Results of Proficiency Test
Preservatives in Skin Care products
(CMIT/MIT & Parabens)
November 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Preservatives may be used in cosmetics to prevent the growth of harmful bacteria and mold. Parabens, Methylisothiazolinone (MIT) and Chloromethylisothiazolinone (CMIT) are widely used as preservatives in liquid cosmetic and personal care products.
MIT and CMIT could be allergenic and cytotoxic, while Parabens are linked to hormonal disrupsion. The mixture of MIT and CMIT as a preservative in rinse-off cosmetic products was authorized in cosmetics products through Annex V of Regulation (EC) No 1223/2009 ("Cosmetics Regulation") at a maximum concentration of 0.0015% (15 mg/kg) in a 3:1 mixture of CMIT : MIT since 16 July 2015.
Parabens are also regulated in cosmetic products through Annex V of Regulation (EC) No 1223/2009 ("Cosmetics Regulation") at a maximum concentration of 0.4% for single ester and 0.8% for mixtures of esters since 16 July 2015. For Phenoxyethanol maximum concentration of 1%M/M is listed.
No reference materials for CMIT or MIT and for individual Parabens in cosmetics are available to optimise the determination of CMIT/MIT or Parabens. As an alternative, participation in a proficiency test may enable the laboratories to check their performance and thus to increase this comparability.

Since 2018 the Institute for Interlaboratory Studies (iis) organizes a proficiency test for the determination of MIT (2-Methyl-4-Isothiazolin-3-one) and CMIT (5-Chloro-2-Methyl-4-Isothiazolin-3-one) in Body Lotion and for Parabens and other preservatives in Body Milk. It was decided to continue the proficiency test on preservatives in skin care products during the annual testing program 2021/2022.

In this interlaboratory study 18 laboratories in 13 different countries registered for participation. See appendix 3 for the number of participants per country. In this report the results of the Preservatives 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 one sample of Body Lotion in a 10 mL bottle labelled #21785, which was artificially fortified with CMIT and MIT and one sample of Body Milk in a 10 mL bottle labelled #21786, which was artificially fortified with Parabens.
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

A body lotion was purchased from a local supermarket and was artificially fortified with CMIT/MIT. After homogenization 35 PE bottles of 10mL were filled with body lotion and labelled #21785.
The homogeneity of the subsamples was checked by determination of CMIT and MIT using an in-house test method on five stratified randomly selected subsamples.

	CMIT in mg/kg	MIT in mg/kg
sample #21785-1	20.316	12.265
sample #21785-2	19.019	11.157
sample #21785-3	20.481	11.356
sample #21785-4	19.785	10.838
sample #21785-5	20.895	11.121

Table 1: homogeneity test results of subsamples #21785

From the above test results the relative standard deviations (RSD) were calculated and compared with 0.3 times the corresponding average relative standard deviation found in previous proficiency tests in agreement with the procedure of ISO13528, Annex B2, in the next table.

	CMIT	MIT
RSD% (observed)	3.6	4.8
reference method	previous iis PTs	previous iis PTs
0.3 x RSD% (ref. method)	3.9	4.8

Table 2: evaluation of the relative standard deviations of subsamples #21785

The calculated relative standard deviations are in agreement with 0.3 times the corresponding average relative standard deviation of the previous iis PTs. Therefore, homogeneity of the subsamples was assumed.

A body milk was purchased from a local supermarket and was artificially fortified with the preservatives: Methylparaben, Propylparaben, Isobutylparaben and Butylparaben. After homogenization 35 PE botlles of 10mL were filled with body milk and labelled #21786.
The homogeneity of the subsamples was checked by determination of Propylparaben and Isobutylparaben by using an in-house test method on four stratified randomly selected subsamples.

	Propylparaben in mg/kg	Isobutylparaben in mg/kg
sample #21786-1	128.7	102.8
sample #21786-2	126.8	103.8
sample #21786-3	128.4	105.5
sample #21786-4	133.4	98.8

Table 3: homogeneity test results of subsamples #21786

From the above test results the relative standard deviations were calculated and compared with 0.3 times the relative standard deviation from previous PTs in agreement with the procedure of ISO13528, Annex B2, in the next table.

	Propylparaben	Isobutylparaben
RSD% (observed)	2.7	2.8
reference method	previous iis PTs	previous iis PTs
0.3 x RSD% (ref. method)	2.6	4.2

Table 4: evaluation of the relative standard deviation of subsamples #21786

The calculated relative standard deviations are in agreement with 0.3 times the corresponding average relative standard deviation from previous iis PTs. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample body lotion labelled #21785 and one sample body milk labelled #21786 were sent on November 3, 2021.

2.5 ANALYZES

The participants were requested to determine on sample #21785 the concentrations of CMIT (5-Chloro-2-Methyl-4-Isothiazolin-3-one) and MIT (2-Methyl-4-Isothiazolin-3-one).
On sample #21786 was requested to determine the concentrations of Methylparaben as ester, Ethylparaben as ester, Propylparaben as ester, Isobutylparaben as ester, Butylparaben as ester, Phenoxyethanol, Formaldehyde and Benzoic acid. applying the analytical procedure that is routinely used in the laboratory.
It was also requested to report if the laboratory was accredited for this determination and to report the amount of sample intake.

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 to 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 evaluation.

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 appendix 1 of this report. The laboratories are presented by their code numbers.

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

3.1 STATISTICS

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

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a 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 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. The kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

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

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

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

The z-scores were calculated according to:

$z_{(target)}$ = (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

During the execution of this proficiency test no problems were encountered with the dispatch of the samples. Two participants did not report any test results. All other participants reported test results before the final reporting date. Not all participants wee able to report all test results requested.
In total 16 participants reported 95 numerical test results. Observed were 7 outlying test results, which is 7.4% of the reported numerical test results. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample and per component. 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 4.

Unfortunately, a suitable reference test method providing the precision data is not available for the determination of preservatives in skin care products. Therefore, the calculated reproducibilities were compared against the estimated reproducibility calculated with the Horwitz equation.

sample #21785

CMIT: The determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation.

MIT: The determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation.

sample #21786

Methylparaben: 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.

Ethylparaben: This determination may not be problematic. All reporting participants agreed on a concentration near or below the limit of detection. Therefore, no z-scores are calculated.

Propylparaben: 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.

Isobutylparaben: 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.

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

Phenoxyethanol: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Formaldehyde: This determination may not be problematic. All reporting participants agreed on a concentration near or below the limit of detection. Therefore, no z-scores are calculated.

Benzoic acid: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the estimated target reproducibility calculated with 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 estimated using the Horwitz equation are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(target)
CMIT	mg/kg	12	20.4	6.0	5.8
MIT	mg/kg	11	7.4	2.6	2.5

Table 5: reproducibilities of tests on sample #21785

Component	unit	n	average	2.8 * sd	R(target)
Methylparaben	mg/kg	11	515	91	90
Ethylparaben	mg/kg	9	<30	n.e.	n.e.
Propylparaben	mg/kg	13	149	26	31
Isobutylparaben	mg/kg	10	111	20	24
Butylparaben	mg/kg	10	75	29	18
Phenoxyethanol	mg/kg	12	6084	713	734
Formaldehyde	mg/kg	5	<30	n.e.	n.e.
Benzoic acid	mg/kg	8	891	173	144

Table 6: reproducibilities of tests on sample #21786

Without further statistical calculations it can be concluded that for a number of components there is a good compliance of the group of participating laboratories with the target reproducibility. See also the discussion in paragraph 4.1.

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

The uncertainties observed in the test results of the determination of CMIT/MIT and Parabens in Skin Care products over the years are listed in the next table.

Component	November 2021	November 2020	November 2019	November 2018
CMIT	11%	8%	20%	10%
MIT	12%	10%	19%	19%
Methylparaben	6%	6%	13%	n.e.
Ethylparaben	n.e.	7%	11%	n.e.
Propylparaben	6%	5%	12%	n.e.
Isobutylparaben	7%	14%	14%	n.e.
Butylparaben	14%	3%	7%	n.e.
Phenoxyethanol	4%	8%	12%	n.e.
Formaldehyde	n.e.	n.e.	n.e.	n.e.
Benzoic acid	7%	n.e.	n.e.	n.e.

Table 7: development of the uncertainties over the years

It is observed that the variation for a few preservatives measured in the PT of this year did improve compared to the previous years. Other preservatives did not improve or were even worse, especially Butylparaben.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested. The reported analytical details are given in appendix 2. Based on the answers given by the participants the following can be summarized:

For the determination of CMIT/MIT, ten participants mentioned that they are accredited for this determination. Eight participants used 1 gram or less for sample intake and four others used an intake of 2 grams or more.

For the determination of Parabens, ten participants mentioned that they are accredited for this determination. Eight participants used 1 gram or less for sample intake and three others used an intake of 2 grams or more.

5 DISCUSSION

It is observed that all of the reporting laboratories, except one, would judge sample #21785 in the same way and reject the sample for too much CMIT/MIT present in accordance with the Annex V of Regulation (EC) No 1223/2009 ("Cosmetics Regulation") limit of 15 mg/kg.
For sample #21786, it is observed that all of the reporting laboratories would judge the sample the same and would accept the sample for too high lever of Parabens and Phenoxyethanol in accordance with the Annex V of Regulation (EC) No 1223/2009 ("Cosmetics Regulation") limit of 0.4 %M/M (single ester) or 0.8%M/M (mixture of esters) for Parabens and 1%M/M for Phenoxyethanol

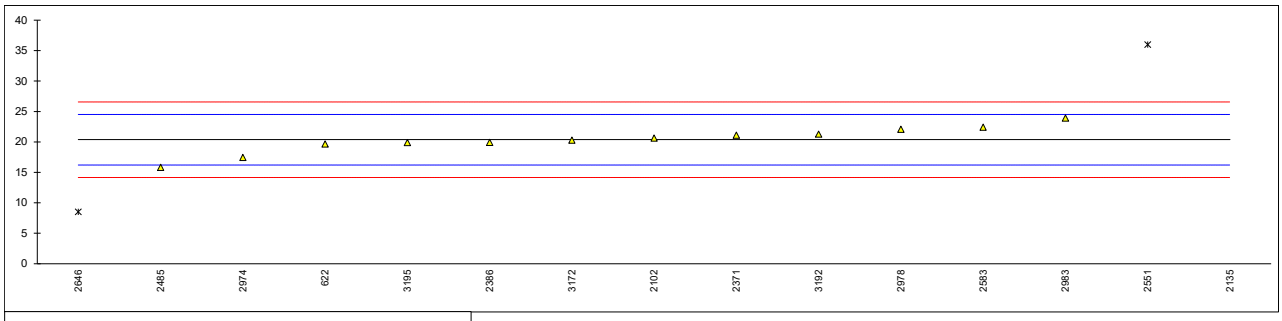
6 CONCLUSION

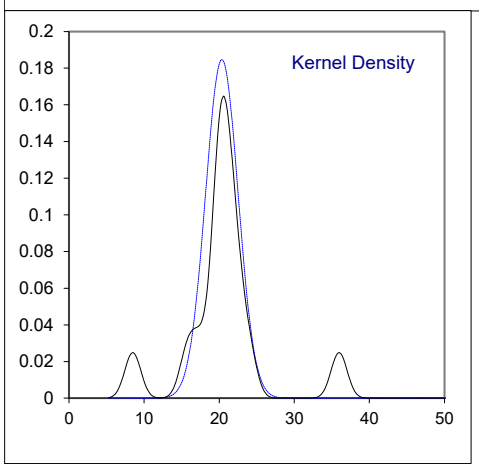
The observed variation in this interlaboratory study may not be caused by just one critical point in the analysis. Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

APPENDIX 1

Determination of CMIT (5-Chloro-2-Methyl-4-Isothiazolin-3-one) CAS No. 26172-55-4 in sample #21785; results in mg/kg

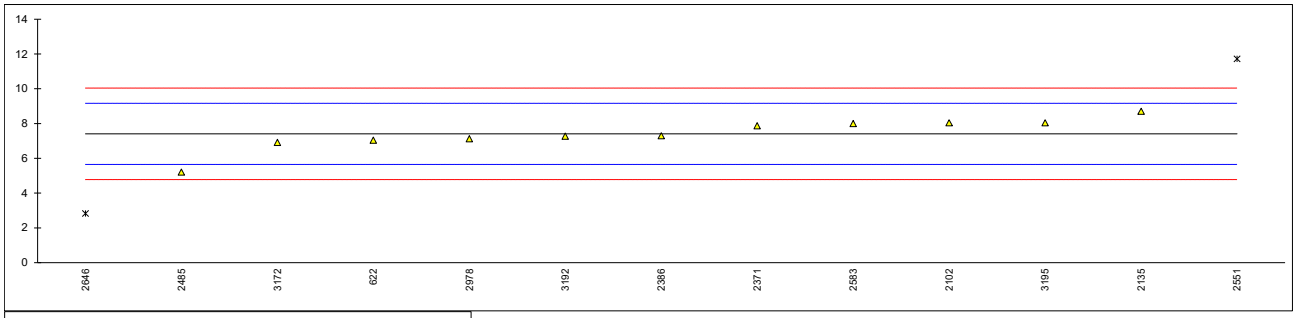
lab	method	value	mark	z(targ)	remarks
339		-----		-----	
622	In house	19.658		-0.34	
2102	In house	20.629		0.13	
2135	In house	85.2	G(0.01)	31.31	
2146		-----		-----	
2371	In house	21.1		0.35	
2375		-----		-----	
2386	In house	19.93		-0.21	
2485	In house	15.8		-2.21	
2551	In house	35.98	C,G(0.05)	7.54	First reported below limit of Quantification
2583	In house	22.4		0.98	
2646	In house	8.50881	C,G(0.01)	-5.73	First reported 2.82914
2974	In house	17.46		-1.40	
2978	In house	22.063		0.82	
2983	In house	23.920		1.72	
3172	In house	20.281		-0.04	
3192	In house	21.281		0.44	
3195	In house	19.8967		-0.23	
	normality	OK			
	n	12			
	outliers	3			
	mean (n)	20.3682			
	st.dev. (n)	2.15965	RSD = 11%		
	R(calc.)	6.0470			
	st.dev.(Horwitz)	2.07041			
	R(Horwitz)	5.7971			

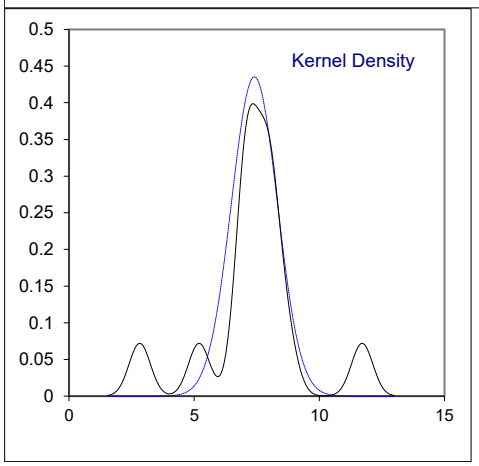




Determination of MIT (2-Methyl-4-Isothiazolin-3-one) CAS No. 2682-20-4 in sample #21785; results in mg/kg

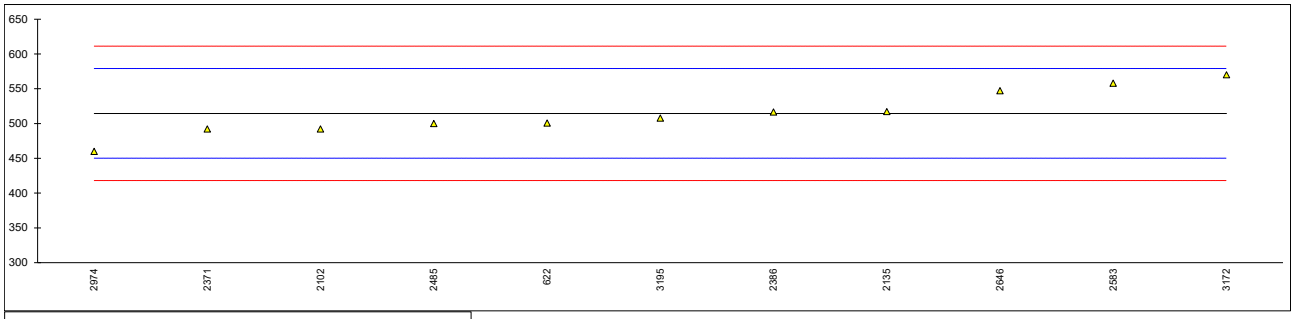
lab	method	value	mark	z(targ)	remarks
339		-----		-----	
622	In house	7.036		-0.42	
2102	In house	8.037		0.72	
2135	In house	8.7		1.47	
2146		-----		-----	
2371	In house	7.87		0.53	
2375		-----		-----	
2386	In house	7.29		-0.13	
2485	In house	5.2		-2.52	
2551	In house	11.71	C,G(0.01)	4.91	First reportedbelow limit of quantification
2583	In house	8.0		0.68	
2646	In house	2.82914	C,G(0.01)	-5.22	First reported 8.50881
2974	In house	<5		-----	
2978	In house	7.128		-0.32	
2983		-----		-----	
3172	In house	6.916		-0.56	
3192	In house	7.265		-0.16	
3195	In house	8.0384		0.72	
	normality	not OK			
	n	11			
	outliers	2			
	mean (n)	7.4073			
	st.dev. (n)	0.91623	RSD = 12%		
	R(calc.)	2.5654			
	st.dev.(Horwitz)	0.87677			
	R(Horwitz)	2.4549			

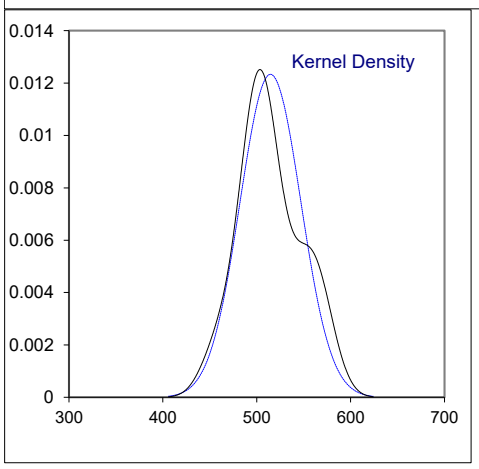




Determination of Methylparaben as ester CAS No. 99-76-3 in sample #21786; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
339		-----		-----	
622	ACM INO 04	500.458		-0.44	
2102	In house	492		-0.70	
2135	In house	517.0		0.08	
2146		-----		-----	
2371	In house	492		-0.70	
2375		-----		-----	
2386	In house	516.5		0.06	
2485	In house	500		-0.45	
2551		-----		-----	
2583	In house	557.94		1.35	
2646	In house	547		1.01	
2974	In house	460.00		-1.70	
2978		-----		-----	
2983		-----		-----	
3172	In house	569.877		1.72	
3192		-----		-----	
3195	In house	507.625		-0.22	
	normality	OK			
	n	11			
	outliers	0			
	mean (n)	514.582			
	st.dev. (n)	32.3381	RSD = 6%		
	R(calc.)	90.547			
	st.dev.(Horwitz)	32.1707			
	R(Horwitz)	90.078			



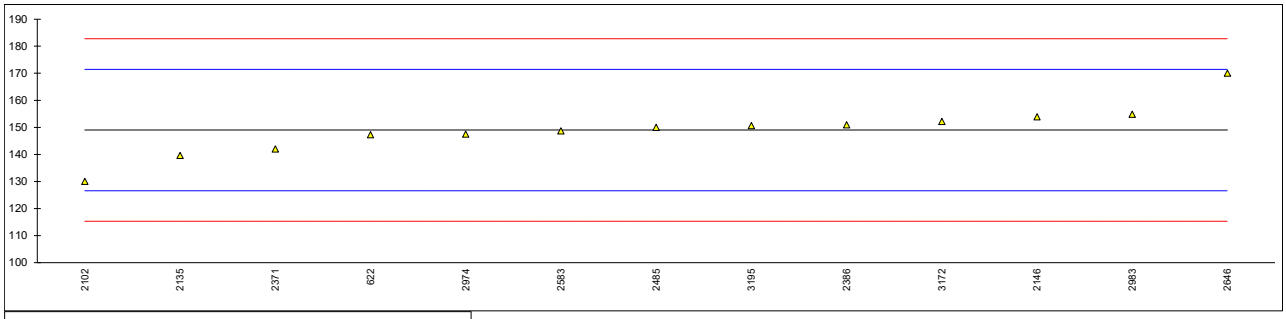


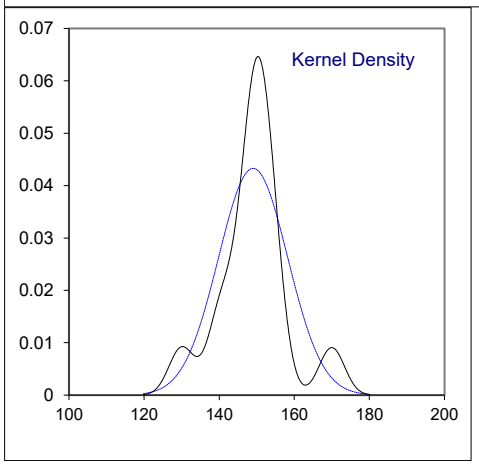
Determination of Ethylparaben as ester CAS No. 120-47-8 in sample #21786; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
622	ACM INO 04	not detected		-----	
2102	In house	not detected		-----	
2135		-----		-----	
2146	In house	not detected		-----	
2371	In house	<5		-----	
2375		-----		-----	
2386	In house	<5		-----	
2485	In house	not detected		-----	
2551		-----		-----	
2583	In house	not detected		-----	
2646	In house	not detected		-----	
2974	In house	<30		-----	
2978		-----		-----	
2983		-----		-----	
3172		-----		-----	
3192		-----		-----	
3195		-----		-----	
	n	9			
	mean (n)	<30			

Determination of Propylparaben as ester CAS No. 94-13-3 in sample #21786; results in mg/kg

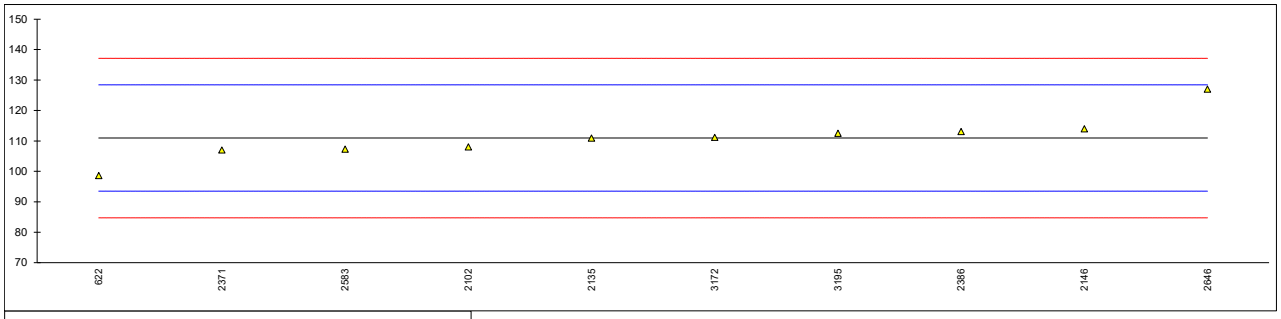
lab	method	value	mark	z(targ)	remarks
339		-----		-----	
622	ACM INO 04	147.250		-0.16	
2102	In house	130		-1.70	
2135	In house	139.6		-0.84	
2146	In house	153.93		0.44	
2371	In house	142		-0.63	
2375		-----		-----	
2386	In house	150.9		0.17	
2485	In house	150	C	0.09	First reported 200
2551		-----		-----	
2583	In house	148.66		-0.03	
2646	In house	170		1.87	
2974	In house	147.50	C	-0.14	First reported 121
2978		-----		-----	
2983	In house	154.856		0.52	
3172	In house	152.145		0.28	
3192		-----		-----	
3195	In house	150.635		0.14	
	normality	not OK			
	n	13			
	outliers	0			
	mean (n)	149.0366	RSD = 6%		
	st.dev. (n)	9.22200			
	R(calc.)	25.8216			
	st.dev.(Horwitz)	11.22793			
	R(Horwitz)	31.4382			

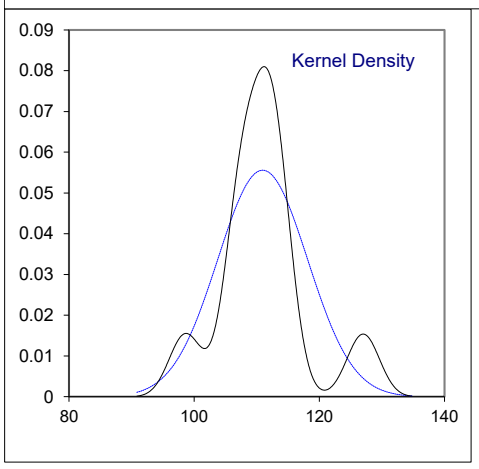




Determination of Isobutylparaben as ester CAS No. 4247-02-3 in sample #21786; results in mg/kg

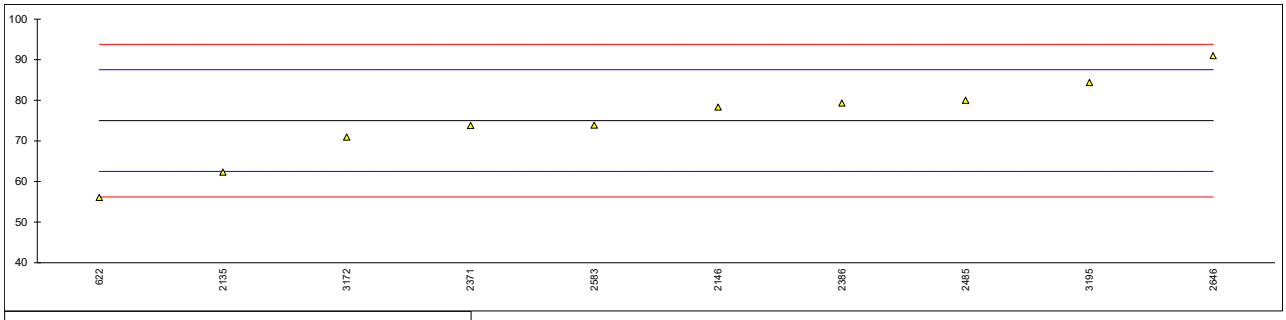
lab	method	value	mark	z(targ)	remarks
339		-----		-----	
622	ACM INO 04	98.618		-1.41	
2102	In house	108		-0.34	
2135	In house	110.9		-0.01	
2146	In house	113.96		0.34	
2371	In house	107		-0.45	
2375		-----		-----	
2386	In house	113.1		0.25	
2485		-----		-----	
2551		-----		-----	
2583	In house	107.27		-0.42	
2646	In house	127		1.84	
2974		-----		-----	
2978		-----		-----	
2983		-----		-----	
3172	In house	111.140		0.02	
3192		-----		-----	
3195	In house	112.485		0.18	
	normality	not OK			
	n	10			
	outliers	0			
	mean (n)	110.9473			
	st.dev. (n)	7.17717	RSD = 7%		
	R(calc.)	20.0961			
	st.dev.(Horwitz)	8.73808			
	R(Horwitz)	24.4666			

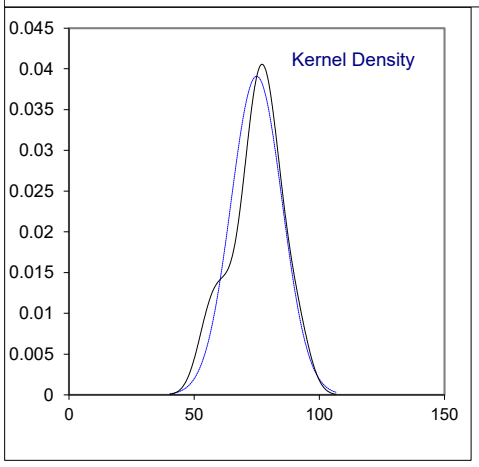




Determination of Butylparaben as ester CAS No. 94-26-8 in sample #21786; results in mg/kg

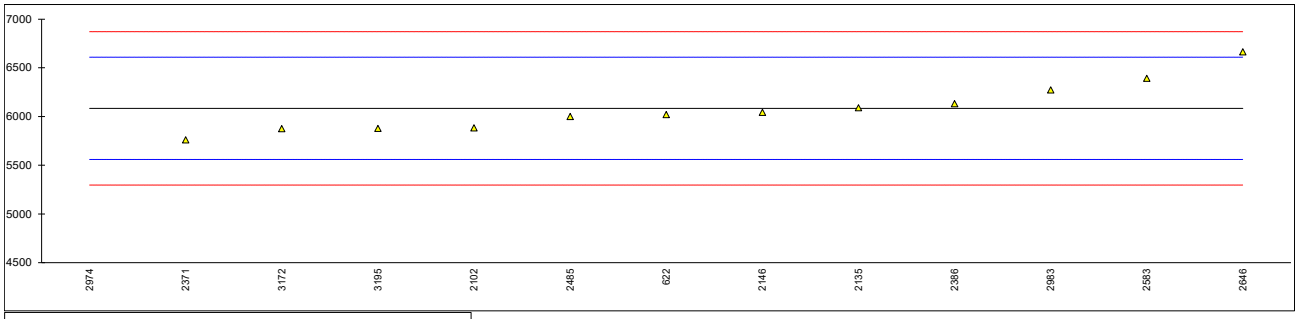
lab	method	value	mark	z(targ)	remarks
339		-----		-----	
622	ACM INO 04	56.060		-3.02	
2102	In house	<125	C	-----	First reported 51
2135	In house	62.3		-2.03	
2146	In house	78.348		0.53	
2371	In house	73.8		-0.19	
2375		-----		-----	
2386	In house	79.3		0.69	
2485	In house	80	C	0.80	First reported 100
2551		-----		-----	
2583	In house	73.86		-0.18	
2646	In house	91		2.55	
2974	In house	<30		<-7.18	Possibly a false negative test result?
2978		-----		-----	
2983		-----		-----	
3172	In house	70.926		-0.65	
3192		-----		-----	
3195	In house	84.38		1.50	
	normality	OK			
	n	10			
	outliers	0			
	mean (n)	74.9974			
	st.dev. (n)	10.20911	RSD = 14%		
	R(calc.)	28.5855			
	st.dev.(Horwitz)	6.26533			
	R(Horwitz)	17.5429			

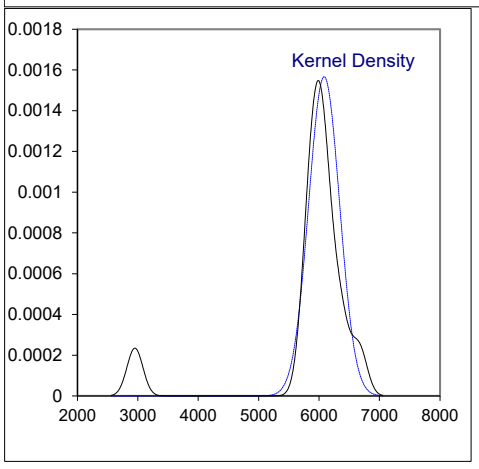




Determination of Phenoxyethanol CAS No. 122-99-6 in sample #21786; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
622	ACM INO 04	6020.635		-0.24	
2102	In house	5884		-0.76	
2135	In house	6090		0.02	
2146	In house	6043.3		-0.16	
2371	In house	5760		-1.24	
2375		-----		-----	
2386	In house	6131.2		0.18	
2485	In house	6000		-0.32	
2551		-----		-----	
2583	In house	6391.5		1.17	
2646	In house	6664		2.21	
2974	In house	2953	C,G(0.01)	-11.94	First reported 2900
2978		-----		-----	
2983	In house	6272.507		0.72	
3172	In house	5874.57		-0.80	
3192		-----		-----	
3195	In house	5877.7		-0.79	
	normality	not OK			
	n	12			
	outliers	1			
	mean (n)	6084.118			
	st.dev. (n)	254.5726	RSD = 4%		
	R(calc.)	712.803			
	st.dev.(Horwitz)	262.2642			
	R(Horwitz)	734.340			



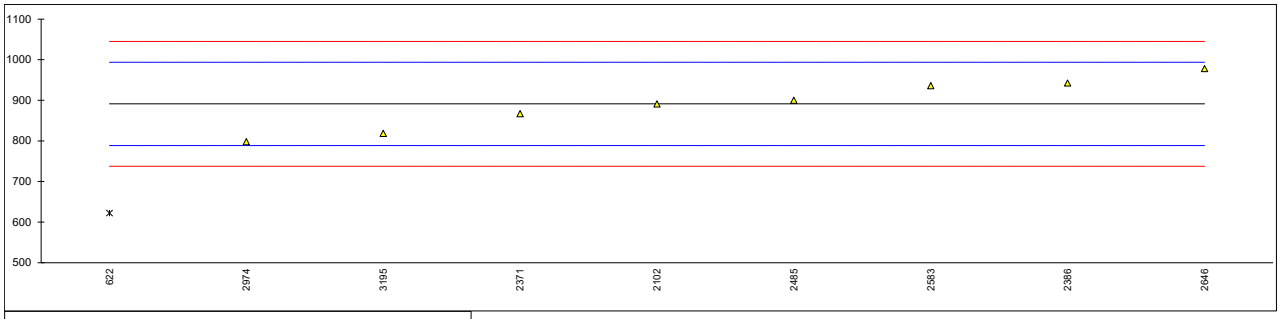


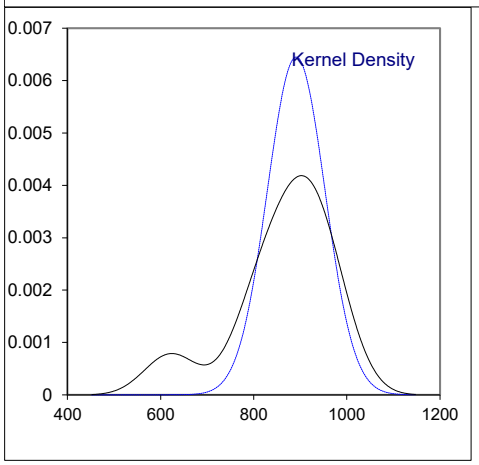
Determination of Formaldehyde in sample #21786; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
622	ACM INO 04	not detected		-----	
2102		-----		-----	
2135		-----		-----	
2146		-----		-----	
2371	In house	8.72		-----	
2375		-----		-----	
2386	In house	<5		-----	
2485		-----		-----	
2551		-----		-----	
2583		-----		-----	
2646		-----		-----	
2974	In house	<30		-----	
2978		-----		-----	
2983		-----		-----	
3172	In house	< 5		-----	
3192		-----		-----	
3195		-----		-----	
	n	5			
	mean (n)	<30			

Determination of Benzoic acid CAS No. 65-85-0 in sample #21786; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
622	ACM INO 04	622.006	G(0.05)	-5.25	
2102	In house	891		-0.01	
2135		-----		-----	
2146		-----		-----	
2371	In house	867		-0.47	
2375		-----		-----	
2386	In house	942.3		0.99	
2485	In house	900		0.17	
2551		-----		-----	
2583	In house	935.73		0.87	
2646	In house	978		1.69	
2974	In house	798.00		-1.82	
2978		-----		-----	
2983		-----		-----	
3172		-----		-----	
3192		-----		-----	
3195	In house	818.38		-1.42	
	normality	OK			
	n	8			
	outliers	1			
	mean (n)	891.301			
	st.dev. (n)	61.8528	RSD = 7%		
	R(calc.)	173.188			
	st.dev.(Horwitz)	51.3005			
	R(Horwitz)	143.641			





APPENDIX 2

Analytical details for sample #21785

lab	Accredited acc ISO1725	Intake amount (g)
339	---	
622	Yes	
2102	No	0.2 gram
2135	Yes	0,5
2146	---	
2371	Yes	5 gram
2375	---	
2386	Yes	2
2485	Yes	1 g
2551	No	1 g in 25 ml
2583	---	
2646	Yes	2,5032g / 2,5064g
2974	Yes	1g
2978	No	1.0 gram
2983	Yes	0.8g
3172	No	
3192	Yes	0,2 g
3195	Yes	6g

Analytical details for sample #21786

lab	Accredited acc ISO1725	Intake amount (g)
339	---	
622	Yes	
2102	Yes	1 gram
2135	Yes	0,5
2146	No	1 g
2371	Yes	8 gram
2375	---	
2386	Yes	0.5
2485	Yes	0.9 g
2551	---	
2583	Yes	2 g
2646	No	0,5175g / 0,5123g
2974	Yes	0.5
2978	---	
2983	Yes	0.8g
3172	No	
3192	---	
3195	Yes	6 g

APPENDIX 3

Number of participants per country

1 lab in FINLAND
1 lab in FRANCE
5 labs in GERMANY
1 lab in INDONESIA
1 lab in ITALY
2 labs in P.R. of CHINA
1 lab in SAUDI ARABIA
1 lab in SLOVENIA
1 lab in SWITZERLAND
1 lab in TAIWAN
1 lab in THE NETHERLANDS
1 lab in TURKEY
1 lab in UNITED ARAB EMIRATES

APPENDIX 4

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
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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