REPORTS OF THE FINNISH ENVIRONMENT INSTITUTE 12| 2016

Interlaboratory Proficiency Test 09/2015

PCB compounds in soil

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Proftest SYKE

FINAS Finnish Accreditation Service PT01 (EN ISO/IEC 17043)

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Finnish Environment Institute

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1 Introduction

Proftest SYKE carried out the proficiency test (PT) for analysis of PCB compounds in soil in November 2015 (PCB 09/15). A synthetic sample (PCBs in isooctane) and a contaminated soil sample were distributed to the participants. The PCB compounds to be measured were: PCB-28, PCB-31, PCB-(28+31), PCB-52, PCB-101, PCB-105, PCB-118, PCB-138, PCB-153, PCB-156, PCB-170 and PCB-180 as well as the sum of seven individual PCB congeners (ΣPCB_7, including 28, 52, 101, 118, 138, 153 and 180, marked as "SigmaPCB_7" in the results) [1].

Finnish Environment Institute (SYKE) is appointed National Reference Laboratory in the environmental sector in Finland. The duties of the reference laboratory include providing interlaboratory proficiency tests and other comparisons for analytical laboratories and other producers of environmental information. This proficiency test has been carried out under the scope of the SYKE reference laboratory and it provides an external quality evaluation between laboratory results, and mutual comparability of analytical reliability. The proficiency test was carried out in accordance with the international guidelines ISO/IEC 17043 [2], ISO 13528 [3] and IUPAC Technical report [4]. The Proftest SYKE has been accredited by the Finnish Accreditation Service as a proficiency testing provider (PT01, ISO/IEC 17043, www.finas.fi/scope/PT01/uk). The organizing of this proficiency test is included in the accreditation scope.

A warm thank you to all the participants of this proficiency test.

2 Organizing the proficiency test

2.1 Responsibilities

Organizer
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The responsibilities in organizing the proficiency test

Jari Nuutinen	coordinator and analytical expert
Riitta Koivikko	substitute for coordinator
Anne Markkanen	technical assistance
Keijo Tervonen	technical assistance
Ritva Väisänen	technical assistance
Markku Ilmakunnas	technical assistance
Sari Lanteri	technical assistance

2.2 Participants

In total 8 laboratories participated in this proficiency test, 7 from Finland and one from Denmark (Appendix 1). All the participants used accredited analytical methods at least for a part of the measurements. For this proficiency test, the organizing laboratory (T003, www.finas.fi/scope/T003/uk) has code 3 (SYKE, Helsinki).

2.3 Samples and delivery

Two samples were delivered to the participants; synthetic sample and soil sample. The synthetic sample A1P was prepared by weighing from the certificated reference materials (PCB-Mix3 from Dr. Ehrenstorfer and custom made mixture of PCBs from UltraScientific). The soil sample M2P was PCB contaminated soil from Tampere region. The dry soil sample was sieved through a 0.5 mm sieve and homogenized by the organizer. Homogenized sample was divided into sub-samples using vibrating feeder distributor by the laboratory of Water Protection Association of the Kokemäenjoki River in Tampere (KVVY, Finland, accredited testing laboratory T064 by the Finnish Accreditation Service, www.finas.fi/scope/T064/uk). The sample preparation was conducted by the organizing laboratory (T003) and is described in details in the Appendix 2.

The samples were delivered on 9 November 2015 to the international participant and 10 November 2015 to the national participants. The samples arrived to the participants on 11 November.

The samples were requested to be measured latest on 30 November 2015. The results were requested to be reported latest on 30 December 2015 and all participants delivered the results accordingly. The preliminary results were delivered to the participants via email on 3 December 2015.

2.4 Homogeneity and stability studies

Homogeneity of the soil sample M2P was tested by analyzing PCB-52, PCB-101, PCB-118, PCB-138, PCB-153, and PCB-180 as duplicate determinations from seven sub samples (Appendix 3). Further, also homogeneity of the sum of ΣPCB_7 was tested. According to the homogeneity test results the sample M2P was considered to be homogenous.

Based on the earlier similar proficiency tests, all compound included in the synthetic as well as the soil samples are known to be persistent.

The stability was checked for the synthetic sample (A1P) during the transport. The synthetic samples (A1P) were weighed at SYKE before the delivery and reweighed by the participants after the sample receiving. The difference of these two measurements was allowed to be < 1 %. The difference was slightly higher for one participant and it was taken into consideration when analyzing the results.

2.5 Feedback from the proficiency test

The feedback from the proficiency test is shown in Appendix 4. The comments from participants dealt only with their reporting errors. The comments from the provider are mainly focused to the lacking conversancy to the given information with the samples. Proftest SYKE is currently updating the results processing program and simultaneously the electronic interface will be improved. All the feedback is valuable and is exploited when improving the activities.

2.6 Processing the data

2.6.1 Pretesting the data

The normality of the data was tested by the Kolmogorov-Smirnov test. The outliers were rejected according to the Grubbs or Hampel test before calculating the mean. The results which differed more than 50 % or 5 times from the robust mean were rejected before the statistical robust results handling. The replicate results were tested using the Cochran-test. If the result has been reported as below detection limit, it has not been included in the statistical calculations.

More information about the statistical handling of the data is available in the Guide for participant [5].

2.6.2 Assigned values

The assigned values and their uncertainties are presented in Appendix 5. The calculated concentrations were used as the assigned values for the analytes of the synthetic sample A1P, which was prepared from reference materials (NIST traceable material from Ultra Scientific Analytical Solutions and Dr. Ehrenstorfer standard with gravimetric certificate). For the calculated assigned values the expanded measurement uncertainty (k=2) was estimated using standard uncertainties associated with individual operations involved in the preparation of the sample. The main individual source of the uncertainty was the uncertainty of the concentration in the stock solutions. The expanded uncertainty of the calculated assigned values varied from 0.5 to 1.2 %.

The means of the reported results were used as the assigned values for the concentrations of the PCBs in the soil sample M2P. The uncertainty of the assigned value was calculated using the standard deviation of the reported results [3, 5]. The mean is not metrologically traceable assigned value. As it was not possible to have metrologically traceable assigned values, the means of the results were the best available values to be used as the assigned values. The reliability of the assigned value was statistically tested according to the IUPAC Technical report [4]. Depending of the PCB congener the uncertainties varied from 6.8 to 21.3 % (Appendix 5). When the number of reported results was low ($n \leq 5$: PCB-110, PCB-170, PCB-(28+31), and PCB-31), the assigned value was not set. After reporting the preliminary results no changes have been done for the assigned values.

2.6.3 Standard deviation for proficiency assesment and z score

The target value for the standard deviation for proficiency assessment was estimated on the basis of the analyte concentration, the results of homogeneity and stability tests, the uncertainty of the assigned value, and the long-term variation in the former proficiency tests. When the number of reported results was low ($n \leq 5$: PCB-110, PCB-170, PCB-(28+31), PCB-31), the target value for the standard deviation was not set. The target value for the standard deviation for proficiency assessment ($2 \times s_{pt}$) was set to 20–30 % depending on the measurements. After reporting the preliminary results no changes have been done for the standard deviations of the proficiency assessment values.

When the means of the reported results were used as the assigned values, the reliability was tested according to the criterion $u_{pt} / s_{pt} \leq 0.3$, where u is the standard uncertainty of the assigned value (the expanded uncertainty of the assigned value (U_{pt}) divided by 2) and s_{pt} is the standard deviation for proficiency assessment [4]. The criterion was fulfilled here only partly.

The reliability of the target value of the standard deviation and the corresponding z score was estimated by comparing the standard deviation (sd) of the reported results with the deviation for proficiency assessment (s_{pt}). The criterion value for the correlation is $sd/s_{pt} < 1.2$, which was here only partly fulfilled.

Due to low number of the results, the criterion for the reliability of the assigned value[a] and for the reliability of the target value for the deviation[b] was not met in the following cases, and, therefore, the evaluation of the performance is weakened in this proficiency test:

Sample	Measurement
M2P	PCB-101[a], PCB-105[a,b], PCB-118[a,b], PCB-156[a,b] PCB-180[a,b], PCB-28[a], PCB-52[a]

3 Results and conclusions

3.1 Results

The terms in the results tables are explained in Appendix 6. The results and the performance of each laboratory are presented in Appendix 7 and the summary of the results in Table 1. The results of the replicate determination are presented in Table 2 (ANOVA statistics). The reported results with their expanded uncertainties (k=2) are presented in Appendix 8. The summary of the z scores is shown in Appendix 9 and z scores in the ascending order in Appendix 10.

Table 1. The summary of the results in the proficiency test PCB 09/2015.

Analyte	Sample	Unit	Assigned value	Mean	Rob. mean	Median	SD	SD %	SD rob	2×s_{pt} %	n (all)	Acc z %
PCB-101	A1P	ng/ml	18.0	18.0		18.5	1.9	10.5		20	8	75
	M2P	µg/kg	49.6	49.6	49.6	47.3	7.8	15.8	8.9	30	8	100
PCB-105	A1P	ng/ml	7.59	7.38		7.48	0.8	10.4		20	6	100
	M2P	µg/kg	2.49	2.49		2.14	0.6	25.9		30	6	83
PCB-110	A1P	ng/ml		7.45		7.45	0.3	4.5		-	2	-
	M2P	µg/kg		38.1		38.1	0.4	0.9		-	2	-
PCB-118	A1P	ng/ml	18.1	18.5		19.1	2.0	10.8		20	8	75
	M2P	µg/kg	15.2	15.2	15.2	15.7	3.4	22.2	3.8	30	8	75
PCB-138	A1P	ng/ml	18.1	18.8		18.9	1.4	7.5		20	8	75
	M2P	µg/kg	121	121	124	120	10.8	9.0	16	30	8	88
PCB-153	A1P	ng/ml	18.1	18.6		18.6	1.5	8.1		20	8	75
	M2P	µg/kg	116	116	120	116	11.6	10.0	17	30	8	88
PCB-156	A1P	ng/ml	7.59	7.24		7.49	1.6	22.0		20	6	67
	M2P	µg/kg	10.5	10.5		11.8	2.7	26.1		30	6	83
PCB-170	A1P	ng/ml		7.16		7.33	0.8	11.7		-	4	-
	M2P	µg/kg		46.9		47.3	14.2	30.3		-	4	-
PCB-180	A1P	ng/ml	18.0	19.0		19.4	2.7	14.2		20	8	63
	M2P	µg/kg	74.9	74.9	74.7	74.4	15.0	20.0	16.6	30	8	75
PCB-28	A1P	ng/ml	18.1	17.4		16.9	3.6	20.8		30	7	71
	M2P	µg/kg	0.104	0.10		0.10	0.0	14.0		30	7	75
PCB-28+31	A1P	ng/ml		24.4		25.6	5.3	21.6		-	5	-
	M2P	µg/kg		0.18		0.18	0.0	17.0		-	5	-
PCB-31	A1P	ng/ml		13.57		7.41	13.6	100.5		-	3	-
	M2P	µg/kg		0.066		0.066	0.0	0.0		-	3	-
PCB-52	A1P	ng/ml	18.0	17.2		17.9	2.6	14.9		20	8	63
	M2P	µg/kg	1.60	1.60	1.62	1.60	0.3	19.8	0.30	30	8	86
SigmaPCB_7	A1P	ng/ml	126	128		130	15.4	12.0		20	7	71
	M2P	µg/kg	375	375	375	371	34.6	9.2	38	25	8	88

Rob. mean: the robust mean, SD rob: the robust standard deviation, SD rob %: the robust standard deviation as percent, 2×s_{pt} %: the total standard deviation for proficiency assessment at the 95 % confidence interval, Acc z %: the results (%), where $|z| \leq 2$, n(all): the number of the participants.

Due to low number of participants, robust standard deviation is calculated only for some analytes. The robust standard deviation was calculated for soil sample M2P when the number of results within the statistical evaluation was ≥ 8 (M2P: PCB-101, PCB-118, PCB-138, PCB-153, PCB-180, PCB-52, and ΣPCB_7). The robust standard deviations of the results varied from 10.2 to 25.2 % (Table 1). The standard deviation was calculated for all congeners and samples and it varied from 7.5 to 26.1 % (Table 1, Appendix 7). The standard deviation was lower than 10 % for 20 % of the results and lower than 20 % for 70 % of the results. Standard deviations higher than 20 % applied mainly to the congeners PCB-105, PCB-118, PCB-156, PCB-180 and PCB-28 (Table 1, Appendix 7). The standard deviations were slightly lower when compared to the previous similar proficiency test Proftest SYKE 11/2011 [6], where 12.5 % of the standard deviations were < 10%.

Table 2. Results of the replicate determinations (ANOVA statistics).

Analyte	Sample	Unit	Ass.val.	Mean	s_w	s_b	s_t	s_w%	s_b%	s_t%	s_b/s_w
PCB-101	A1P	ng/ml	18.0	18.0	0.361	1.87	1.90	2.0	10	11	5.2
	M2P	µg/kg	49.6	49.6	8.16	5.31	9.74	16	11	20	0.65
PCB-105	A1P	ng/ml	7.59	7.38	0.549	0.660	0.859	7.4	8.9	12	1.2
	M2P	µg/kg	2.49	2.49	0.104	0.641	0.649	4.2	26	26	6.2
PCB-118	A1P	ng/ml	18.1	18.5	0.450	1.97	2.02	2.4	11	11	4.4
	M2P	µg/kg	15.2	15.2	1.11	3.29	3.47	7.3	22	23	3.0
PCB-138	A1P	ng/ml	18.1	18.8	0.753	1.32	1.52	4.0	7.0	8.1	1.7
	M2P	µg/kg	121	121	7.08	15.8	17.3	5.7	13	14	2.2
PCB-153	A1P	ng/ml	18.1	18.6	0.581	1.44	1.55	3.1	7.8	8.4	2.5
	M2P	µg/kg	116	116	7.73	20.6	22.0	6.3	17	18	2.7
PCB-156	A1P	ng/ml	7.59	7.24	0.440	1.56	1.62	6.1	22	22	3.5
	M2P	µg/kg	10.5	10.5	1.40	2.56	2.92	13	24	28	1.8
PCB-170	A1P	ng/ml		7.16	0.292	0.810	0.861	4.1	11	12	2.8
	M2P	µg/kg		46.9	0.803	14.2	14.2	1.7	30	30	18
PCB-180	A1P	ng/ml	18.0	19.0	0.564	2.67	2.73	3.0	14	14	4.7
	M2P	µg/kg	74.9	74.9	4.59	14.6	15.3	6.1	20	20	3.2
PCB-28	A1P	ng/ml	18.1	17.4	0.428	3.61	3.64	2.5	21	21	8.4
	M2P	µg/kg	0.104	0.10	0.0222	0	0.0222	21	0	21	0
PCB-28+31	A1P	ng/ml		24.4	0.679	5.24	5.29	2.8	22	22	7.7
PCB-31	A1P	ng/ml		13.57	0.770	13.6	13.6	5.7	100	100	18
PCB-52	A1P	ng/ml	18.0	17.2	0.543	2.54	2.60	3.2	15	15	4.7
	M2P	µg/kg	1.60	1.60	0.122	0.304	0.327	7.6	19	21	2.5
$SigmaPCB_7$	A1P	ng/ml	126	128	3.31	15.2	15.6	2.6	12	12	4.6
	M2P	µg/kg	375	375	15.2	32.9	36.3	4.1	8.8	9.7	2.2

Ass.val.: assigned value; s_w: repeatability standard error; s_b: between participants standard error; s_t: reproducibility standard error.

The participants were requested to report duplicate results for all measurements. The participants reported the replicates with the exception of the participant 7 (sample A1P). The results of the replicate determinations based on the ANOVA statistical handling are presented in Table 2. The estimation of the robustness of the methods could be done by the ratio s_b/s_w, which should not exceed 3 for robust methods. However, in many cases the robustness exceeded the value 3; it varied between 0 and 8.4 and exceeded value 3 in 45 % of the results (Table 2). In the previous similar proficiency test Proftest SYKE 11/2011 [6], the ratio exceeded value 3 in 52 % of the results.

3.2 Analytical methods

The participants were allowed to use different analytical methods for the measurements in the PT. The used analytical methods and results of the participants grouped by methods are shown in more detail in Appendices 11 and 12. The statistical comparison of the analytical methods was possible for the data where the number of the results was ≥ 5. However, in this PT there were not enough results for statistical comparison. Thus, the comparison is based on the graphical result evaluation.

PCB compounds in soil

Determination of PCB compounds from soil samples based on several standard methods or EPAs methods [7, 8]. Half of the participants used methods which were internal modifications of the standard methods. Extraction was mostly done by shaking, followed by ultrasound and accelerated solvent extraction (ASE) techniques. One participant used soxhlet extraction. All participants used splitless-injection technique. Five participants used GC-MS (or GC-HRMS) technique and three used GC-ECD technique.

Method comparison for the samples A1P and M2P was done based on graphical result evaluation between measurement techniques (Appendix 12). No significant differences between used analysis methods were observed. According to the graphical evaluation, the unsatisfactory results were not due to the applied technique (Appendix 12).

In proficiency test Proftest SYKE 3/2005 [9], the determination of the total PCB from soil was tested. There, several different ways to calculate the total PCB were reported: the selected congeners and number of congeners included into the calculation varied, and some participants used a constant factor in the calculations. Due to the low number of reported results, the performance was not evaluated [9]. Thereafter, the Environmental Administration in Finland has published the report *Risk assessment and sustainable risk management of contaminated land* [1, in Finnish]. There the recommendation is given that total PCB should be reported as the sum of seven individual PCB congeners, ΣPCB_7, including congeners PCB-28, PCB-52, PCB-101, PCB-118, PCB-138, PCB-153, and PCB-180.

In this proficiency test all participants reported the ΣPCB_7 (except participant 2 for sample A1P) and the performance was satisfactory for 71 % (A1P) and 88 % (M2P) of the results. 75 % of the participants calculated the ΣPCB_7 according to the recommendation. For the synthetic sample, only the results calculated according to the recommendation were satisfactory, whereas for the soil sample, the similar trend was not seen.

3.3 Uncertainties of the results

All the participants reported the expanded uncertainties (k=2) for at least some of their results (Table 3, Appendix 8). The range of the reported expanded uncertainties varied between the measurements and the sample types.

Several approaches were used for estimating of measurement uncertainty (Appendix 13). The most used approach was based on data from method validation. One participant used MUkit measurement uncertainty software for the estimation of their uncertainties [10]. The free software is available on the webpage: www.syke.fi/envical/en. Generally, the used approach for estimating measurement uncertainty did not make definite impact on the uncertainty estimates.

Table 3. The ranges of the expanded measurement uncertainties (k=2, U_i) reported by the participants.

Analyte	A1P %	M2P %
PCB-101	10-40	20-43
PCB-105	15-30	20-35
PCB-110	15-20	20-35
PCB-118	15-40	20-53
PCB-138	15-40	20-60
PCB-153	10-40	20-48
PCB-156	15-30	20-35
PCB-170	15-30	20-35
PCB-180	15-40	20-59
PCB-28	20-40	20-46
PCB-28+31	20-30	20-35
PCB-31	20	20-35
PCB-52	10-40	20-40
SigmaPCB$_7$	15-30	20-35

4 Evaluation of the results

The evaluation of the participants was based on the z scores, which were calculated using the assigned values and the target values of the standard deviation for the proficiency assessment (Appendix 6). The z scores were interpreted as follows:

Criteria	Performance
$\lvert z \rvert \leq 2$	Satisfactory
$2 < \lvert z \rvert < 3$	Questionable
$\lvert z \rvert \geq 3$	Unsatisfactory

In total, 79 % of the results were satisfactory when total deviation of 20–30 % from the assigned values was accepted. All the participants used accredited analytical methods at least for a part of the measurements and 79 % of the results from accredited methods were satisfactory. In average, 74 % of the results for the synthetic sample A1P and 84 % of the results for the soil sample M2P were satisfactory. The summary of the performance evaluation and comparison to the previous performance is presented in Table 4. Proftest SYKE carried out the similar proficiency test in 2011 where 76 % of the results were satisfactory [6].

The sample stability during the transport was checked by weighing to control the possible evaporation of the solvent. In such case, the results would be systematically too high. Here, this was not the case for the participant who reported the highest weight difference for the synthetic sample vial. Therefore the samples were considered stable.

Table 4. Summary of the performance evaluation in the proficiency test PCB 09/2015.

Sample	$2 \cdot s_{pt}$, %	Satisfactory results, %	Assessment
A1P	20	74	The performance was better that in the previous similar PT. In the PT SYKE 11/2011 the performance was satisfactory for 62 % of the results [6].
M2P	30	84	High uncertainty of the assigned value for PCB-101, PCB-105, PCB-118, PCB-156 PCB-180 PCB-28, and PCB-52. In the PT SYKE 11/2011 the performance was satisfactory for 85 % of the results [6].
A1P, ΣPCB_7	20	71	Five satisfactory results and two unsatisfactory.
M2P, ΣPCB_7	25	88	Good performance. Seven satisfactory results and one unsatisfactory.

5 Summary

Proftest SYKE carried out the proficiency test (PT) for analysis of PCB compounds in soil in November-December 2015 (PCB 09/2015). Two types of samples were delivered to the participants, synthetic and soil samples. In total, 8 laboratories participated in the PT.

For the synthetic sample the calculated concentration and for the soil sample the mean of the results reported by the participants was chosen to be the assigned value. The uncertainty for the assigned value was estimated at the 95 % confidence interval and for calculated assigned values it was 0.5–1.2 %, for assigned values based on the mean it varied from 6.8 to 21.3 %.

The evaluation of the performance was based on the z scores, which were calculated using the standard deviation for proficiency assessment at 95 % confidence level. In this proficiency test 79 % of the data was regarded to be satisfactory when the standard deviation of 20 to 30 % from the assigned value was accepted.

6 Summary in Finnish

Proftest SYKE järjesti marras-joulukuussa 2015 pätevyyskokeen PCB-yhdisteitä maasta analysoiville laboratorioille (PCB 09/2015). Pätevyyskokeen osallistujille toimitettiin kaksi näytettä, synteettinen ja maanäyte. Pätevyyskokeeseen osallistui yhteensä 8 laboratoriota.

Mittaussuureen vertailuarvona käytettiin laskennallista pitoisuutta tai osallistujien tulosten keskiarvoa. Vertailuarvolle laskettiin mittausepävarmuus 95 % luottamusvälillä. Vertailuarvon laajennettu epävarmuus oli 0,5–1,2 % laskennallista pitoisuutta vertailuarvona käytettäessä ja kun vertailuarvo määritettiin osallistujien tulosten keskiarvona, sen laajennettu epävarmuus vaihteli välillä 6,8–21,3 %.

Pätevyyden arviointi tehtiin z-arvon avulla ja tulosten sallittiin poiketa vertailuarvosta 20–30 %. Koko aineistossa hyväksyttäviä tuloksia oli 79 %.

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APPENDIX 1: Participants in the proficiency test

Country	Participant
Denmark	ALS Denmark
Finland	Kokemäenjoen vesistön vesiensuojeluyhdistys ry, Tampere Metropolilab Oy Nab Labs Oy / Ambiotica Jyväskylä Ramboll Finland Oy, Ramboll Analytics, Lahti SGS Inspection Services Oy, Kotka SYKE Ympäristökemia Helsinki THL/ Ympäristöterveyden osasto, Kuopio

APPENDIX 2: Preparation of the samples

Sample preparation was conducted by the organizing laboratory (T003, ww.finas.fi/scope/T003/uk). All the dilutions were made gravimetrically.

Stock solutions:
CUS-13245 (NIST traceable, certified reference material, Ultra Scientific Analytical Solutions), concentrations for each PCB are shown in Table 1. PCB-Mix 3 (Gravimetric certificate, Dr.Ehrenstorfer): PCB congeners: PCB-28, 52, 101, 110, 118, 138, 153, and 180, each 10 µg/ml in isooctane.

PCB congeners in the synthetic sample A1P:
PCB-28, 31, 52, 101, 105, 110, 118, 138, 153, 156, 170, 180

The PCB congeners included to the ΣPCB_7 are the **bold** numbers in the table below.

Preparing the sample A1P:
0.50 ml (0.34843 g) of PCB-Mix 3 was diluted with 4.39 ml (3.02825 g) of isooctane (*Dilution 1*).

Sample A1P was prepared by diluting 1.011 ml (0.69777 g) of *Dilution 1* and 0.757 ml (0.52246 g) of CUS-13245 with 97.94 ml (67.58028 g) isooctane.

Concentration of PCB congeners in stock solutions and the sample A1P:

PCB	Concentration in stock solution CUS-13245	Concentration of CUS-13245 PCBs in sample A1P	Concentration in stock solution PCB-Mix3	Concentration of PCB-Mix3 PCBs in sample A1P	Final PCB concentrations in the sample A1P
	[ng/ml]				
28	1004	7.624	10000	10.41	**18.04**
31	1000	7.594	-	-	7.59
52	997.0	7.571	10000	10.36	**17.93**
101	997.7	7.576	10000	10.36	**17.94**
105	1000	7.594	-	-	7.59
110	1000	7.594	-	-	7.59
118	1001	7.601	10000	10.36	**17.96**
138	1004	7.624	10000	10.31	**17.93**
153	1001	7.601	10000	10.41	**18.01**
156	1000	7.594	-	-	7.59
170	1000	7.594	-	-	7.59
180	996.7	7.569	10000	10.41	**17.98**
ΣPCB_7					**125.80**

APPENDIX 3: Homogeneity of the samples

The soil sample M2P: The sample was divides to vessels and the homogeneity of the samples was tested by analyzing as duplicate determination from the seven sub samples. Homogeneity testing was carried out in July 2015.

Criteria for homogeneity

$$\mathbf{s_a/s_h < 0.5} \quad \textbf{and}$$

$$\mathbf{s_{sam}^2 < c} \quad \text{, where}$$

s_h = standard deviation for testing of homogeneity
s_a = analytical deviation, standard deviation of the results within sub samples
s_{sam} = between-sample deviation, standard deviation of the results between sub samples

$c = F1 \cdot s_{all}^2 + F2 \cdot s_a^2$, where
$s_{all}^2 = (0.3 \cdot s_h)^2$
F1 and F2 are constants of F distribution derived from the standard statistical tables for the tested number of samples [4].

Analyte/Sample	Concentration [µg/kg]	n	s_{pt} %	s_h %	s_h	s_a	s_a/s_h	s_a/s_h<0,5?	s_{sam}	s_{sam}^2	c	s_{sam}^2<c?
PCB-101 / M2P	48.0	7	15	11	5.3	2.4	0.46	Yes	0	0	13.7	Yes
PCB-118 / M2P	15.9	7	15	10	1.59	0.6	0.38	Yes	0.22	0.046	1.0	Yes
PCB-138 / M2P	98.7	7	15	10	9.9	3.7	0.37	Yes	0	0	37.9	Yes
PCB-153 / M2P	121	7	15	10	12.1	5.6	0.46	Yes	0	0	71.9	Yes
PCB-180 / M2P	52.6	7	15	10	5.3	2.2	0.41	Yes	0	0	11.9	Yes
PCB-52 / M2P	1.53	7	15	11	0.17	0.08	0.48	Yes	0.057	0.0033	0.015	Yes
ΣPCB_7 / M2P	338	7	12.5	10	33.8	14.6	0.43	Yes	0	0	520	Yes

s_{pt} % = standard deviation for proficiency assessment

Conclusion: The criteria for homogeneity were fulfilled and the sample considered homogenous.

APPENDIX 4: Feedback from the proficiency test

FEEDBACK FROM THE PARTICIPANTS

Participant	Comments to the results	Action / Proftest
4	The participant reported the results for the synthetic sample erroneously. The correct results were: 17.6 ng/ml PCB-28 19.6 ng/ml PCB-52 19.1 ng/ml PCB-101 18.4 ng/ml PCB-118 18.2 ng/ml PCB-138 19.3 ng/ml PCB-153 17.3 ng/ml PCB-180 131 ng/ml ΣPCB_7	The results were outliers in statistical treatment and thus did not affect the performance evaluation. If the results would have been reported correctly they would have been satisfactory. The participant can re-calculate z scores according to the guide for participating laboratories [5].

FEEDBACK TO THE PARTICIPANTS

Participant	Comments
1, 5, 7	PCB-28+31 and PCB-31: The reported results <1 for sample M2P are valid according to the results reported by the participant(s) 3 and/or 6.
2, 7	For these participants the deviation of replicate measurements for some measurands of the sample M2P was high and those results were Cochran outliers (totally 7 cases). The provider recommends the participants to validate their accepted deviation of replicate measurements.
6	The bottle numbers were filled in to the result sheet by the provider.
7	The participant reported only one result in their dataset for the synthetic sample when replicate results were requested for the PT. These results were not included in the calculation of assigned values. The provider recommends the participants to follow the given guidelines.

APPENDIX 5: Evaluation of the assigned values and their uncertainties

Analyte	Sample	Unit	Assigned value	U_{pt}	U_{pt}, %	Evaluation method of assigned value	u_{pt}/s_{pt}
PCB-101	A1P	ng/ml	18.0	0.2	1.2	Calculated value	0.06
	M2P	µg/kg	49.6	5.6	11.2	Mean	0.37
PCB-105	A1P	ng/ml	7.59	0.05	0.6	Calculated value	0.03
	M2P	µg/kg	2.49	0.53	21.2	Mean	0.71
PCB-110	A1P	ng/ml					
	M2P	µg/kg					
PCB-118	A1P	ng/ml	18.1	0.1	0.6	Calculated value	0.03
	M2P	µg/kg	15.2	2.4	15.7	Mean	0.52
PCB-138	A1P	ng/ml	18.1	0.2	1.2	Calculated value	0.06
	M2P	µg/kg	121	8	6.8	Mean	0.23
PCB-153	A1P	ng/ml	18.1	0.2	1.2	Calculated value	0.06
	M2P	µg/kg	116	9	7.6	Mean	0.25
PCB-156	A1P	ng/ml	7.59	0.05	0.6	Calculated value	0.03
	M2P	µg/kg	10.5	2.2	21.3	Mean	0.71
PCB-170	A1P	ng/ml					
	M2P	µg/kg					
PCB-180	A1P	ng/ml	18.0	0.2	1.2	Calculated value	0.06
	M2P	µg/kg	74.9	10.6	14.1	Mean	0.47
PCB-28	A1P	ng/ml	18.1	0.2	1.2	Calculated value	0.04
	M2P	µg/kg	0.104	0.02	16.2	Mean	0.54
PCB-28+31	A1P	ng/ml					
	M2P	µg/kg					
PCB-31	A1P	ng/ml					
	M2P	µg/kg					
PCB-52	A1P	ng/ml	18.0	0.2	1.2	Calculated value	0.06
	M2P	µg/kg	1.60	0.24	15.0	Mean	0.50
$SigmaPCB_7$	A1P	ng/ml	126	1	0.5	Calculated value	0.03
	M2P	µg/kg	375	26	7.0	Mean	0.28

U_{pt} = Expanded uncertainty of the assigned value
Criterion for reliability of the assigned value $u_{pt}/s_{pt} \leq 0.3$, where
s_{pt}= target value of the standard deviation for proficiency assessment
u_{pt}= standard uncertainty of the assigned value

If $u_{pt}/s_{pt} \leq 0.3$, the assigned value is reliable and the z scores are qualified.

APPENDIX 6: Terms in the results tables

Results of each participant

Analyte	The tested parameter
Sample	The code of the sample
z score	Calculated as follows: $z = (x_i - x_{pt})/s_{pt}$, where x_i = the result of the individual participant x_{pt} = the assigned value s_{pt} = the target value of the standard deviation for proficiency assessment
Assigned value	The value attributed to a particular property of a proficiency test item
2×s_{pt} %	The target value of total standard deviation for proficiency assessment (s_{pt}) at the 95 % confidence level
Lab's result	The result reported by the participant (the mean value of the replicates)
Md	Median
Mean	Mean
SD	Standard deviation
SD%	Standard deviation, %
n (stat)	Number of results in statistical processing

Summary on the z scores

S – satisfactory ($-2 \le z \le 2$)
Q – questionable ($2 < z < 3$), positive error, the result deviates more than 2 × s_{pt} from the assigned value
q – questionable ($-3 < z < -2$), negative error, the result deviates more than 2 × s_{pt} from the assigned value
U – unsatisfactory ($z \ge 3$), positive error, the result deviates more than 3 × s_{pt} from the assigned value
u – unsatisfactory ($z \le -3$), negative error, the result deviates more than 3 × s_{pt} from the assigned value

Robust analysis

The items of data are sorted into increasing order, $x_1, x_2, x_i, \ldots, x_p$.
Initial values for x^* and s^* are calculated as:
x^* = median of x_i ($i = 1, 2, \ldots, p$)
s^* = 1,483 · median of $|x_i - x^*|$ ($i = 1, 2, \ldots, p$)

The mean x^* and s^* are updated as follows:
Calculate $\varphi = 1.5 \cdot s^*$. A new value is then calculated for each result x_i $(i = 1, 2, \ldots, p)$:

$$x_i^* = \begin{cases} x^* - \varphi, & \text{if } x_i < x^* - \varphi \\ x^* + \varphi, & \text{if } x_i > x^* + \varphi, \\ x_i & \text{otherwise} \end{cases}$$

The new values of x^* and s^* are calculated from:

$$x^* = \sum x_i^* / p$$

$$s^* = 1.134\sqrt{\sum (x_i^* - x^*)^2 / (p-1)}$$

The robust estimates x^* and s^* can be derived by an iterative calculation, i.e. by updating the values of x^* and s^* several times, until the process convergences [3].

APPENDIX 7: Results of each participant

Participant 1												
Analyte	Unit	Sample	-3 0 3	z score	Assigned value	2×s_{pt} %	Lab's result	Md	Mean	SD	SD%	n (stat)
PCB-101	ng/ml	A1P		1.33	18.0	20	20.4	18.5	18.0	1.9	10.5	6
	µg/kg	M2P		0.92	49.6	30	56.5	47.3	49.6	7.8	15.8	8
PCB-105	ng/ml	A1P		-1.26	7.59	20	6.63	7.48	7.38	0.8	10.4	5
	µg/kg	M2P		-1.08	2.49	30	2.09	2.14	2.49	0.6	25.9	6
PCB-118	ng/ml	A1P		0.55	18.1	20	19.1	19.1	18.5	2.0	10.8	6
	µg/kg	M2P		0.64	15.2	30	16.7	15.7	15.2	3.4	22.2	8
PCB-138	ng/ml	A1P		0.75	18.1	20	19.5	18.9	18.8	1.4	7.5	6
	µg/kg	M2P		-0.55	121	30	111	120	121	10.8	9.0	7
PCB-153	ng/ml	A1P		0.33	18.1	20	18.7	18.6	18.6	1.5	8.1	6
	µg/kg	M2P		-0.03	116	30	116	116	116	11.6	10.0	7
PCB-156	ng/ml	A1P		-1.63	7.59	20	6.36	7.49	7.24	1.6	22.0	5
	µg/kg	M2P		0.41	10.5	30	11.2	11.8	10.5	2.7	26.1	6
PCB-180	ng/ml	A1P		0.67	18.0	20	19.2	19.4	19.0	2.7	14.2	6
	µg/kg	M2P		-0.63	74.9	30	67.9	74.4	74.9	15.0	20.0	8
PCB-28	ng/ml	A1P		4.09	18.1	30	29.2	16.9	17.4	3.6	20.8	5
	µg/kg	M2P			0.104	30	*<1,00*	0.10	0.10	0.0	14.0	3
PCB-28+31	ng/ml	A1P					29.2	25.6	24.4	5.3	21.6	4
	µg/kg	M2P					*<1,00*	0.18	0.18	0.0	17.0	2
PCB-31	ng/ml	A1P					29.20	7.41	13.57	13.6	100.5	3
	µg/kg	M2P					*<1,00*	0.066	0.066	0.0	0.0	1
PCB-52	ng/ml	A1P		0.50	18.0	20	18.9	17.9	17.2	2.6	14.9	6
	µg/kg	M2P		1.21	1.60	30	1.89	1.60	1.60	0.3	19.8	7
SigmaPCB$_7$	ng/ml	A1P		1.47	126	20	145	130	128	15.4	12.0	5
	µg/kg	M2P		-0.14	375	25	369	371	375	34.6	9.2	7

Participant 2												
Analyte	Unit	Sample	-3 0 3	z score	Assigned value	2×s_{pt} %	Lab's result	Md	Mean	SD	SD%	n (stat)
PCB-101	ng/ml	A1P		0.17	18.0	20	18.3	18.5	18.0	1.9	10.5	6
	µg/kg	M2P		1.37	49.6	30	59.8	47.3	49.6	7.8	15.8	8
PCB-118	ng/ml	A1P		0.69	18.1	20	19.4	19.1	18.5	2.0	10.8	6
	µg/kg	M2P		0.75	15.2	30	16.9	15.7	15.2	3.4	22.2	8
PCB-138	ng/ml	A1P		0.97	18.1	20	19.9	18.9	18.8	1.4	7.5	6
	µg/kg	M2P		2.04	121	30	158	120	121	10.8	9.0	7
PCB-153	ng/ml	A1P		0.55	18.1	20	19.1	18.6	18.6	1.5	8.1	6
	µg/kg	M2P		3.00	116	30	168	116	116	11.6	10.0	7
PCB-180	ng/ml	A1P		1.61	18.0	20	20.9	19.4	19.0	2.7	14.2	6
	µg/kg	M2P		2.32	74.9	30	101.0	74.4	74.9	15.0	20.0	8
PCB-28	ng/ml	A1P		1.84	18.1	30	23.1	16.9	17.4	3.6	20.8	5
	µg/kg	M2P		-6.67	0.104	30	0.00	0.10	0.10	0.0	14.0	3
PCB-52	ng/ml	A1P		-0.75	18.0	20	16.7	17.9	17.2	2.6	14.9	6
	µg/kg	M2P		0.00	1.60	30	1.60	1.60	1.60	0.3	19.8	7
SigmaPCB$_7$	µg/kg	M2P		2.79	375	25	506	371	375	34.6	9.2	7

Participant 3												
Analyte	Unit	Sample	-3 . . 0 . . 3	z score	Assigned value	2×s_{pt} %	Lab's result	Md	Mean	SD	SD%	n (stat)
PCB-101	ng/ml	A1P		-0.73	18.0	20	16.7	18.5	18.0	1.9	10.5	6
	µg/kg	M2P		-0.67	49.6	30	44.6	47.3	49.6	7.8	15.8	8
PCB-105	ng/ml	A1P		-0.15	7.59	20	7.48	7.48	7.38	0.8	10.4	5
	µg/kg	M2P		1.82	2.49	30	3.17	2.14	2.49	0.6	25.9	6
PCB-110	ng/ml	A1P					7.22	7.45	7.45	0.3	4.5	2
	µg/kg	M2P					37.8	38.1	38.1	0.4	0.9	2
PCB-118	ng/ml	A1P		-0.70	18.1	20	16.8	19.1	18.5	2.0	10.8	6
	µg/kg	M2P		-0.24	15.2	30	14.7	15.7	15.2	3.4	22.2	8
PCB-138	ng/ml	A1P		-0.74	18.1	20	16.8	18.9	18.8	1.4	7.5	6
	µg/kg	M2P		0.07	121	30	122	120	121	10.8	9.0	7
PCB-153	ng/ml	A1P		-0.87	18.1	20	16.5	18.6	18.6	1.5	8.1	6
	µg/kg	M2P		-0.20	116	30	113	116	116	11.6	10.0	7
PCB-156	ng/ml	A1P		-0.13	7.59	20	7.49	7.49	7.24	1.6	22.0	5
	µg/kg	M2P		1.29	10.5	30	12.5	11.8	10.5	2.7	26.1	6
PCB-170	ng/ml	A1P					7.33	7.33	7.16	0.8	11.7	3
	µg/kg	M2P					40.5	47.3	46.9	14.2	30.3	4
PCB-180	ng/ml	A1P		-1.00	18.0	20	16.2	19.4	19.0	2.7	14.2	6
	µg/kg	M2P		0.09	74.9	30	75.9	74.4	74.9	15.0	20.0	8
PCB-28	ng/ml	A1P		-0.51	18.1	30	16.7	16.9	17.4	3.6	20.8	5
	µg/kg	M2P		-0.83	0.104	30	0.09	0.10	0.10	0.0	14.0	3
PCB-28+31	ng/ml	A1P					24.1	25.6	24.4	5.3	21.6	4
	µg/kg	M2P					0.16	0.18	0.18	0.0	17.0	2
PCB-31	ng/ml	A1P					7.41	7.41	13.57	13.6	100.5	3
	µg/kg	M2P					0.066	0.066	0.066	0.0	0.0	1
PCB-52	ng/ml	A1P		-0.57	18.0	20	17.0	17.9	17.2	2.6	14.9	6
	µg/kg	M2P		-0.74	1.60	30	1.42	1.60	1.60	0.3	19.8	7
SigmaPCB$_7$	ng/ml	A1P		-0.74	126	20	117	130	128	15.4	12.0	5
	µg/kg	M2P		-0.09	375	25	371	371	375	34.6	9.2	7

Participant 4												
Analyte	Unit	Sample	-3 . . 0 . . 3	z score	Assigned value	2×s_{pt} %	Lab's result	Md	Mean	SD	SD%	n (stat)
PCB-101	ng/ml	A1P		43.14	18.0	20	95.7	18.5	18.0	1.9	10.5	6
	µg/kg	M2P		-0.57	49.6	30	45.4	47.3	49.6	7.8	15.8	8
PCB-118	ng/ml	A1P		40.69	18.1	20	91.8	19.1	18.5	2.0	10.8	6
	µg/kg	M2P		-0.55	15.2	30	14.0	15.7	15.2	3.4	22.2	8
PCB-138	ng/ml	A1P		40.33	18.1	20	91.1	18.9	18.8	1.4	7.5	6
	µg/kg	M2P		-0.77	121	30	107	120	121	10.8	9.0	7
PCB-153	ng/ml	A1P		43.18	18.1	20	96.3	18.6	18.6	1.5	8.1	6
	µg/kg	M2P		0.32	116	30	122	116	116	11.6	10.0	7
PCB-180	ng/ml	A1P		38.00	18.0	20	86.4	19.4	19.0	2.7	14.2	6
	µg/kg	M2P		-0.78	74.9	30	66.1	74.4	74.9	15.0	20.0	8
PCB-28	ng/ml	A1P		25.75	18.1	30	88.0	16.9	17.4	3.6	20.8	5
	µg/kg	M2P			0.104	30	*<1*	0.10	0.10	0.0	14.0	3
PCB-52	ng/ml	A1P		44.42	18.0	20	98.0	17.9	17.2	2.6	14.9	6
	µg/kg	M2P		1.15	1.60	30	1.88	1.60	1.60	0.3	19.8	7
SigmaPCB$_7$	ng/ml	A1P		42.14	126	20	657	130	128	15.4	12.0	5
	µg/kg	M2P		-0.41	375	25	356	371	375	34.6	9.2	7

Participant 5												
Analyte	**Unit**	**Sample**	-3 0 3	**z score**	**Assigned value**	**2×s_{pt} %**	**Lab's result**	**Md**	**Mean**	**SD**	**SD%**	**n (stat)**
PCB-101	ng/ml	A1P		-1.67	18.0	20	15.0	18.5	18.0	1.9	10.5	6
	µg/kg	M2P		-1.63	49.6	30	37.5	47.3	49.6	7.8	15.8	8
PCB-105	ng/ml	A1P		-1.17	7.59	20	6.70	7.48	7.38	0.8	10.4	5
	µg/kg	M2P		-0.91	2.49	30	2.15	2.14	2.49	0.6	25.9	6
PCB-118	ng/ml	A1P		-1.44	18.1	20	15.5	19.1	18.5	2.0	10.8	6
	µg/kg	M2P		-2.46	15.2	30	9.6	15.7	15.2	3.4	22.2	8
PCB-138	ng/ml	A1P		-0.06	18.1	20	18.0	18.9	18.8	1.4	7.5	6
	µg/kg	M2P		-0.06	121	30	120	120	121	10.8	9.0	7
PCB-153	ng/ml	A1P		0.22	18.1	20	18.5	18.6	18.6	1.5	8.1	6
	µg/kg	M2P		-1.21	116	30	95	116	116	11.6	10.0	7
PCB-156	ng/ml	A1P		-3.28	7.59	20	5.10	7.49	7.24	1.6	22.0	5
	µg/kg	M2P		-1.94	10.5	30	7.5	11.8	10.5	2.7	26.1	6
PCB-170	ng/ml	A1P					6.25	7.33	7.16	0.8	11.7	3
	µg/kg	M2P					30.5	47.3	46.9	14.2	30.3	4
PCB-180	ng/ml	A1P		-1.39	18.0	20	15.5	19.4	19.0	2.7	14.2	6
	µg/kg	M2P		-2.13	74.9	30	51.0	74.4	74.9	15.0	20.0	8
PCB-28	ng/ml	A1P		-1.88	18.1	30	13.0	16.9	17.4	3.6	20.8	5
	µg/kg	M2P			0.104	30	*<1,0*	0.10	0.10	0.0	14.0	3
PCB-28+31	ng/ml	A1P					17.1	25.6	24.4	5.3	21.6	4
	µg/kg	M2P					*<1,0*	0.18	0.18	0.0	17.0	2
PCB-31	ng/ml	A1P					4.10	7.41	13.57	13.6	100.5	3
	µg/kg	M2P					*<1,0*	0.066	0.066	0.0	0.0	1
PCB-52	ng/ml	A1P		-3.06	18.0	20	12.5	17.9	17.2	2.6	14.9	6
	µg/kg	M2P		-2.50	1.60	30	1.00	1.60	1.60	0.3	19.8	7
SigmaPCB$_7$	ng/ml	A1P		-1.43	126	20	108	130	128	15.4	12.0	5
	µg/kg	M2P		-1.29	375	25	315	371	375	34.6	9.2	7

Participant 6												
Analyte	**Unit**	**Sample**	-3 0 3	**z score**	**Assigned value**	**2×s_{pt} %**	**Lab's result**	**Md**	**Mean**	**SD**	**SD%**	**n (stat)**
PCB-101	ng/ml	A1P		0.39	18.0	20	18.7	18.5	18.0	1.9	10.5	6
	µg/kg	M2P		-0.42	49.6	30	46.5	47.3	49.6	7.8	15.8	8
PCB-105	ng/ml	A1P		1.21	7.59	20	8.51	7.48	7.38	0.8	10.4	5
	µg/kg	M2P		-1.45	2.49	30	1.95	2.14	2.49	0.6	25.9	6
PCB-118	ng/ml	A1P		1.66	18.1	20	21.1	19.1	18.5	2.0	10.8	6
	µg/kg	M2P		-1.40	15.2	30	12.0	15.7	15.2	3.4	22.2	8
PCB-138	ng/ml	A1P		1.41	18.1	20	20.7	18.9	18.8	1.4	7.5	6
	µg/kg	M2P		0.22	121	30	125	120	121	10.8	9.0	7
PCB-153	ng/ml	A1P		1.60	18.1	20	21.0	18.6	18.6	1.5	8.1	6
	µg/kg	M2P		0.60	116	30	127	116	116	11.6	10.0	7
PCB-156	ng/ml	A1P		2.23	7.59	20	9.28	7.49	7.24	1.6	22.0	5
	µg/kg	M2P		-2.41	10.5	30	6.7	11.8	10.5	2.7	26.1	6
PCB-170	ng/ml	A1P					28.30	7.33	7.16	0.8	11.7	3
	µg/kg	M2P					54.0	47.3	46.9	14.2	30.3	4
PCB-180	ng/ml	A1P		2.50	18.0	20	22.5	19.4	19.0	2.7	14.2	6
	µg/kg	M2P		-0.17	74.9	30	73.0	74.4	74.9	15.0	20.0	8
PCB-28	ng/ml	A1P		-0.46	18.1	30	16.9	16.9	17.4	3.6	20.8	5
	µg/kg	M2P		1.03	0.104	30	0.12	0.10	0.10	0.0	14.0	3

Participant 6												
Analyte	**Unit**	**Sample**	-3 0 3	**z score**	**Assigned value**	**2×s_{pt} %**	**Lab's result**	**Md**	**Mean**	**SD**	**SD%**	**n (stat)**
PCB-28+31	ng/ml	A1P					27.0	25.6	24.4	5.3	21.6	4
	µg/kg	M2P					0.20	0.18	0.18	0.0	17.0	2
PCB-52	ng/ml	A1P		0.58	18.0	20	19.1	17.9	17.2	2.6	14.9	6
	µg/kg	M2P		-0.15	1.60	30	1.57	1.60	1.60	0.3	19.8	7
SigmaPCB$_7$	ng/ml	A1P		1.11	126	20	140	130	128	15.4	12.0	5
	µg/kg	M2P		0.21	375	25	385	371	375	34.6	9.2	7

Participant 7												
Analyte	**Unit**	**Sample**	-3 0 3	**z score**	**Assigned value**	**2×s_{pt} %**	**Lab's result**	**Md**	**Mean**	**SD**	**SD%**	**n (stat)**
PCB-101	ng/ml	A1P		3.83	18.0	20	24.9	18.5	18.0	1.9	10.5	6
	µg/kg	M2P		-0.22	49.6	30	48.0	47.3	49.6	7.8	15.8	8
PCB-105	ng/ml	A1P		-0.22	7.59	20	7.42	7.48	7.38	0.8	10.4	5
	µg/kg	M2P		2.57	2.49	30	3.45	2.14	2.49	0.6	25.9	6
PCB-118	ng/ml	A1P		2.65	18.1	20	22.9	19.1	18.5	2.0	10.8	6
	µg/kg	M2P		2.11	15.2	30	20.0	15.7	15.2	3.4	22.2	8
PCB-138	ng/ml	A1P		5.86	18.1	20	28.7	18.9	18.8	1.4	7.5	6
	µg/kg	M2P		1.07	121	30	141	120	121	10.8	9.0	7
PCB-153	ng/ml	A1P		5.52	18.1	20	28.1	18.6	18.6	1.5	8.1	6
	µg/kg	M2P		-0.26	116	30	112	116	116	11.6	10.0	7
PCB-156	ng/ml	A1P		0.80	7.59	20	8.20	7.49	7.24	1.6	22.0	5
	µg/kg	M2P		1.17	10.5	30	12.4	11.8	10.5	2.7	26.1	6
PCB-180	ng/ml	A1P		5.39	18.0	20	27.7	19.4	19.0	2.7	14.2	6
	µg/kg	M2P		0.08	74.9	30	75.9	74.4	74.9	15.0	20.0	8
PCB-28+31	ng/ml	A1P					35.5	25.6	24.4	5.3	21.6	4
	µg/kg	M2P					*<1*	0.18	0.18	0.0	17.0	2
PCB-52	ng/ml	A1P		2.11	18.0	20	21.8	17.9	17.2	2.6	14.9	6
	µg/kg	M2P			1.60	30	*<3*	1.60	1.60	0.3	19.8	7
SigmaPCB$_7$	ng/ml	A1P		6.27	126	20	205	130	128	15.4	12.0	5
	µg/kg	M2P		0.79	375	25	412	371	375	34.6	9.2	7

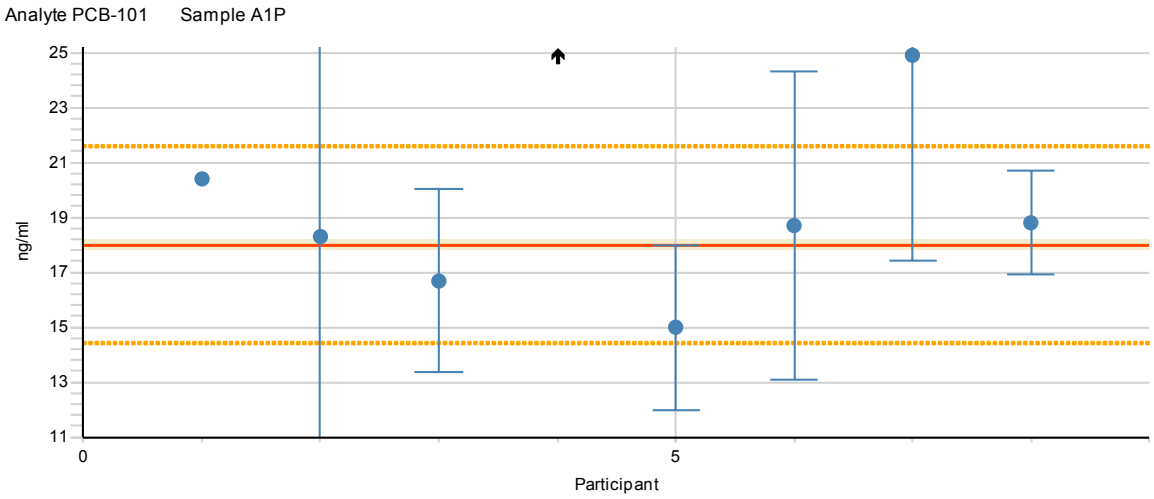
Participant 8												
Analyte	**Unit**	**Sample**	-3 0 3	**z score**	**Assigned value**	**2×s_{pt} %**	**Lab's result**	**Md**	**Mean**	**SD**	**SD%**	**n (stat)**
PCB-101	ng/ml	A1P		0.44	18.0	20	18.8	18.5	18.0	1.9	10.5	6
	µg/kg	M2P		1.19	49.6	30	58.5	47.3	49.6	7.8	15.8	8
PCB-105	ng/ml	A1P		-0.03	7.59	20	7.57	7.48	7.38	0.8	10.4	5
	µg/kg	M2P		-0.95	2.49	30	2.14	2.14	2.49	0.6	25.9	6
PCB-110	ng/ml	A1P					7.69	7.45	7.45	0.3	4.5	2
	µg/kg	M2P					38.4	38.1	38.1	0.4	0.9	2
PCB-118	ng/ml	A1P		0.52	18.1	20	19.1	19.1	18.5	2.0	10.8	6
	µg/kg	M2P		1.29	15.2	30	18.2	15.7	15.2	3.4	22.2	8
PCB-138	ng/ml	A1P		0.08	18.1	20	18.3	18.9	18.8	1.4	7.5	6
	µg/kg	M2P		-0.14	121	30	119	120	121	10.8	9.0	7
PCB-153	ng/ml	A1P		-0.25	18.1	20	17.7	18.6	18.6	1.5	8.1	6
	µg/kg	M2P		0.80	116	30	130	116	116	11.6	10.0	7
PCB-156	ng/ml	A1P		0.49	7.59	20	7.97	7.49	7.24	1.6	22.0	5
	µg/kg	M2P		1.56	10.5	30	13.0	11.8	10.5	2.7	26.1	6
PCB-170	ng/ml	A1P					7.90	7.33	7.16	0.8	11.7	3
	µg/kg	M2P					62.5	47.3	46.9	14.2	30.3	4

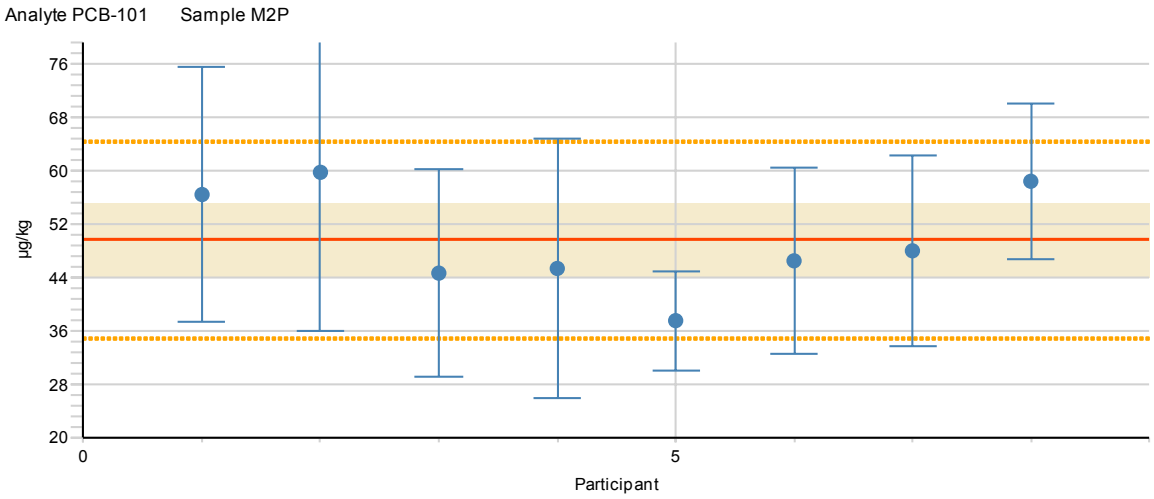
Participant 8												
Analyte	**Unit**	**Sample**	-3 0 3	**z score**	**Assigned value**	**2×s_{pt} %**	**Lab's result**	**Md**	**Mean**	**SD**	**SD%**	**n (stat)**
PCB-180	ng/ml	A1P		0.92	18.0	20	19.7	19.4	19.0	2.7	14.2	6
	µg/kg	M2P		1.21	74.9	30	88.5	74.4	74.9	15.0	20.0	8
PCB-28	ng/ml	A1P		-0.20	18.1	30	17.6	16.9	17.4	3.6	20.8	5
	µg/kg	M2P		-0.13	0.104	30	0.10	0.10	0.10	0.0	14.0	3
PCB-52	ng/ml	A1P		0.69	18.0	20	19.3	17.9	17.2	2.6	14.9	6
	µg/kg	M2P		0.90	1.60	30	1.82	1.60	1.60	0.3	19.8	7
SigmaPCB$_7$	ng/ml	A1P		0.32	126	20	130	130	128	15.4	12.0	5
	µg/kg	M2P		0.86	375	25	416	371	375	34.6	9.2	7

APPENDIX 8: Results of participants and their uncertainties

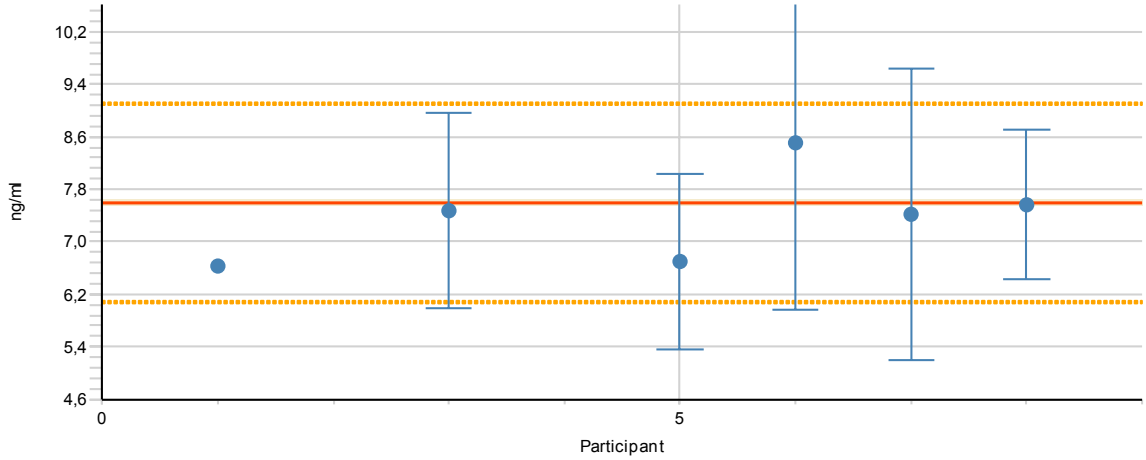
In figures:

- The dashed lines describe the standard deviation for the proficiency assessment, the red solid line shows the assigned value, the shaded area describes the expanded measurement uncertainty of the assigned value, and the arrow describes the value outside the scale.

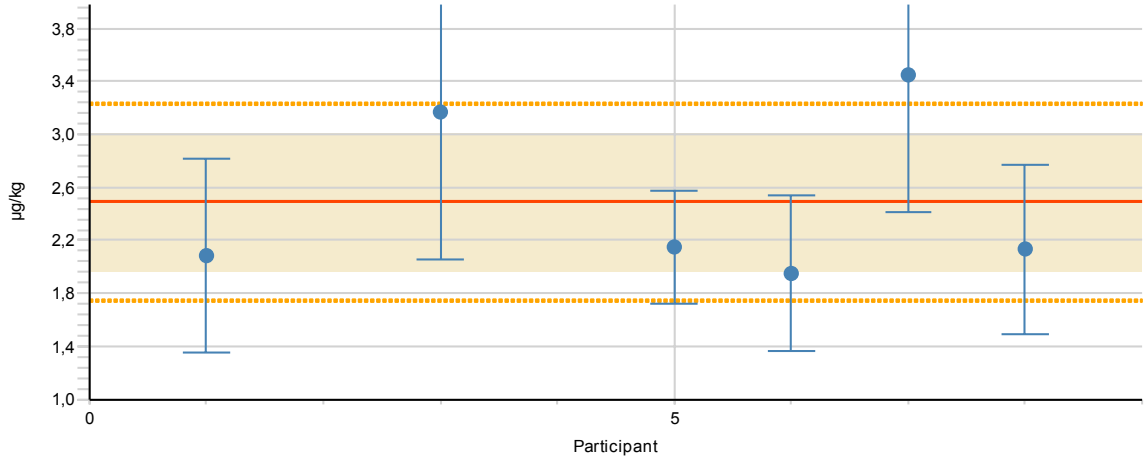




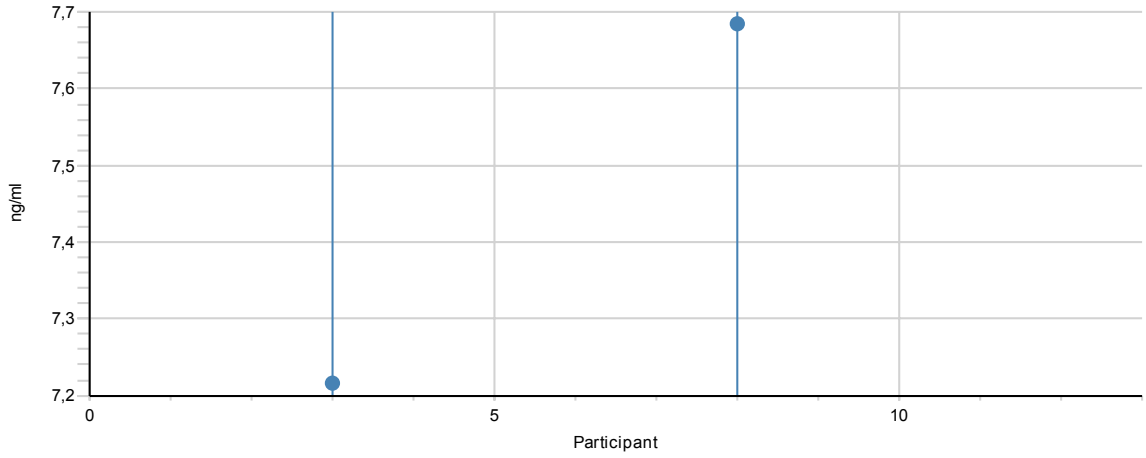
Analyte PCB-105 Sample A1P



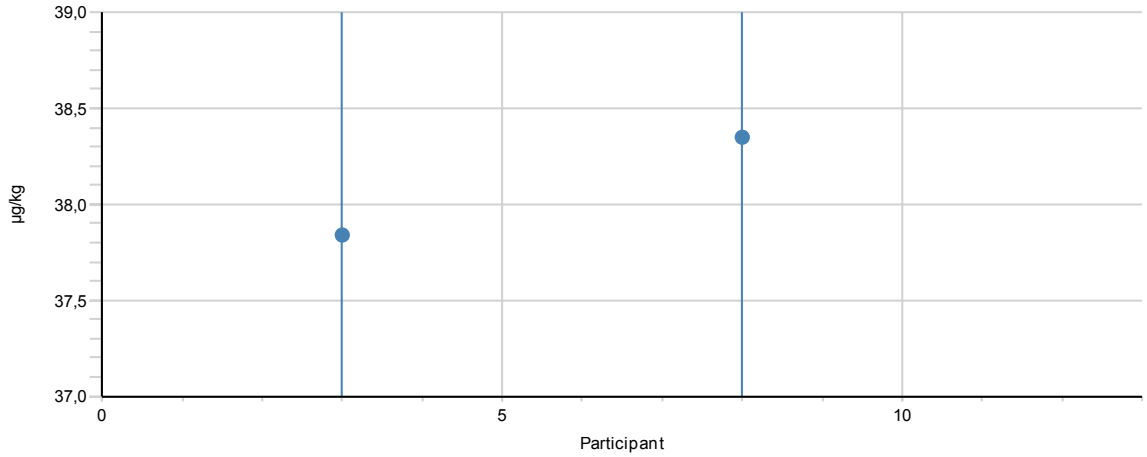
Analyte PCB-105 Sample M2P



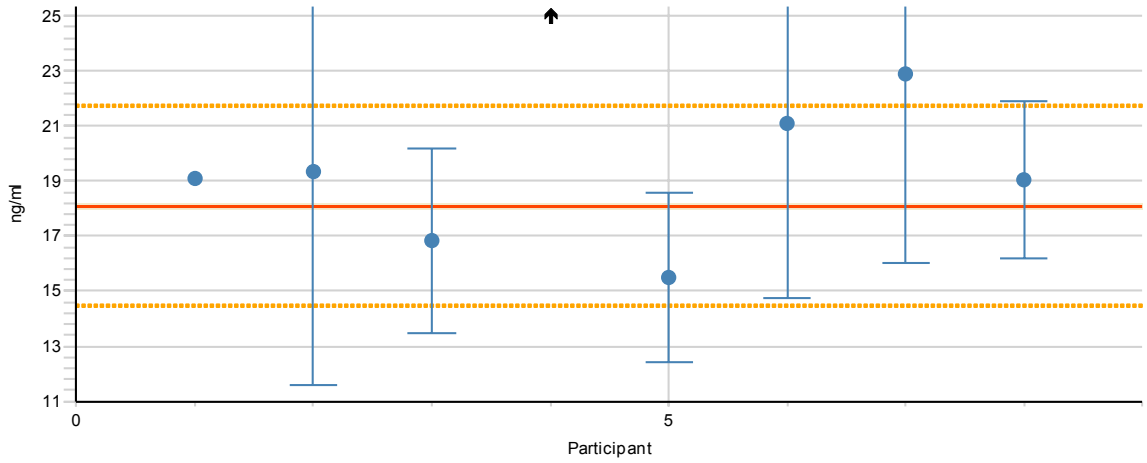
Analyte PCB-110 Sample A1P



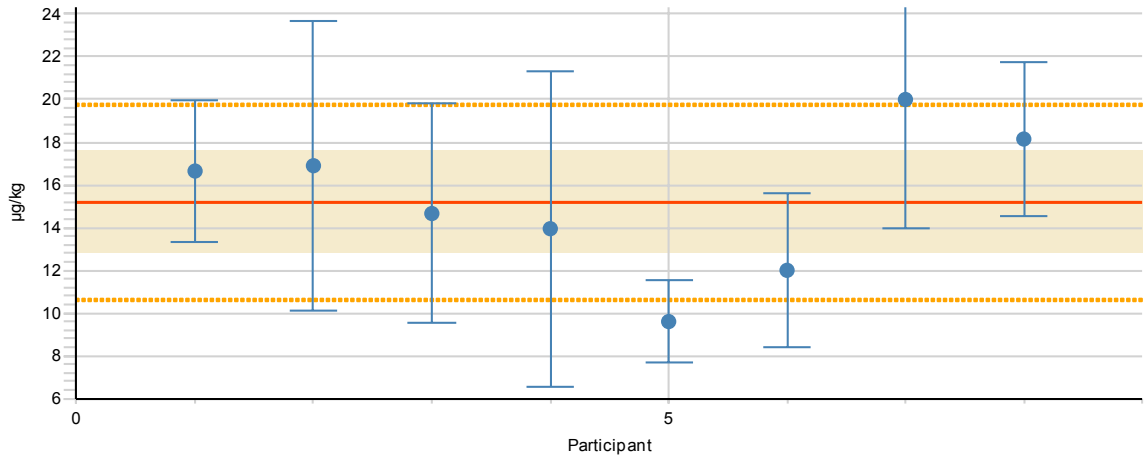
Analyte PCB-110 Sample M2P



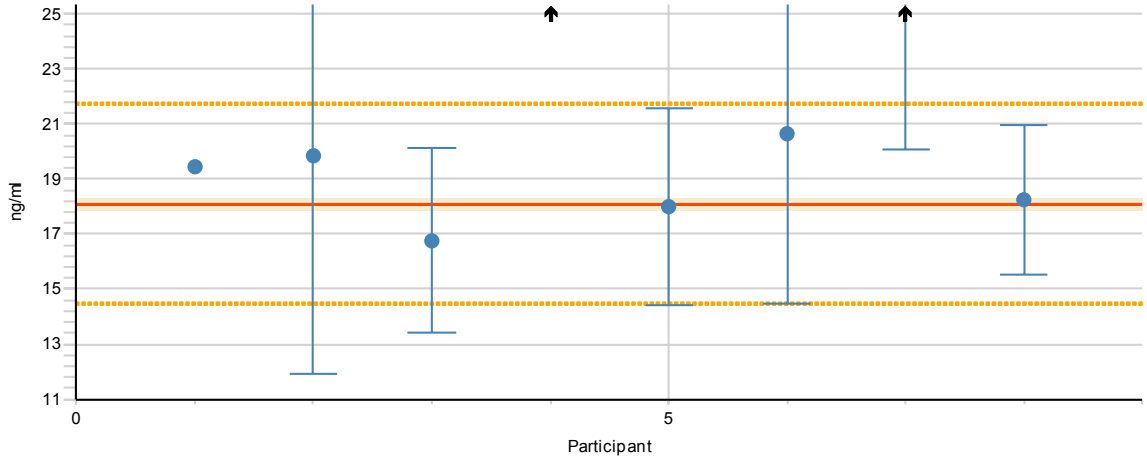
Analyte PCB-118 Sample A1P



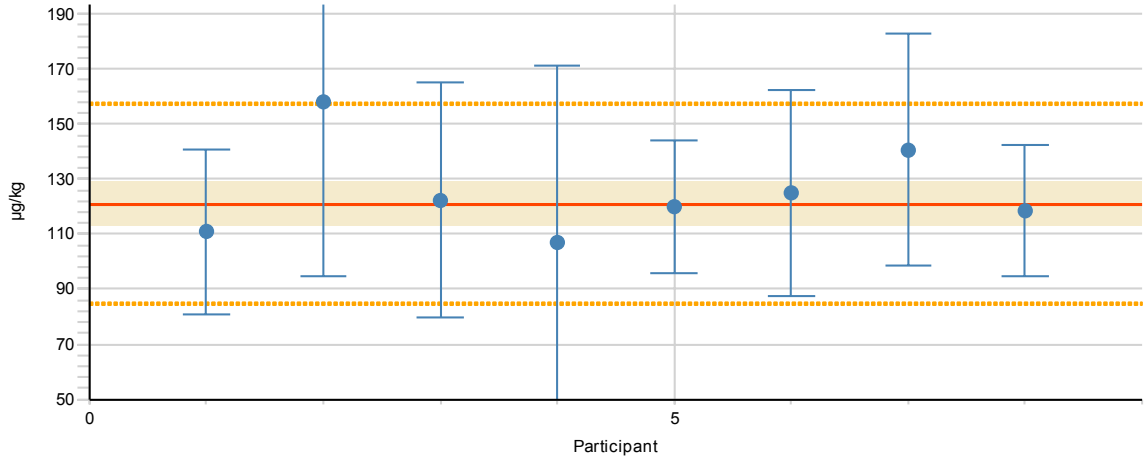
Analyte PCB-118 Sample M2P



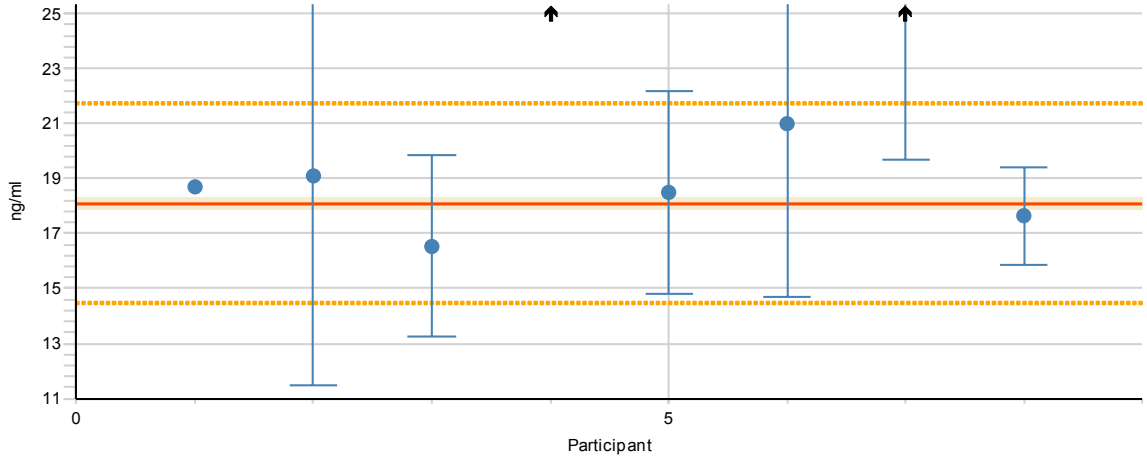
Analyte PCB-138 Sample A1P



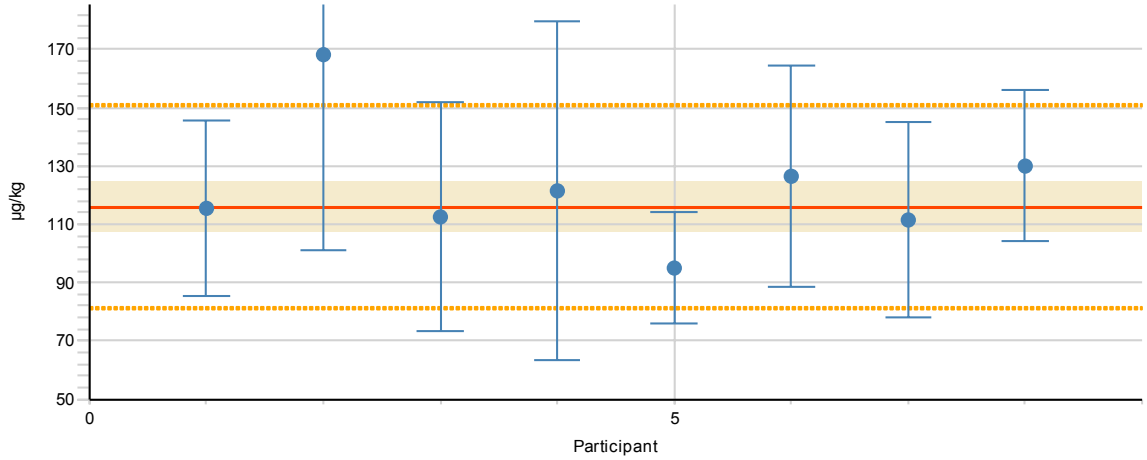
Analyte PCB-138 Sample M2P



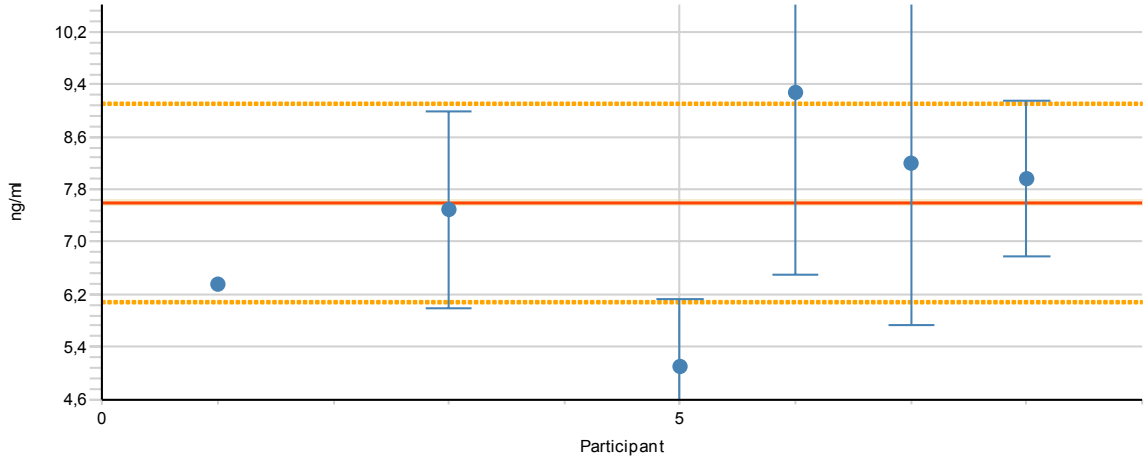
Analyte PCB-153 Sample A1P



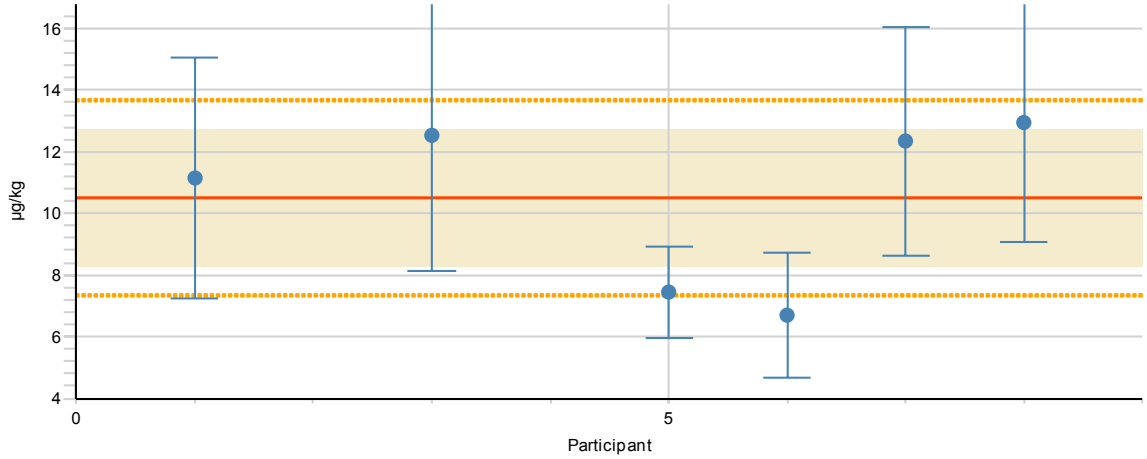
Analyte PCB-153 Sample M2P



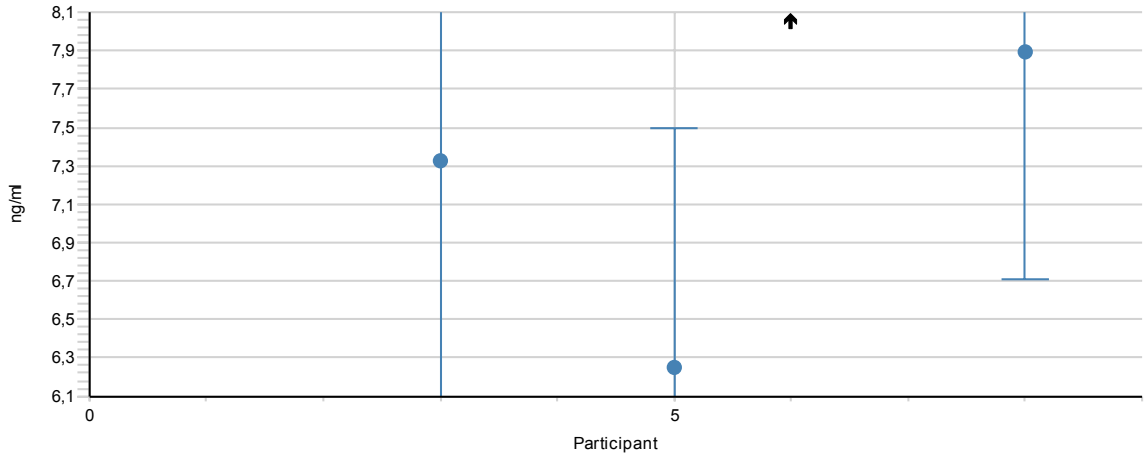
Analyte PCB-156 Sample A1P



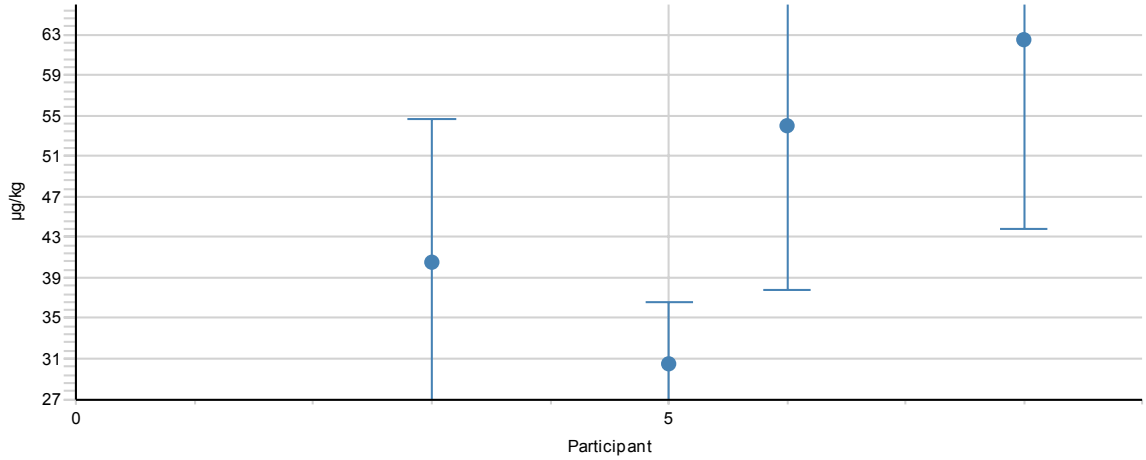
Analyte PCB-156 Sample M2P



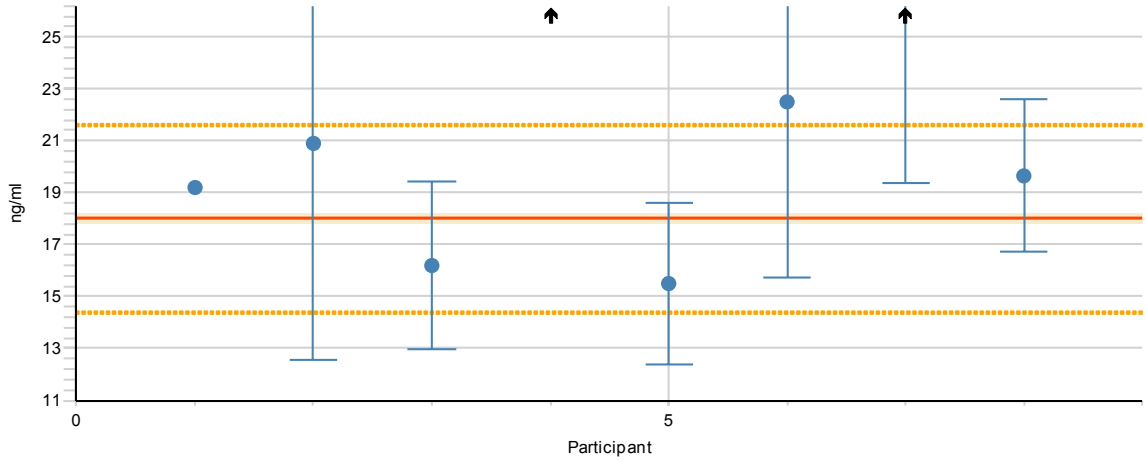
Analyte PCB-170 Sample A1P



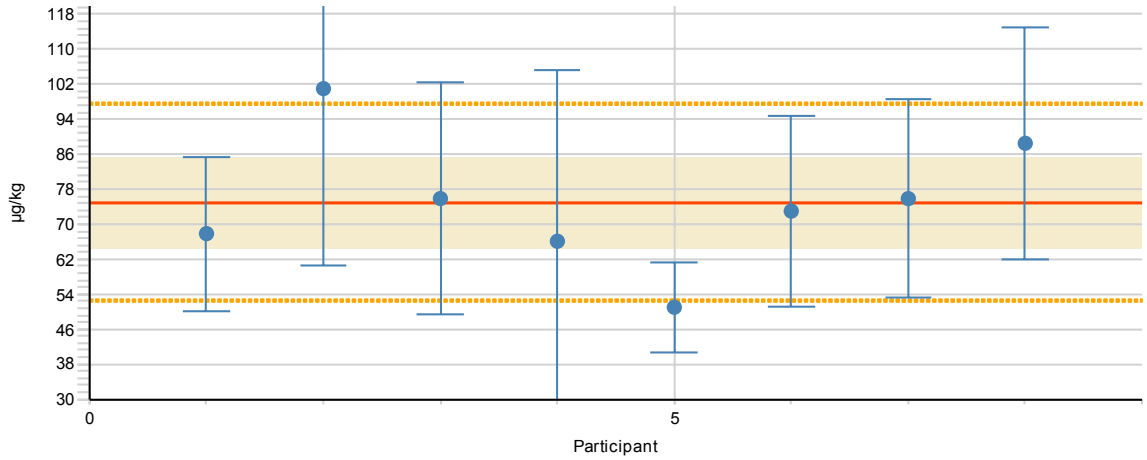
Analyte PCB-170 Sample M2P



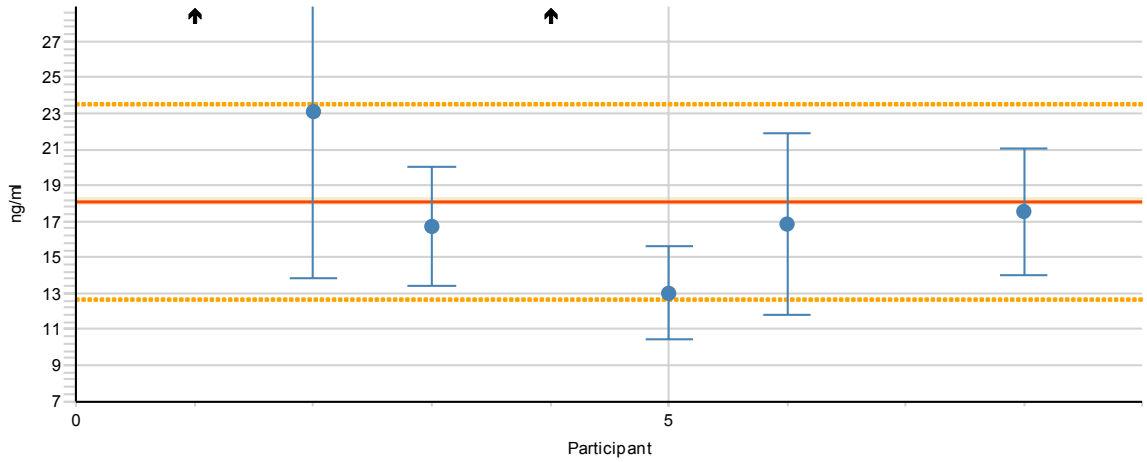
Analyte PCB-180 Sample A1P



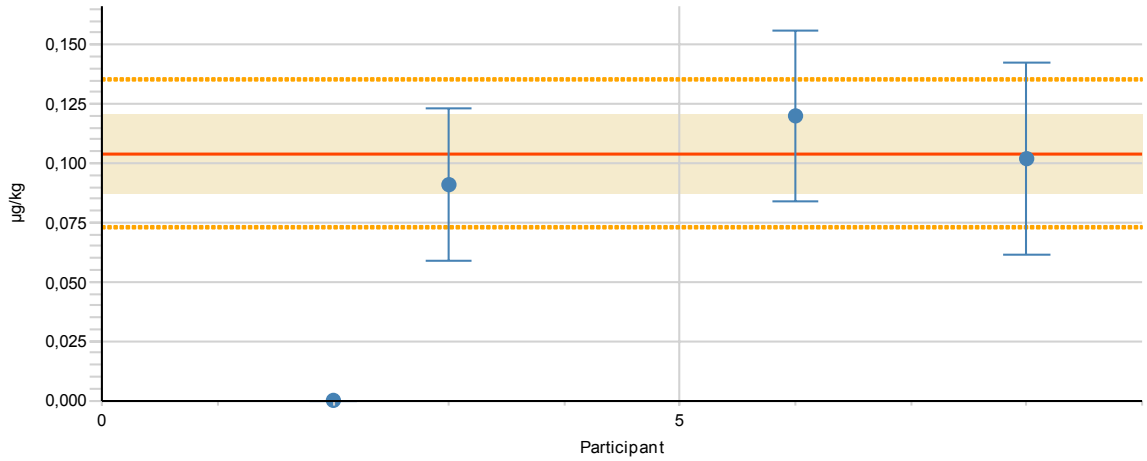
Analyte PCB-180 Sample M2P



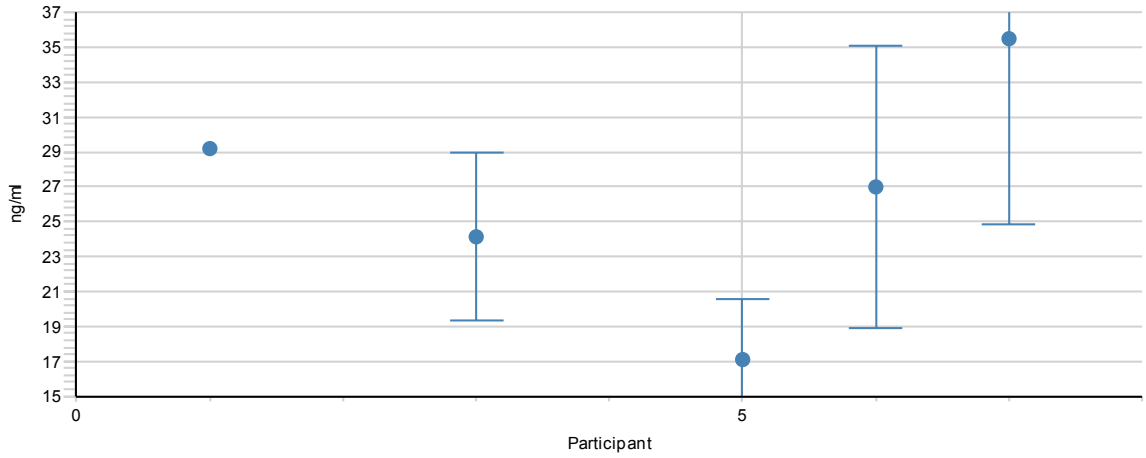
Analyte PCB-28 Sample A1P



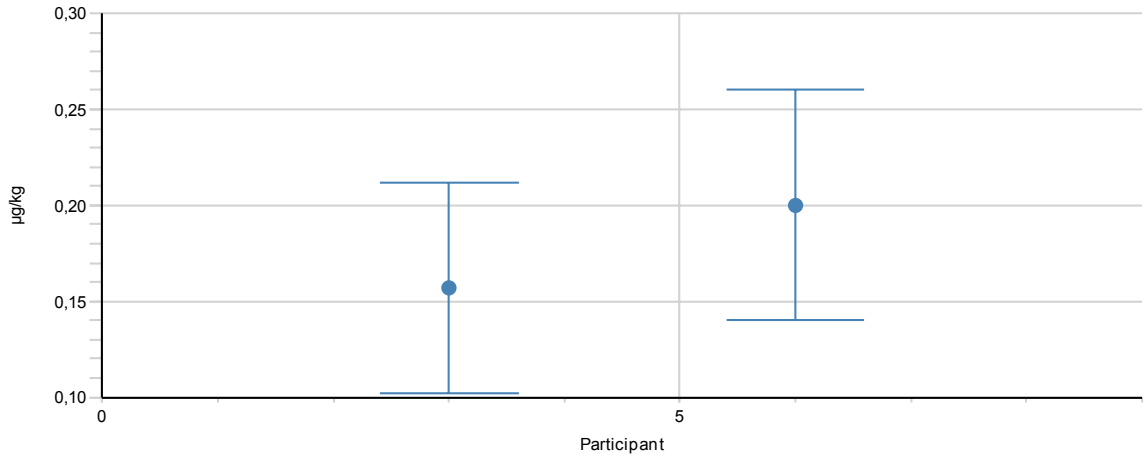
Analyte PCB-28 Sample M2P



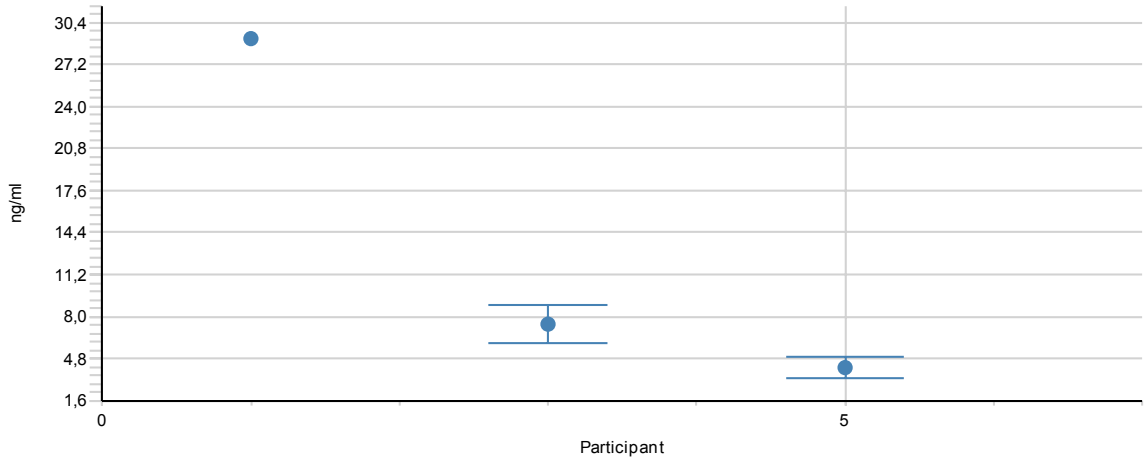
Analyte PCB-28+31 Sample A1P



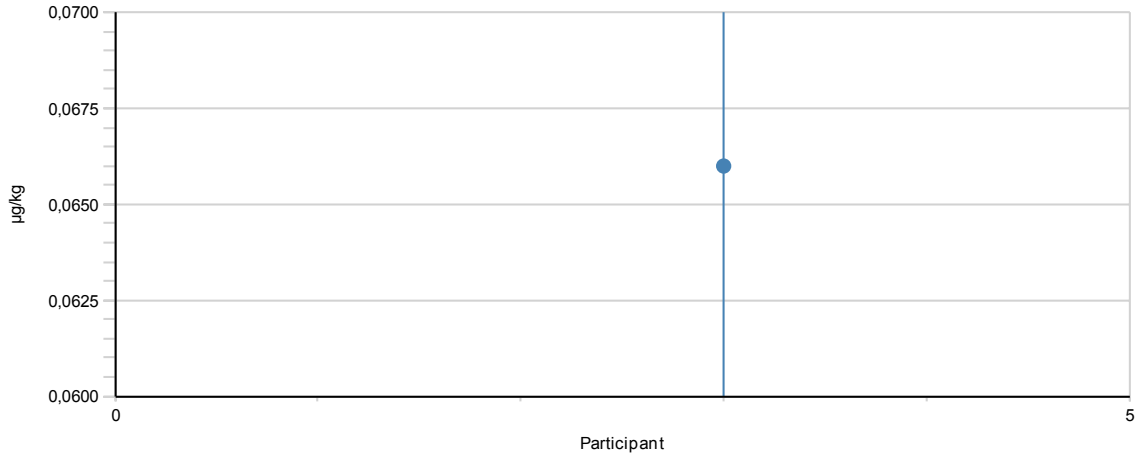
Analyte PCB-28+31 Sample M2P



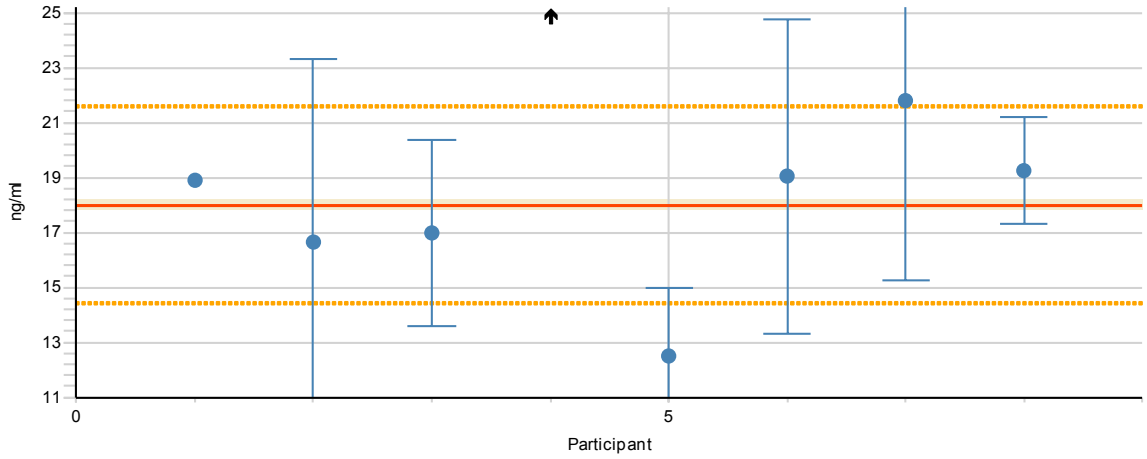
Analyte PCB-31 Sample A1P



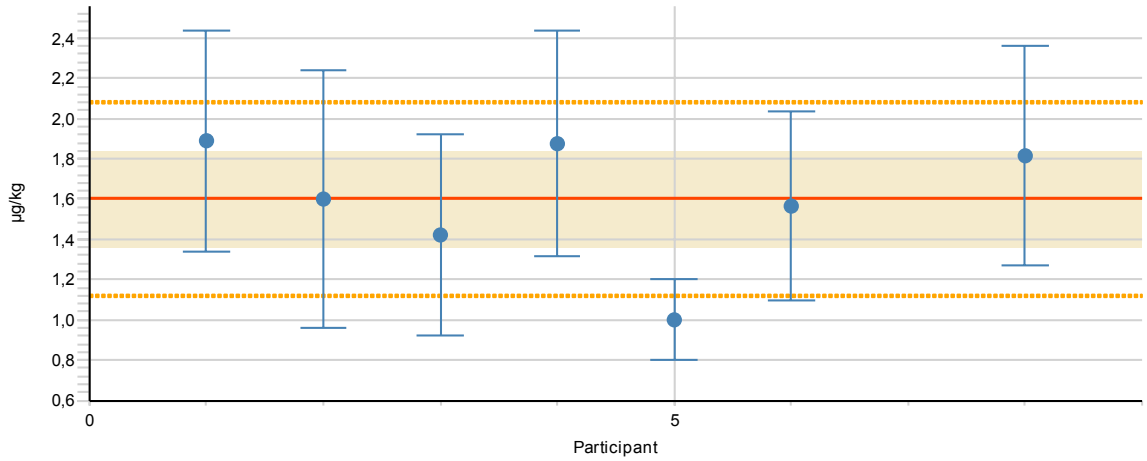
Analyte PCB-31 Sample M2P

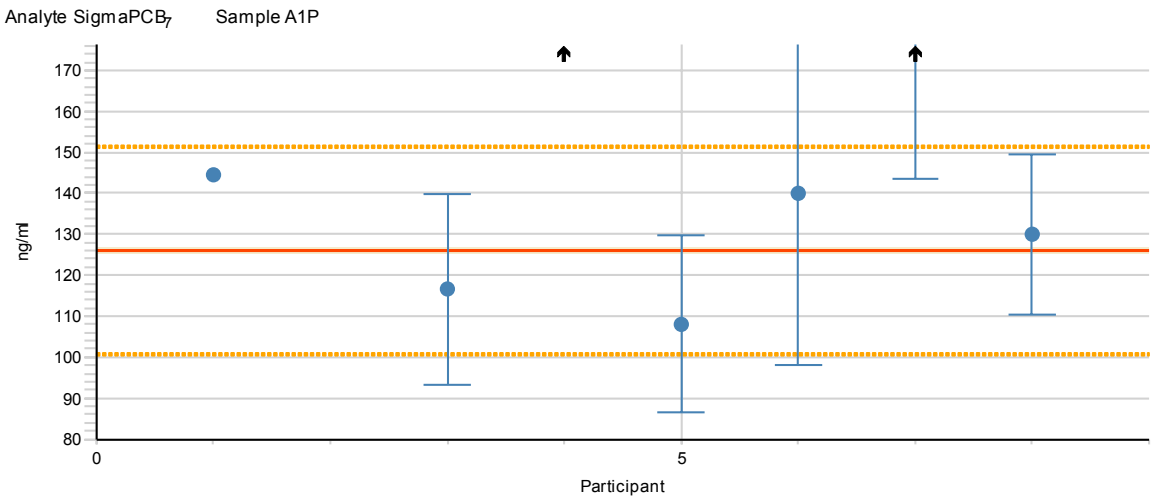


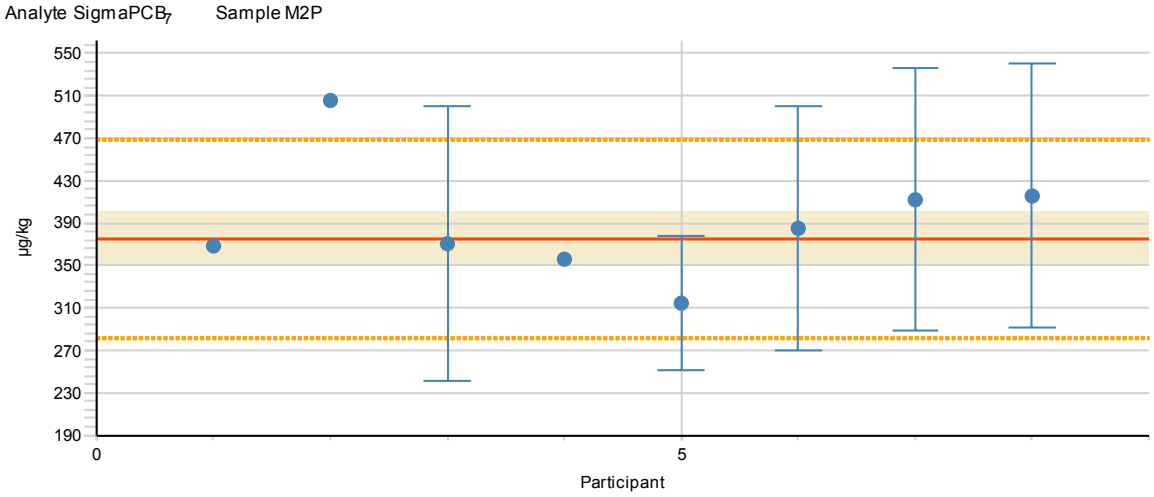
Analyte PCB-52 Sample A1P



Analyte PCB-52 Sample M2P




Analyte SigmaPCB7
Sample A1P
ng/ml
170
160
150
140
130
120
110
100
90
80
0
5
Participant


Analyte SigmaPCB7
Sample M2P
µg/kg
550
510
470
430
390
350
310
270
230
190
0
5
Participant

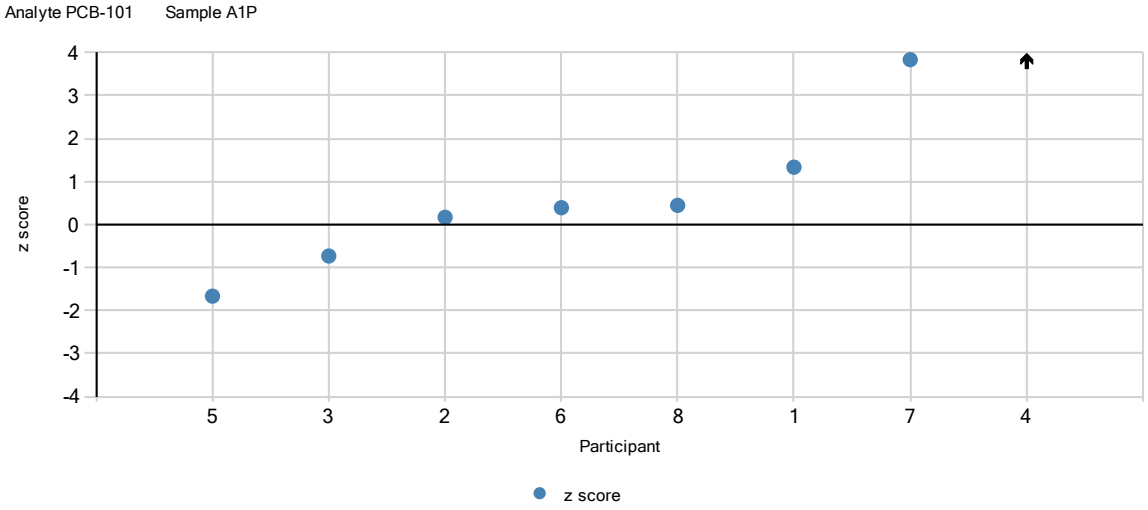
APPENDIX 9: Summary of the z scores

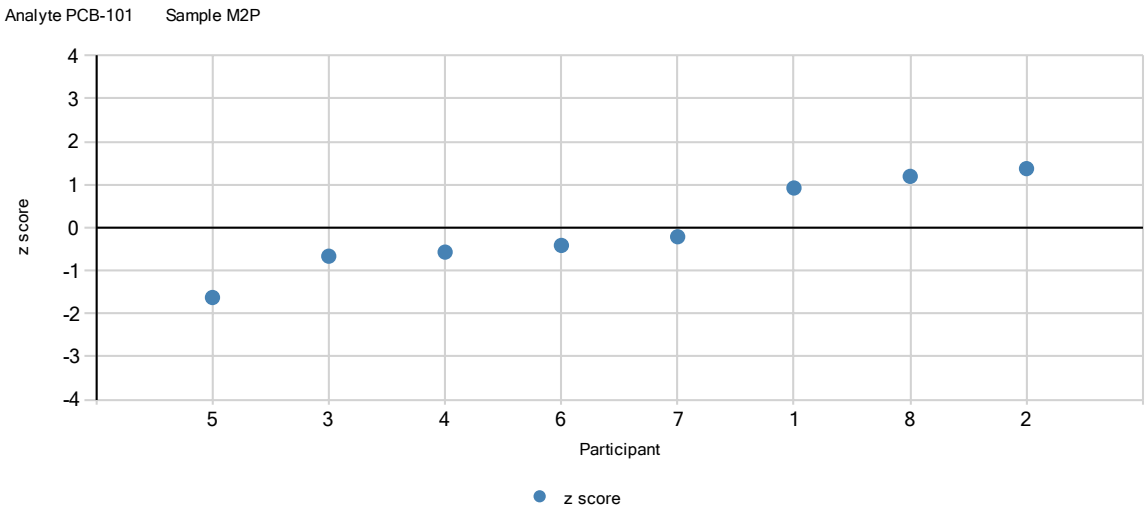
Analyte	Sample	1	2	3	4	5	6	7	8		%
PCB-101	A1P	**S**	**S**	**S**	**U**	*S*	*S*	**U**	**S**		75.0
	M2P	**S**	**S**	**S**	**S**	**S**	**S**	**S**	**S**		100
PCB-105	A1P	**S**	.	**S**	.	*S*	*S*	**S**	**S**		100
	M2P	**S**	.	**S**	.	**S**	*S*	**Q**	**S**		83.3
PCB-110	A1P	.	.	.	.	.	.	.	.		
	M2P	.	.	.	.	.	.	.	.		
PCB-118	A1P	**S**	**S**	**S**	**U**	*S*	*S*	**Q**	**S**		75.0
	M2P	**S**	**S**	**S**	**S**	**q**	**S**	**Q**	**S**		75.0
PCB-138	A1P	**S**	**S**	**S**	**U**	*S*	*S*	**U**	**S**		75.0
	M2P	**S**	**Q**	**S**	**S**	**S**	**S**	**S**	**S**		87.5
PCB-153	A1P	**S**	**S**	**S**	**U**	*S*	*S*	**U**	**S**		75.0
	M2P	**S**	**U**	**S**	**S**	**S**	**S**	**S**	**S**		87.5
PCB-156	A1P	**S**	.	**S**	.	*u*	*Q*	**S**	**S**		66.7
	M2P	**S**	.	**S**	.	**S**	*q*	**S**	**S**		83.3
PCB-170	A1P	.	.	.	.	.	.	.	.		
	M2P	.	.	.	.	.	.	.	.		
PCB-180	A1P	**S**	**S**	**S**	**U**	*S*	*Q*	**U**	**S**		62.5
	M2P	**S**	**Q**	**S**	**S**	**q**	**S**	**S**	**S**		75.0
PCB-28	A1P	**U**	**S**	**S**	**U**	*S*	*S*	.	**S**		71.4
	M2P	.	**u**	**S**	.	.	**S**	.	**S**		75.0
PCB-28+31	A1P	.	.	.	.	.	.	.	.		
	M2P	.	.	.	.	.	.	.	.		
PCB-31	A1P	.	.	.	.	.	.	.	.		
	M2P	.	.	.	.	.	.	.	.		
PCB-52	A1P	**S**	**S**	**S**	**U**	*u*	*S*	**Q**	**S**		62.5
	M2P	**S**	**S**	**S**	**S**	**q**	**S**	.	**S**		85.7
SigmaPCB$_7$	A1P	**S**	.	**S**	*U*	*S*	*S*	**U**	**S**		71.4
	M2P	**S**	**Q**	**S**	*S*	**S**	**S**	**S**	**S**		87.5
%		95	67	100	47	74	85	47	100		
accredited		19	15	20	13	9	8	17	20		

S - satisfactory (-2 ≤ z ≤ 2), Q - questionable (2 < z < 3), q - questionable (-3 < z < -2),
U - unsatisfactory (z ≥ 3), u - unsatisfactory (z ≤ -3), and z'-scores, respectively
bold - accredited, *italics - non-accredited*, normal - other
% - percentage of satisfactory results

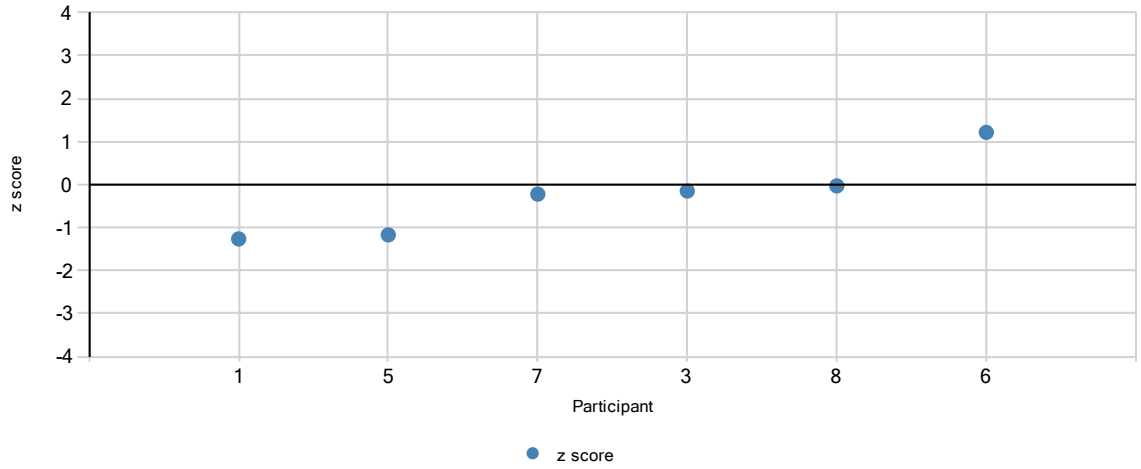
Totally satisfactory, % in all: 79 % in accredited: 79 % in non-accredited: 75

APPENDIX 10: z scores in ascending order

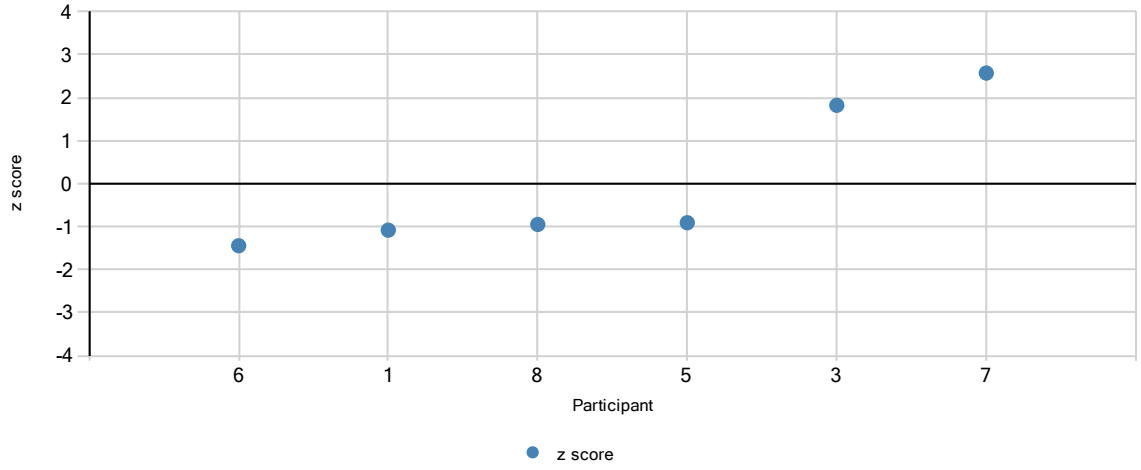




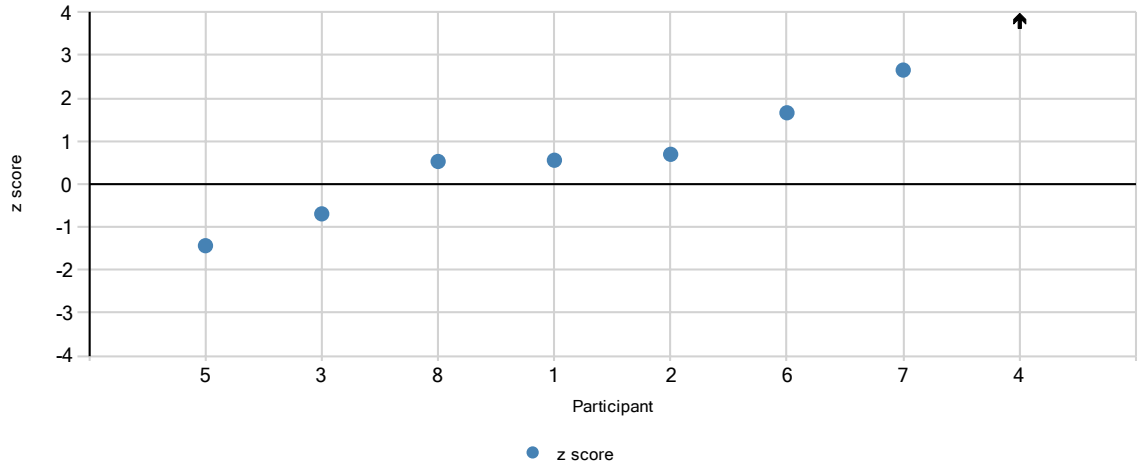
Analyte PCB-105 Sample A1P



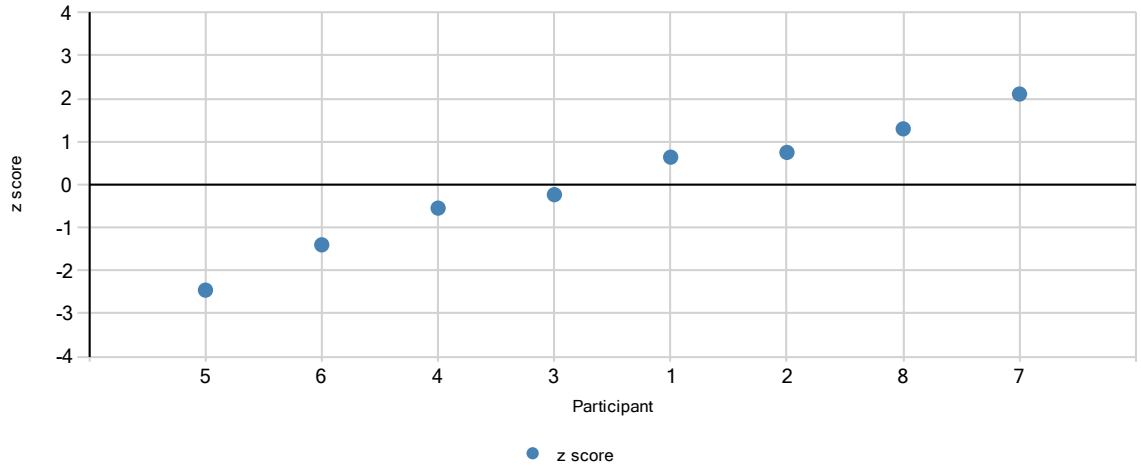
Analyte PCB-105 Sample M2P



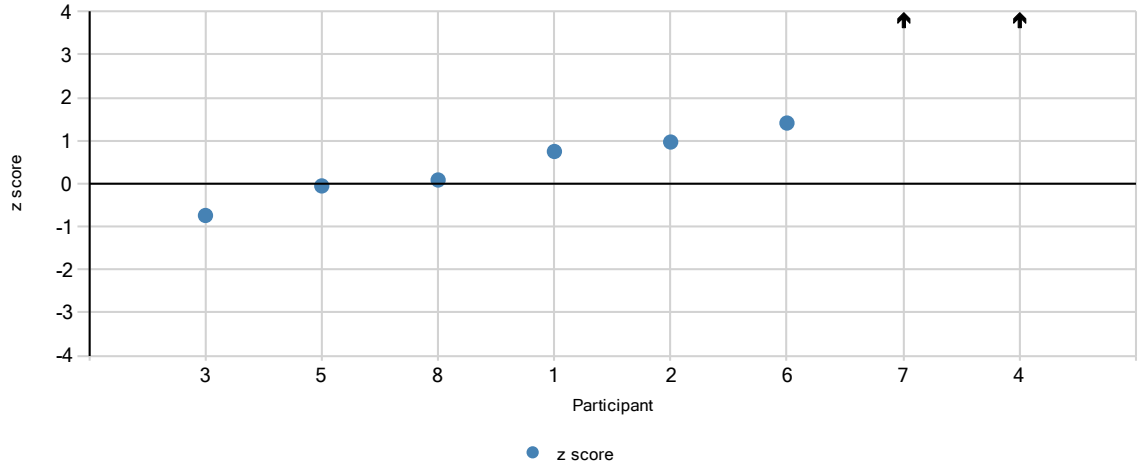
Analyte PCB-118 Sample A1P



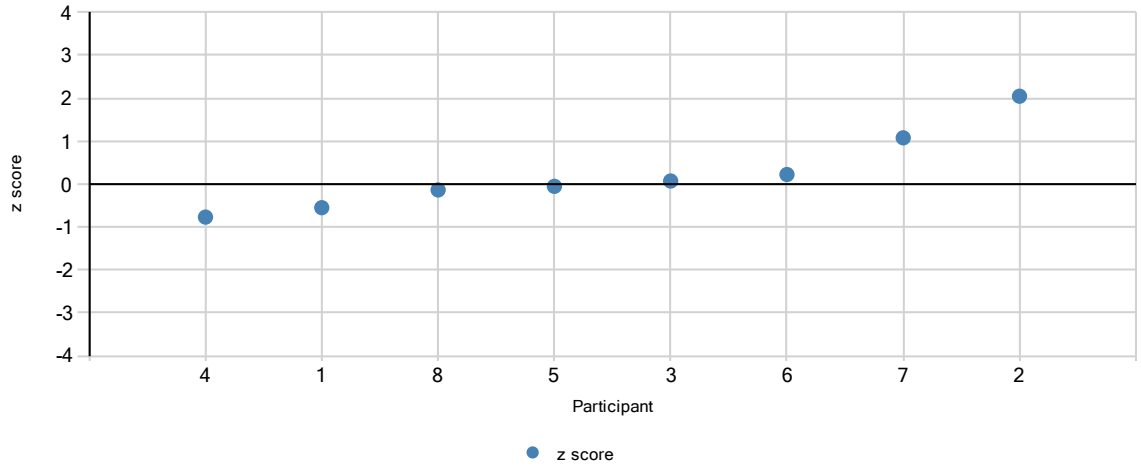
Analyte PCB-118 Sample M2P



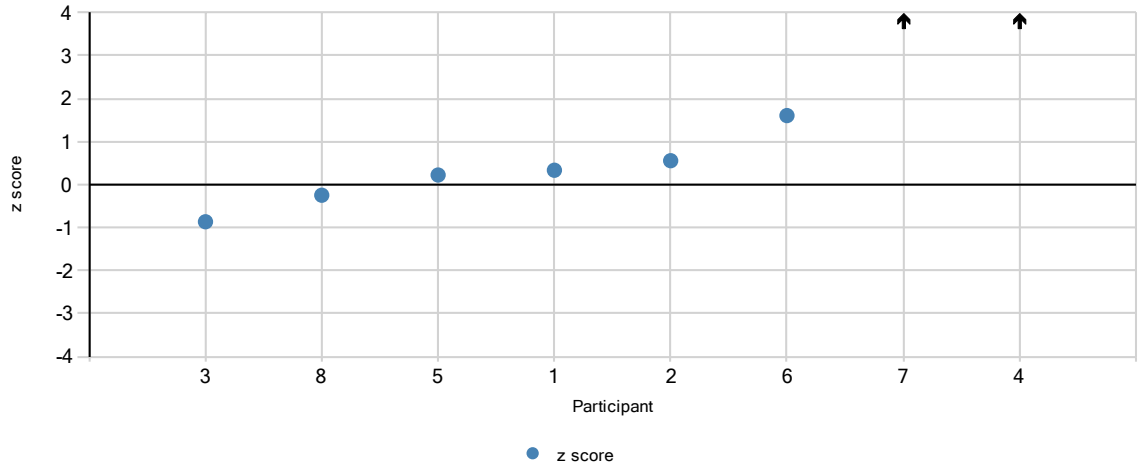
Analyte PCB-138 Sample A1P



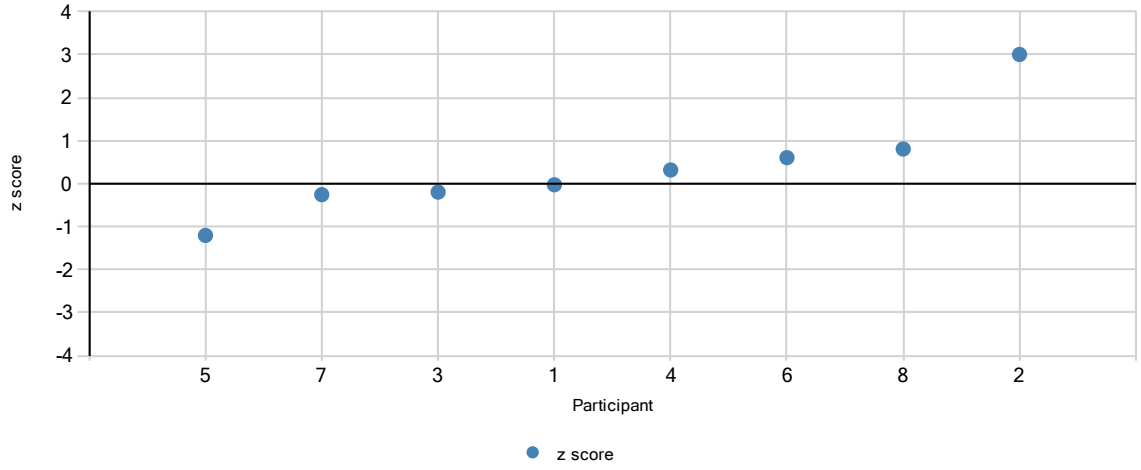
Analyte PCB-138 Sample M2P



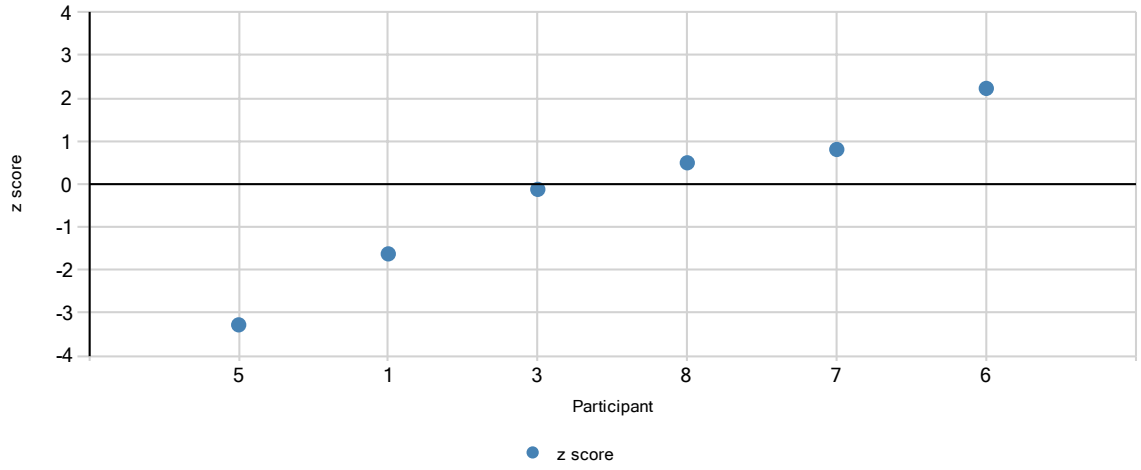
Analyte PCB-153 Sample A1P



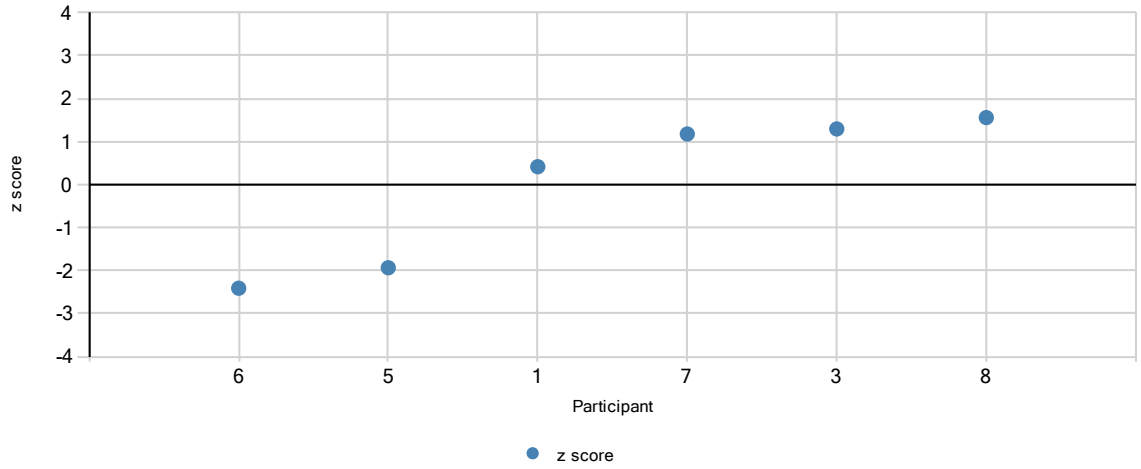
Analyte PCB-153 Sample M2P



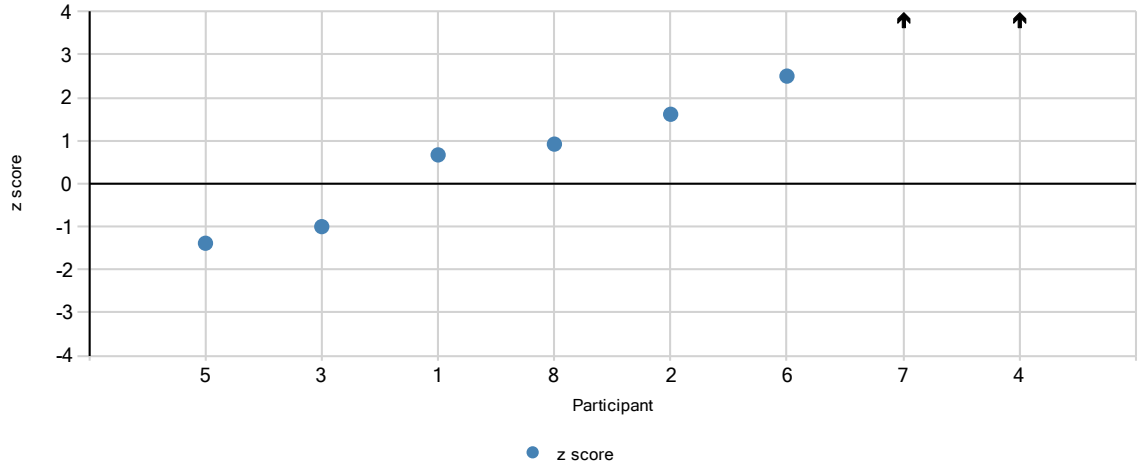
Analyte PCB-156 Sample A1P



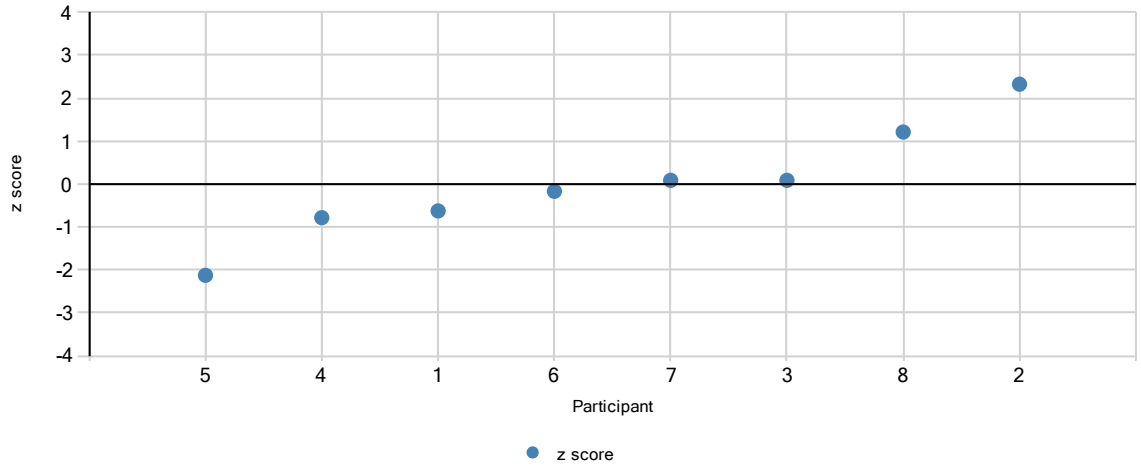
Analyte PCB-156 Sample M2P



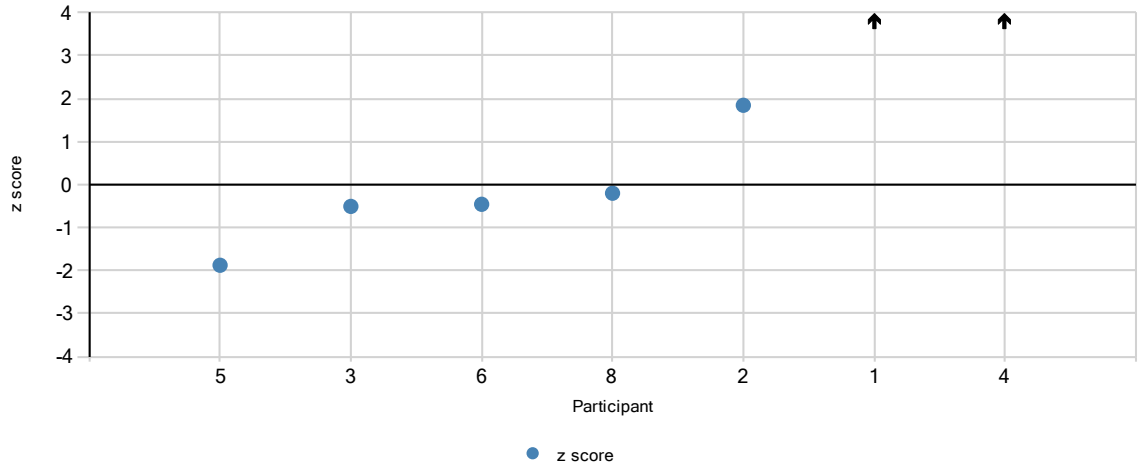
Analyte PCB-180 Sample A1P



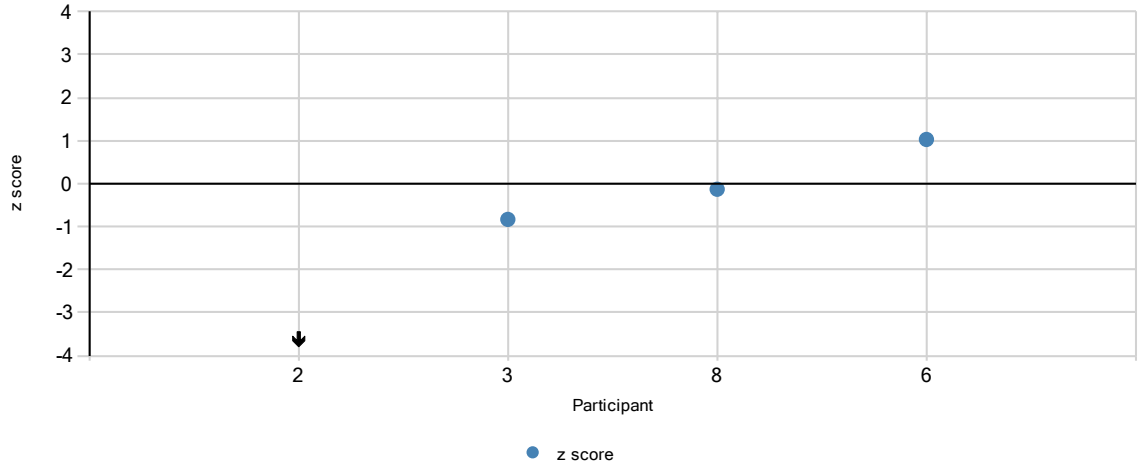
Analyte PCB-180 Sample M2P



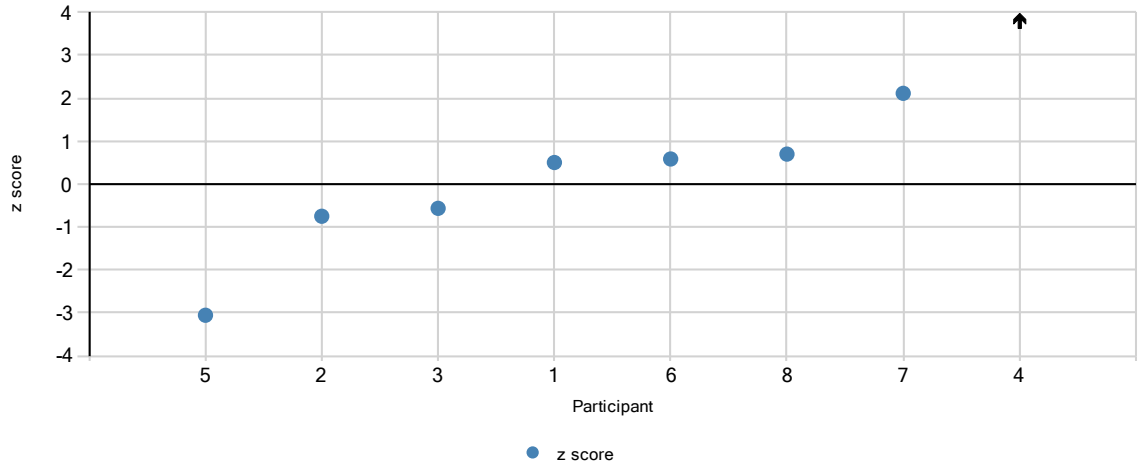
Analyte PCB-28 Sample A1P



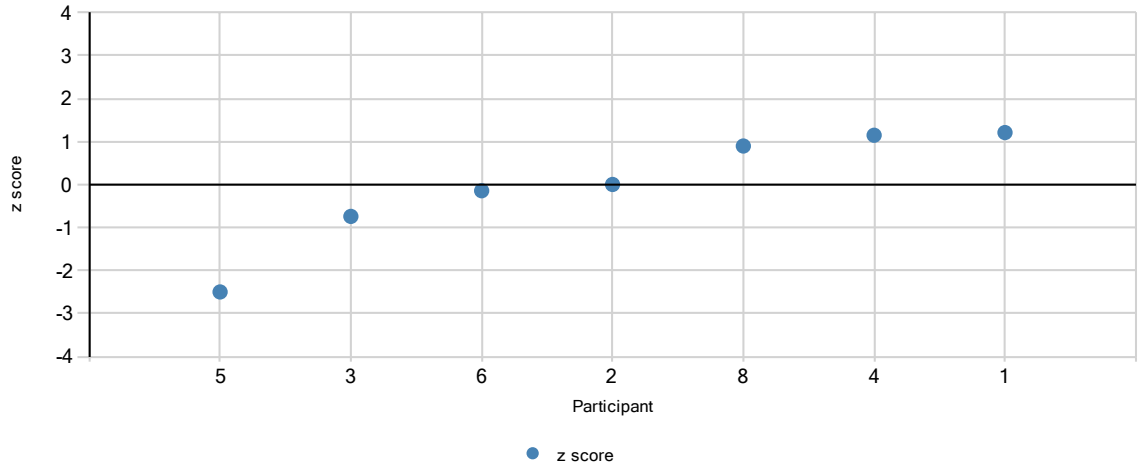
Analyte PCB-28 Sample M2P



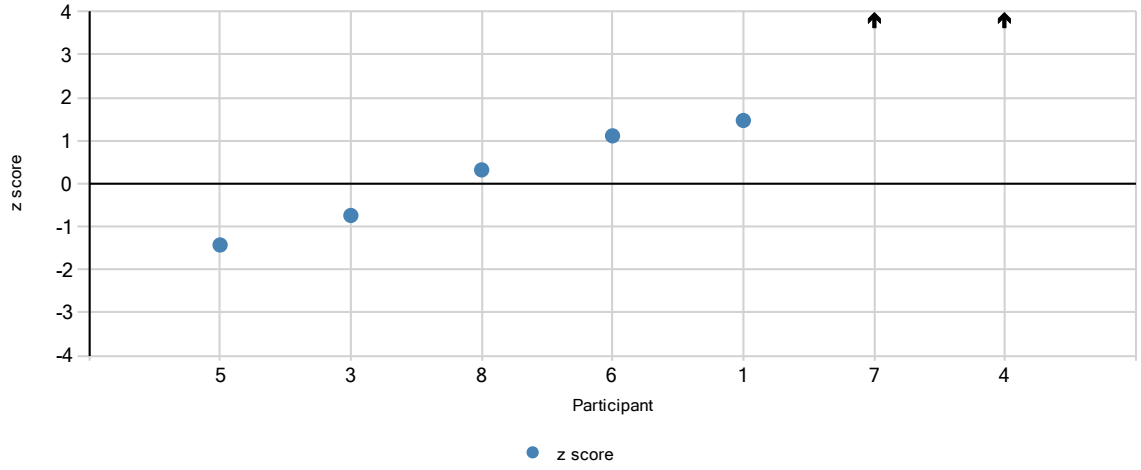
Analyte PCB-52 Sample A1P



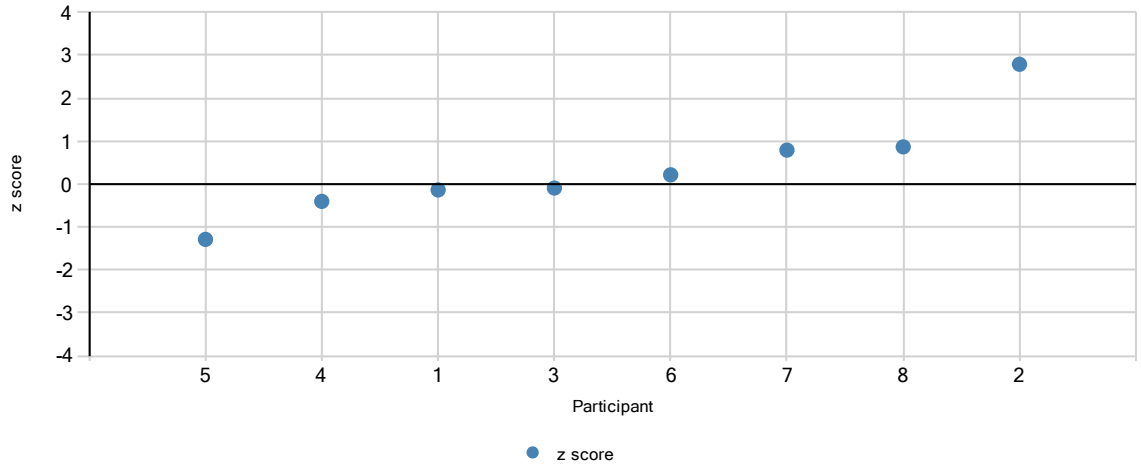
Analyte PCB-52 Sample M2P



Analyte SigmaPCB$_7$ Sample A1P



Analyte SigmaPCB$_7$ Sample M2P



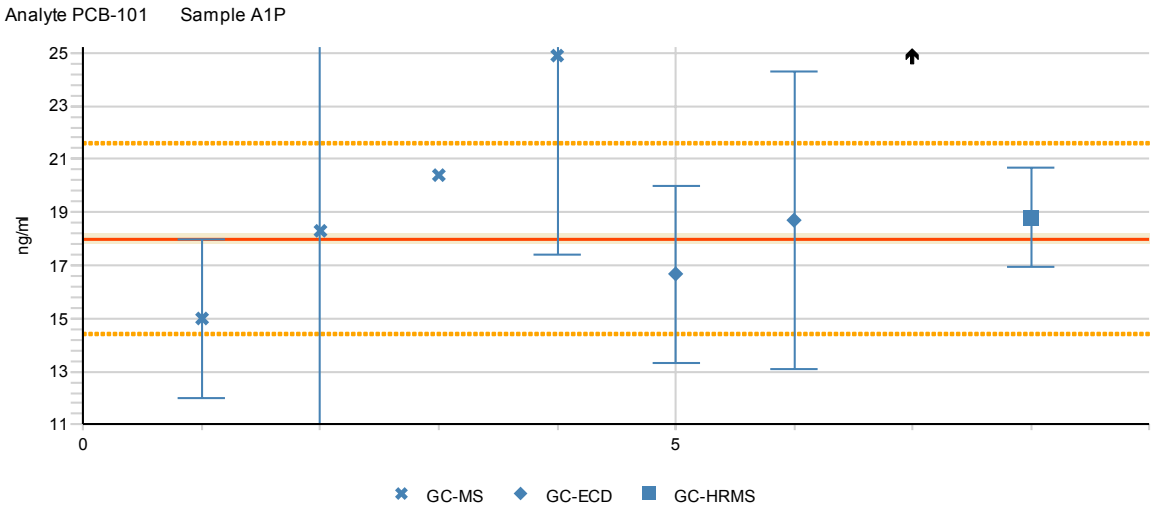
APPENDIX 11: Analytical methods

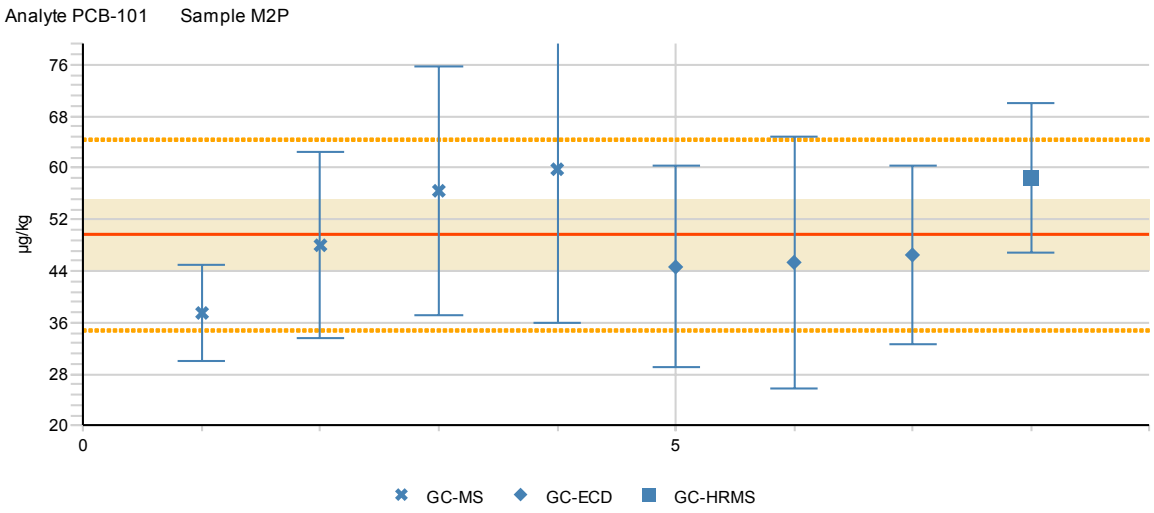
Soil – M2P

Participant	Reference	Sample amount [g]	Extraction	Injection	Column
1	Mod. Nordtest Techn Report 329, Mod. EPA 1668 method	5	Shaking, toluene 10 ml / ASE, toluene 40 ml	Splitless, 1 µl	GC-MS: 60m×0.25mm×0.25µm GC-HRMS: 40 m×0.18mm×0.18µm
3	In house -method	10	ASE, dichloromethane 40 ml	Splitless, 1 µl	CG-ECD: HP-5 60 m×0.25mm×0.25µm DB-1701: 60m×0.25m×0.25µm
4	ISO 10382	3 – 5	Ultrasound, 10 ml acetone, 5 ml hexane	Splitless, 1 µl	GC-ECD: (2 columns) 60 m×0.25mm×0.25µm
5	EPA 8082	10	Shaking, hexane 5 ml + 0.1 M Na-pyrophosphate 5 ml	Splitless, 2 µl	GC-MS: 30m×0.25mm×0.25µm
6	Internal method, based on ISO 10383:2002 and SFS-EN 15308:2008	5	Ultrasound, mixture of petroleum ether, acetone, hexane, ether, V = 40 ml	Splitless, 1 µl	GC-ECD: HP5 30m×0.32mm×0.25µm HP 50+ 30m×0.32mm×0.25µm
7	ISO 10382:2002	20	Shaking, solvent mixture, V = 60 ml	Splitless, 1 µl	GC-MS: HP5-MSUI 50m×0.25mm×0.25µm
8	In house –method	5	Soxhlet, 300 ml toluene-ethanol mixture (70/30 v/v)	Splitless, 1 µl	GC-HRMS: 60m×0.25mm×0.25µm

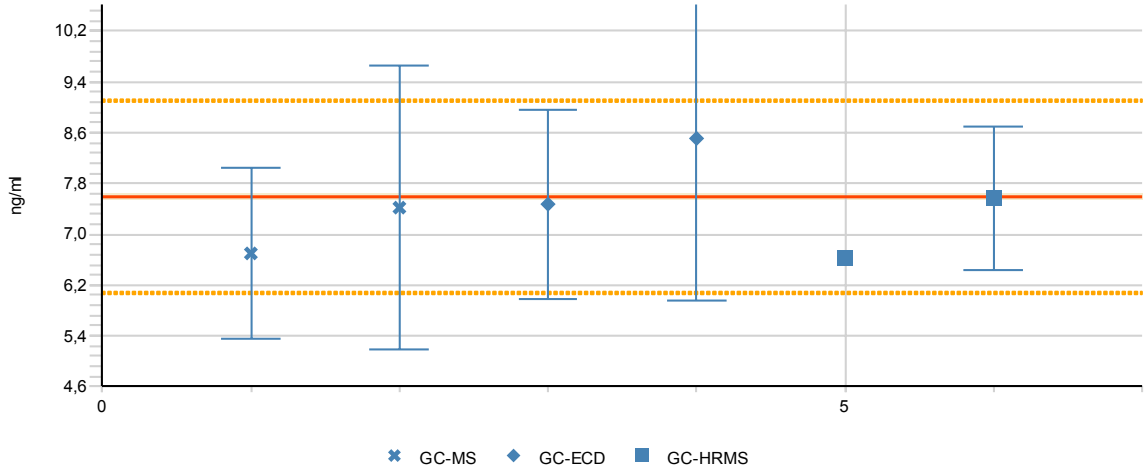
APPENDIX 12: Results grouped according to the methods

The explanations for the figures are described in the Appendix 8.

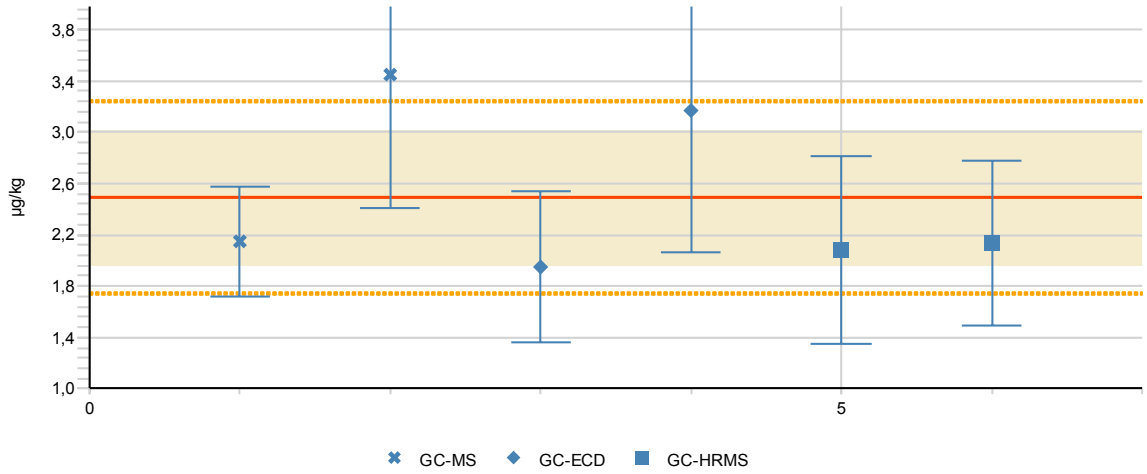




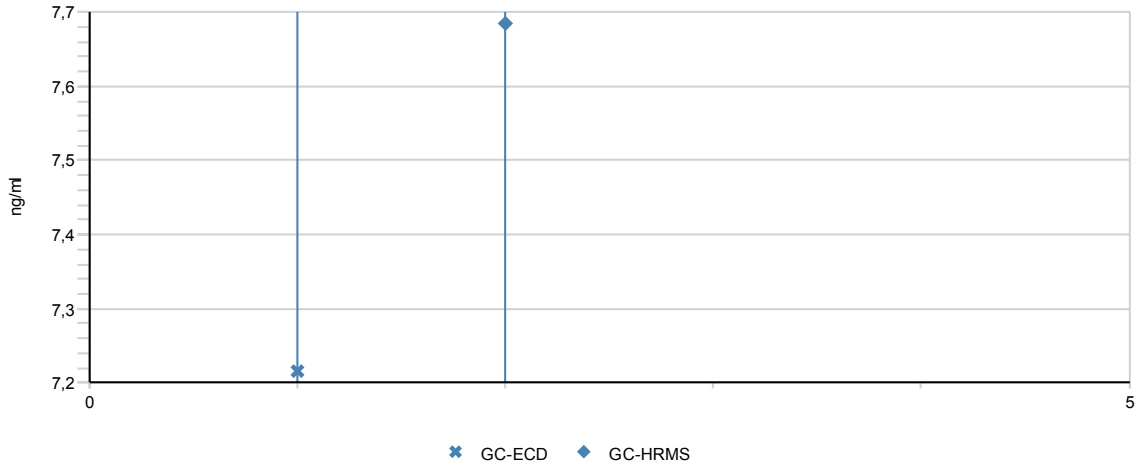
Analyte PCB-105 Sample A1P



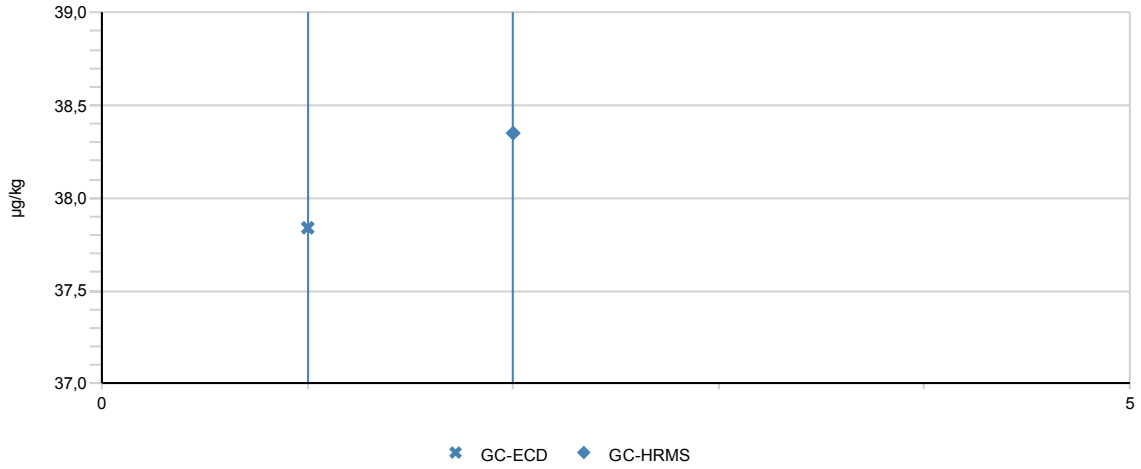
Analyte PCB-105 Sample M2P



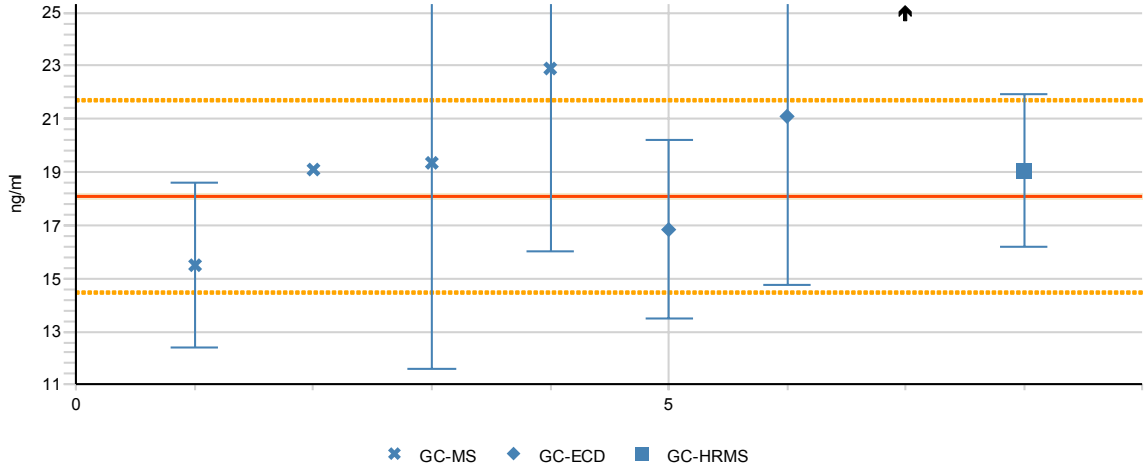
Analyte PCB-110 Sample A1P



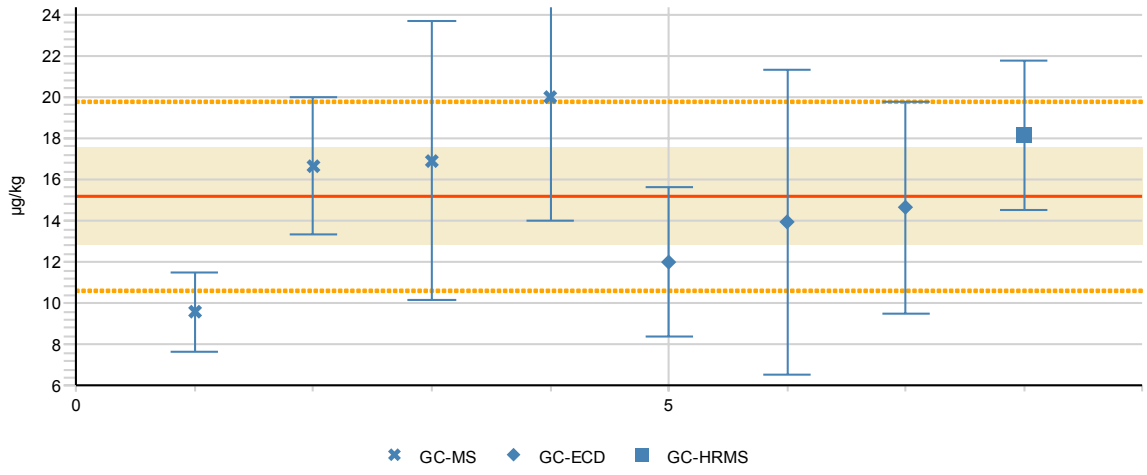
Analyte PCB-110 Sample M2P



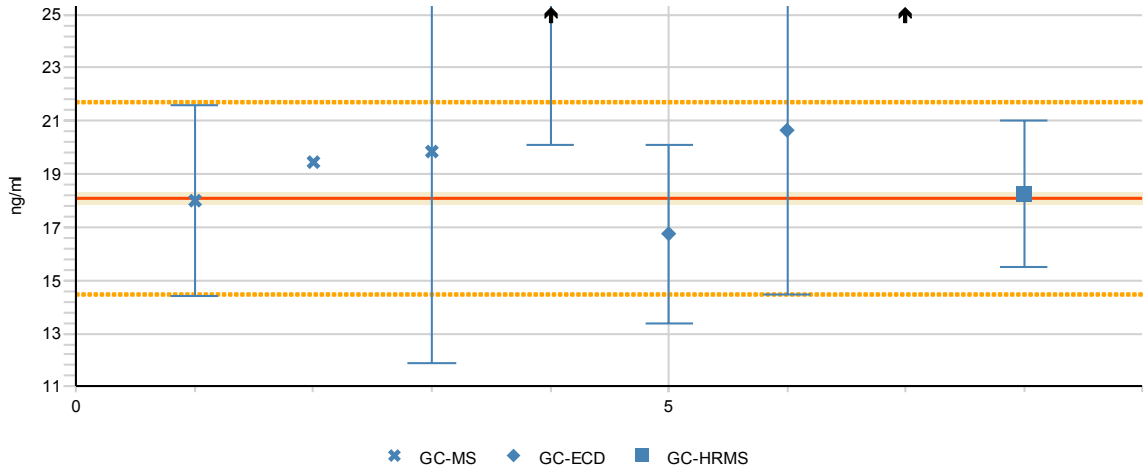
Analyte PCB-118 Sample A1P



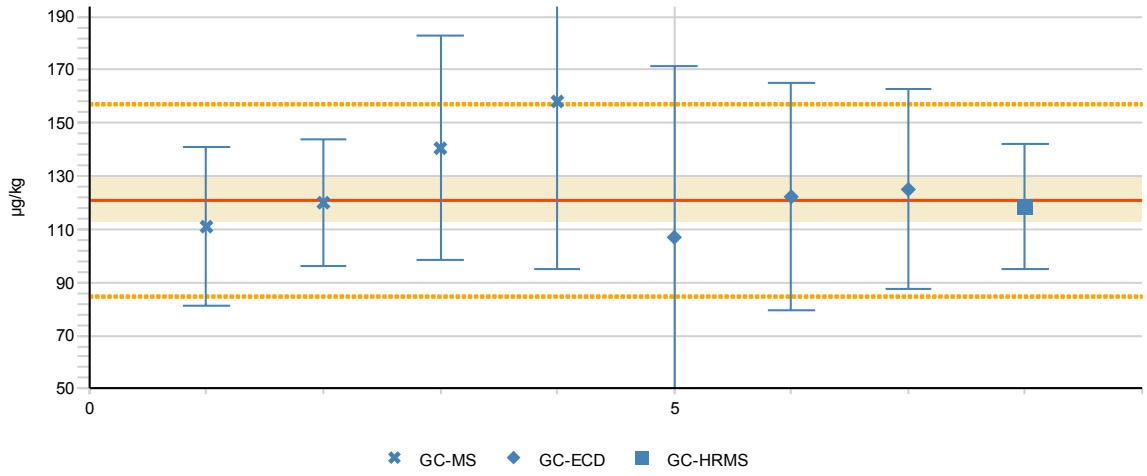
Analyte PCB-118 Sample M2P



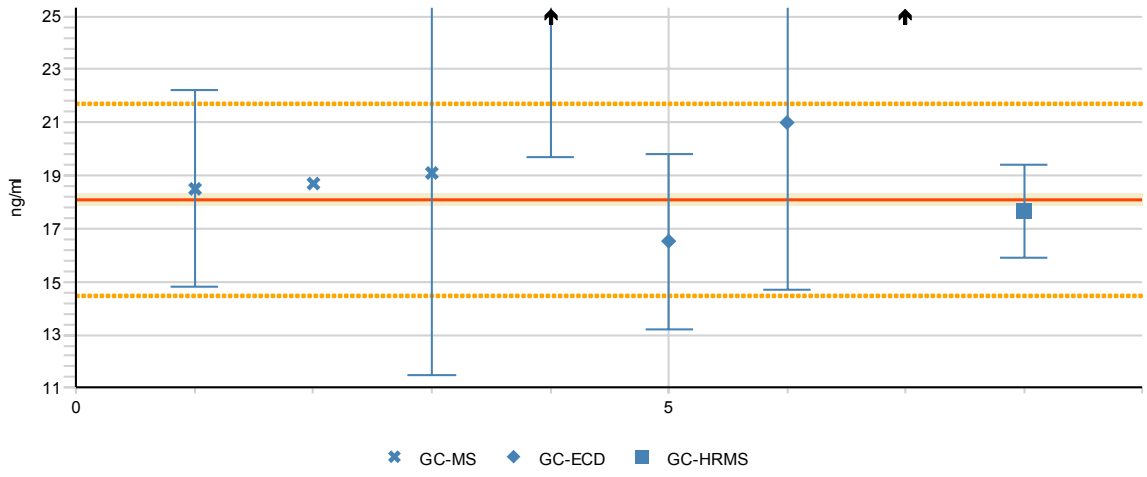
Analyte PCB-138 Sample A1P



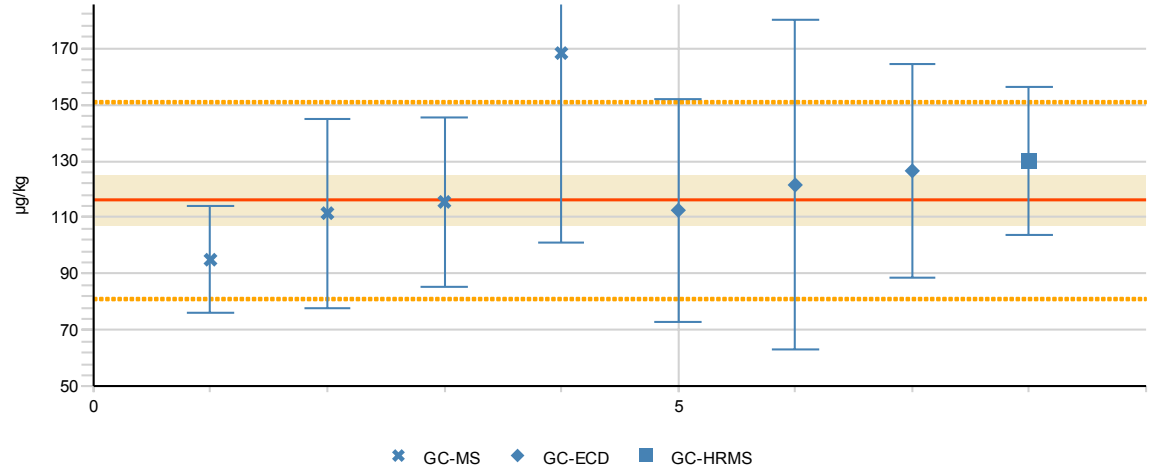
Analyte PCB-138 Sample M2P



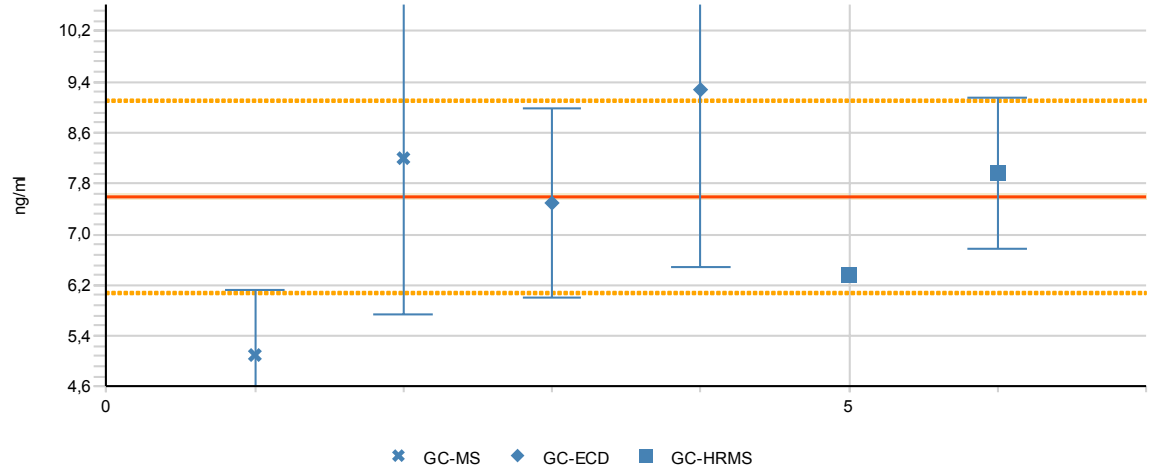
Analyte PCB-153 Sample A1P



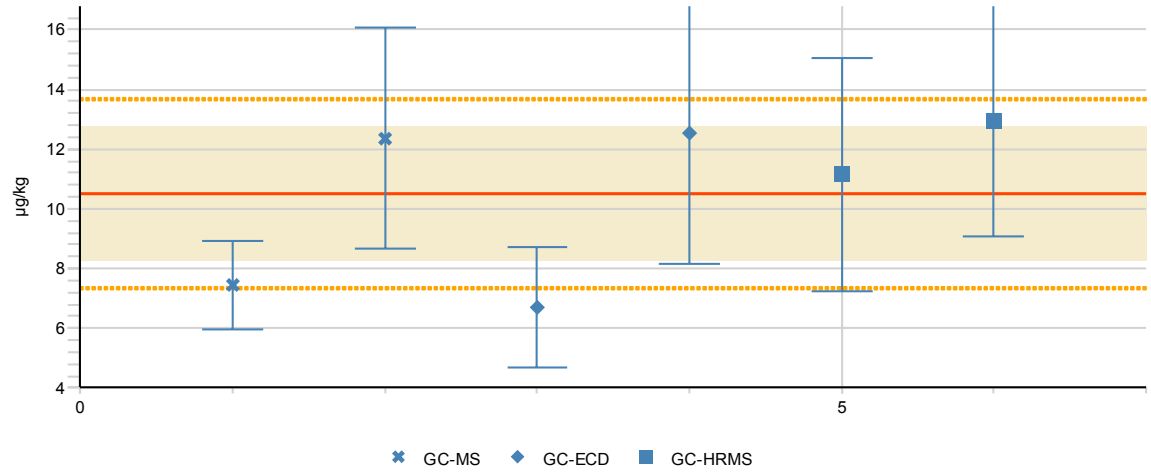
Analyte PCB-153 Sample M2P



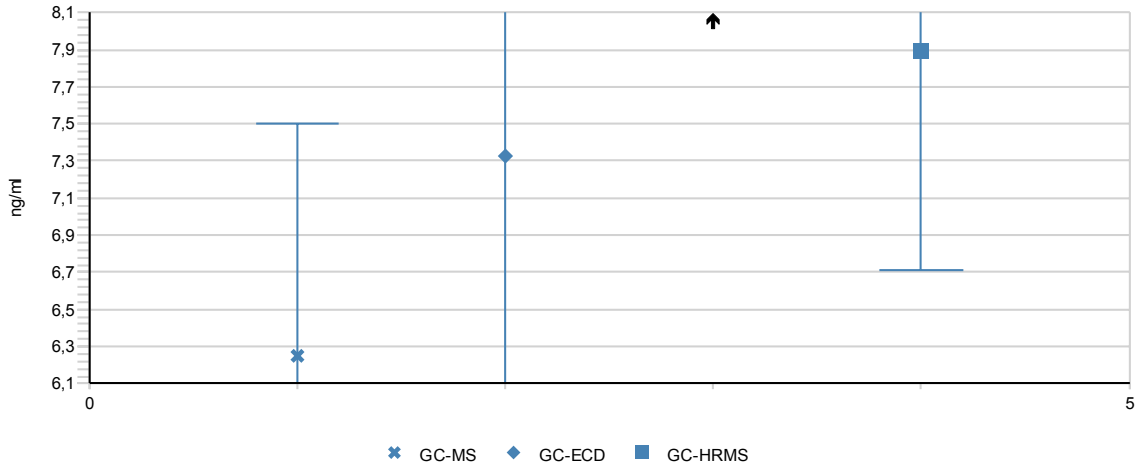
Analyte PCB-156 Sample A1P



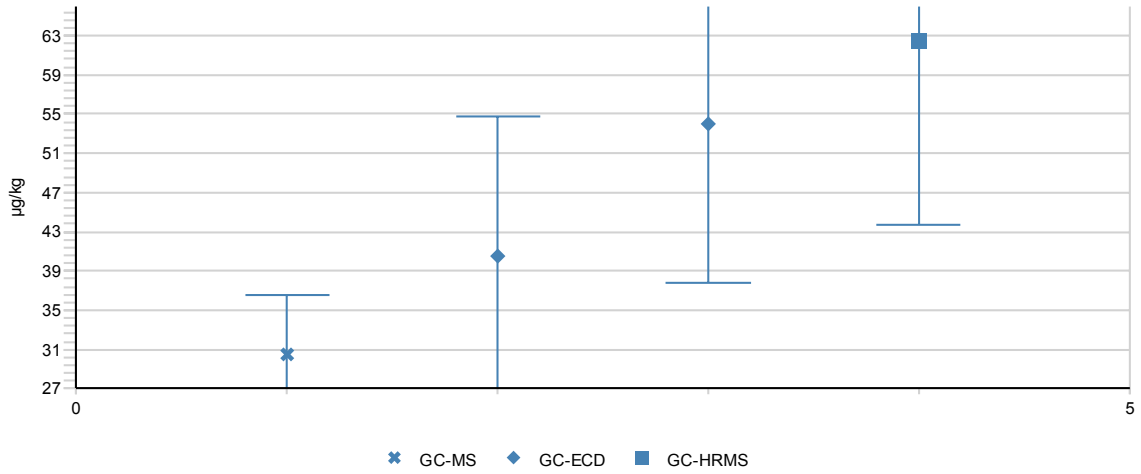
Analyte PCB-156 Sample M2P



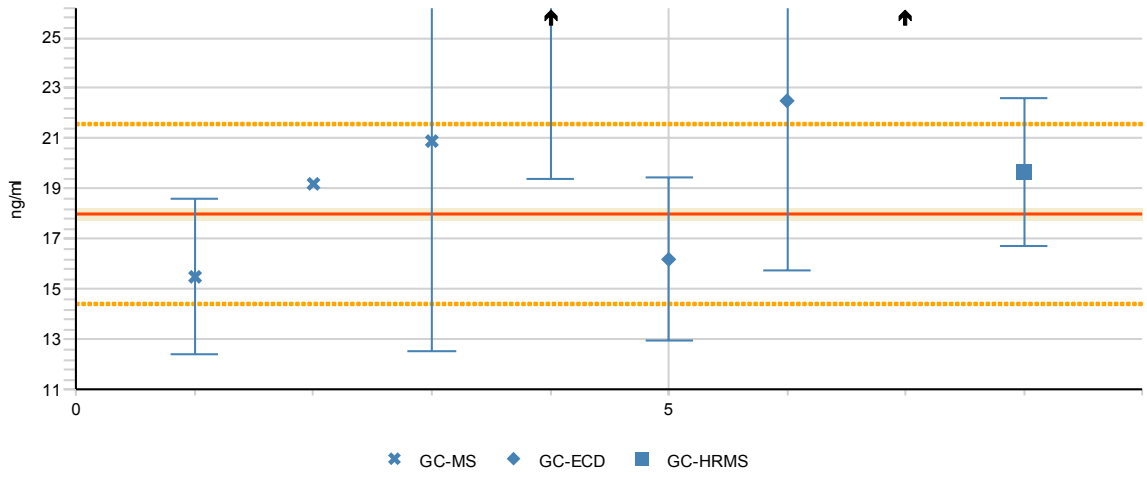
Analyte PCB-170 Sample A1P



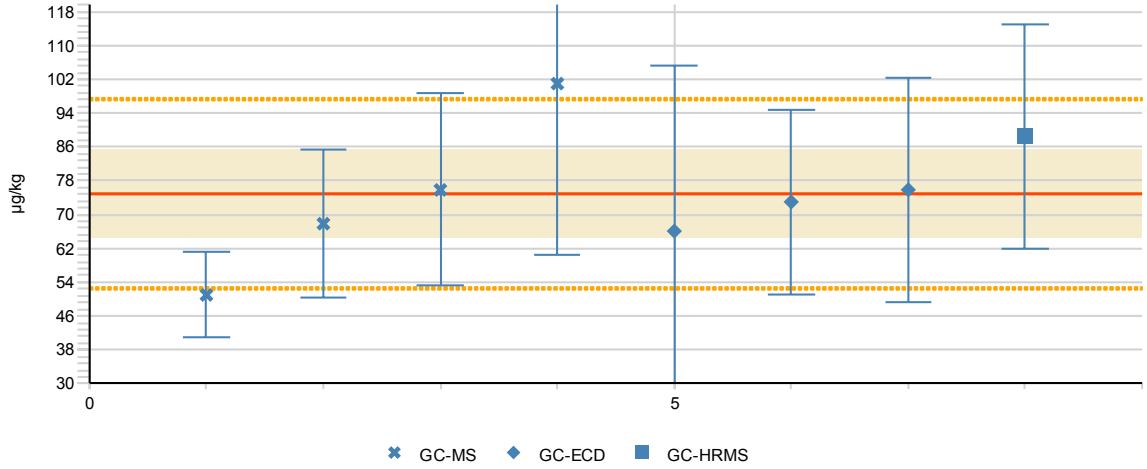
Analyte PCB-170 Sample M2P



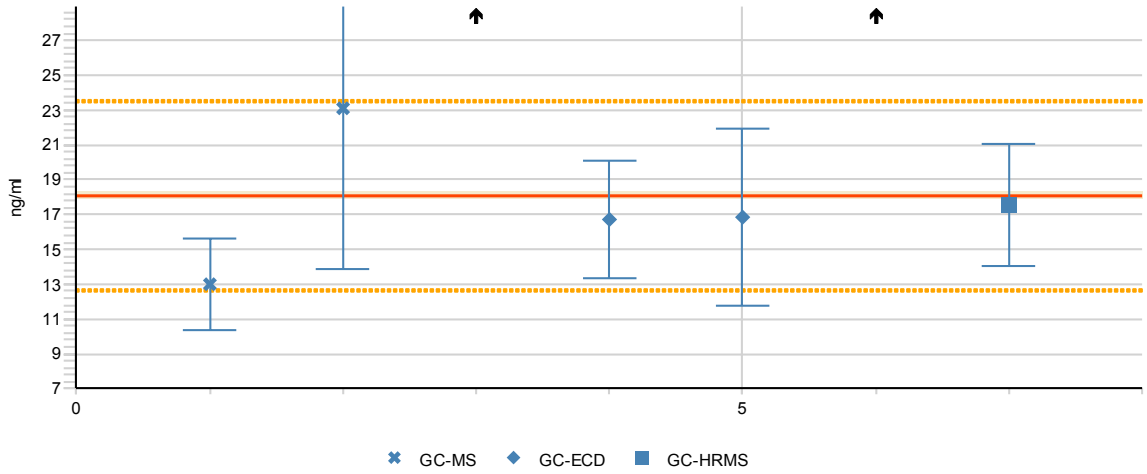
Analyte PCB-180 Sample A1P



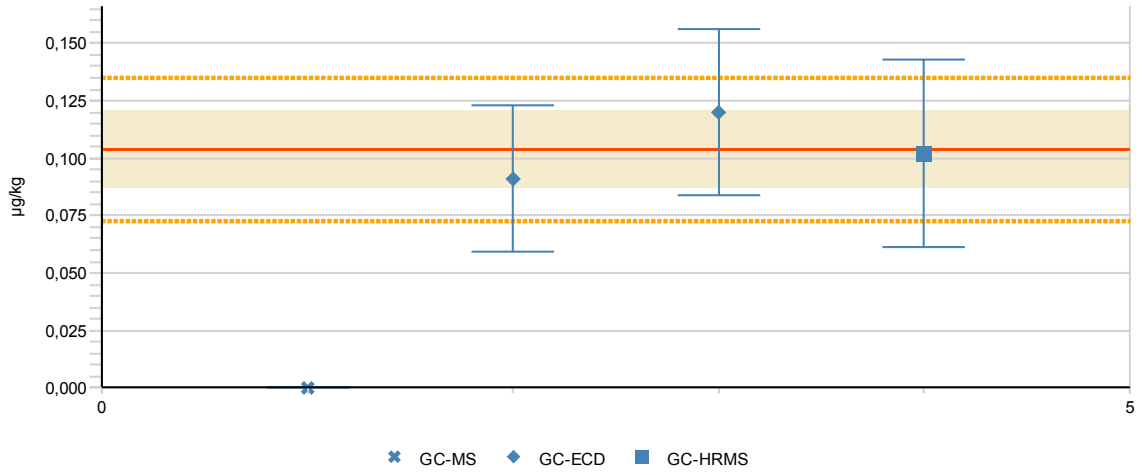
Analyte PCB-180 Sample M2P



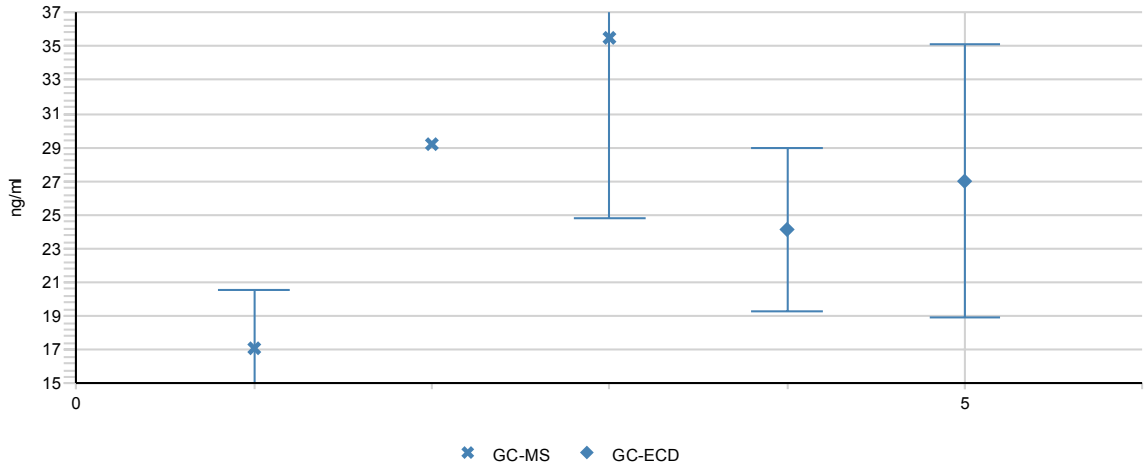
Analyte PCB-28 Sample A1P



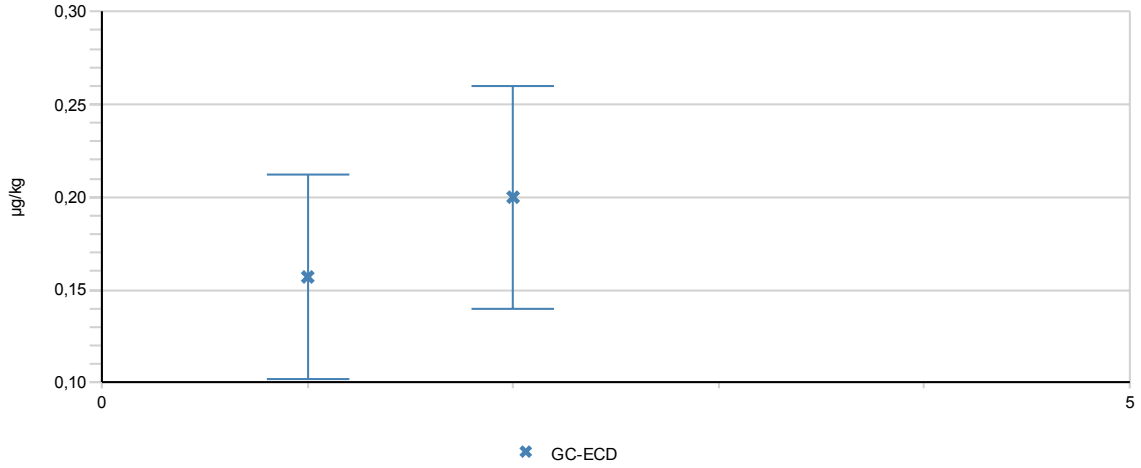
Analyte PCB-28 Sample M2P



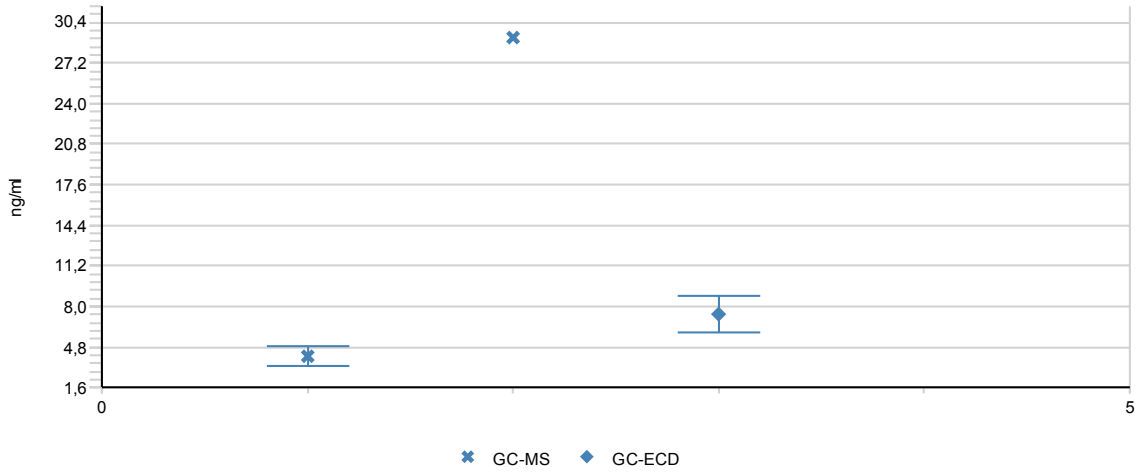
Analyte PCB-28+31 Sample A1P



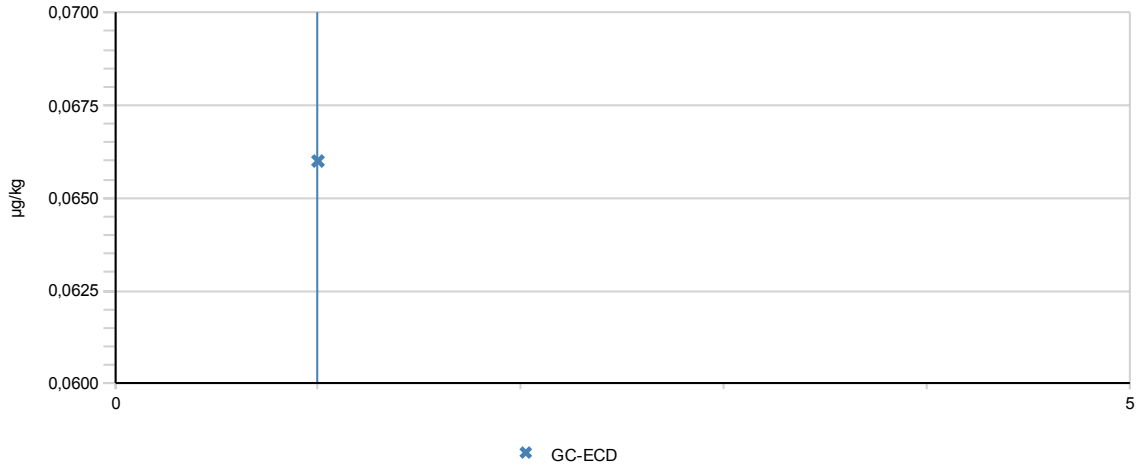
Analyte PCB-28+31 Sample M2P



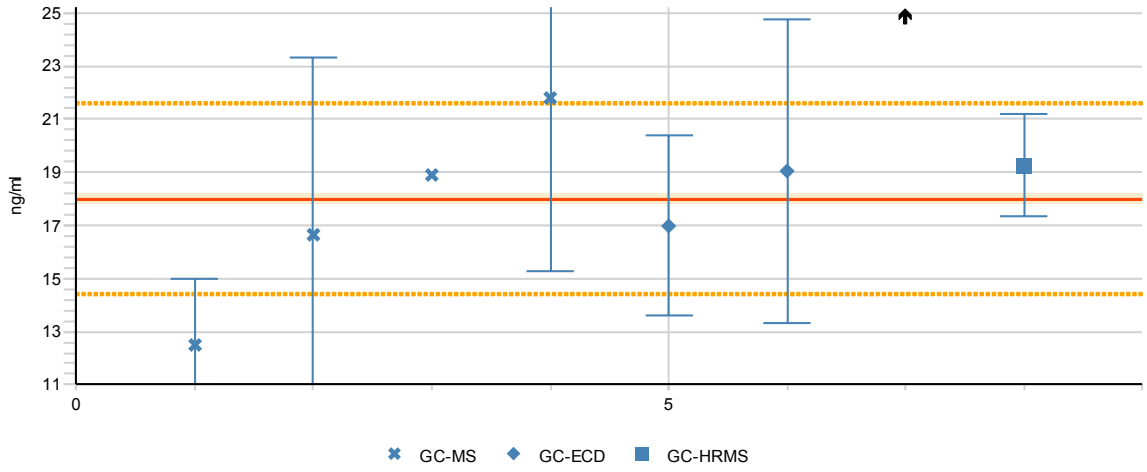
Analyte PCB-31 Sample A1P



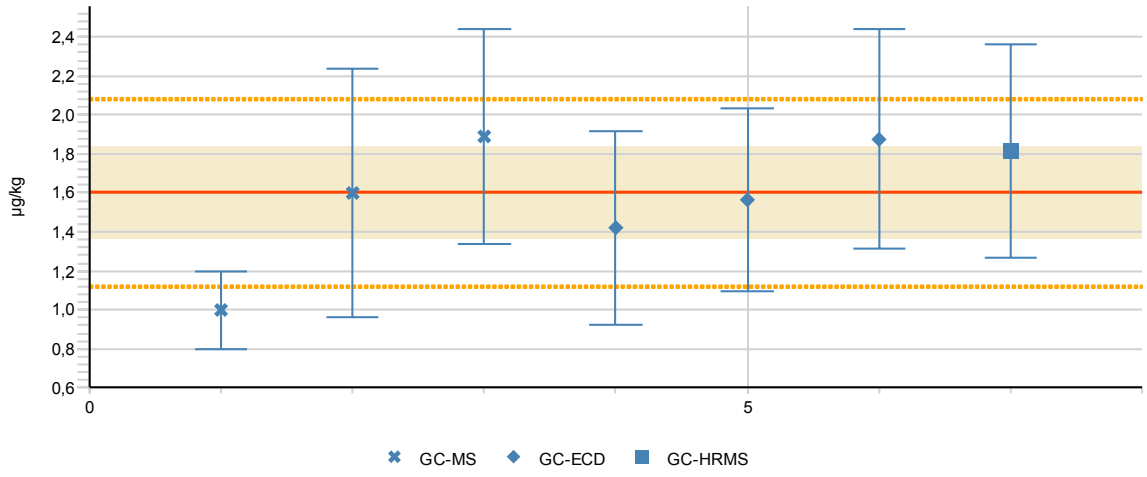
Analyte PCB-31 Sample M2P

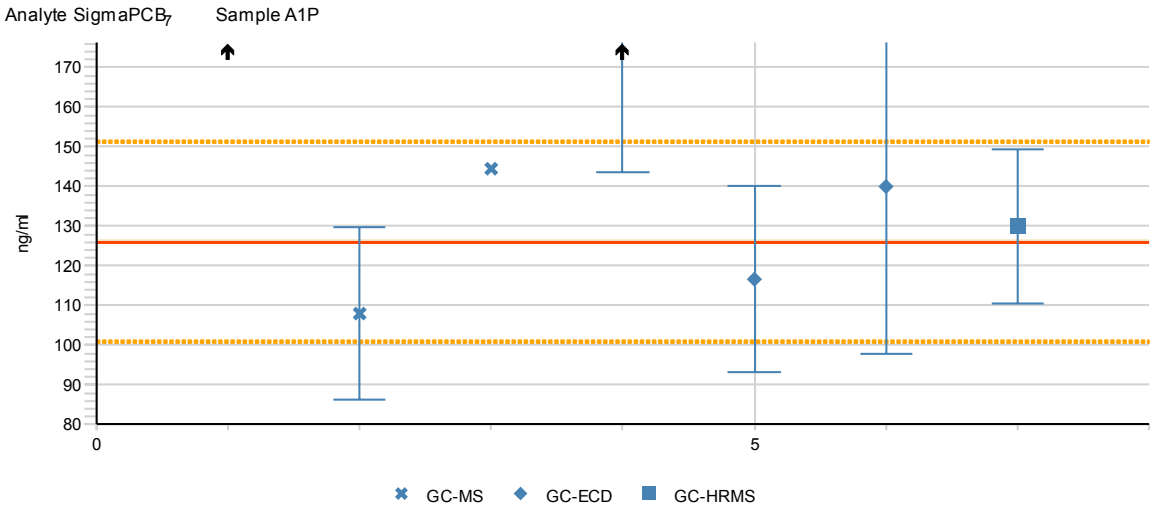


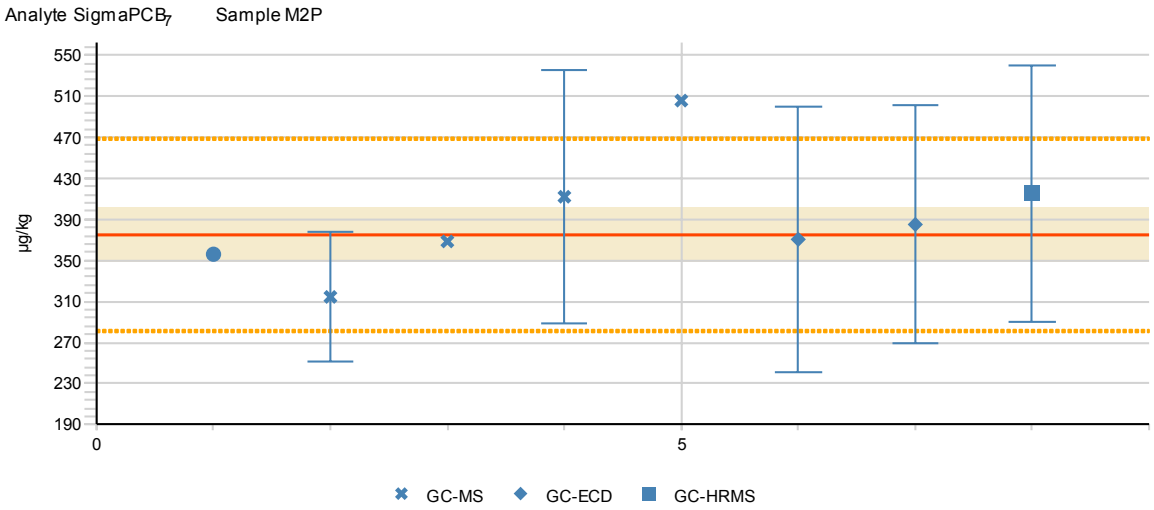
Analyte PCB-52 Sample A1P



Analyte PCB-52 Sample M2P




Analyte SigmaPCB7
Sample A1P
ng/ml
170
160
150
140
130
120
110
100
90
80
0
5
GC-MS
GC-ECD
GC-HRMS


Analyte SigmaPCB7
Sample M2P
µg/kg
550
510
470
430
390
350
310
270
230
190
0
5
GC-MS
GC-ECD
GC-HRMS

APPENDIX 13: Examples of measurement uncertainties reported by the participants

In figures, the presented measurement uncertainties are grouped according to the method of estimation. The following procedures are used for the estimation of the expanded measurement uncertainty at 95 % confidence level (k=2). In figures, the corresponding method numbers are used.

1. Using the IQC data only from synthetic control sample and/or CRM (X-chart). **Using MUkit measurement uncertainty software**. [10, 11]

2. Using the IQC data only from synthetic control sample and/or CRM (X-chart). **Without MUkit measurement uncertainty software.** [11]

3. Using the IQC data from synthetic sample (X-chart) together with the IQC data from routine sample replicates (R-chart or r%-chart). **Using MUkit software.** [10, 11]

4. Using the IQC data from synthetic sample (X-chart) together with the IQC data from routine sample replicates (R-chart or r%-chart). **Without MUkit software.** [11]

5. Using the IQC data and the results obtained in proficiency tests. **Using MUkit software.** [10, 11]

6. Using the IQC data and the results obtained in proficiency tests. **Without MUkit software.** [11]

7. Using the data obtained in method validation. **Using MUkit software.** [10]

8. Using the data obtained in method validation. **Without MUkit software.** [11]

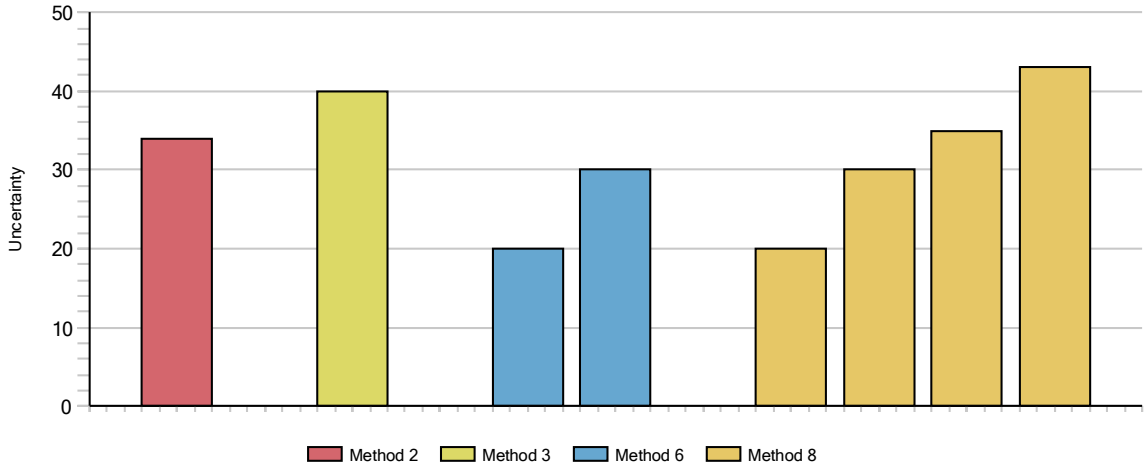
9. Using the "modeling approach". [12, 13]

10. Other procedure, please specify

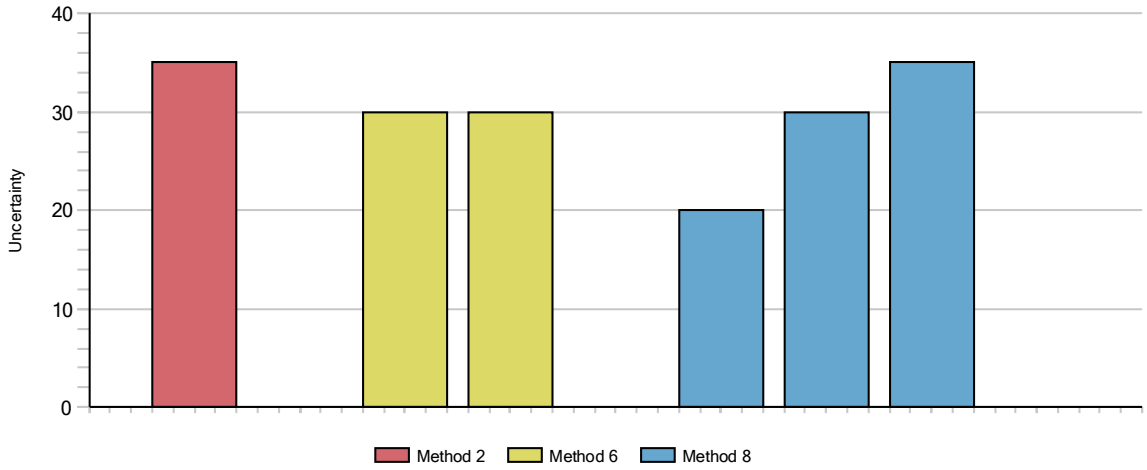
11. No uncertainty estimation

IQC = internal quality control

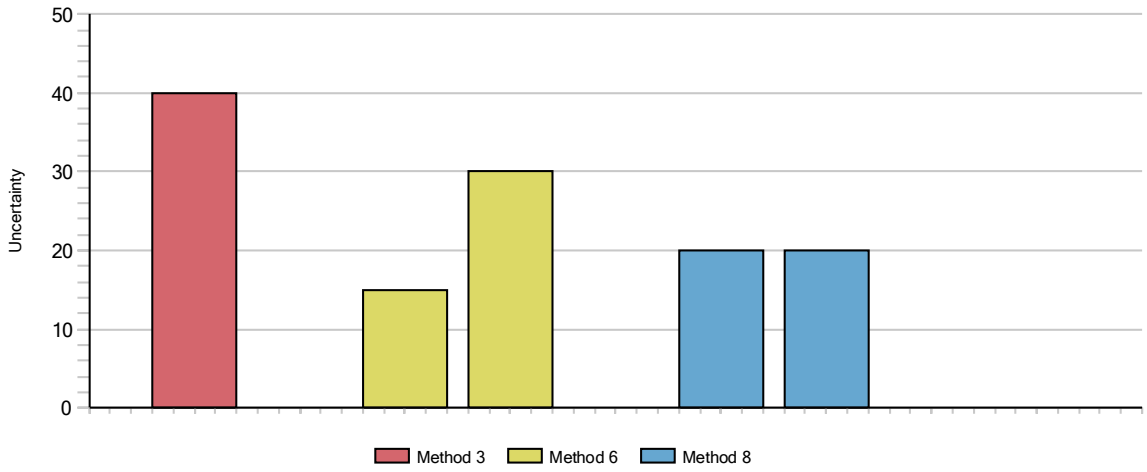
Analyte PCB-101 Sample M2P



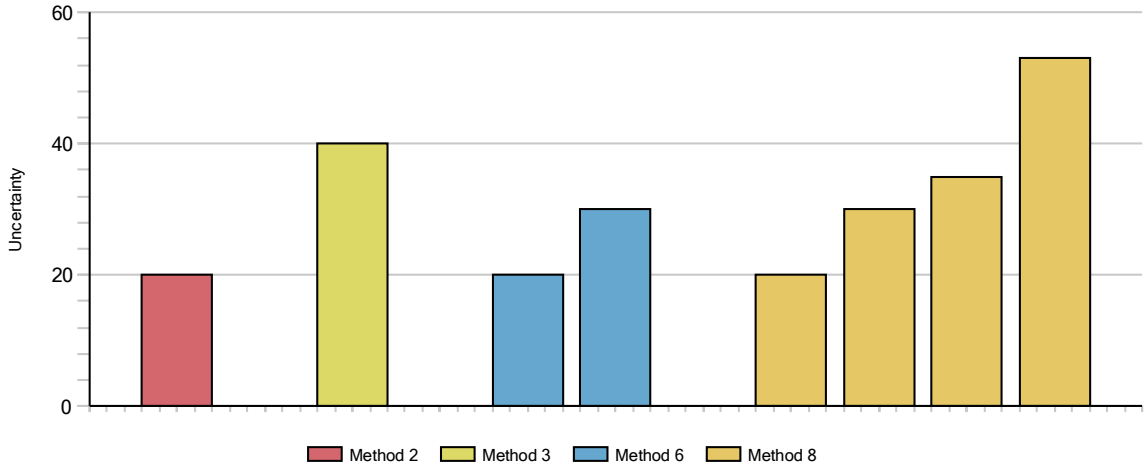
Analyte PCB-105 Sample M2P



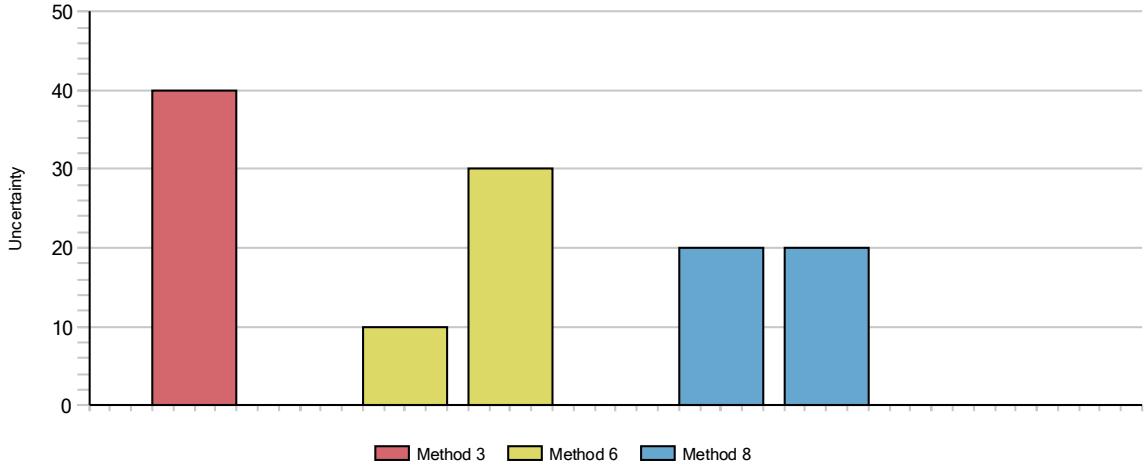
Analyte PCB-118 Sample A1P



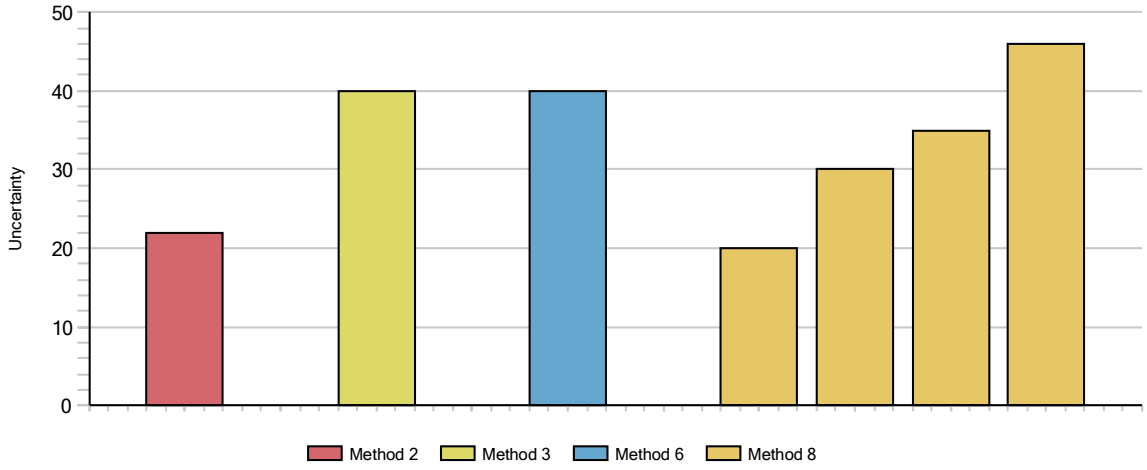
Analyte PCB-118 Sample M2P



Analyte PCB-153 Sample A1P



Analyte PCB-28 Sample M2P



DOCUMENTATION PAGE

Publisher	Finnish Environment Institute	Date March 2016
Author(s)	Riitta Koivikko, Jari Nuutinen and Markku Ilmakunnas	
Title of publication	Interlaboratory Proficiency Test 09/2015 PCB compounds in soil	
Publication series and number	Reports of the Finnish Environment Institute 12/2016	
Theme of publication		
Parts of publication/ other project publications	The publication is available in the internet: www.syke.fi/publications | helda.helsinki.fi/syke	
Abstract	Proftest SYKE carried out the proficiency test (PT) for analysis of PCB compounds in soil in November-December 2015 (PCB 09/2015). Two types of samples were delivered to the participants, synthetic and soil samples. In total, 8 laboratories participated in the PT. The evaluation of the performance was based on the z scores, which were calculated using the standard deviation for proficiency assessment at 95 % confidence level. In this proficiency test 79 % of the data was regarded to be satisfactory when the standard deviation of 20 to 30 % from the assigned value was accepted. For the synthetic sample the calculated concentration and for the soil sample the mean of the results reported by the participants was chosen to be the assigned value. The uncertainty for the assigned value was estimated at the 95 % confidence interval and for calculated assigned values it was 0.5–1.2 %, for assigned values based on the mean it varied from 6.8 to 21.3 %.	
Keywords	PCB compounds, soil analysis, proficiency test, interlaboratory comparison	
Financier/ commissioner		
	ISSN (pdf) 1796-1726 (pdf)	ISBN (online) 978-952-11-4561-2
	No. of pages 59	Language English
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Printing place and year	Helsinki 2016	

KUVAILULEHTI

Julkaisija	Suomen ympäristökeskus	Julkaisuaika Maaliskuu 2016
Tekijä(t)	Riitta Koivikko, Jari Nuutinen ja Markku Ilmakunnas	
Julkaisun nimi	Laboratorioiden välinen pätevyyskoe 09/2015 PCB-yhdisteet maasta	
Julkaisusarjan nimi ja numero	Suomen ympäristökeskuksen raportteja 12/2016	
Julkaisun teema		
Julkaisun osat/ muut saman projektin tuottamat julkaisut	Julkaisu on saatavana vain internetistä: www.syke.fi/julkaisut | helda.helsinki.fi/syke	
Tiivistelmä	Proftest SYKE järjesti marras-joulukuussa 2015 pätevyyskokeen PCB-yhdisteitä maasta analysoiville laboratorioille (PCB 09/2015). Pätevyyskokeen osallistujille toimitettiin kaksi näytettä, synteettinen ja maanäyte. Pätevyyskokeeseen osallistui yhteensä 8 laboratoriota. Pätevyyden arviointi tehtiin z-arvon avulla ja tulosten sallittiin poiketa vertailuarvosta 20–30 %. Koko aineistossa hyväksyttäviä tuloksia oli 79 %. Mittaussuureen vertailuarvona käytettiin laskennallista pitoisuutta tai osallistujien tulosten keskiarvoa. Vertailuarvolle laskettiin mittausepävarmuus 95 % luottamusvälillä. Vertailuarvon laajennettu epävarmuus oli 0,5–1,2 % laskennallista pitoisuutta vertailuarvona käytettäessä ja kun vertailuarvo määritettiin osallistujien tulosten keskiarvona, sen laajennettu epävarmuus vaihteli välillä 6,8–21,3 %.	
Asiasanat	maa-analyysi, PCB-yhdisteet, vesi- ja ympäristölaboratoriot, pätevyyskoe, laboratorioiden välinen vertailumittaus	
Rahoittaja/ toimeksiantaja		
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	Luottamuksellisuus julkinen	
Julkaisun jakelu	Suomen ympäristökeskus (SYKE), neuvonta PL 140, 00251, Helsinki Sähköposti: neuvonta.syke@ymparisto.fi	
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Painopaikka ja -aika	Helsinki 2016	

PRESENTATIONSBLAD

Utgivare	Finlands miljöcentral	Datum Mars 2016
Författare	Riitta Koivikko, Jari Nuutinen och Markku Ilmakunnas	
Publikationens titel	Provningsjämförelse 09/2015 PCB ämnen i marken	
Publikationsserie och nummer	Finlands miljöcentrals rapporter 12/2016	
Publikationens tema		
Publikationens delar/ andra publikationer inom samma projekt	Publikationen finns tillgänglig på internet: www.syke.fi/publikationer | helda.helsinki.fi/syke	
Sammandrag	Under november-december 2015 genomförde Proftest SYKE en provningsjämförelse, som omfattade bestämningen av PCB ämnen i jord. Totalt 8 laboratorier deltog i provningsjämförelsen. Som referensvärde för analyternas koncentration användes det teoretiska värdet eller det medelvärdet av deltagarnas resultat. Resultaten värderades med hjälp av z-värden. I jämfördelsen var 79 % av alla resultaten tillfredställande, när en totalavvikelse på 20–30 % från referensvärdet accepterades.	
Nyckelord	jordanalyser, PCB ämnen, provningsjämförelse, interkalibrering, vatten- och miljölaboratorier	
Finansiär/ uppdragsgivare		
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