Results of Proficiency Test
Trace Metals in oral care
Mouthwash & Toothpaste
October 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Heavy metals like Arsenic, Cadmium, Chromium, Lead, Mercury and Nickel 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 Council Directive 76/768/EEC. In Annex II there is a list of substances that cosmetics must not contain like Arsenic, Cadmium, Chromium, Lead and Mercury. Based on this European regulation China issued the Hygienic Standard for Cosmetics (HSC2007) with limit levels for certain heavy metals in 2007. In 2015 this standard was superseded by the Chinese Technical Safety Standards for Cosmetics (TSSC2015) which was implemented in 2016 (see table 1).

Element	HSC 2007	TSSC 2015
Arsenic	≤10mg/kg	≤2mg/kg
Cadmium	Not Specified	≤5mg/kg
Lead	≤40mg/kg	≤10mg/kg
Mercury	≤1mg/kg	≤1mg/kg

Table 1: Limits for different Elements

No certified reference materials (CRMs) for Trace Metals in cosmetics are available to optimize 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 the comparability between laboratories.

Since 2019 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Trace Metals in oral care Mouth Wash and Toothpaste. During the annual proficiency testing program 2020/2021 it was decided to continue the proficiency test for the analysis of Trace Metals in oral care Mouth Wash and Toothpaste.
In this interlaboratory study 9 laboratories from 8 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the 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 oral care samples; a sample based on Mouthwash labelled #20685 of approximately 10 mL and a sample based on Toothpaste labelled #20686 of approximately 13 grams. Both samples were made positive with several 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 a regular mouthwash was purchased from a local supermarket and was artificially fortified with Arsenic, Cadmium, Chromium, Nickel and Lead. After homogenization 23 bottles of 10 mL were filled with moutwash and labelled #20685.
The homogeneity of the subsamples was checked by determination of Arsenic, Cadmium, Chromium, Lead and Nickel by using ICP-MS on five stratified randomly selected subsamples.

	Arsenic in mg/kg	Cadmium in mg/kg	Chromium in mg/kg	Lead in mg/kg	Nickel in mg/kg
sample #20685-1	6.124	6.124	6.350	17.010	6.124
sample #20685-2	6.203	5.973	6.203	16.771	5.973
sample #20685-3	6.436	5.771	5.993	16.868	5.771
sample #20685-4	6.455	5.830	6.038	16.657	5.830
sample #20685-5	6.501	5.851	5.851	16.685	5.634

Table 2: homogeneity test results of subsamples #20685

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

	Arsenic in mg/kg	Cadmium in mg/kg	Chromium in mg/kg	Lead in mg/kg	Nickel in mg/kg
r (observed)	0.472	0.393	0.541	0.404	0.528
reference method	Horwitz	Horwitz	Horwitz	Horwitz	Horwitz
0.3 * R (reference method)	0.646	0.608	0.623	1.477	0.604

Table 3: evaluation of the repeatabilities of subsamples #20685

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

For the second sample a batch of a regular toothpaste was purchased from a local supermarket and was artificially fortified with Cadmium, Chromium, Lead and Nickel. After homogenization 25 bottles of 10 mL were filled with approximately 13 grams toothpaste and labelled #20686.
The homogeneity of the subsamples was checked by determination of Cadmium by using ICP-MS on five stratified randomly selected subsamples.

	Cadmium in mg/kg
sample #20686-1	5.295
sample #20686-2	5.464
sample #20686-3	5.303
sample #20686-4	5.094
sample #20686-5	5.254

Table 4: homogeneity test results of subsamples #20686

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

	Cadmium in mg/kg
r (observed)	0.370
reference method	Horwitz
0.3 * R (reference method)	0.553

Table 5: evaluation of the repeatability of subsamples #20686

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 labelled #20685 and one sample labelled #20686 were sent on September 23, 2020.

2.5 ANALYZES

The participants were requested to determine on samples #20685 and #20686 the concentrations of: Arsenic as As, Cadmium as Cd, Chromium as Cr, Lead as Pb, Mercury as Hg, Nickel as Ni, Aluminum as Al, Antimony as Sb, Iron as Fe 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 re-analysis). Additional or corrected test results are used for the data analysis and the 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 in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ (iis-protocol, version 3.5) of June 2018. 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.

According to ISO5725 the original test results per determination were submitted to Dixon’s, Grubbs' 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.

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 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 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. 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 interlaboratory study some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the final reporting date was extended with one week. One participant reported test results after the final reporting date. All participants reported test results. Not all participants were able to report all tests requested. In total the 9 reporting laboratories submitted 65 numerical test results. No outlying test results were observed. 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.

4.1 EVALUATION PER SAMPLE AND PER ELEMENT

In this section the 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 reported test results 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 determinations of heavy metals in personal care products. Therefore, the calculated reproducibilities were compared against the estimated reproducibility calculated from the Horwitz equation.

Sample #20685, Mouthwash

Arsenic as As: 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.

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

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

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

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

Other Elements: The participants agreed on a concentration near or below the limit of detection for the other elements requested. Therefore, no z-scores were calculated. The reported test values are given in appendix 2.

Sample #20686, Toothpaste

Cadmium as Cd: 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.

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

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

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

Other Elements: The participants agreed on a concentration near or below the limit of detection for the other elements requested. Therefore, no z-scores were calculated. The reported test values 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 or as declared by the estimated target reproducibility using the Horwitz equation 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 literature reference test methods (in casu Horwitz Equation) are presented in the next table.

Element	unit	n	average	2.8 * sd	R(target)
Arsenic as As	mg/kg	9	5.8	1.9	2.0
Cadmium as Cd	mg/kg	9	5.2	1.6	1.8
Chromium as Cr	mg/kg	6	5.4	2.7	1.9
Lead as Pb	mg/kg	9	16.1	8.9	4.8
Nickel as Ni	mg/kg	5	5.2	3.4	1.8

Table 6: performance overview on sample #20685

Element	unit	n	average	2.8 * sd	R(target)
Cadmium as Cd	mg/kg	8	4.8	1.6	1.7
Chromium as Cr	mg/kg	6	5.7	3.0	2.0
Lead as Pb	mg/kg	8	14.8	9.1	4.4
Nickel as Ni	mg/kg	5	5.2	2.9	1.8

Table 7: performance overview on sample #20686

Without further statistical calculations, it can be concluded that there is a good compliance of the group of participating laboratories with the reference target for the elements Arsenic and Cadmium but not a good compliance for Chromium, Lead, Nickel and Iron. See also paragraphs 4.1 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2020 WITH THE PREVIOUS PT

	October 2020	November 2019
Number of reporting laboratories	9	8
Number of test results	65	40
Number of statistical outliers	0	1
Percentage of statistical outliers	0.0%	2.5%

Table 8: 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 2020	November 2019	Target	Conc in mg.kg
Arsenic as As	12%	n.e.	12-13%	5-6
Cadmium as Cd	11-12%	8-9%	12-13%	5-6
Chromium as Cr	18-19%	n.e.	12-13%	5-6
Lead as Pb	20-22%	9-10%	10-11%	15-20
Mercury as Hg	n.e.	10-14%	14-16%	1-2
Nickel as Ni	20-23%	n.e.	12-13%	5-6

Table 9: 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 8 participants the following can be summarized:

- Five participants mentioned that they are ISO/IEC17025 accredited to determine the reported elements.
- Six participants used a sample intake between 0.1 - 0.5 grams and two participants around 10 grams. Presummable amount of intake is dependent on matrix.
- Seven participants used ICP-MS to determine the metal content and one participant used ICP-OES and AAS.

5 DISCUSSION

In this proficiency test the added metals in two different types of cosmetic products were correctly identified. Elements like Cadmium and Arsenic seems to be determined more easily and therefore more precise.

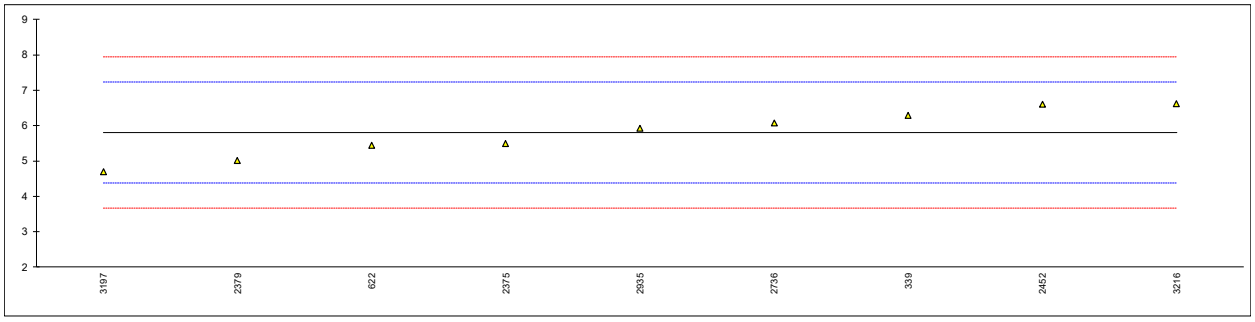
6 CONCLUSION

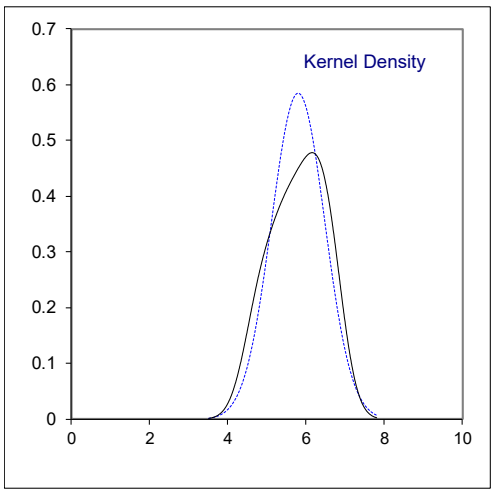
Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of Arsenic as As in Mouthwash, sample #20685; results in mg/kg

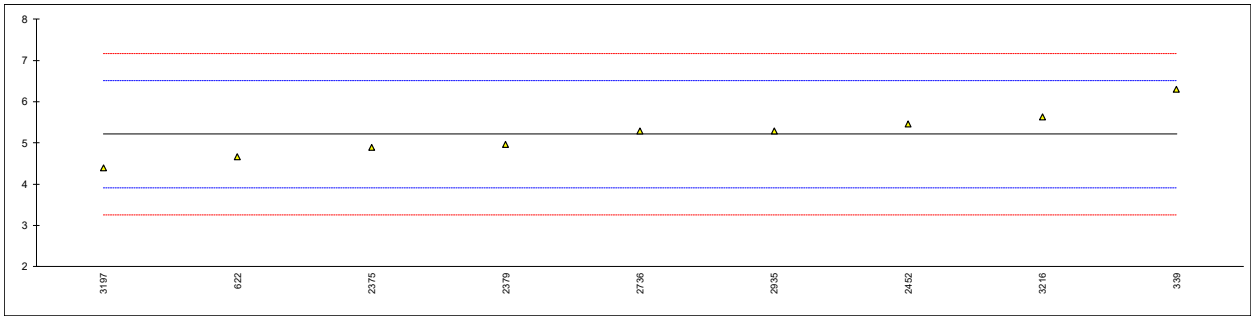
lab	method	value	mark	z(targ)	remarks
339		6.3		0.70	
622	INH-05	5.453		-0.49	
2375	In house	5.5		-0.42	
2379	INH-005	5.01		-1.11	
2452	ISO17276	6.61		1.14	
2736	In house	6.072		0.38	
2935	In house	5.933		0.19	
3197	In house	4.7		-1.54	
3216	In house	6.620		1.15	
	normality	OK			
	n	9			
	outliers	0			
	mean (n)	5.800			
	st.dev. (n)	0.6814	RSD=12%		
	R(calc.)	1.908			
	st.dev.(Horwitz)	0.7122			
	R(Horwitz)	1.994			

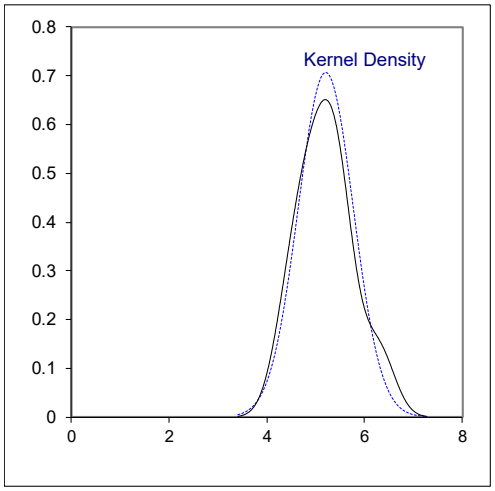




Determination of Cadmium as Cd in Mouthwash, sample #20685; results in mg/kg

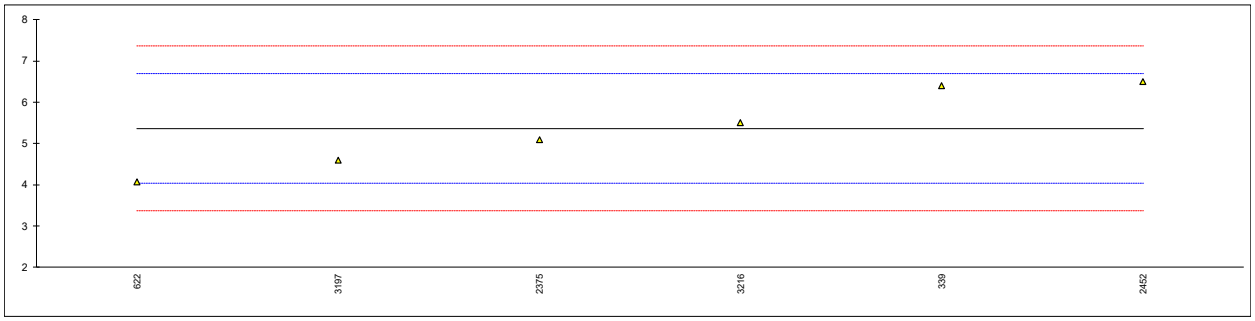
lab	method	value	mark	z(targ)	remarks
339		6.3		1.67	
622	INH-05	4.67		-0.84	
2375	In house	4.9		-0.48	
2379	INH-005	4.97		-0.38	
2452	ISO17276	5.465		0.38	
2736	In house	5.301		0.13	
2935	In house	5.302		0.13	
3197	In house	4.4		-1.25	
3216	In house	5.632		0.64	
	normality	OK			
	n	9			
	outliers	0			
	mean (n)	5.216			
	st.dev. (n)	0.5645	RSD=11%		
	R(calc.)	1.581			
	st.dev.(Horwitz)	0.6508			
	R(Horwitz)	1.822			





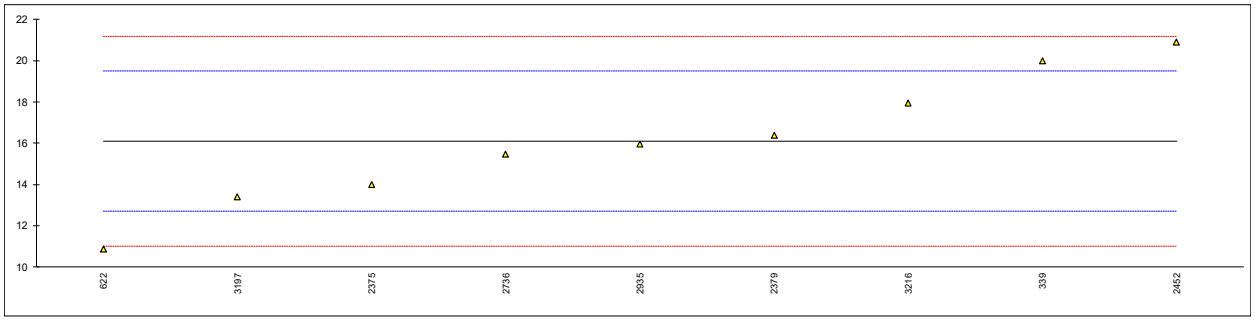
Determination of Chromium as Cr in Mouthwash, sample #20685; results in mg/kg

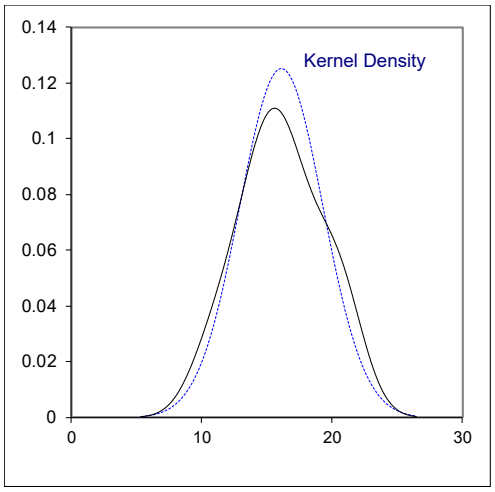
lab	method	value	mark	z(targ)	remarks
339		6.4		1.55	
622	INH-05	4.08		-1.93	
2375	In house	5.1		-0.40	
2379		-----		-----	
2452	ISO17276	6.505		1.71	
2736		-----		-----	
2935		-----		-----	
3197	In house	4.6		-1.15	
3216	In house	5.515		0.22	
	normality	unknown			
	n	6			
	outliers	0			
	mean (n)	5.367			
	st.dev. (n)	0.9695	RSD=18%		
	R(calc.)	2.715			
	st.dev.(Horwitz)	0.6668			
	R(Horwitz)	1.867			



Determination of Lead as Pb in Mouthwash, sample #20685; results in mg/kg

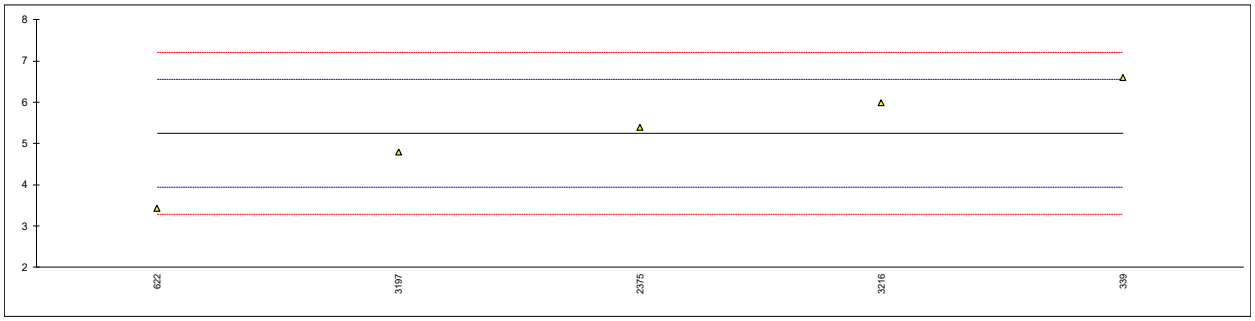
lab	method	value	mark	z(targ)	remarks
339		20		2.29	
622	INH-05	10.89		-3.08	
2375	In house	14.0		-1.24	
2379	INH-005	16.39		0.17	
2452	ISO17276	20.91		2.83	
2736	In house	15.485		-0.37	
2935	In house	15.964		-0.09	
3197	In house	13.4		-1.60	
3216	In house	17.946		1.08	
	normality	OK			
	n	9			
	outliers	0			
	mean (n)	16.109			
	st.dev. (n)	3.1835	RSD=20%		
	R(calc.)	8.914			
	st.dev.(Horwitz)	1.6964			
	R(Horwitz)	4.750			





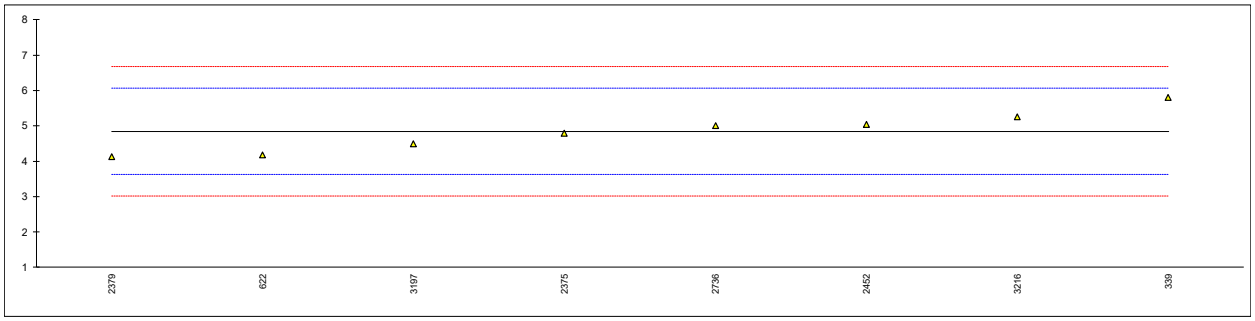
Determination of Nickel as Ni in Mouthwash, sample #20685; results in mg/kg

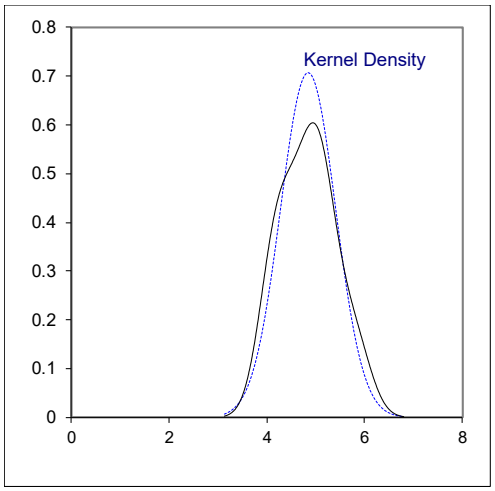
lab	method	value	mark	z(targ)	remarks
339		6.6		2.07	
622	INH-05	3.43		-2.78	
2375	In house	5.4		0.24	
2379		-----		-----	
2452	ISO17276	Not analysed		-----	
2736		-----		-----	
2935		-----		-----	
3197	In house	4.8		-0.68	
3216	In house	6.000		1.15	
	normality	unknown			
	n	5			
	outliers	0			
	mean (n)	5.246			
	st.dev. (n)	1.2168	RSD=23%		
	R(calc.)	3.407			
	st.dev.(Horwitz)	0.6540			
	R(Horwitz)	1.831			



Determination of Cadmium as Cd in Toothpaste, sample #20686; results in mg/kg

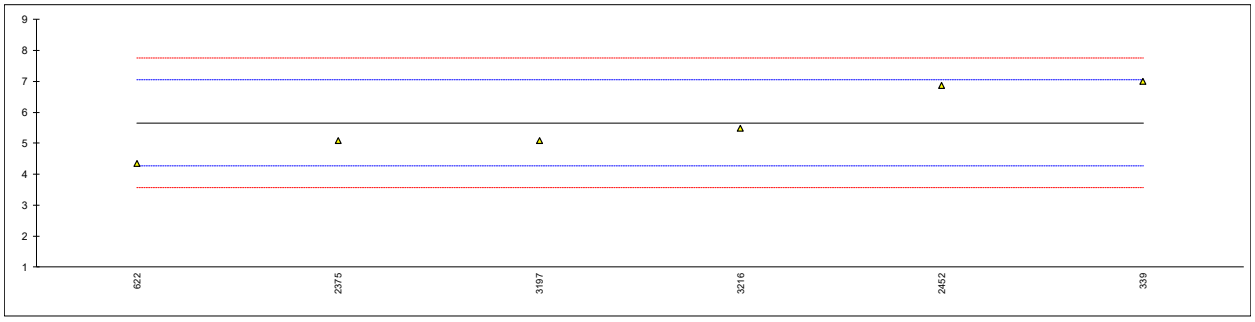
lab	method	value	mark	z(targ)	remarks
339		5.8		1.57	
622	INH-05	4.18		-1.08	
2375	In house	4.8		-0.07	
2379	INH-005	4.13		-1.17	
2452	ISO17276	5.05		0.34	
2736	In house	5.018		0.29	
2935		-----		-----	
3197	In house	4.5		-0.56	
3216	In house	5.263		0.69	
	normality	unknown			
	n	8			
	outliers	0			
	mean (n)	4.843			
	st.dev. (n)	0.5648	RSD=12%		
	R(calc.)	1.581			
	st.dev.(Horwitz)	0.6111			
	R(Horwitz)	1.711			





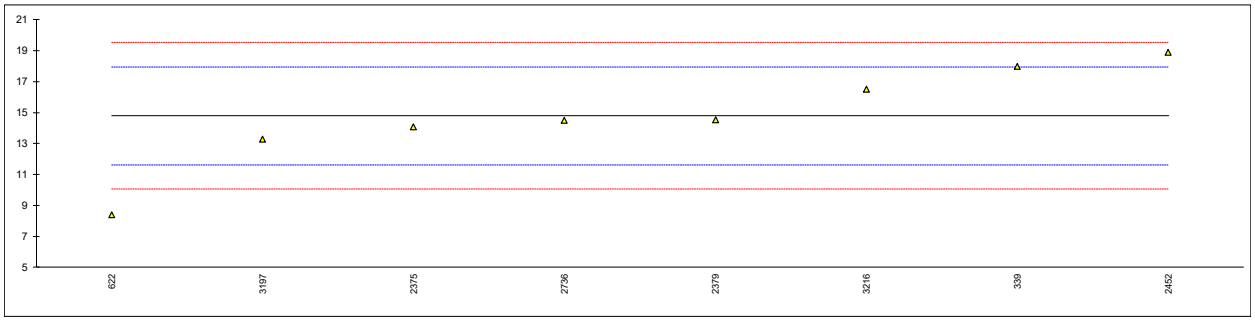
Determination of Chromium as Cr in Toothpaste, sample #20686; results in mg/kg

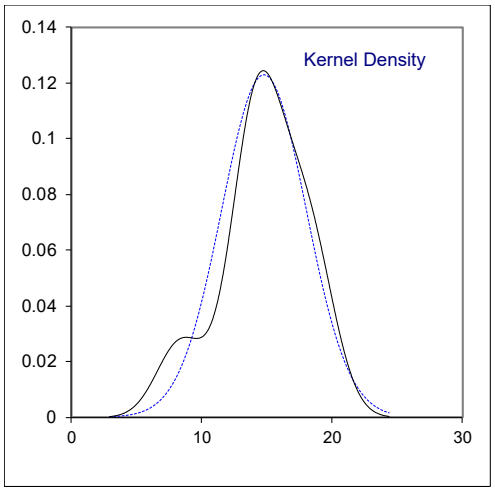
lab	method	value	mark	z(targ)	remarks
339		7.0		1.93	
622	INH-05	4.36		-1.86	
2375	In house	5.1		-0.80	
2379		-----		-----	
2452	ISO17276	6.88		1.76	
2736		-----		-----	
2935		-----		-----	
3197	In house	5.1		-0.80	
3216	In house	5.488		-0.24	
	normality	unknown			
	n	6			
	outliers	0			
	mean (n)	5.655			
	st.dev. (n)	1.0612	RSD=19%		
	R(calc.)	2.971			
	st.dev.(Horwitz)	0.6971			
	R(Horwitz)	1.952			



Determination of Lead as Pb in Toothpaste, sample #20686; results in mg/kg

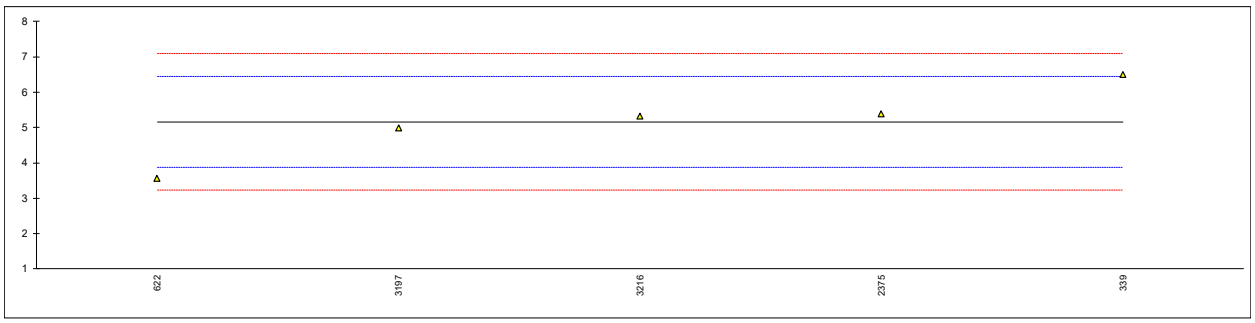
lab	method	value	mark	z(targ)	remarks
339		18		2.04	
622	INH-05	8.41		-4.04	
2375	In house	14.1		-0.43	
2379	INH-005	14.55		-0.15	
2452	ISO17276	18.92		2.62	
2736	In house	14.499		-0.18	
2935		-----		-----	
3197	In house	13.3		-0.94	
3216	In house	16.507		1.09	
	normality	unknown			
	n	8			
	outliers	0			
	mean (n)	14.786			
	st.dev. (n)	3.2481	RSD=22%		
	R(calc.)	9.095			
	st.dev.(Horwitz)	1.5772			
	R(Horwitz)	4.416			





Determination of Nickel as Ni in Toothpaste, sample #20686; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		6.5		2.08	
622	INH-05	3.57		-2.46	
2375	In house	5.4		0.37	
2379		-----		-----	
2452	ISO17276	Not analysed		-----	
2736		-----		-----	
2935		-----		-----	
3197	In house	5.0		-0.25	
3216	In house	5.327		0.26	
	normality	unknown			
	n	5			
	outliers	0			
	mean (n)	5.159			
	st.dev. (n)	1.0529	RSD=20%		
	R(calc.)	2.948			
	st.dev.(Horwitz)	0.6449			
	R(Horwitz)	1.806			



APPENDIX 2

Other reported Elements in sample #20685; results in mg/kg

lab	Hg	Al	Sb	Fe	Zn
339	<0.1	-----	<0.1	-----	-----
622	0.0074	13.43	0.72	14.35	0.89
2375	<0.083	-----	<0.083	-----	-----
2379	-----	-----	-----	-----	-----
2452	0.018	Not analysed	0	Not analysed	Not analysed
2736	<0.100	-----	-----	-----	-----
2935	-----	-----	-----	-----	-----
3197	<0,1	<1	<1	<1	<1
3216	Not detected	0.236	0.159	0.516	Not detected

Other reported Elements in sample #20686; results in mg/kg

lab	As	Hg	Al	Sb	Fe	Zn
339	<0.1	<0.1	-----	<0.1	-----	-----
622	0.126	0.0099	48.44	0.63	28.79	0.56
2375	<0.083	<0.083	-----	<0.083	-----	-----
2379	Not detected	Not detected	-----	-----	-----	-----
2452	0	0	Not analysed	0.038	Not analysed	Not analysed
2736	0.264	<0.114	-----	-----	-----	-----
2935	-----	-----	-----	-----	-----	-----
3197	<1	<0,1	191.8	<1	24.2	<1
3216	Not detected	Not detected	76.055	Not detected	15.463	Not detected

APPENDIX 3

Analytical details

lab	ISO17025 accredited	Sample intake	Technique used	remarks
339	No	0.1 g for elements by ICP-MS 0.06g for Mercury	ICP-MS	Mercury determined by DMA
622	Yes	9 grams	ICP-OES and AAS	ISO/IEC17025 accredited for Pb, Cd, As and Hg, not accredited yet for Cr, Ni, Al, Sb, Fe, Zn.
2375	---	---	---	
2379	No	0.25 grams	ICP-MS	
2452	Yes	100mg	ICP-MS	
2736	Yes	0.25g	ICP-MS	
2935	Yes	10 mL	ICP-MS	
3197	Yes	0,2 g	ICP-MS	
3216	No	0.5 g	ICP-MS	

APPENDIX 4

Number of participants per country

1 lab in	FRANCE
1 lab in	INDONESIA
1 lab in	P.R. of CHINA
1 lab in	SPAIN
1 lab in	THAILAND
1 lab in	TUNISIA
2 labs in	TURKEY
1 lab in	U.S.A.
1 lab in	FRANCE

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

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