Institute for
Interlaboratory Studies

Results of Proficiency Test
Trace Metals in Skin Care products
October 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Heavy metals are found in a wide variety of cosmetics and personal care products like lipstick, 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.

In Europe the current regulation for cosmetics is EC 1223/2009 with the latest consolidation in December 2022. This regulation has replaced the council directive of 76/768/EEC.
In Annex II there is a list of substances that states that cosmetics shall not contain certain heavy metals like Antimony, Arsenic, Cadmium, Chromium, Lead, Mercury and Nickel.
Based on this European regulation China issued the Hygienic Standard for Cosmetics (HSC 2007). In 2015 this standard was superseded by the Chinese Safety and Technical Standards for Cosmetics (STSC 2015) which was implemented in 2016 limits for Arsenic, Cadmium, Lead and Mercury. The Association of South East Asean Nations (ASEAN) developed a test method for the same heavy metals (ACMTHA05) and has published limits for test results from this method. The Food and Drug Administration of the USA has set a limit for Mercury in cosmetics.

Since 2019 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Trace Metals in Skin Care products. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of Trace Metals in Skin Care products.

In this interlaboratory study 10 laboratories in 8 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Trace Metals in Skin Care products proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send two different skin care samples of approximately 10 mL each: a Body Cream labelled #22745 and a Foundation labelled #22746.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant’s data. Feedback from the participants on the reported data is encouraged and customer’s satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

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

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

For the first sample a batch of regular body cream was purchased from a local supermarket and was artificially fortified with Cadmium, Lead and Nickel. After homogenization 30 bottles of 10 mL were filled and labelled #22745.
The homogeneity of the subsamples was checked by determination of Lead by using an in house test method on 4 stratified randomly selected subsamples.

	Lead as Pb in mg/kg
sample #22745-1	15.6
sample #22745-2	14.9
sample #22745-3	15.7
sample #22745-4	15.7

Table 1: homogeneity test results of subsamples #22745

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 as Pb in mg/kg
r (observed)	1.1
reference method	Horwitz
0.3 x R (reference method)	1.4

Table 2: evaluation of the repeatability of subsamples #22745

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 second sample a batch of regular foundation was purchased from a local supermarket and was artificially fortified with Cadmium, Chromium, Lead and Nickel. After homogenization 30 bottles of 10 mL were filled and labelled #22746.
The homogeneity of the subsamples was checked by determination of Cadmium by using an in house test method on 4 stratified randomly selected subsamples.

	Cadmium as Cd in mg/kg
sample #22746-1	13.7
sample #22746-2	13.1
sample #22746-3	13.4
sample #22746-4	13.8

Table 3: homogeneity test results of subsamples #22746

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.

	Cadmium as Cd in mg/kg
r (observed)	0.9
reference method	Horwitz
0.3 x R (reference method)	1.2

Table 4: evaluation of the repeatabilities of subsamples #22746

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 one sample of Body Cream labelled #22745 and one sample of Foundation labelled #22746 were sent on September 28, 2022.

2.5 ANALYZES

The participants were requested to determine on both samples the concentrations of: Antimony as Sb, Arsenic as As, Cadmium as Cd, Chromium as Cr, Lead as Pb, Mercury as Hg and Nickel as Ni.
It was also requested to report if the laboratory was accredited for the determined elements and to report some analytical details.

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 reference test methods (when applicable) 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 appendices 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 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 appendices 1 and 2. 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 (derived from e.g. ISO or ASTM test methods), 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:

$z_{(target)}$ = (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. 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

In this proficiency test no problems were encountered with the dispatch of the samples. Two participants did not report any test results after the final reporting date. All other participants reported test results in time. Not all participants were able to report all tests requested.

In total 8 participants reported 52 numerical test results. No outlying test result were observed. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

None of the data sets proved to have a normal Gaussian distribution. These are referred to as “unknown”. 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 ELEMENT

In this section the reported test results are discussed per sample and per element. 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 in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

Unfortunately, a suitable reference test method, providing the precision data, is not available for the determination of Metals in Skin Care products. For the evaluation in this PT the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

In this PT the uncertainty of the assigned value of the data in appendix 1 was investigated. It was concluded that the effect of the uncertainty on the calculation of the z-score was negligible and therefore not included in the z-score calculation.

sample #22745

Cadmium as Cd: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the estimated reproducibility calculated with the Horwitz equation.

Lead as Pb: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the estimated reproducibility calculated with the Horwitz equation.

Nickel as Ni: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

The participants agreed on a concentration near or below the limit of detection for all other elements mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these elements. The reported test results are given in appendix 2.

sample #22746

Cadmium as Cd: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the estimated reproducibility calculated with the Horwitz equation.

Chromium as Cr: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Lead as Pb: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Nickel as Ni: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

The participants agreed on a concentration near or below the limit of detection for all other elements mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these elements. The reported test results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from the reference method are presented in the next tables.

Element	unit	n	average	2.8 * sd	R(target)
Cadmium as Cd	mg/kg	8	9.5	3.1	3.0
Lead as Pb	mg/kg	8	14.9	4.1	4.4
Nickel as Ni	mg/kg	7	4.8	1.2	1.7

Table 5: reproducibilities of tests on sample #22745

Element	unit	n	average	2.8 * sd	R(target)
Cadmium as Cd	mg/kg	8	11.6	3.8	3.6
Chromium as Cr	mg/kg	6	6.4	1.4	2.2
Lead as Pb	mg/kg	8	17.8	4.6	5.2
Nickel as Ni	mg/kg	7	6.3	1.4	2.1

Table 6: reproducibilities of tests on sample #22746

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2022 WITH PREVIOUS PTS

	October 2022	October 2021	October 2020	November 2019
Number of reporting laboratories	8	17	16	18
Number of test results	52	68	106	155
Number of statistical outliers	0	1	0	6
Percentage of statistical outliers	0.0%	1.5%	0.0%	3.9%

Table 7: comparison with previous proficiency tests

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

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTS, see next table.

The uncertainties observed in this PT is in line with the uncertainties found in previous iis PTs.

Element	October 2022	October 2021	October 2020	February 2019	Target
Cadmium as Cd	11 - 12%	9%	7 - 11%	8 - 11%	10-11%
Chromium as Cr	8%	---	10 - 16%	9 - 14%	10-11%
Lead as Pb	9 - 10%	8 - 9%	---	13%	11-13%
Mercury as Hg	---	31%	17 - 19%	54%	12-15%
Nickel as Ni	8 - 9%	13%	8 - 15%	7 - 10%	10-18%

Table 8: development of the uncertainties over the years

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested and are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

- Seven participants mentioned that they are ISO/IEC17025 accredited to determine the reported elements.
- All participants mentioned to have used a sample intake between 0.1 - 0.3 grams.
- Six participants used ICP-MS to quantify the elements and one participant used ICP-OES.

The influence of these analytical details could not be determined because the group of participants is too small for further sub analyzes.

5 DISCUSSION

The participants identified all added metals in both Skin Care products correctly: the Body Cream sample #22745 contained Cadmium, Lead and Nickel and the Foundation sample #22746 contained Cadmium, Chromium, Lead and Nickel.

Limits for metals in cosmetics have been set by the EU, China, South East Asia and the USA (see Table 9). Other elements like Aluminum, Iron and Zinc can be present in the cosmetics, because they are introduced in the matrix as Fluoride salt or Oxide coloring (e.g. Fe). The limits of these elements are dependent on the use and higher than those of the other elements.

Element	EU 1223/09	STSC 2015	ASEAN	FDA
Antimony	not present	-----	-----	-----
Arsenic	not present	≤2mg/kg	<5 mg/kg	-----
Cadmium	not present	≤5mg/kg	<5 mg/kg	-----
Chromium	not present	-----	-----	-----
Lead	not present	≤10mg/kg	<20 mg/kg	-----
Mercury	not present	≤1mg/kg	<1 mg/kg	< 1 mg/kg
Nickel	not present	-----	-----	-----

Table 9: Limits for different Elements

Based on the Mercury limit set by the FDA all participants would have accepted both samples, however all participants would have rejected both samples based on EU 1223/09, STSC 2015 or ASEAN limits.

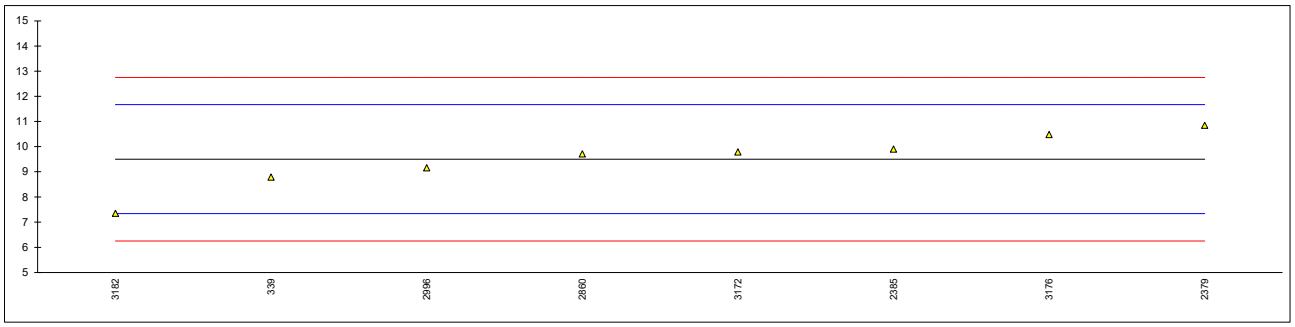
6 CONCLUSION

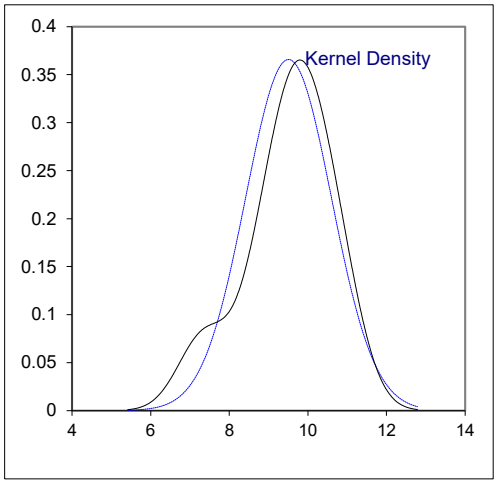
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of Cadmium as Cd in Body Cream, sample #22745; results in mg/kg

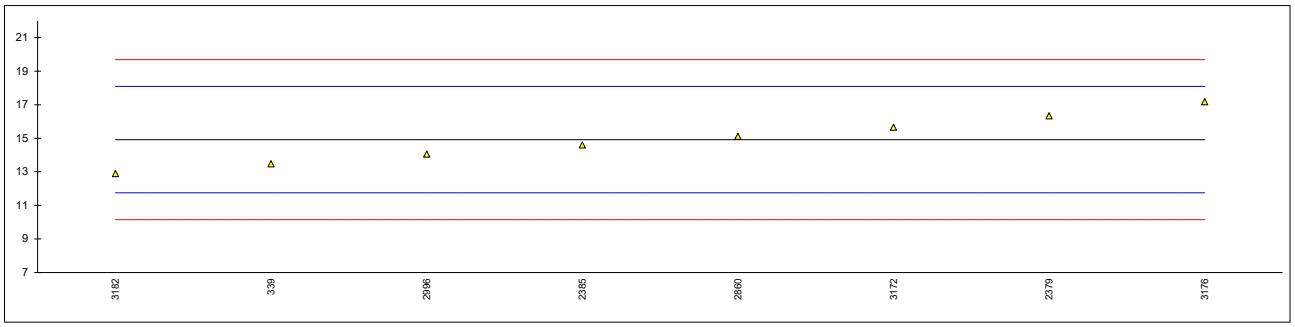
lab	method	value	mark	z(targ)	remarks
339	In house	8.79		-0.66	
2379	In house	10.85		1.24	
2385		9.9		0.36	
2762		-----		-----	
2860	In house	9.7165		0.19	
2996	K84.00-31/ISO21392	9.16		-0.32	
3166		-----		-----	
3172	In house	9.7925		0.27	
3176	In house	10.483		0.90	
3182	In house	7.35	C	-1.99	first reported 6.61
	normality	unknown			
	n	8			
	outliers	0			
	mean (n)	9.505			
	st.dev. (n)	1.0904	RSD = 11%		
	R(calc.)	3.053			
	st.dev.(Horwitz)	1.0836			
	R(Horwitz)	3.034			

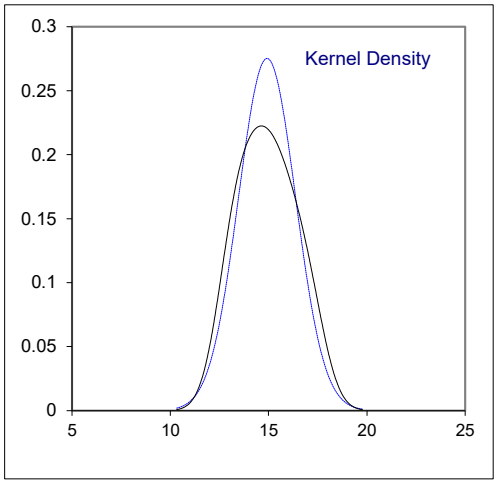




Determination of Lead as Pb in Body Cream, sample #22745; results in mg/kg

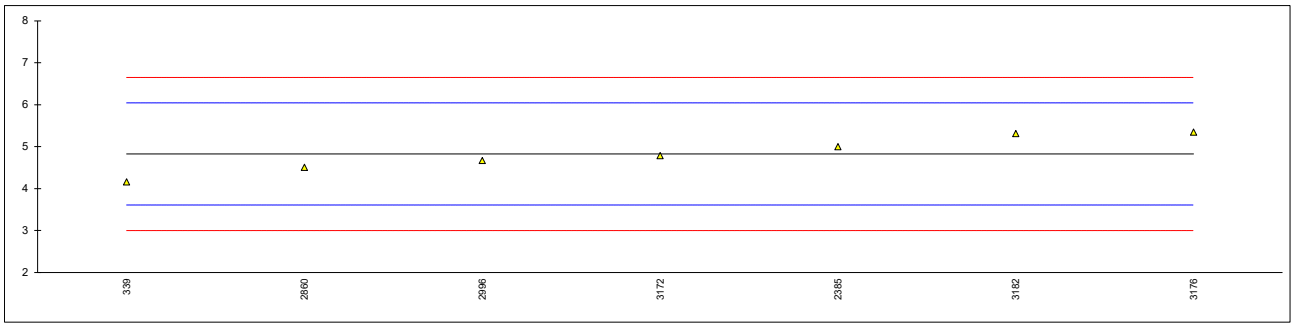
lab	method	value	mark	z(targ)	remarks
339	In house	13.48		-0.90	
2379	In house	16.34		0.90	
2385		14.6		-0.20	
2762		-----		-----	
2860	In house	15.1166		0.13	
2996	K84.00-31/ISO21392	14.06		-0.54	
3166		-----		-----	
3172	In house	15.655		0.46	
3176	In house	17.185		1.43	
3182	In house	12.90		-1.27	
	normality	unknown			
	n	8			
	outliers	0			
	mean (n)	14.917			
	st.dev. (n)	1.4502	RSD = 10%		
	R(calc.)	4.061			
	st.dev.(Horwitz)	1.5891			
	R(Horwitz)	4.449			

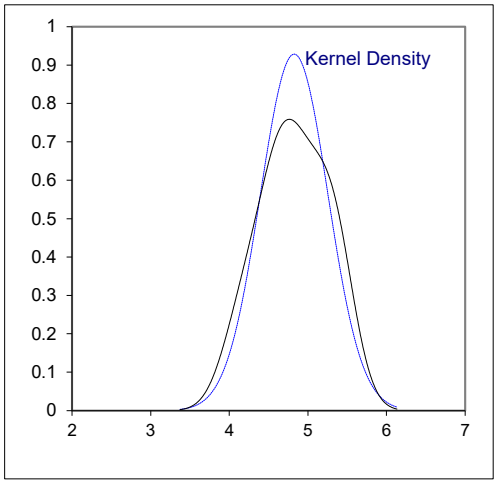




Determination of Nickel as Ni in Body Cream, sample #22745; results in mg/kg

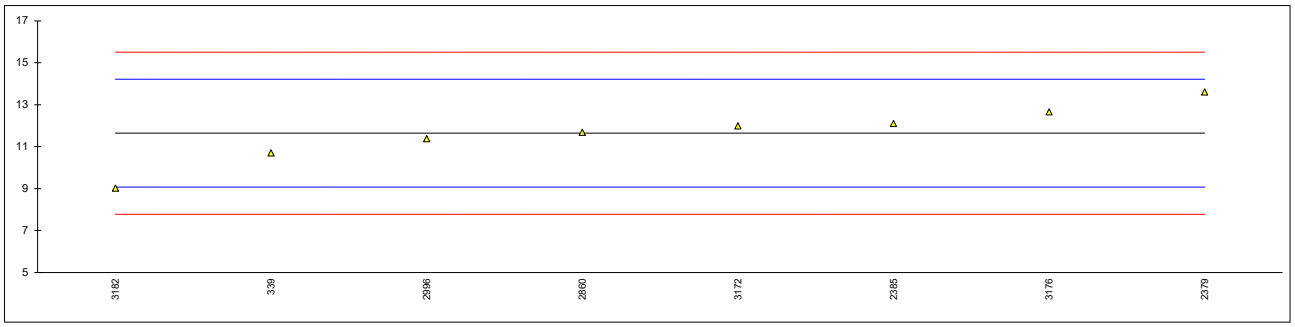
lab	method	value	mark	z(targ)	remarks
339	In house	4.16		-1.09	
2379		not analyzed		-----	
2385		5.0		0.29	
2762		-----		-----	
2860	In house	4.5068		-0.52	
2996	K84.00-31/ISO21392	4.67		-0.26	
3166		-----		-----	
3172	In house	4.785		-0.07	
3176	In house	5.347		0.86	
3182	In house	5.31		0.80	
	normality	unknown			
	n	7			
	outliers	0			
	mean (n)	4.826			
	st.dev. (n)	0.4296	RSD = 9%		
	R(calc.)	1.203			
	st.dev.(Horwitz)	0.6092			
	R(Horwitz)	1.706			

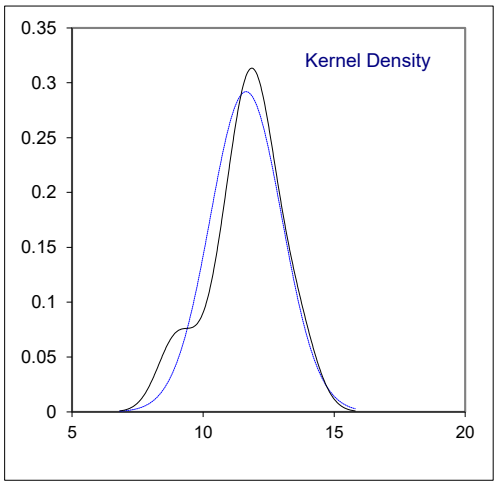




Determination of Cadmium as Cd in Foundation, sample #22746; results in mg/kg

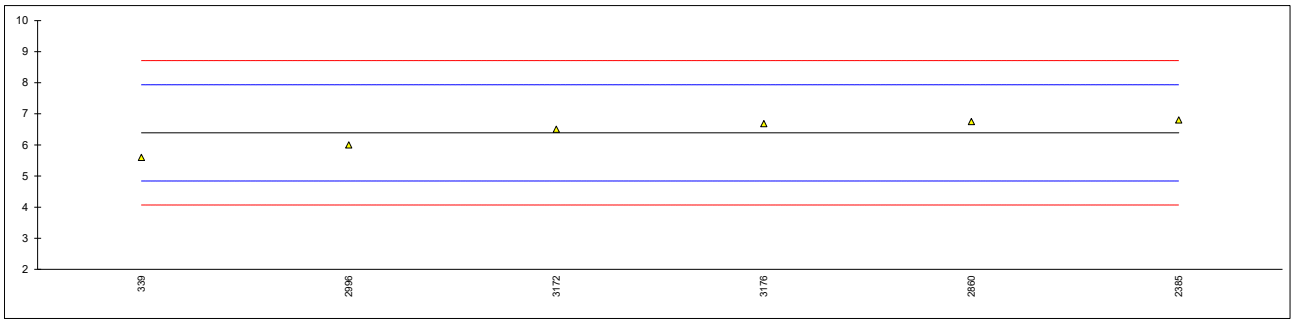
lab	method	value	mark	z(targ)	remarks
339	In house	10.70		-0.73	
2379	In house	13.61		1.53	
2385		12.1		0.35	
2762		-----		-----	
2860	In house	11.6848		0.03	
2996	K84.00-31/ISO21392	11.38		-0.20	
3166		-----		-----	
3172		11.99		0.27	
3176	In house	12.663		0.79	
3182	In house	9.02	C	-2.04	first reported 8.06
	normality	unknown			
	n	8			
	outliers	0			
	mean (n)	11.643			
	st.dev. (n)	1.3669	RSD = 12%		
	R(calc.)	3.827			
	st.dev.(Horwitz)	1.2875			
	R(Horwitz)	3.605			





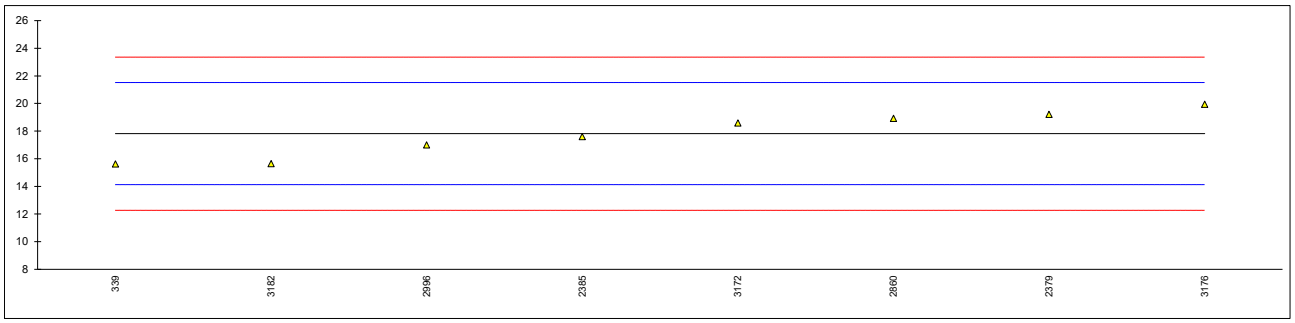
Determination of Chromium as Cr in Foundation, sample #22746; results in mg/kg

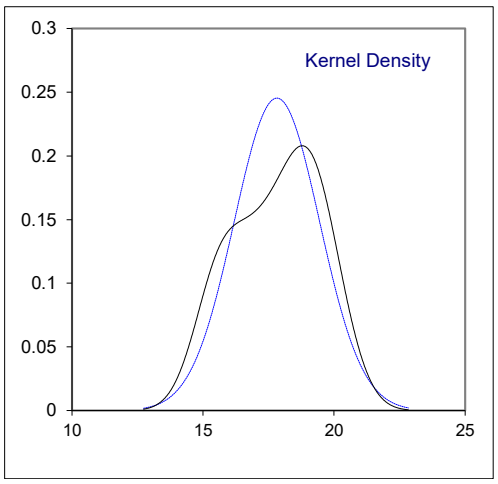
lab	method	value	mark	z(targ)	remarks
339	In house	5.60		-1.02	
2379		not analyzed		-----	
2385		6.8		0.53	
2762		-----		-----	
2860	In house	6.7511		0.47	
2996	K84.00-31/ISO21392	6.00		-0.50	
3166		-----		-----	
3172		6.50		0.14	
3176	In house	6.683		0.38	
3182		Not analyzed		-----	
	normality	unknown			
	n	6			
	outliers	0			
	mean (n)	6.389			
	st.dev. (n)	0.4843	RSD = 8%		
	R(calc.)	1.356			
	st.dev.(Horwitz)	0.7733			
	R(Horwitz)	2.165			



Determination of Lead as Pb in Foundation, sample #22746; results in mg/kg

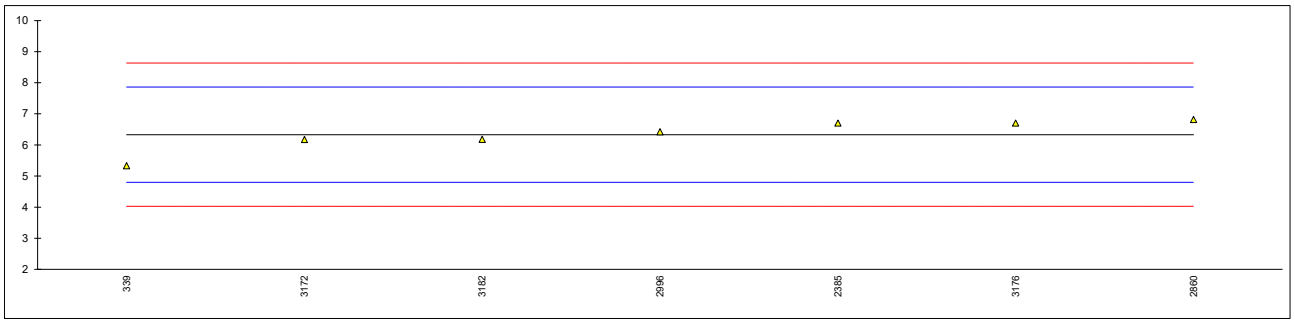
lab	method	value	mark	z(targ)	remarks
339	In house	15.62		-1.19	
2379	In house	19.21		0.75	
2385		17.6		-0.12	
2762		-----		-----	
2860	In house	18.9252		0.60	
2996	K84.00-31/ISO21392	16.99		-0.45	
3166		-----		-----	
3172		18.59		0.42	
3176	In house	19.935		1.15	
3182	In house	15.65		-1.17	
	normality	unknown			
	n	8			
	outliers	0			
	mean (n)	17.815			
	st.dev. (n)	1.6259	RSD = 9%		
	R(calc.)	4.553			
	st.dev.(Horwitz)	1.8478			
	R(Horwitz)	5.174			

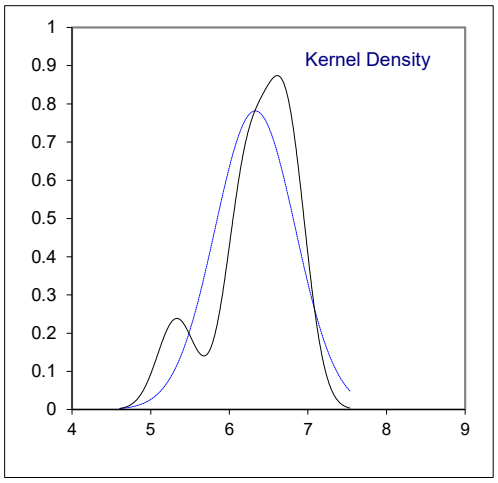




Determination of Nickel as Ni in Foundation, sample #22746; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	5.33		-1.30	
2379		not analyzed		-----	
2385		6.7		0.48	
2762		-----		-----	
2860	In house	6.8144		0.63	
2996	K84.00-31/ISO21392	6.42		0.12	
3166		-----		-----	
3172		6.1725		-0.21	
3176	In house	6.70	C	0.48	first reported as Hg
3182	In house	6.18		-0.20	
	normality	unknown			
	n	7			
	outliers	0			
	mean (n)	6.331			
	st.dev. (n)	0.5105	RSD = 8%		
	R(calc.)	1.429			
	st.dev.(Horwitz)	0.7673			
	R(Horwitz)	2.148			





APPENDIX 2 Other reported elements

Other reported elements in sample #22745; results in mg/kg

lab	Sb	As	Cr	Hg
339	<0.1	<0.1	<1	<0.1
2379	not analyzed	not detected	not analyzed	not detected
2385	<0.5	<0.5	<0.5	<0.05
2762	-----	-----	-----	-----
2860	<0,5	<0,5	<0,5	<0,5
2996	<LOQ (<0.075)	< LOQ (<0.100)	<LOQ (<0.070)	<LOQ (<0.030)
3166	-----	-----	-----	-----
3172	< 0.25	< 0.25	< 0.5	< 0.05
3176	-----	-----	-----	-----
3182	<0.50	<0.50	Not analyzed	<0.10

Other reported elements in sample #22746; results in mg/kg

lab	Sb	As	Hg
339	0.38	<0.1	<0.1
2379	not analyzed	not detected	not detected
2385	<0.5	<0.5	<0.05
2762	-----	-----	-----
2860	<0,5	<0,5	<0,5
2996	<LOQ (<0.075)	<LOQ (<0.100)	<LOQ (<0.030)
3166	-----	-----	-----
3172	< 0.25	< 0.25	< 0.05
3176	-----	-----	ND C
3182	<0.50	<0.50	<0.10

Lab 3176 first reported 6.700 for Hg

APPENDIX 3 Analytical details

lab	ISO/IEC17025 accredited	Sample intake (g)	Technique used
339	No	0.1	ICP-MS
2379	Yes	0.2	ICP-MS
2385	Yes	0,2 - 0,3	ICP-MS
2762	---		---
2860	Yes	0,25	ICP-OES
2996	Yes	0,1	ICP-MS
3166	---		---
3172	Yes		---
3176	Yes	0,1	ICP-MS
3182	Yes	0.25	ICP-MS

APPENDIX 4

Number of participants per country

1 lab in	CZECH REPUBLIC
1 lab in	FRANCE
2 labs in	GERMANY
1 lab in	ITALY
1 lab in	SERBIA
2 labs in	THAILAND
1 lab in	TURKEY
1 lab in	U.S.A.

APPENDIX 5

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
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

Literature

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