Results of Proficiency Test Preservatives in Skin Care products (CMIT/MIT & Parabens) November 2020

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 Methylchloroisothiazolinone (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 (RMs) 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 proficiency tests 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 in Body Milk. It was decided to continue these proficiency tests on preservatives in skin care products during the annual testing program 2020/2021.
In the interlaboratory study on CMIT/MIT in Body Lotion 11 laboratories in 9 different countries registered for participation and in the interlaboratory study on Parabens in Body Milk 12 laboratories in 11 countries registered for participation. In total 16 laboratories in 12 countries registered in the proficiency test on preservatives in skin care products. See appendix 3 for the number of participants per country.
In this report the results of the proficiency test on preservatives in skin care products 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 depending on the registration: one sample of Body Lotion of 10 grams and labelled #20730, which was artificially fortified with CMIT and MIT and/or one sample of Body Milk of 10 grams and labelled #20735, which was artificially fortified with a number of 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 (iis) 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 a CMIT/MIT standard. After homogenization 26 PE botlles of 15mL were filled with approximately 10 grams of body lotion and labelled #20730.
The homogeneity of the subsamples was checked by determination of CMIT using an in-house test method on five stratified randomly selected subsamples.

	CMIT in mg/kg
sample #20730-1	20.820
sample #20730-2	20.952
sample #20730-3	21.074
sample #20730-4	20.929
sample #20730-5	21.176

Table 1: homogeneity test results of subsamples #20730

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

	CMIT in mg/kg
r (observed)	0.385
reference method	Horwitz
0.3 x R (reference method)	1.784

Table 2: evaluation of the repeatability of subsamples #20730

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

A body milk was purchased from a supermarket and was artificially fortified with the preservatives: Methylparaben, Ethylparaben, Propylparaben, Isobutylparaben, Butylparaben and Phenoxyethanol. After homogenization 30 PE botlles of 15mL were filled with approximately 10 grams of body milk and labelled #20735.
The homogeneity of the subsamples was checked by determination of Methylparaben and Phenoxyethanol by using an in-house test method on five stratified randomly selected subsamples.

	Methylparaben in mg/kg	Phenoxyethanol in mg/kg
sample #20735-1	392.6	4859
sample #20735-2	392.4	4863
sample #20735-3	392.3	4912
sample #20735-4	392.4	4890
sample #20735-5	393.4	4926

Table 3: homogeneity test results of subsamples #20735

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

	Methylparaben in mg/kg	Phenoxyethanol in mg/kg
r (observed)	1.3	82
reference method	Horwitz	Horwitz
0.3 x R (reference method)	21.5	183

Table 4: evaluation of the repeatabilities of subsamples #20735

The calculated repeatabilities are in agreement with 0.3 times the reproducibilities of the reference method. Therefore, homogeneity of the subsamples was assumed.

Dependent on the registration of the participant the appropriate set of PT samples was dispatched on November 4, 2020.

2.5 ANALYSES

The participants were requested to determine on sample #20730 the concentrations of CMIT (5-Chloro-2-methyl-4-isothiazolin-3-one) and MIT (2-Methyl-4-isothiazolin-3-one) and on sample #20735 to determine the concentrations of Methylparaben, Ethylparaben, Propylparaben, Isobutylparaben, Butylparaben and Phenoxyethanol 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 test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

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

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a dataset does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to 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 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 was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

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

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. 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. All participants reported test results before the final reporting date. Thus 16 laboratories reported in total 82 numerical test results. Eight outlying test results were observed, which is 9.8% of the reported numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the 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 in appendix 1 together with the original data. The abbreviations used in these tables are explained in appendix 3.

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.

#20730

CMIT: The determination of this component was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

MIT: The determination of this component was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

#20735

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

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

Propylparaben: This 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.

Isopropylparaben: 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.

Butylparaben: This 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.

Phenoxyethanol: 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 estimated target reproducibility and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from the reference method (in casu Horwitz Equation) are compared in the next tables.

Component	unit	n	average	2.8 * sd	R(target)
CMIT	mg/kg	10	22.6	4.9	6.3
MIT	mg/kg	10	7.9	2.2	2.6

Table 5: reproducibilities of tests on sample #20730

Component	unit	n	average	2.8 * sd	R(target)
Methylparaben	mg/kg	10	487	76	86
Ethylparaben	mg/kg	10	182	36	53
Propylparaben	mg/kg	8	90	14	20
Isobutylparaben	mg/kg	9	88	35	20
Butylparaben	mg/kg	8	108	8	24
Phenoxyethanol	mg/kg	9	7607	1624	888

Table 6: reproducibilities of tests on sample #20735

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 2020 WITH PREVIOUS PT S

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

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

Table 7: comparison of uncertainties in iis proficiency tests.

It is observed that the variation of preservatives measured in the PTs of this year did improve compared to the previous year.

4.4 EVALUATION 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 CMIT/MIT, six of the ten reporting participants mentioned that they are accredited for this determination.

For the determination Parabens, nine of the eleven reporting participants mentioned that they are accredited for this determination.
For the determination CMIT/MIT, five of the ten reporting participants used 1 gram for intake and three others used an intake of less 1 gram. One participant did not report the intake used.
For the determination Parabens, four of the eleven reporting participants used 1 gram for intake and four others used an intake of less 1 gram. Three participants did not report the intake used.

5 DISCUSSION

It is observed that all of the reporting laboratories would judge sample #20730 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 #20735, it is observed that all of the reporting laboratories would judge the sample the same and would accept the sample for not exceeding Parabens and Phenoxyethanol present 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

One of the participants mentioned that it was not clear if the test results should be reported as ester or as acid. In the next PT iis will add this question to the PT. For next PT eventually deviations in the reported test results could be possibly explained due to this difference.

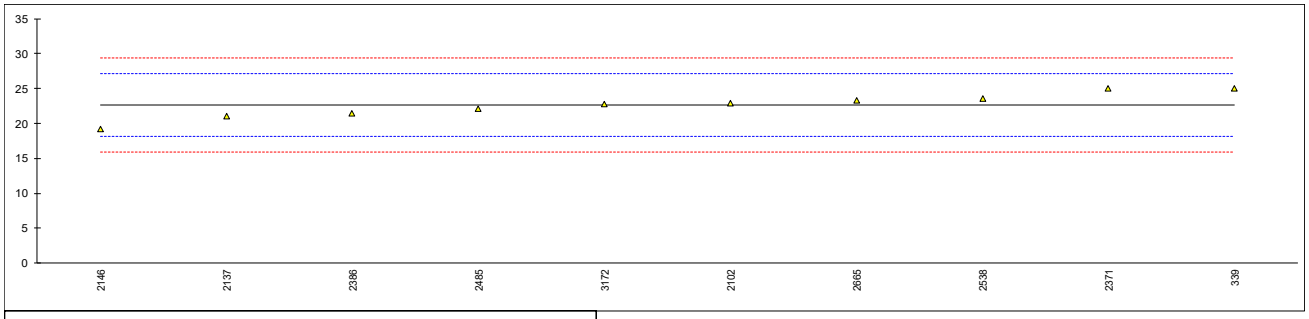
6 CONCLUSION

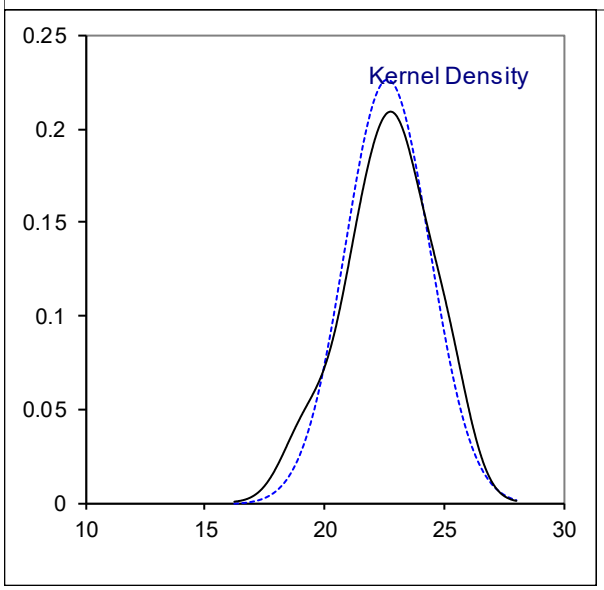
The observed variation in this interlaboratory study may not be caused by just one critical point in the analysis. Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this 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 #20730; results in mg/kg

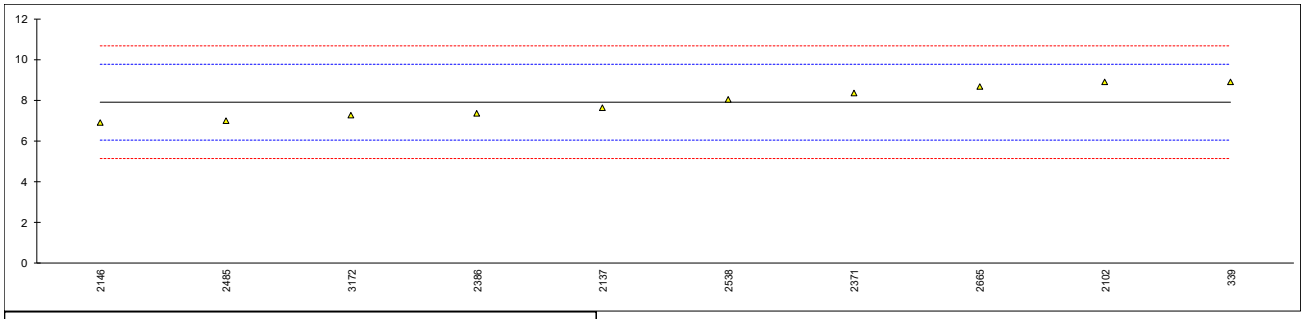
lab	method	value	mark	z(targ)	remarks
339	In house	25.0	C	1.05	Reported 8.90 (possibly switched CMIT and MIT test results)
2102	In house	22.934		0.14	
2137	In house	21.097		-0.68	
2146	In house	19.241		-1.50	
2371	In house	24.967		1.03	
2375		-----		-----	
2386	In house	21.46		-0.52	
2485	In house	22.05		-0.25	
2538	In house	23.516		0.39	
2665	In house	23.2784		0.29	
3172	In house	22.72		0.04	
	normality	OK			
	n	10			
	outliers	0			
	mean (n)	22.626			
	st.dev. (n)	1.7615	RSD = 8%		
	R(calc.)	4.932			
	st.dev.(Horwitz)	2.2638			
	R(Horwitz)	6.339			

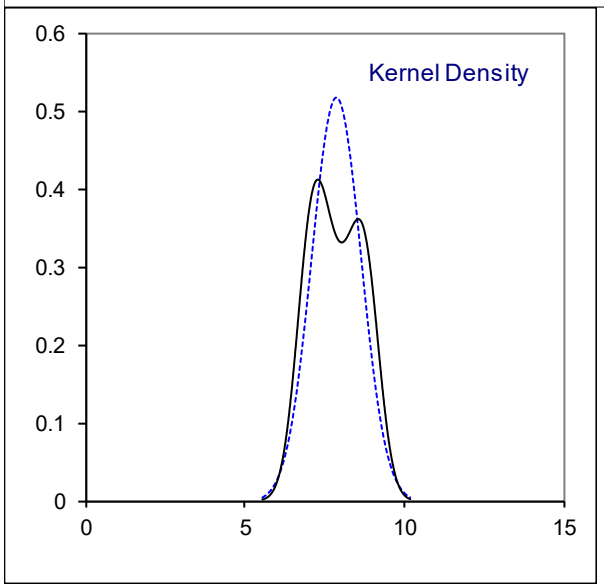




Determination of MIT (2-Methyl-4-isothiazolin-3-one) CAS no. 2682-20-4 in sample #20730; results in mg/kg

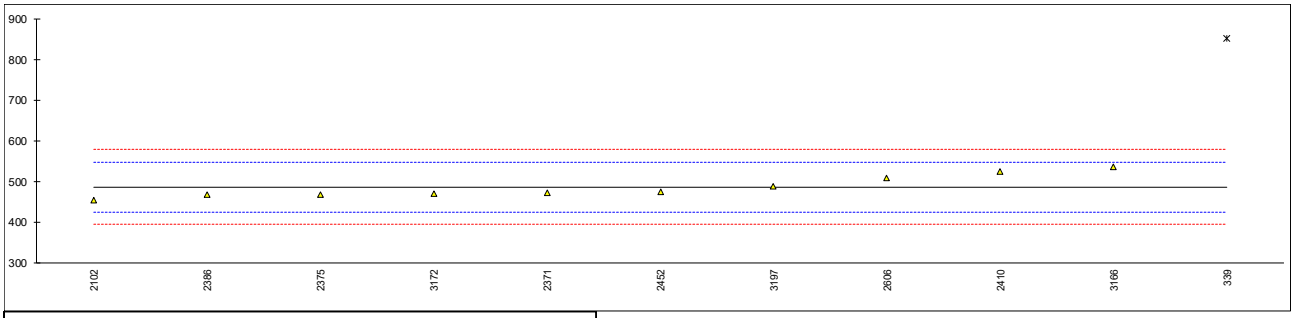
lab	method	value	mark	z(targ)	remarks
339	In house	8.90	C	1.08	Reported 25.0 (possibly switched CMIT and MIT test results)
2102	In house	8.881		1.06	
2137	In house	7.613		-0.31	
2146	In house	6.894		-1.09	
2371	In house	8.357		0.49	
2375		-----		-----	
2386	In house	7.36		-0.58	
2485	In house	7.01		-0.96	
2538	In house	8.026		0.14	
2665	In house	8.6831		0.85	
3172	In house	7.271		-0.68	
	normality	OK			
	n	10			
	outliers	0			
	mean (n)	7.900			
	st.dev. (n)	0.7727	RSD = 10%		
	R(calc.)	2.164			
	st.dev.(Horwitz)	0.9260			
	R(Horwitz)	2.593			

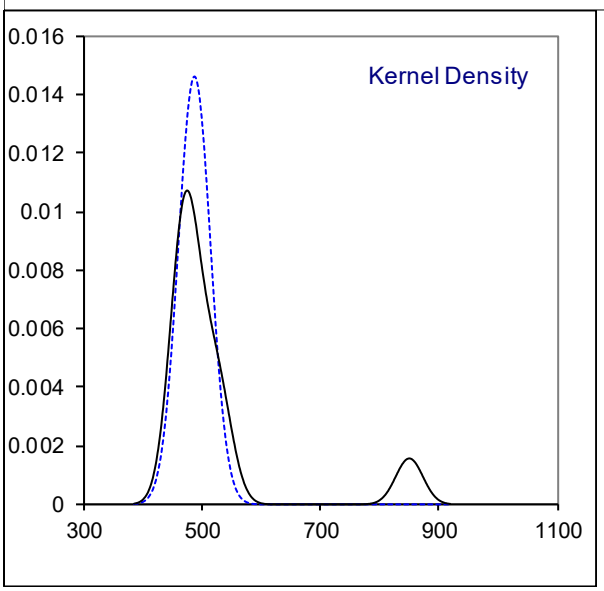




Determination of Methylparaben CAS No. 99-76-3 in sample #20735; results in mg/kg

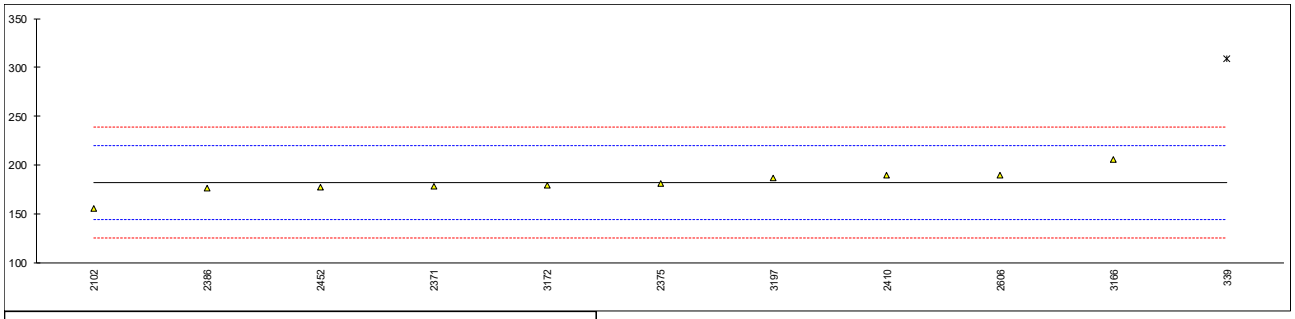
lab	method	value	mark	z(targ)	remarks
339	In house	851	D(0.01)	11.86	
2102	In house	453.57	C	-1.09	First reported 411.75 mg/kg (as acid)
2146		-----		-----	
2371	In house	473.341		-0.44	
2375	In house	468.53		-0.60	
2386	In house	468.14		-0.61	
2410	In house	523.9		1.21	
2452	In house	476.059		-0.35	
2606	In house	510		0.75	
3166	In house	536		1.60	
3172	In house	470.54		-0.53	
3197	In house	489		0.07	
	normality	OK			
	n	10			
	outliers	1			
	mean (n)	486.908			
	st.dev. (n)	27.2583	RSD = 6%		
	R(calc.)	76.324			
	st.dev.(Horwitz)	30.6949			
	R(Horwitz)	85.946			

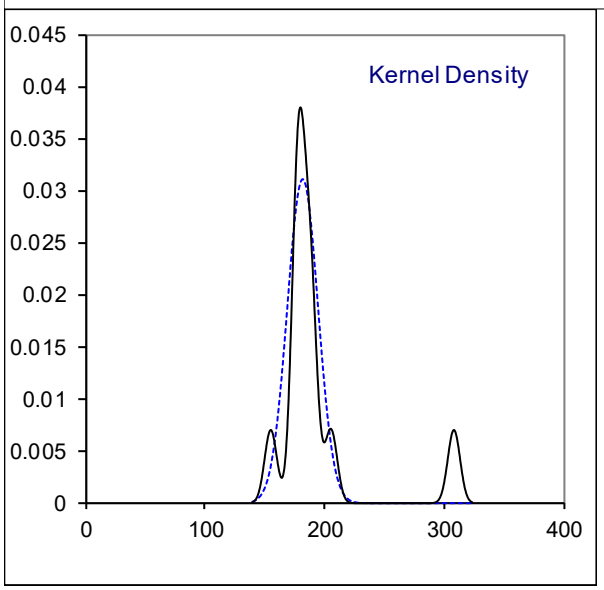




Determination of Ethylparaben CAS No. 120-47-8 in sample #20735; results in mg/kg

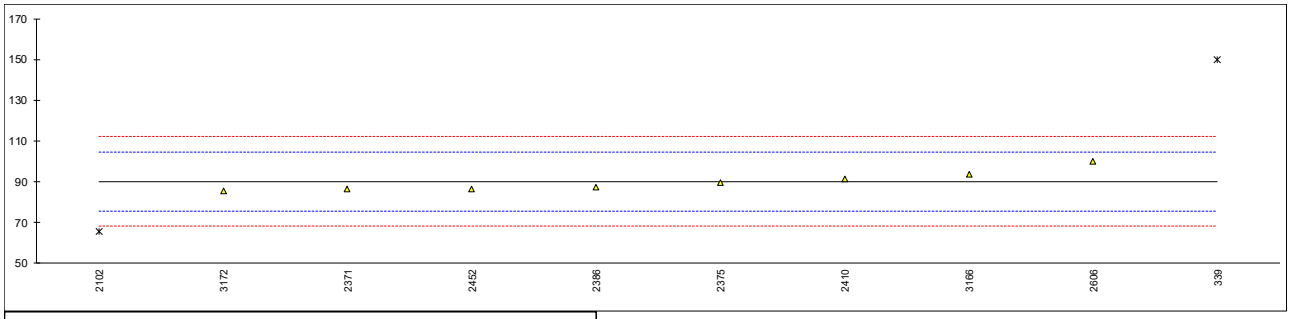
lab	method	value	mark	z(targ)	remarks
339	In house	309	G(0.01)	6.73	
2102	In house	155.48	C	-1.42	First reported 129.23 mg/kg (as acid)
2146		-----		-----	
2371	In house	178.628		-0.19	
2375	In house	181.29		-0.05	
2386	In house	177.06		-0.27	
2410	In house	189.6		0.39	
2452	In house	177.615		-0.25	
2606	In house	190		0.41	
3166	In house	206		1.26	
3172	In house	179.68		-0.14	
3197	In house	187		0.25	
	normality	not OK			
	n	10			
	outliers	1			
	mean (n)	182.235			
	st.dev. (n)	12.8513	RSD = 7%		
	R(calc.)	35.984			
	st.dev.(Horwitz)	18.8369			
	R(Horwitz)	52.743			

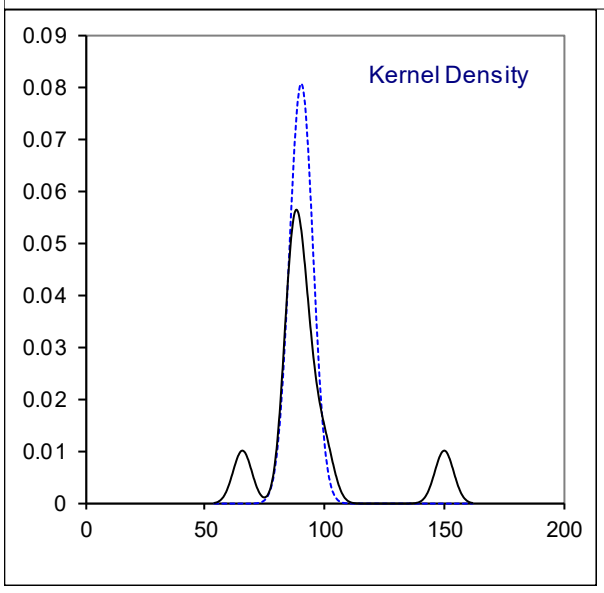




Determination of Propylparaben CAS No. 94-13-3 in sample #20735; results in mg/kg

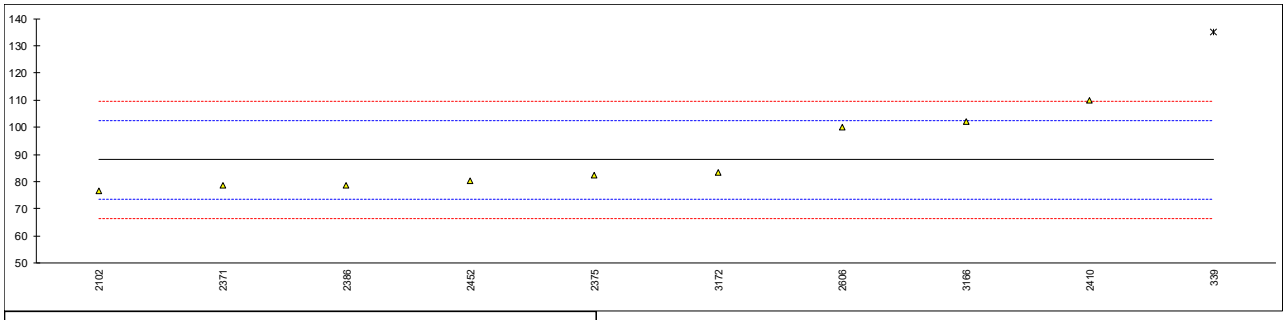
lab	method	value	mark	z(targ)	remarks
339	In house	150	G(0.01)	8.19	
2102	In house	65.40	C,G(0.05)	-3.37	First reported 50.13 mg/kg (as acid)
2146		-----		-----	
2371	In house	86.289		-0.51	
2375	In house	89.65		-0.05	
2386	In house	87.38		-0.36	
2410	In house	91.5		0.20	
2452	In house	86.332		-0.51	
2606	In house	100		1.36	
3166	In house	93.8		0.51	
3172	In house	85.45		-0.63	
3197	In house	<100		-----	
	normality	not OK			
	n	8			
	outliers	2			
	mean (n)	90.050			
	st.dev. (n)	4.9510	RSD = 5%		
	R(calc.)	13.863			
	st.dev.(Horwitz)	7.3186			
	R(Horwitz)	20.492			

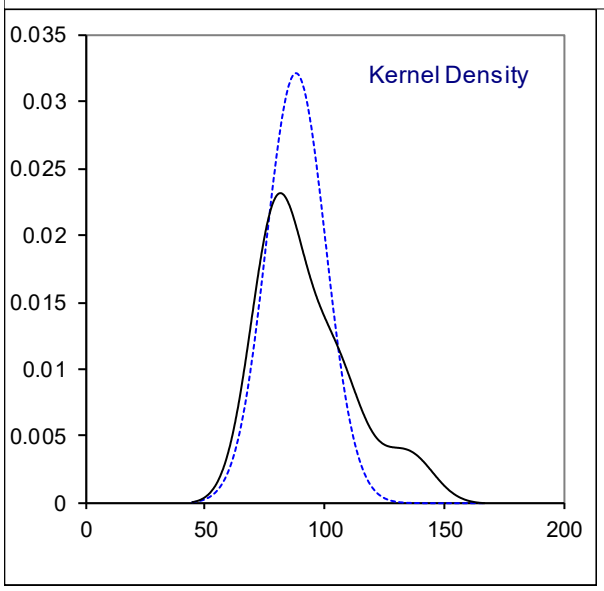




Determination of Isobutylparaben CAS No. 4247-02-3 in sample #20735; results in mg/kg

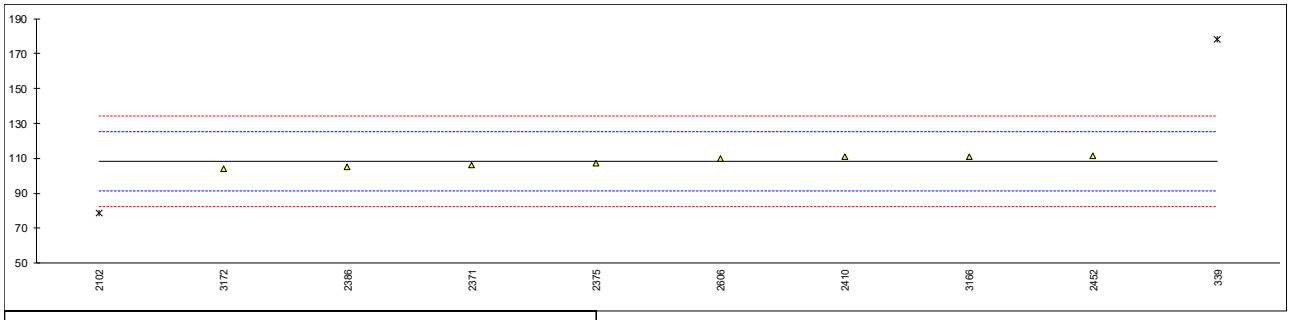
lab	method	value	mark	z(targ)	remarks
339	In house	135	G(0.05)	6.55	
2102	In house	76.61	C	-1.59	First reported 54.48 mg/kg (as acid)
2146		-----		-----	
2371	In house	78.667		-1.30	
2375	In house	82.4		-0.78	
2386	In house	78.76		-1.29	
2410	In house	110.0		3.06	
2452	In house	80.234		-1.08	
2606	In house	100		1.67	
3166	In house	102		1.95	
3172	In house	83.408		-0.64	
3197	In house	<100		-----	
	normality	OK			
	n	9			
	outliers	1			
	mean (n)	88.009			
	st.dev. (n)	12.4447	RSD = 14%		
	R(calc.)	34.845			
	st.dev.(Horwitz)	7.1774			
	R(Horwitz)	20.097			

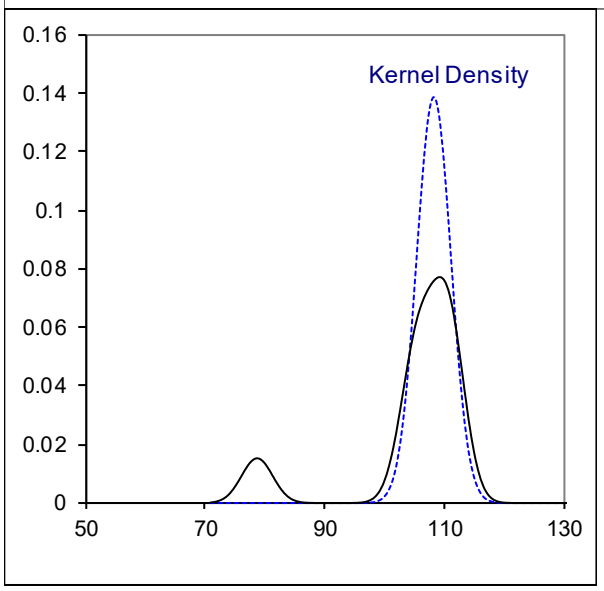




Determination of Butylparaben CAS No. 94-26-8 in sample #20735; results in mg/kg

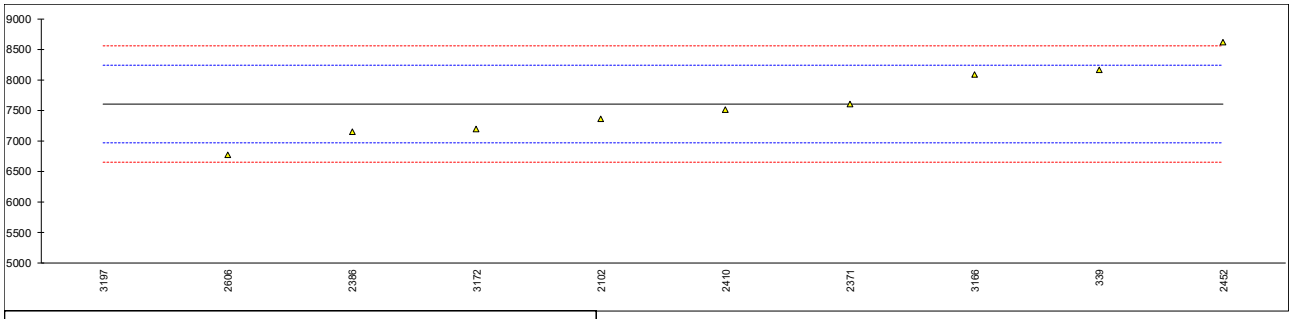
lab	method	value	mark	z(targ)	remarks
339	In house	178	G(0.01)	8.15	
2102	In house	78.64	C,G(0.01)	-3.46	First reported 55.92 mg/kg (as acid)
2146		-----		-----	
2371	In house	106.151		-0.25	
2375	In house	107.47		-0.09	
2386	In house	105.16		-0.36	
2410	In house	110.8		0.30	
2452	In house	111.379		0.36	
2606	In house	110		0.20	
3166	In house	111		0.32	
3172	In house	104.19		-0.48	
3197	In house	<100		-----	
	normality	OK			
	n	8			
	outliers	2			
	mean (n)	108.269			
	st.dev. (n)	2.8775	RSD = 3%		
	R(calc.)	8.057			
	st.dev.(Horwitz)	8.5585			
	R(Horwitz)	23.964			

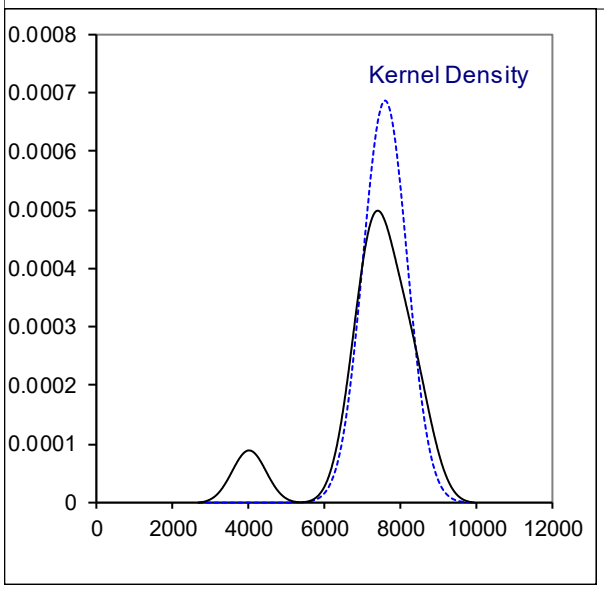




Determination of Phenoxyethanol CAS No. 122-99-6 in sample #20735; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	8160		1.74	
2102	In house	7356.70		-0.79	
2146		-----		-----	
2371	In house	7607.983		0.00	
2375		-----		-----	
2386	In house	7150.28		-1.44	
2410	In house	7516.2		-0.29	
2452	In house	8617.094		3.18	
2606	In house	6777.8		-2.62	
3166	In house	8086		1.51	
3172	In house	7194.71		-1.30	
3197	In house	4041.9	C,G(0.01)	-11.24	First reported 6265
	normality	OK			
	n	9			
	outliers	1			
	mean (n)	7607.418			
	st.dev. (n)	580.1490	RSD = 8%		
	R(calc.)	1624.417			
	st.dev.(Horwitz)	317.0829			
	R(Horwitz)	887.832			





APPENDIX 2

Analytical details for iis20H07A

lab	Accredited acc ISO1725	Intake amount (g)
339	No	0.8g x 2
2102	No	0.175 gram
2137	No	1
2146	Yes	-
2371	Yes	1 g
2375	---	--
2386	Yes	1
2485	Yes	1 g
2538	Yes	1 g
2665	Yes	0,5g
3172	---	-

Analytical details for iis20H07B

lab	Accredited acc ISO1725	Intake amount (g)
339	No	1.1 g
2102	Yes	1 gram
2146	---	-
2371	Yes	0.5 g
2375	Yes	-
2386	Yes	1
2410	Yes	0.5 g
2452	No	0.5
2606	Yes	1
3166	Yes	0.5
3172	Yes	-
3197	Yes	-

APPENDIX 3

Number of participants per country

1 lab in	FINLAND
1 lab in	FRANCE
3 labs in	GERMANY
1 lab in	ITALY
2 labs in	SOUTH KOREA
1 lab in	SWITZERLAND
1 lab in	TAIWAN
1 lab in	THE NETHERLANDS
1 lab in	TUNISIA
2 labs in	TURKEY
1 lab in	U.S.A.
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
ex = test result excluded from statistical evaluation
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected

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