.
The average recovery of Nickel (theoretical increment of 21.8 mg Nickel/kg) may be good (<100%), the actual Nickel content is not known.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibilities as declared by the relevant reference method and the reproducibilities as found for the group of participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8*sd) and the target reproducibility derived from the reference method (in casu Horwitz Equation) are presented in the next table.

Component	unit	n	average	2.8 * sd	R (target)
Arsenic as As	mg/kg	10	<0.5	n.a.	n.a.
Cadmium as Cd	mg/kg	15	20.8	6.3	5.9
Chromium as Cr	mg/kg	14	21.1	8.2	6.0
Lead as Pb	mg/kg	17	5.4	1.9	1.9
Mercury as Hg	mg/kg	15	n.a.	n.a.	n.a.
Nickel as Ni	mg/kg	16	21.2	5.9	6.0

Table 5: reproducibilities of tests on sample #19505

Component	unit	n	average	2.8 * sd	R (target)
Arsenic as As	mg/kg	9	<0.5	n.a.	n.a.
Cadmium as Cd	mg/kg	15	21.1	5.0	6.0
Chromium as Cr	mg/kg	15	22.4	5.5	6.3
Lead as Pb	mg/kg	17	5.2	1.9	1.8
Mercury as Hg	mg/kg	8	0.10	0.14	0.06
Nickel as Ni	mg/kg	17	21.8	4.3	6.1

Table 6: reproducibilities of tests on sample #19506

From the table above, it can be concluded that, without statistical calculations, the group of participating laboratories do not have difficulties with the analysis of Metals in Body Cream or Foundation when compared with the target reproducibility, except for Mercury. See also the discussions in paragraphs 4.1.

4.3 UNCERTAINTIES OF THE PROFICIENCY TEST OF FEBRUARY 2019

The uncertainties observedy in the test results of the determination of Metals in Body Cream in the PT: iis19H01 are listed in the next table:

Component	February 2019	Target (Horwitz)
Cadmium as Cd	8 - 11%	10%
Chromium as Cr	9 - 14%	10%
Lead as Pb	13%	12%
Mercury as Hg	54%	17 - 22%
Nickel asNi	7 - 10%	10%

Table 7: overview of relative uncertainties (RSD).

4.4 EVALUATION ANALYTICAL DETAILS

For this PT some analytical details were requested, see appendix 2. Based on the answers given by the participants the following can be summarized:
Twelve of the eighteen reporting participants (= 67%) mentioned that they are accredited for determination of Heavy Metals in Body Cream and/or Foundation.
The other questions were about the intake of the sample used for the analyzes. Thirteen of the eighteen reporting laboratories used between 0.2 – 0.5 gram, one participant used 1 gram and another used 3.3 grams.
Eleven participants used ICP-MS to determine the metalcontent, five used ICP-OES and one used EDXRF to determine the metals. Two participants reported also to use a different method to determine Arsenic (Cold Vapour AAS) and Mercury (Hg-analyser).

5 DISCUSSION

The evaluation of Mercury in both samples (#19505 and #19506) was problematic. During the preparation of both batches, the same spike solution was used. Therefore, the same mean was expected to be found. Surprisingly, the mean of sample #19506 Foundation is much smaller than the mean of sample #19505 Body Cream (0.10 vs 0.73 mg/kg). This might be caused by interference in the analysis.
All other determined elements showed a recovery of approximately 100% which is good.
The test results obtained from sample #19506 9Foundation) show a better reproducibility.

6 CONCLUSION

In this proficiency test the metals content in two different types of cosmetics were identified. Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

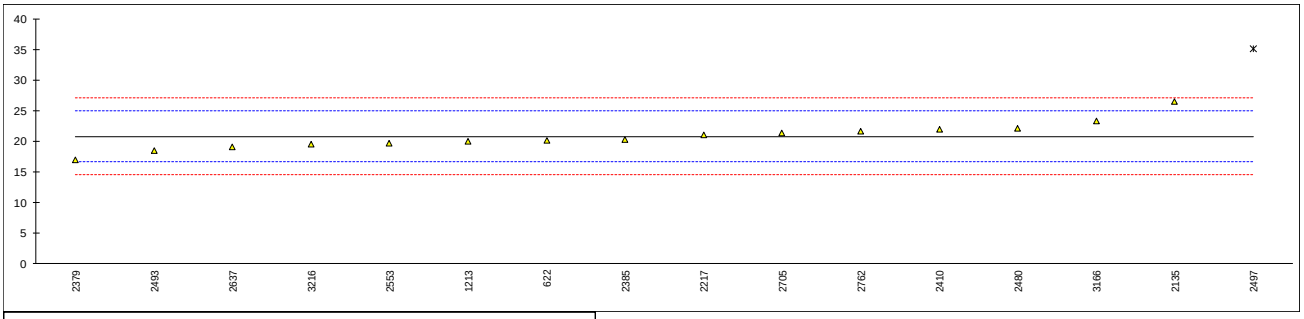
APPENDIX 1

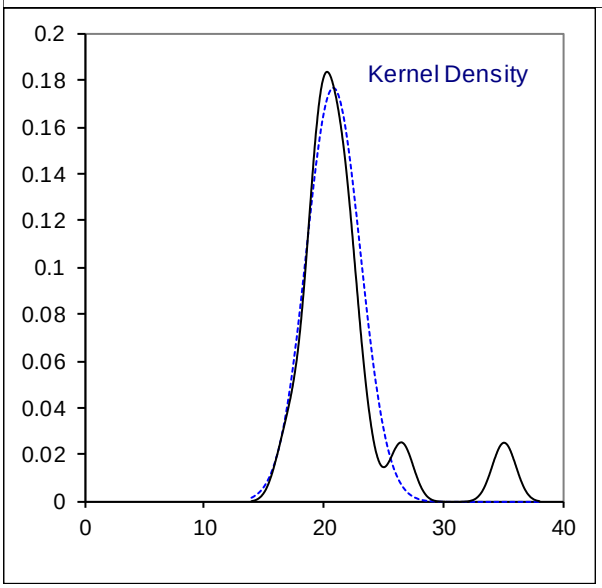
Determination of Arsenic as As in Body Cream, sample #19505; results in mg/kg

lab	method	value	mark	z(targ)	remarks
622		-----		-----	
1128	In house	nd		-----	
1213		0.03		-----	
2135		-----		-----	
2217	ISO TR 17276	0.022		-----	
2379		Not detected		-----	
2385	In house	<0,5		-----	
2410	In house	< 2		-----	
2480	In house	<0.1		-----	
2493	EPA6020A	0.004		-----	
2497		2.805		-----	False positive test result?
2538		-----		-----	
2553	In house	ND		-----	
2637		<0,05		-----	
2705	In house	0.00		-----	
2762		<0.5		-----	
3166	In house	<0.007		-----	
3172	In house	< 1.0		-----	
3216	In house	nd		-----	
	normality	n.a.			
	n	10			
	outliers	n.a.			
	mean (n)	<0.5			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	st.dev.(lit)	n.a.			
	R(lit)	n.a.			

Determination of Cadmium as Cd in Body Cream, sample #19505; results in mg/kg

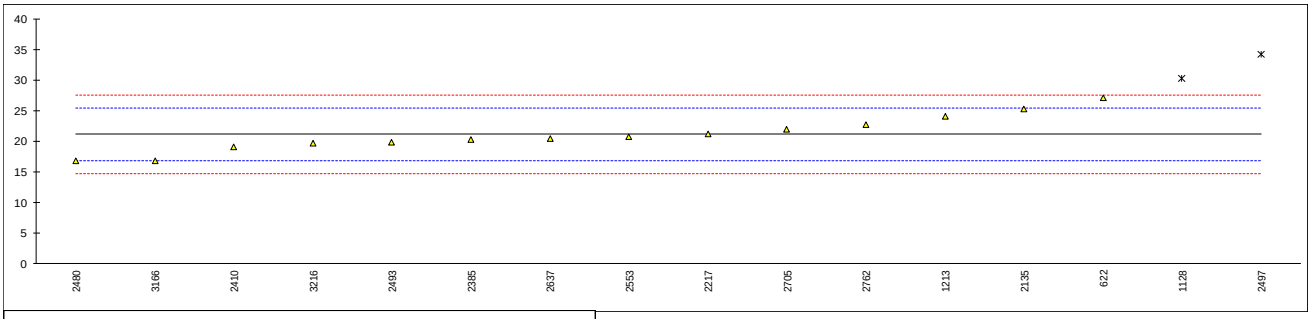
lab	method	value	mark	z(targ)	remarks
622	In house	20.11		-0.33	
1128		-----		-----	
1213		20		-0.38	
2135		26.50	C	2.70	First reported 32.205
2217	ISO TR 17276	20.993		0.09	
2379		16.92		-1.84	
2385	In house	20.3		-0.24	
2410	In house	22		0.57	
2480	In house	22.158		0.64	
2493	EPA6020A	18.4		-1.14	
2497		35.09	G(0.01)	6.78	
2538		-----		-----	
2553	In house	19.72		-0.51	
2637		19.1		-0.81	
2705	In house	21.34		0.25	
2762		21.7		0.42	
3166	In house	23.33		1.20	
3172		-----		-----	
3216	In house	19.5028		-0.62	
	normality	not OK			
	n	15			
	outliers	1	Spike		
	mean (n)	20.805	20.0		Recovery: <104%
	st.dev. (n)	2.2557	RSD	= 11%	
	R(calc.)	6.316			
	st.dev.(Horwitz)	2.1081			
	R(Horwitz)	5.903			

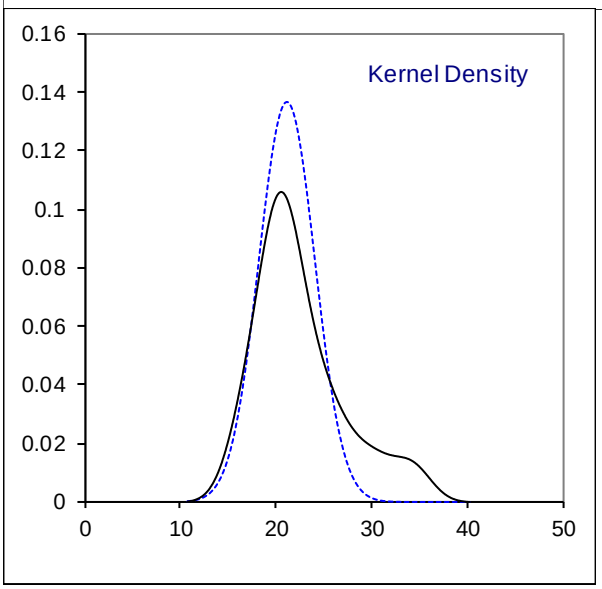




Determination of Chromium as Cr in Body Cream, sample #19505; results in mg/kg

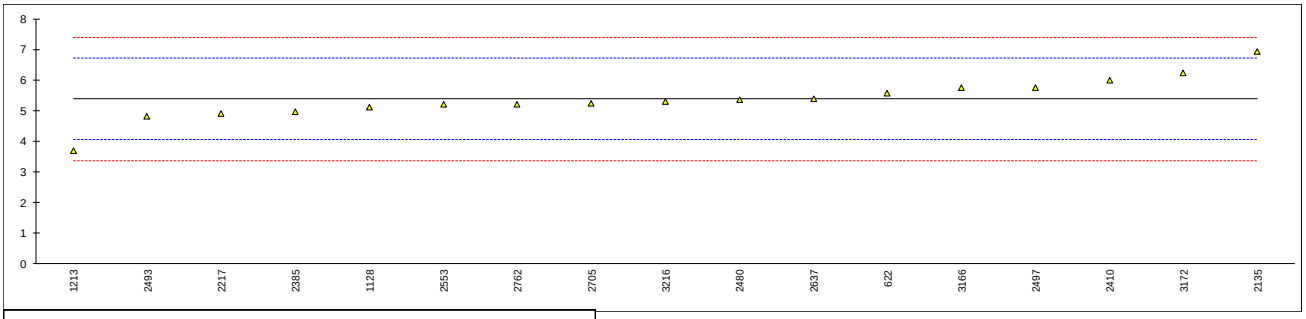
lab	method	value	mark	z(targ)	remarks
622	In house	27.06		2.79	
1128	In house	30.2	DG(0.05)	4.26	
1213		24		1.35	
2135		25.21	C	1.92	First reported 29.755
2217	ISO TR 17276	21.131		0.01	
2379		-----		-----	
2385	In house	20.3		-0.38	
2410	In house	19		-0.99	
2480	In house	16.788		-2.03	
2493	EPA6020A	19.9		-0.57	
2497		34.118	DG(0.05)	6.09	
2538		-----		-----	
2553	In house	20.75		-0.17	
2637		20.4		-0.33	
2705	In house	21.92		0.38	
2762		22.7		0.74	
3166	In house	16.8		-2.02	
3172		-----		-----	
3216	In house	19.6067		-0.71	
	normality	OK			
	n	14			
	outliers	2	Spike		
	mean (n)	21.112	20.0		Recovery: <106%
	st.dev. (n)	2.9151	RSD	= 14%	
	R(calc.)	8.162			
	st.dev.(Horwitz)	2.1344			
	R(Horwitz)	5.976			

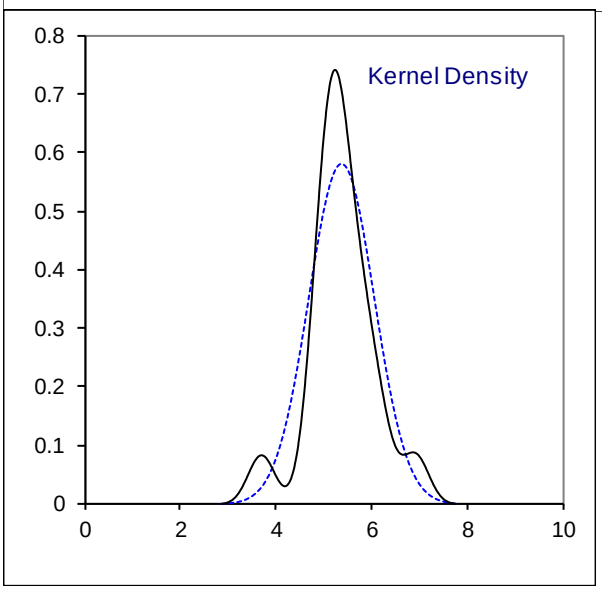




Determination of Lead as Pb in Body Cream, sample #19505; results in mg/kg

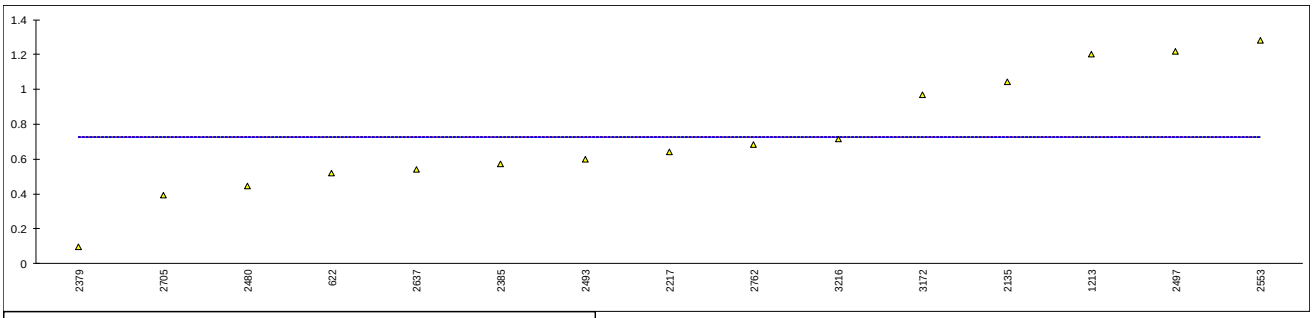
lab	method	value	mark	z(targ)	Remarks
622	In house	5.58		0.30	
1128	In house	5.1		-0.42	
1213		3.7		-2.51	
2135		6.93	C	2.32	First reported 7.996
2217	ISO TR 17276	4.905		-0.71	
2379		Not detected		-----	
2385	In house	4.95		-0.64	
2410	In house	6		0.93	
2480	In house	5.368		-0.02	
2493	EPA6020A	4.82		-0.84	
2497		5.761		0.57	
2538		-----		-----	
2553	In house	5.19		-0.28	
2637		5.4		0.03	
2705	In house	5.239		-0.21	
2762		5.21		-0.25	
3166	In house	5.76		0.57	
3172	In house	6.23		1.27	
3216	In house	5.3072		-0.11	
	normality	not OK			
	n	17			
	outliers	0	Spike		
	mean (n)	5.379	5.0		Recovery: <108%
	st.dev. (n)	0.6871	RSD	= 13%	
	R(calc.)	1.924			
	st.dev.(Horwitz)	0.6681			
	R(Horwitz)	1.871			

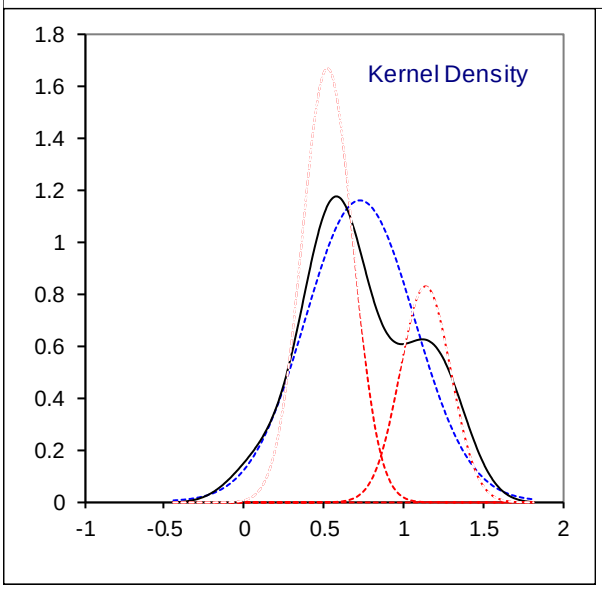




Determination of Mercury as Hg in Body Cream, sample #19505; results in mg/kg

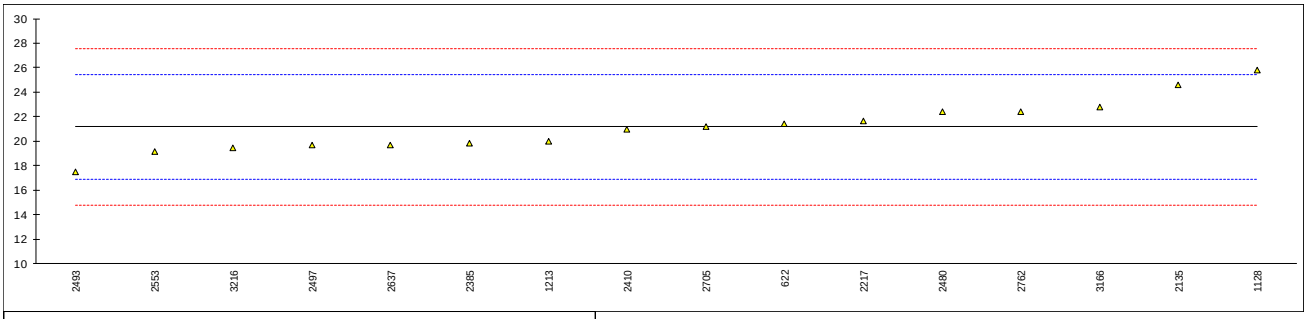
lab	method	value	mark	z(targ)	remarks		
622	In house	0.522		-----			
1128	In house	nd		-----			
1213		1.2		-----			
2135		1.041		-----			
2217	ISO TR 17276	0.642		-----			
2379		0.094		-----			
2385	In house	0.57		-----			
2410	In house	< 1		-----			
2480	In house	0.445		-----			
2493	EPA6020A	0.60		-----			
2497		1.218		-----			
2538		-----		-----			
2553	In house	1.28		-----			
2637		0.54		-----			
2705	In house	0.393		-----			
2762		0.683		-----			
3166		-----		-----			
3172	In house	0.97		-----			
3216	In house	0.7146		-----			
						Group 1	Group 2
	normality	OK				not OK	OK
	n	15				10	5
	outliers	0	Spike			0	0
	mean (n)	(0.7275)	1.007		Recovery:<72%	0.5204	1.1418
	st.dev. (n)	(0.34300)				0.18008	0.13035
	R(calc.)	(0.9604)				0.5042	0.3650
	st.dev.(Horwitz)	(0.12211)				0.09186	0.17908
	R(Horwitz)	(0.3419)				0.2572	0.5014

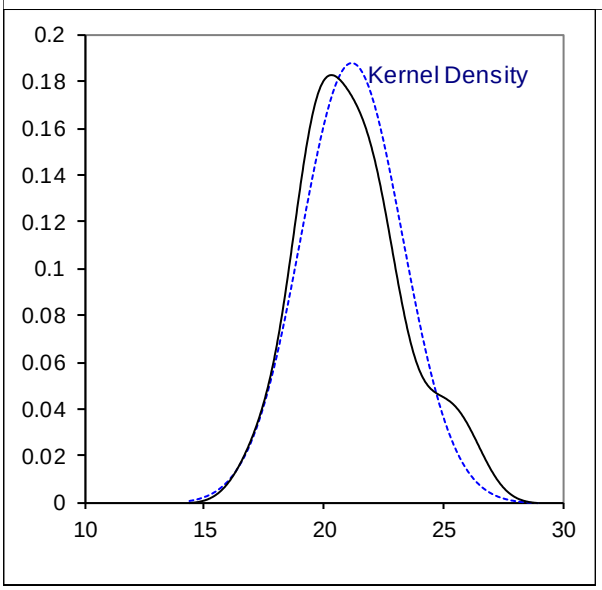




Determination of Nickel as Ni in Body Cream, sample #19505; results in mg/kg

lab	method	value	mark	z(targ)	remarks
622	In house	21.39		0.11	
1128	In house	25.8		2.17	
1213		20		-0.54	
2135		24.63	C	1.62	First reported 30.88
2217	ISO TR 17276	21.675		0.24	
2379		-----		-----	
2385	In house	19.8		-0.64	
2410	In house	21		-0.08	
2480	In house	22.385		0.57	
2493	EPA6020A	17.5		-1.71	
2497		19.693		-0.69	
2538		-----		-----	
2553	In house	19.14		-0.95	
2637		19.7		-0.68	
2705	In house	21.198		0.02	
2762		22.4		0.58	
3166	In house	22.82		0.78	
3172		-----		-----	
3216	In house	19.4527		-0.80	
	normality	OK			
	n	16			
	outliers	0	Spike		
	mean (n)	21.161	20.0		Recovery: <106%
	st.dev. (n)	2.1226	RSD	= 10%	
	R(calc.)	5.943			
	st.dev.(Horwitz)	2.1387			
	R(Horwitz)	5.988			



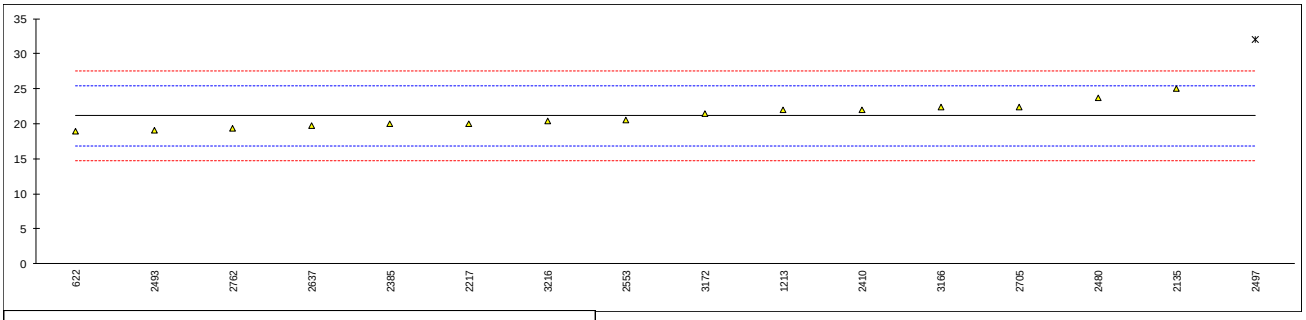


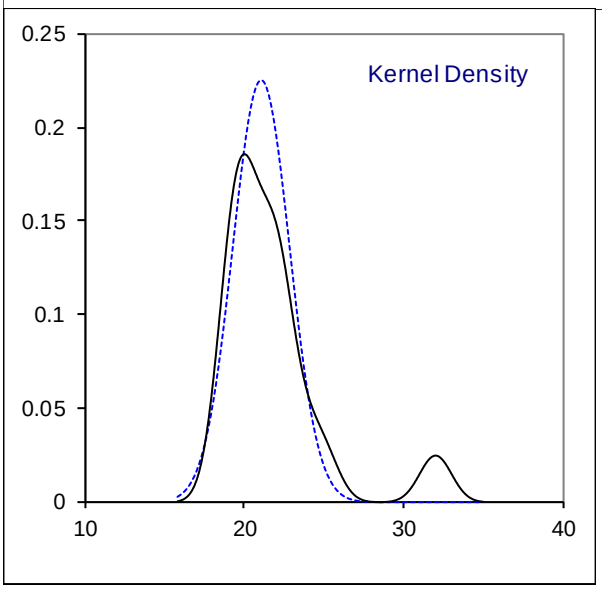
Determination of Arsenic as As in Foundation, sample #19506; results in mg/kg

lab	method	value	mark	z(targ)	remarks
622		-----		-----	
1128	In house	nd		-----	
1213		0.06		-----	
2135		-----		-----	
2217	ISO TR 17276	0.024		-----	
2379		-----		-----	
2385	In house	<0,5		-----	
2410	In house	< 2		-----	
2480	In house	0.092		-----	
2493	EPA6020A	0.05		-----	
2497		3.138		-----	False positive test result?
2538		-----		-----	
2553	In house	ND		-----	
2637		0.05		-----	
2705	In house	0.043		-----	
2762		<0.5		-----	
3166	In house	0.072		-----	
3172	In house	< 1.0		-----	
3216	In house	0.0591		-----	
	normality	n.a.			
	n	9			
	outliers	n.a.			
	mean (n)	<0.5			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	st.dev.(lit)	n.a.			
	R(lit)	n.a.			

Determination of Cadmium as Cd in Foundation, sample #19506; results in mg/kg

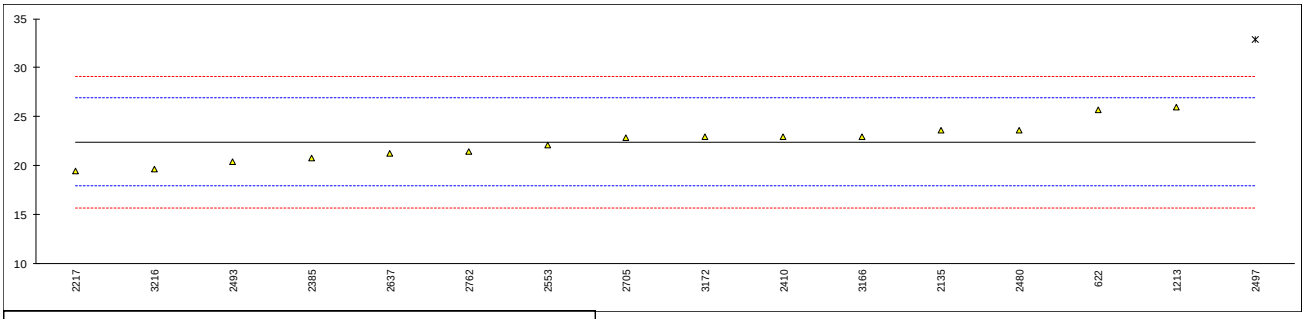
lab	method	value	mark	z(targ)	Remarks
622	In house	18.88		-1.05	
1128		-----		-----	
1213		22		0.41	
2135		25.010		1.82	
2217	ISO TR 17276	20.027		-0.51	
2379		-----		-----	
2385	In house	20.0		-0.52	
2410	In house	22		0.41	
2480	In house	23.640		1.18	
2493	EPA6020A	19.1		-0.95	
2497		32.027	G(0.01)	5.11	
2538		-----		-----	
2553	In house	20.56		-0.26	
2637		19.7		-0.67	
2705	In house	22.37		0.59	
2762		19.3		-0.85	
3166	In house	22.34		0.57	
3172	In house	21.45		0.15	
3216	In house	20.4259		-0.33	
	normality	OK			
	n	15			
	outliers	1	Spike		
	mean (n)	21.120	21.8		Recovery: <97%
	st.dev. (n)	1.7700	RSD	= 8%	
	R(calc.)	4.956			
	st.dev.(Horwitz)	2.1352			
	R(Horwitz)	5.978			

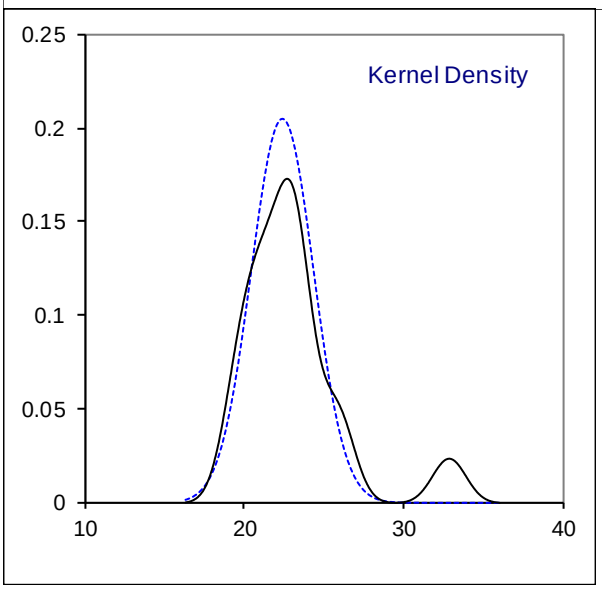




Determination of Chromium as Cr in Foundation, sample #19506; results in mg/kg

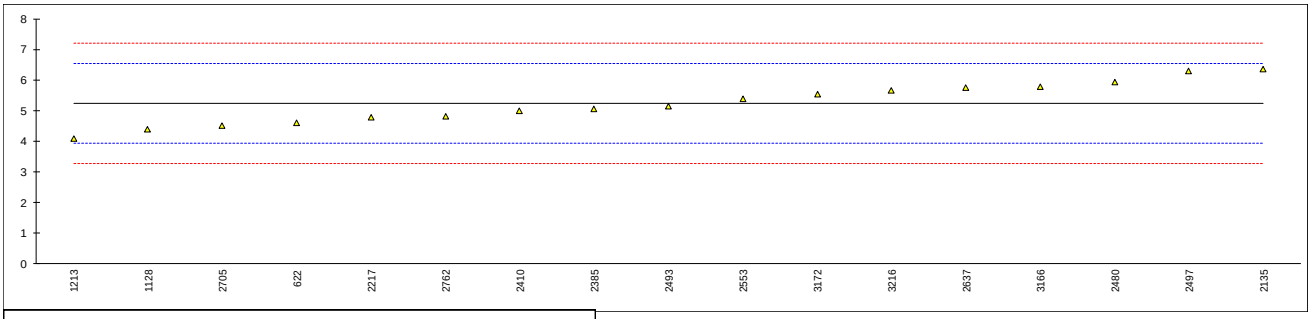
lab	method	value	mark	z(targ)	remarks
622	In house	25.71		1.48	
1128	In house	nd		-----	
1213		26		1.61	
2135		23.635		0.55	
2217	ISO TR 17276	19.464		-1.31	
2379		-----		-----	
2385	In house	20.8		-0.71	
2410	In house	23		0.27	
2480	In house	23.657		0.56	
2493	EPA6020A	20.4		-0.89	
2497		32.87	G(0.01)	4.67	
2538		-----		-----	
2553	In house	22.11		-0.13	
2637		21.3		-0.49	
2705	In house	22.90		0.22	
2762		21.4		-0.44	
3166	In house	23.0		0.27	
3172	In house	22.92		0.23	
3216	In house	19.6512		-1.22	
	normality	OK			
	n	15			
	outliers	1	Spike		
	mean (n)	22.396	21.8		Recovery: <103%
	st.dev. (n)	1.9510	RSD	=9%	
	R(calc.)	5.463			
	st.dev.(Horwitz)	2.2443			
	R(Horwitz)	6.284			

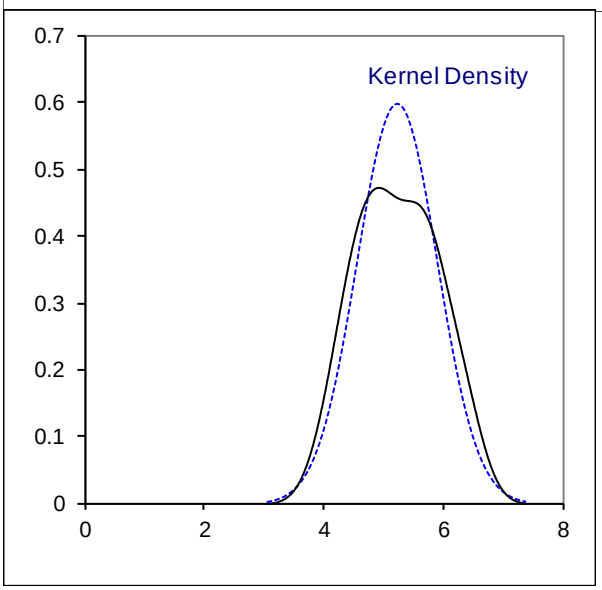




Determination of Lead as Pb in Foundation, sample #19506; results in mg/kg

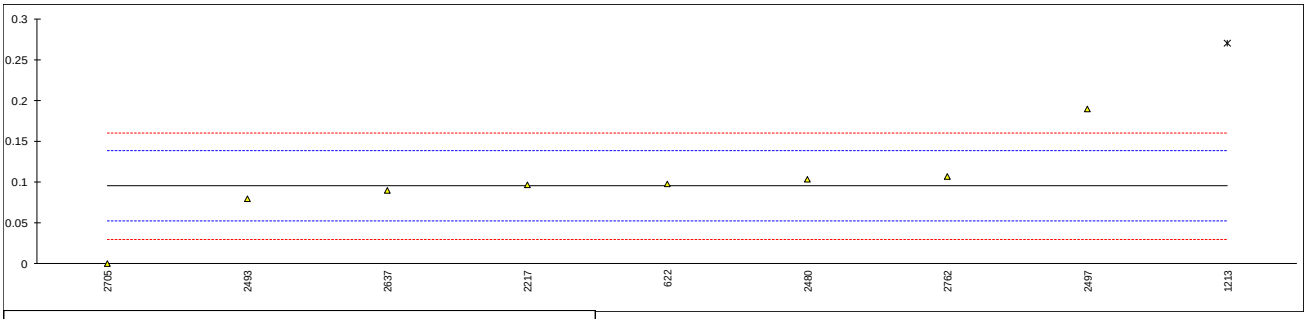
lab	method	value	mark	z(targ)	remarks
622	In house	4.59		-1.00	
1128	In house	4.4		-1.29	
1213		4.1		-1.74	
2135		6.355		1.71	
2217	ISO TR 17276	4.794		-0.68	
2379		-----		-----	
2385	In house	5.05		-0.29	
2410	In house	5		-0.37	
2480	In house	5.928		1.05	
2493	EPA6020A	5.14		-0.15	
2497		6.3		1.62	
2538		-----		-----	
2553	In house	5.40		0.24	
2637		5.75		0.78	
2705	In house	4.495		-1.14	
2762		4.82		-0.64	
3166	In house	5.77		0.81	
3172	In house	5.53		0.44	
3216	In house	5.6607		0.64	
	normality	OK			
	n	17			
	outliers	0	Spike		
	mean (n)	5.240	5.4		Recovery: <97%
	st.dev. (n)	0.6675	RSD	= 13%	
	R(calc.)	1.869			
	st.dev.(Horwitz)	0.6534			
	R(Horwitz)	1.830			

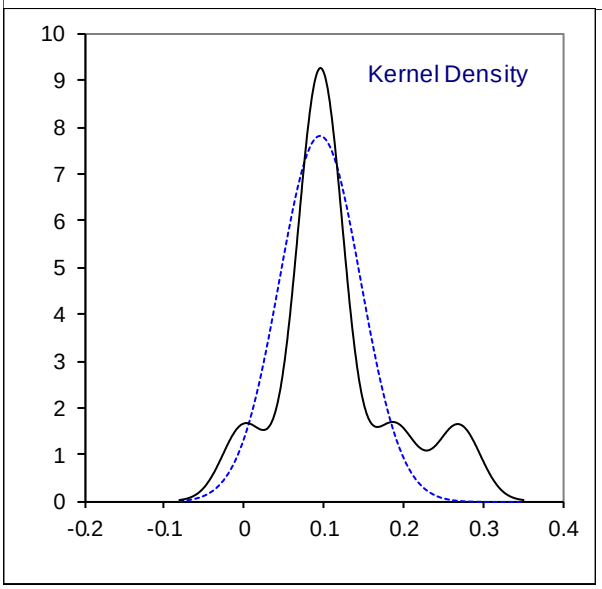




Determination of Mercury as Hg in Foundation, sample #19506; results in mg/kg

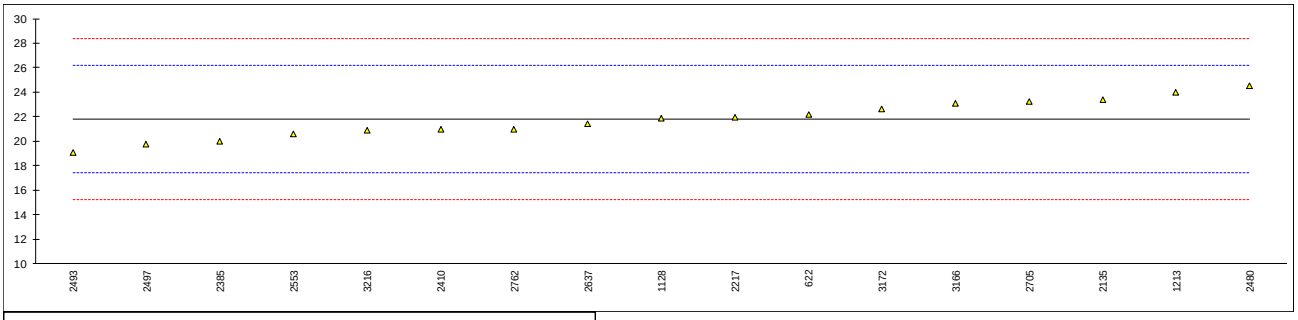
lab	method	value	mark	z(targ)	remarks
622	In house	0.098		0.12	
1128	In house	nd		-----	
1213		0.27	G(0.05)	8.03	
2135		-----		-----	
2217	ISO TR 17276	0.096		0.03	
2379		-----		-----	
2385	In house	<0,1		-----	
2410	In house	< 1		-----	
2480	In house	0.103		0.35	
2493	EPA6020A	0.08		-0.71	
2497		0.189		4.31	
2538		-----		-----	
2553	In house	ND		-----	
2637		0.09		-0.25	
2705	In house	0.00		-4.39	
2762		0.107		0.53	
3166		-----		-----	
3172	In house	< 0.5		-----	
3216	In house	nd		-----	
	normality	not OK			
	n	8			
	outliers	1	Spike		
	mean (n)	0.0954	1.099		Recovery: <9%
	st.dev. (n)	0.05117	RSD	= 54%	
	R(calc.)	0.1433			
	st.dev.(Horwitz)	0.02174			
	R(Horwitz)	0.0609			

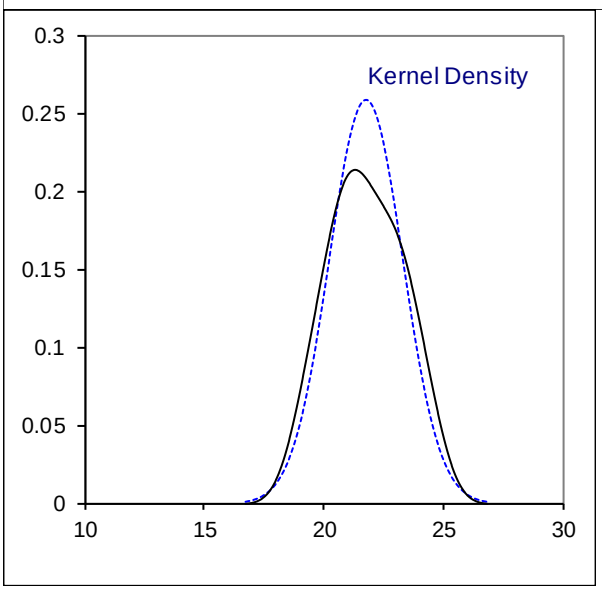




Determination of Nickel as Ni in Foundation, sample #19506; results in mg/kg

lab	method	value	mark	z(targ)	remarks
622	In house	22.19		0.18	
1128	In house	21.9		0.04	
1213		24		1.00	
2135		23.390		0.72	
2217	ISO TR 17276	21.933		0.06	
2379		-----		-----	
2385	In house	20.0		-0.82	
2410	In house	21		-0.37	
2480	In house	24.541		1.25	
2493	EPA6020A	19.1		-1.23	
2497		19.778		-0.92	
2538		-----		-----	
2553	In house	20.56		-0.57	
2637		21.4		-0.18	
2705	In house	23.252		0.66	
2762		21.0		-0.37	
3166	In house	23.08		0.58	
3172	In house	22.65		0.39	
3216	In house	20.8630		-0.43	
	normality	OK			
	n	17			
	outliers	0	Spike		
	mean (n)	21.802	21.8		Recovery:<100%
	st.dev. (n)	1.5412	RSD	= 7%	
	R(calc.)	4.315			
	st.dev.(Horwitz)	2.1936			
	R(Horwitz)	6.142			





APPENDIX 2

Analytical details

lab	ISO17025 accredited	Intake in gram	Technique used
622	Yes	1	ICP-OES / Cold vapour AAS
1128	No	3.3	EDXRF
1213	Yes	---	ICP-OES
2135	Yes	0.3	ICP-OES
2217	Yes	0.5	ICP-MS
2379	Yes	0.25	ICP-MS / Hg analyzer
2385	Yes	0.2	ICP-MS
2410	Yes	0.2	ICP-OES
2480	No	0.25	ICP-MS
2493	Yes	0.5	ICP-MS
2497	---	---	---
2538	---	---	---
2553	Yes	0.5	ICP-OES
2637	Yes	0.25	ICP-MS
2705	No	0.21	ICP-MS
2762	Yes	0.5	ICP-MS
3166	Yes	0.2	ICP-MS
3172	No	---	ICP-MS
3216	No	0.5	ICP-MS

APPENDIX 3

Number of participants per country

1 lab in CZECH REPUBLIC
1 lab in FRANCE
4 labs in GERMANY
2 labs in HUNGARY
1 lab in INDONESIA
2 labs in ITALY
1 lab in KOREA
1 lab in LUXEMBOURG
1 lab in SPAIN
1 lab in SRI LANKA
1 lab in THAILAND
1 lab in THE NETHERLANDS
1 lab in U.S.A.
1 lab in VIETNAM

APPENDIX 4

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
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected

Literature:

1 iis-Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation, June 2018

2 P.L. Davies, Fr Z. Anal. Chem, 351, 513, (1988)

3 W.J. Conover, Practical; Nonparametric Statistics, J. Wiley&Sons, NY, p.302, (1971)

4 ISO 5725, (1986)

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 G. Rohm, J. Bohnen & H. Kruessmann, GIT Labor-Fachzeitschrift, p 1080, 11, (1997)

10 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, T*echnometrics*, 25(2), 165-172, (1983)

11 Analytical Methods Committee Technical brief, No 4, January 2001

12 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)

13 Horwitz, W and Albert, R, J. AOAC Int, 79, 3, 589, (1996)