Results of Proficiency Test
Trace Metals in skin care
Body Cream and Foundation
October 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. C.M. Nijssen-Wester
Correctors: ing. R.J. Starink & ing. A.S. Noordman-de Neef
Report: iis21H05

January 2022

CONTENTS

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 October 2021. 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 (HSC2007). In 2015 this standard was superseded by the Chinese Technical Safety Standards for Cosmetics (TSSC2015) which was implemented in 2016 limits for Arsenic, Cadmium, Lead and Mercury.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 Body Cream and Foundation. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test for the determination of Trace Metals in Body Cream and Foundation.

In this interlaboratory study 17 laboratories in 15 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the Trace Metals in Body Cream and Foundation 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; a Body Cream of approximately 10 mL labelled #21735 and a Foundation of approximately 10 mL labelled #21736. Both samples were made positive with a few 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/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 and Lead. After homogenization 35 bottles of 10 mL were filled and labelled #21735.
The homogeneity of the subsamples was checked by determination of Cadmium by using the Safety and Technical Standard for Cosmetics (2015 version) on five stratified randomly selected subsamples.

	Cadmium as Cd in mg/kg
sample #21735-1	11.61
sample #21735-2	11.05
sample #21735-3	11.72
sample #21735-4	11.33
sample #21735-5	11.52

Table 1: homogeneity test results of subsamples #21735

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)	0.74
reference method	Horwitz
0.3 x R (reference method)	1.07

Table 2: evaluation of the repeatability of subsamples #21735

The calculated repeatability was in agreement with 0.3 times the target reproducibility. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of a regular foundation was purchased from a local supermarket and was artificially fortified with Lead and Mercury. After homogenization 35 bottles of 10 mL were filled and labelled #21736.
The homogeneity of the subsamples was checked by determination of Mercury by using the Safety and Technical Standard for Cosmetics (2015 version) on five stratified randomly selected subsamples.

	Mercury as Hg in mg/kg
sample #21736-1	8.47
sample #21736-2	8.90
sample #21736-3	9.05
sample #21736-4	8.71
sample #21736-5	8.55

Table 3: homogeneity test results of subsamples #21736

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 as Hg in mg/kg
r (observed)	0.67
reference method	Horwitz
0.3 x R (reference method)	0.85

Table 4: evaluation of the repeatabilities of subsamples #21736

The calculated repeatability was in agreement with 0.3 times the target reproducibility. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample of Body Cream labelled #21735 and one sample of Foundation labelled #21736 were sent on September 22, 2021.

2.5 ANALYZES

The participants were requested to determine on both samples the concentrations of: Aluminum as Al, Antimony as Sb, Arsenic as As, Cadmium as Cd, Chromium as Cr, Iron as Fe, Lead as Pb, Mercury as Hg, Nickel as Ni and Zinc as Zn.
It was also requested to report if the laboratory was accredited for the requested elements that were determined 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 appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus 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 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.

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

This 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

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. One participant did not report any test results after the extended reporting date. All other participants reported test results in time. Not all participants were able to report all tests requested.

In total 17 participants reported 68 numerical test results. Observed was 1 outlying test result, which is 1.5%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

All data sets proved to have a normal Gaussian distribution.

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 all determinations. For these tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

sample #21735

Cadmium as Cd: 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.

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

sample #21736

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.

Mercury as Hg: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility is not at all in agreement with the estimated reproducibility calculated from the Horwitz equation. Due to the large variation in reported test results, no z-scores were calculated.

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.

For all other elements the participants agreed on a concentration near or below the limit of detection. 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 estimated using the Horwitz equation are presented in the next table.

Element	unit	n	average	2.8 * sd	R(target)
Cadmium as Cd	mg/kg	16	10.5	2.7	3.3
Lead as Pb	mg/kg	16	10.6	2.5	3.3

Table 5: reproducibilities of tests on sample #21735

Element	unit	n	average	2.8 * sd	R(target)
Lead as Pb	mg/kg	16	10.5	2.6	3.3
Mercury as Hg	mg/kg	14	7.6	6.6	(2.5)
Nickel as Ni	mg/kg	6	0.7	0.3	0.3

Table 6: reproducibilities of tests on sample #21736

Values between brackets were not used for the calculation of z-scores

Without further statistical calculations, it can be concluded that there is a good compliance of the group of participating laboratories with the reference target. The problematic tests have been discussed in paragraph 4.1 and 5.

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

	October 2021	October 2020	November 2019
Number of reporting laboratories	17	16	18
Number of test results	68	106	155
Number of statistical outliers	1	0	6
Percentage of statistical outliers	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 tests was compared, expressed as relative standard deviation (RSD) of the PTs, see next table.

Element	October 2021	October 2020	February 2019	Target	Conc. in mg/kg
Cadmium as Cd	9%	7-11%	8-11%	10-11%	10-21
Chromium as Cr	n.e.	10-16%	9-14%	10-11%	16-22
Lead as Pb	8-9%	n.e.	13%	11-12%	5-10
Mercury as Hg	31%	17-19%	54%	12-15%	1.5-7.5
Nickel as Ni	13%	8-15%	7-10%	10-18%	0.5-22
Aluminum as Al	n.e.	16%	n.a.	5%	2050
Iron as Fe	n.e.	6%	n.a.	4%	15746

Table 8: development of the uncertainties over the years

4.4 EVALUATION ANALYTICAL DETAILS

The participants were asked to provide some analytical details which are listed in appendix 3. Based on the reported answers by sixteen participants the following can be summarized:

- Thirteen participants mentioned that they are ISO/IEC17025 accredited to determine the reported elements.
- Fourteen participants used a sample intake between 0.1 - 0.5 grams and one participant around 2 grams. Presummable amount of intake is dependent on matrix.
- Fourteen participants used ICP-MS to determine the metal content. Two participants 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

In this proficiency test the added metals in Body Cream and Foundation, two different types of Skin cosmetic products, were correctly identified.

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. All participants would have rejected both samples based on one of these limits.

Element	EU 1223/09	TSSC 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

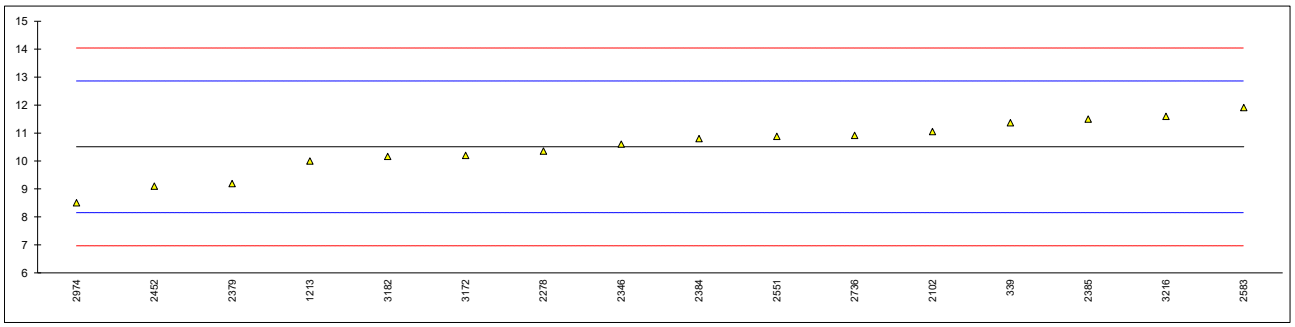
6 CONCLUSION

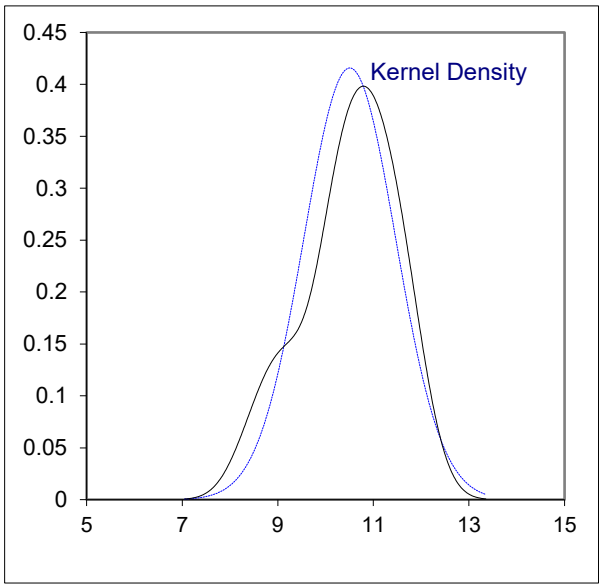
Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of Cadmium as Cd in Body Cream, sample #21735; results in mg/kg

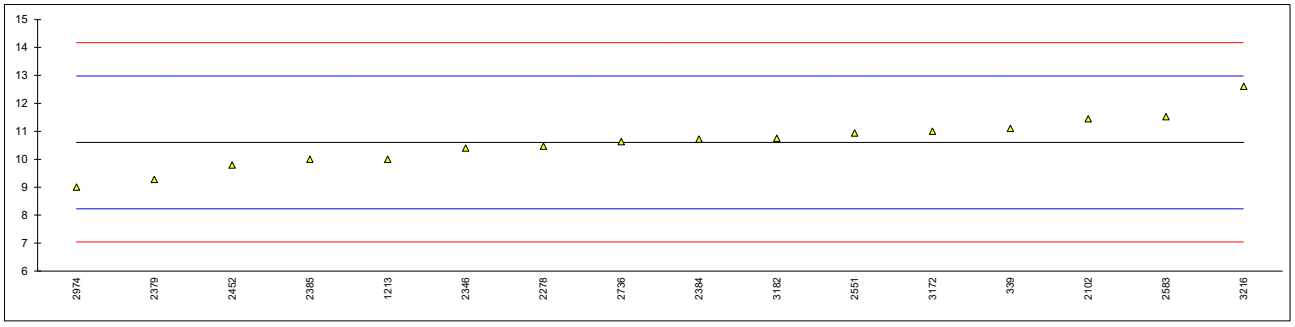
lab	method	value	mark	z(targ)	remarks
339		11.37		0.73	
1213	ACM THA 05	10	C	-0.43	first reported: 8.18
2102	In house	11.051		0.46	
2278	STSC	10.3498		-0.13	
2346	In house	10.6		0.08	
2375		-----		-----	
2379	ACM 005	9.19		-1.12	
2384	In house	10.80		0.25	
2385		11.5		0.84	
2452	In house	9.10		-1.19	
2551	In house	10.879		0.32	
2583	§LFGB K80.00-31	11.91		1.19	
2736	In house	10.909		0.34	
2974	In house	8.50		-1.70	
3172	In house	10.2		-0.26	
3182	in house	10.159		-0.30	
3216	In house	11.596		0.92	
	normality	OK			
	n	16			
	outliers	0			
	mean (n)	10.507			
	st.dev. (n)	0.9597	RSD = 9%		
	R(calc.)	2.687			
	st.dev.(Horwitz)	1.1800			
	R(Horwitz)	3.304			

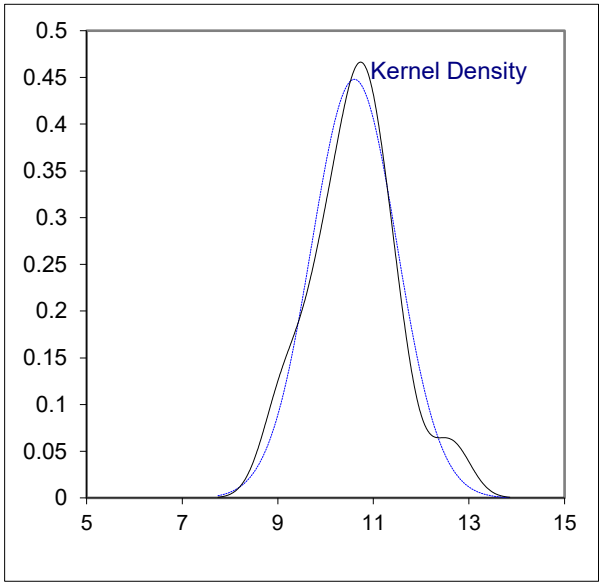




Determination of Lead as Pb in Body Cream, sample #21735; results in mg/kg

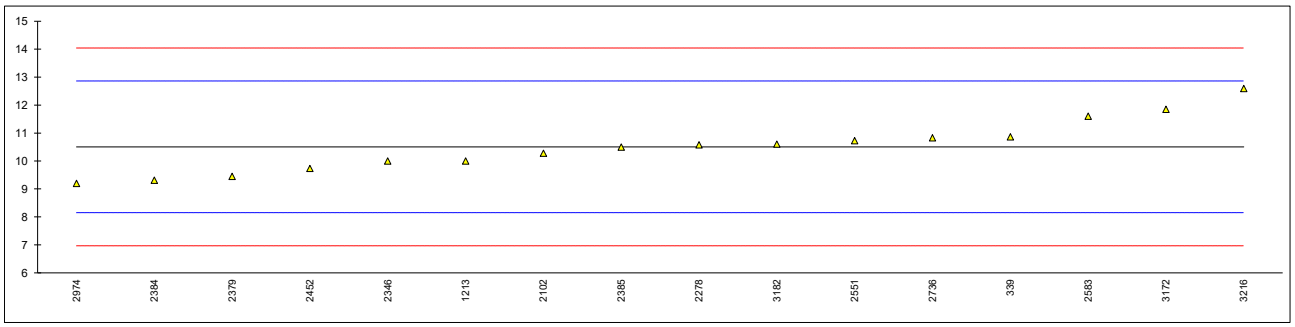
lab	method	value	mark	z(targ)	remarks
339		11.104		0.42	
1213	ACM THA 05	10	C	-0.51	first reported: 7.49
2102	In house	11.445		0.71	
2278	STSC	10.4680		-0.11	
2346	In house	10.4		-0.17	
2375		-----		-----	
2379	ACM 005	9.28		-1.11	
2384	In house	10.72		0.10	
2385		10		-0.51	
2452	In house	9.79		-0.68	
2551	In house	10.939		0.28	
2583	§LFGB K80.00-31	11.52		0.77	
2736	In house	10.631		0.02	
2974	In house	9.00		-1.35	
3172	In house	11.0		0.33	
3182	in house	10.749		0.12	
3216	In house	12.609		1.69	
	normality	OK			
	n	16			
	outliers	0			
	mean (n)	10.603			
	st.dev. (n)	0.8906	RSD = 8%		
	R(calc.)	2.494			
	st.dev.(Horwitz)	1.1891			
	R(Horwitz)	3.330			

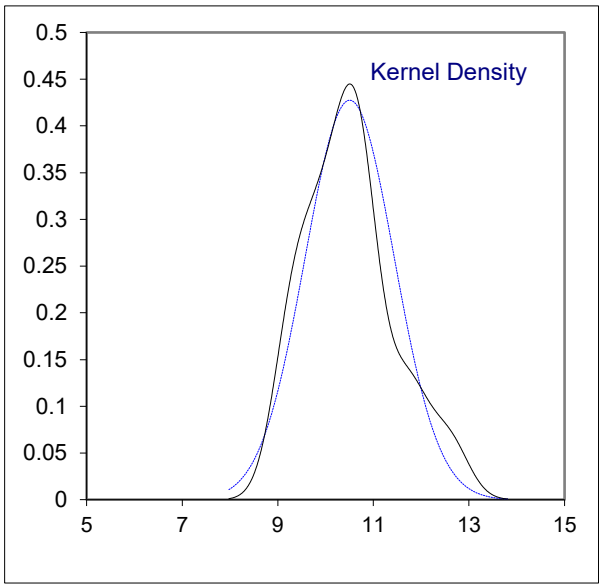




Determination of Lead as Pb in Foundation, sample #21736; results in mg/kg

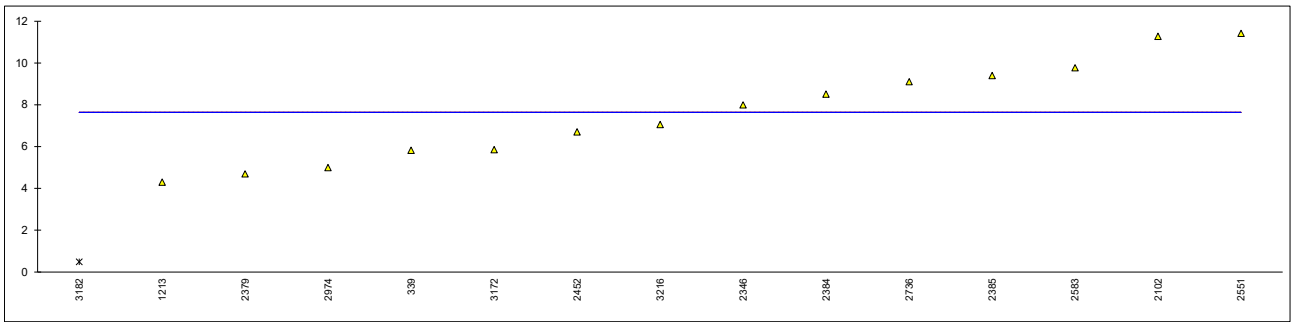
lab	method	value	mark	z(targ)	remarks
339		10.861		0.30	
1213	ACM THA 05	10	C	-0.43	first reported: 7.34
2102		10.277		-0.19	
2278	STSC	10.5724		0.06	
2346	In house	10.0		-0.43	
2375		-----		-----	
2379	ACM 005	9.45		-0.90	
2384	In house	9.31		-1.01	
2385		10.5		-0.01	
2452	In house	9.73		-0.66	
2551	In house	10.726		0.19	
2583	§LFGB K80.00-31	11.60		0.93	
2736	In house	10.829		0.27	
2974	In house	9.20		-1.11	
3172	In house	11.85		1.14	
3182	In house	10.6		0.08	
3216	In house	12.590		1.77	
	normality	OK			
	n	16			
	outliers	0			
	mean (n)	10.506			
	st.dev. (n)	0.9330	RSD = 9%		
	R(calc.)	2.612			
	st.dev.(Horwitz)	1.1798			
	R(Horwitz)	3.303			

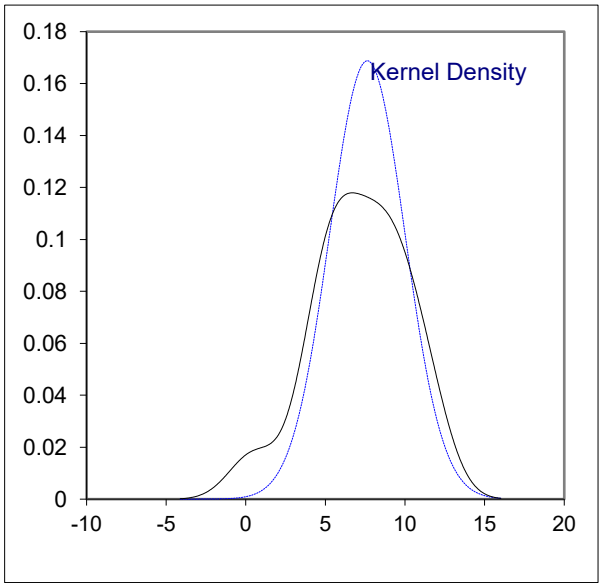




Determination of Mercury as Hg in Foundation, sample #21736; results in mg/kg

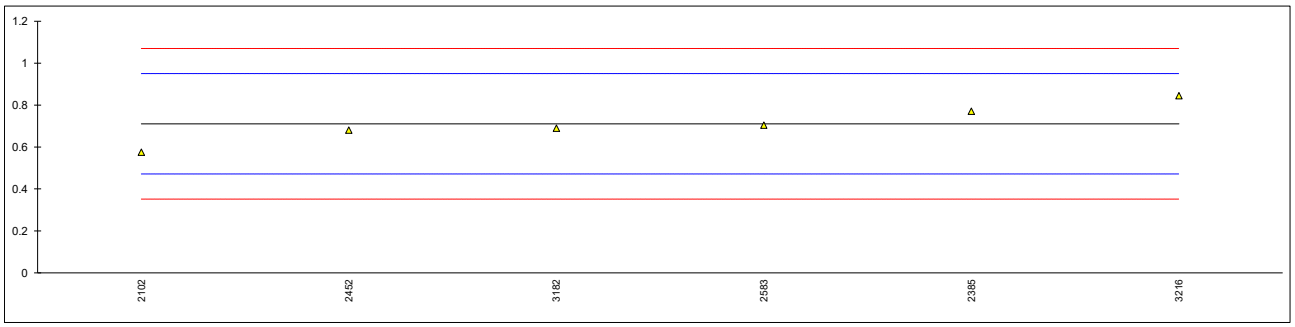
lab	method	value	mark	z(targ)	remarks
339		5.823		-----	
1213	ACM THA 05	4.30		-----	
2102		11.269		-----	
2278		-----		-----	
2346	In house	7.99		-----	
2375		-----		-----	
2379	ACM 005	4.69		-----	
2384	In house	8.51		-----	
2385		9.4		-----	
2452	In house	6.70		-----	
2551	In house	11.419		-----	
2583	§LFGB K80.00-33	9.773		-----	
2736	In house	9.102		-----	
2974	In house	5.00		-----	
3172	In house	5.85		-----	
3182	In house	0.485	G(0.05)	-----	
3216	In house	7.055		-----	
	normality	OK			
	n	14			
	outliers	1			
	mean (n)	7.634			
	st.dev. (n)	2.3644	RSD = 31%		
	R(calc.)	6.620			
	st.dev.(Horwitz)	(0.900)			
	R(Horwitz)	(2.519)			





Determination of Nickel as Ni in Foundation, sample #21736; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		<1		-----	
1213		-----		-----	
2102		0.575		-1.13	
2278		-----		-----	
2346		-----		-----	
2375		-----		-----	
2379		-----		-----	
2384		-----		-----	
2385		0.77		0.50	
2452	In house	0.68		-0.25	
2551		-----		-----	
2583	§LFGB K80.00-31	0.704		-0.05	
2736		-----		-----	
2974	In house	Not analysed		-----	
3172	In house	< 1		-----	
3182	In house	0.690		-0.17	
3216	In house	0.844		1.12	
	normality	unknown			
	n	6			
	outliers	0			
	mean (n)	0.711			
	st.dev. (n)	0.0907	RSD = 13%		
	R(calc.)	0.254			
	st.dev.(Horwitz)	0.1197			
	R(Horwitz)	0.335			



APPENDIX 2 Other reported elements

Other reported elements in sample #21735; results in mg/kg

lab	Al	Sb	As	Cr
339	-----	<0.1	<0.1	<1
1213	-----	-----	<0.20 C	-----
2102	Not analysed	Not detected	Not detected	<0.125
2278	-----	-----	＜0.0033	-----
2346	-----	-----	-----	-----
2375	-----	<0.083	<0.12	-----
2379	-----	-----	Not detected	-----
2384	-----	not detected	not detected	-----
2385	11.3	<0.5	<0.5	<0.5
2452	8.80	not detected	not detected	not detected
2551	-----	< LOQ	< LOQ	-----
2583	11.89	below det. limit	below det. limit	0.038
2736	-----	-----	<0.125	<0.125
2974	Not analysed	<LOQ (0.8 ppm)	<LOQ (0.8 ppm)	Not analysed
3172	-----	< 1	< 1	< 1
3182	Not analysed	<0.5	Not detected	Not analysed
3216	17.202	not detected	not detected	not detected

lab	Fe	Hg	Ni	Zn
339	-----	<0.1	<1	-----
1213	-----	<0.03	-----	-----
2102	Not analysed	Not detected	Not detected	Not detected
2278	-----	-----	-----	-----
2346	-----	-----	-----	-----
2375	-----	-----	-----	-----
2379	-----	Not detected	-----	-----
2384	-----	not detected	-----	-----
2385	11.8	<0.05	<0.5	<0.5
2452	10.49	not detected	not detected	not detected
2551	-----	< LOQ	-----	-----
2583	12.54	below det. limit	0.017	1.674
2736	-----	<0.125	-----	-----
2974	Not analysed	<LOQ (0.8 ppm)	Not analysed	Not analysed
3172	-----	< 0.5	< 1	-----
3182	Not analysed	<0.1	<0.5	Not analysed
3216	11.171	0.051	not detected	not detected

Labcode 1213 first reported for As: 0.56

Other reported elements in sample #21736; results in mg/kg

lab	Al	Sb	As	Cd	Cr	Fe	Zn
339	-----	<0.1	0.155	<0.1	<1	-----	-----
1213	-----	-----	<0.20 C	<0.30	-----	-----	-----
2102	Not analysed	Not detected	0.124	Not detected	0.455	Not analysed	11.376
2278	-----	-----	0.1086	＜0.0033	-----	-----	-----
2346	-----	-----	0.134	-----	-----	-----	-----
2375	-----	<0.083	-----	-----	-----	-----	-----
2379	-----	-----	Not detected	Not detected	-----	-----	-----
2384	-----	not detected	not detected	not detected	-----	-----	-----
2385	1460	<0.5	<0.5	<0.05	<0.5	7795	13.1
2452	1214.00	Not detected	Not detected	Not detected	0.23	6713.07	10.89
2551	-----	< LOQ	< LOQ	< LOQ	-----	-----	-----
2583	1139	below det. limit	0.168	0.003	0.353	7984	15.29
2736	-----	-----	<0.122	<0.024	0.348	-----	-----
2974	Not analysed	<LOQ (0.8 ppm)	<LOQ (0.8 ppm)	<LOQ (0.8 ppm)	Not analysed	Not analysed	Not analysed
3172	-----	< 1	< 1	< 0.5	< 1	-----	-----
3182	Not analysed	Not detected	<0.5	Not detected	Not analysed	Not analysed	Not analysed
3216	394.796 C	1.636	0.113	not detected	0.360	9761.285	19.660

Labcode 1213 first reported for As: 0.54
Labcode 3216 first reported for Al: 355.884

APPENDIX 3 Analytical details

lab	ISO17025 accredited	Sample intake (g) #21735	Sample intake (g) #21736	Technique used	remarks
339	No	0.1g	0.1g	ICP-MS	Hg was performed by DMA.
1213	Yes	0.5 gram	0.5 gram	ICP-OES:Pb, Cd	Hg-AAS: As CV-AAS: Hg
2102	Yes	0.1	0.1	ICP-MS	
2278	Yes	About 2g	About 2g	ICP-MS	
2346	Yes	0.5 g	0.5 g	ICP-MS	
2375	Yes			ICP-MS	
2379	Yes	0.2 g	0.2 g	ICP-MS	
2384	Yes	0.25	0.25	ICP-MS	
2385	Yes	ca. 0.3 g	ca. 0.3 g	ICP-MS	some results by ICP-OES
2452	Yes	0.1	0.1	ICP-MS	
2551	No	0.2 - 0.25 gm	0.2 - 0.25 gm	ICP-MS	*)
2583	Yes	0,14 - 0,34	0,14-0,33	ICP-MS	For Hg CV-AAS was used.
2736	Yes	0.2g	0.2g	ICP-MS	
2974	Yes	0.25	0.25	ICP-OES	
3172	---			---	
3182	Yes	0.25	0.25	ICP-MS	
3216	No	0,3g	0,3g	ICP-MS	-

*) Remarks lab 2551: our scope in 5 elements (As, Cd, Sb, Hg and Pb) LOQ (mg/Kg) for these element are as following: AS = 0.224 , Cd = 0.184 , Sb = 0.242 , Hg = 0.188

APPENDIX 4

Number of participants per country

1 lab in	FRANCE
2 labs in	GERMANY
1 lab in	HONG KONG
1 lab in	ITALY
1 lab in	MALAYSIA
1 lab in	P.R. of CHINA
1 lab in	SAUDI ARABIA
1 lab in	SPAIN
2 labs in	THAILAND
1 lab in	THE NETHERLANDS
1 lab in	TUNISIA
1 lab in	TURKEY
1 lab in	U.S.A.
1 lab in	UNITED ARAB EMIRATES
1 lab in	VIETNAM

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
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 & 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)