REPORTS OF FINNISH ENVIRONMENT INSTITUTE 12| 2012

Proficiency Test SYKE 11/2011

PCB compounds in soil

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Helsinki 2012

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Finnish Environment Institute SYKE

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ALKUSANAT

Suomen ympäristökeskus (SYKE) on toiminut ympäristöalan kansallisena vertailulaboratoriona vuodesta 2001 lähtien. Toiminta perustuu ympäristöministeriön määräykseen, mikä on annettu ympäristönsuojelulain (86/2000) nojalla. Vertailulaboratorion tarjoamista palveluista yksi tärkeimmistä on pätevyyskokeiden ja muiden vertailumittausten järjestäminen. SYKEn laboratoriot on FINAS-akkreditointipalvelun akkreditoima testauslaboratorio T003 ja kalibrointilaboratorio K054 (SFS-EN ISO/IEC 17025) sekä vertailumittausten järjestäjä Proftest SYKE PT01 (SFS-EN ISO/IEC 17043, www.finas.fi).

Tämä pätevyyskoe on toteutettu SYKEn vertailulaboratorion pätevyysalueella ja se antaa tietoa osallistujien pätevyyden lisäksi tulosten vertailukelpoisuudesta myös yleisemmällä tasolla. Pätevyyskokeen onnistumisen edellytys on järjestäjän ja osallistujien välinen luottamuksellinen yhteistyö.

Parhaat kiitokset yhteistyöstä kaikille osallistujille!

PREFACE

Finnish Environment Institute (SYKE) is appointed National Reference Laboratory in the environmental sector by the Ministry of the Environment according to section 24 of the Environment Protection Act (86/2000) since 2001. The duties of the reference laboratory service include providing proficiency tests and other interlaboratory comparisons for analytical laboratories and other producers of environmental information. SYKE laboratories has been accredited by the Finnish Accreditation service as the testing laboratory T003 and the calibration laboratory K054 (EN ISO/IEC 17025) and as the proficiency testing provider Proftest SYKE PT01 (EN ISO/IEC 17043, www.finas.fi).

This proficiency test has been carried out under the scope of the SYKE reference laboratory and it provides information about performance of the participants as well as comparability of the results at a more general level. The success of the proficiency test requires confidential co-operation between the provider and participants.

Thank you for your co-operation!

Helsingissä 10. huhtikuuta 2012 / Helsinki 10 April 2012

Marja Luotola

Laboratorionjohtaja / Chief of Laboratory

1 INTRODUCTION

Proftest SYKE carried out the proficiency test (PT) for analysis of PCB compounds in soil in November 2011 – January 2012. An artificial sample (PCBs in isooctane) and a contaminated soil sample were distributed to the participants. In this PT the results of Finnish laboratories providing environmental data for Finnish environmental authorities were evaluated. Additionally, other water and environmental laboratories were welcomed in the proficiency test.

The test was carried out in accordance with the international guidelines, ISO/IEC 17043 [1], ISO 13528 [2] and IUPAC Technical Report [3]. Proftest SYKE has been accredited by the Finnish Accreditation Service as a proficiency testing provider PT01 (ISO/IEC 17043, www.finas.fi). Proftest SYKE is the accredited proficiency test provider on the field of the present test.

2 ORGANIZING THE PROFICIENCY TEST

2.1 Responsibilities

Organizing laboratory:
Proftest SYKE, Finnish Environment Institute (SYKE), Laboratory Centre
Hakuninmaantie 6, 00430 Helsinki, Finland
Phone: +358 20 610 123
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The responsibilities in organizing the PT were as follows:

Kaija Korhonen-Ylönen, coordinator
Jari Nuutinen, substitute of coordinator, analytical expert
Anne Markkanen, technical assistant
Markku Ilmakunnas, technical assistant, layout of the report
Keijo Tervonen, technical assistant
Ritva Väisänen, technical assistant
Sari Lanteri, technical assistant

2.2 Participants

In total, 10 laboratories from Denmark, Finland, Sweden and Turkey participated in this proficiency test (Appendix 1). From the participants eight laboratories used an accredited method for most of PCB compounds. The organizing laboratory (SYKE) had the codes 3 (ASE extraction) and 4 (Ultrasonic extraction) in the result tables.

2.3 Samples and delivery

The sample A1 was a synthetic sample prepared by diluting the certificated reference PCB solutions (Dr. Ehrenstorfer) with isooctane. The preparation of the samples is presented in more detail in Appendix 2. The sample A1 contained also the PCB congeners 77, 126, 128 and 169 though these congeners were not requested to be analysed. The sample A1 did not contained PCB 110 but it was requested to be analysed.

The sample M1 was the PCB contaminated soil containing sealant. The soil sample was dried at the room temperature and sieved through a 0.5 mm sieve before homogenization and subsampling. The same sample was distributed earlier also in the PT SYKE 5 / 2001 and PT SYKE 3 / 2005.

The samples were delivered 21 November 2011. They were requested to be analysed and reported at the latest 27 January 2012.

2.4 Homogeneity studies

Homogeneity of the soil sample M1 was tested by analyzing PCB-52, PCB-101, PCB-118, PCB-138, PCB-156 and PCB-180 as duplicate determinations from six sub samples (Appendix 3). According to the homogeneity test results the sample M1 was considered to be homogenous.

2.5 Feedback from the proficiency test

Appendix 4 contains the comments sent by the participants and feedback from the provider to the participants.

2.6 Processing of the data

2.6.1 Pretesting of the data

Before the statistical treatment, the data was tested according to the Kolmogorov-Smirnov normality test and the possible extreme values were rejected as the outliers according to the Hampel test (marked as H in the results sheets). Also before the robust calculation some extreme outliers were rejected in case that the results deviated from the robust mean more than 50 %.

The replicate results were tested using the Cochran test (marked as C in the results sheets). In case that the result had been reported to be lower than detection limit, it had not been included in the statistical calculation of the results. More detailed information of the testing and statistical treatment of the PT data is available on the internet in the guide for participating laboratories in SYKE proficiency testing schemes (www.environment.fi/syke/proftest).

2.6.2 Assigned values and their uncertainties

The assigned values and their uncertainties are presented in Appendix 5. The calculated concentrations were used as the assigned values for the concentrations of all measurements in the artificial sample A1. The uncertainty of the assigned value given is the expanded combined uncertainty based on the combination of uncertainties associated with individual operations involved in the preparation of the sample. In all cases the uncertainty was less than 3 % and the main individual resource of the uncertainty was the uncertainty of the impurity in the stock solution.

The robust means of the reported results were used as the assigned value for the concentrations of the PCBs in the soil sample M1. The uncertainty of the assigned value was calculated using the robust standard deviation of the reported results. Depending of the PCB congener the uncertainties varied from 5 to 20 % (Appendix 5).

After reporting of the preliminary results no significant correction of the assigned value has been done.

2.6.3 Standard deviation for proficiency assessment and z score

The evaluation of the participants was based on z scores, which were calculated using the estimated standard deviation for proficiency assessment. The estimation of the target standard deviation based on the type of sample, the concentration of the analyte in the sample, the results of homogeneity test and the uncertainty of the assigned value.

The reliability of the assigned value was tested according the criterion u / $s_p \leq 0.3$, where u is the standard uncertainty (U / 2) of the assigned value and s_p the target value (target value for total deviation / 2). Due to low number of the participants and the large deviation of the results the criterion was not fulfilled in every case, which indicated that the following assigned values in the soil sample had high uncertainty: PCB-138, PCB-156, PCB-170, PCB-28, PCB-28+31, PCB-31.

The reliability of the standard deviation for proficiency assessment (s_p) and correspondingly the z score were estimated by comparing the target value (s_p) with the robust standard deviation of the reported results (s_{rob}). Due to low number of the results the criterion $s_{rob} < 1.2 \cdot s_p$ was not fulfilled in every cases (PCB-138, PCB-156, PCB-28, PCB-31), which weakened the evaluation of performance.

The performance evaluation was not carried out in the case where the assigned value based on the consensus value and the number of the participants was below 6. Due to low number of participants the results of PCB-110, PCB-170 and PCB-31 in the soil sample were not evaluated.

3 RESULTS

3.1 Results

Explanations for the result sheets are presented 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 and their uncertainties are presented graphically in Appendix 8 and the summary of z scores is presented in Appendix 9.

The robust standard deviation of the results was lower than 10 % for 16 % of the results and lower than 20 % for 60 % of the results. Standard deviations higher than 20 % applied mainly to the congeners PCB-138, PCB-28, PCB-28+31 and PCB-31.

Table 1. Summary of the proficiency test 11 /2011.

Analyte	Sample	Unit	Ass. val.	Mean	Mean rob.	Md	SD rob	SD rob, %	Num. of labs	2*Targ SD%	Ac-cepted z-val%
PCB-101	A1	ng/ml	27,74	24.94	24.88	24.40	4.42	17,7	10	20	70
	M1	µg/kg	282	288.15	282.27	280.00	38.84	13,8	11	30	91
PCB-105	A1	ng/ml	9,17	8.44	8.44	8.04	1.87	22,2	5	20	60
	M1	µg/kg	91,5	91.53	91.53	94.22	8.67	9,5	6	30	83
PCB-110	A1	ng/ml				0.000			1		
	M1	µg/kg	309	308.63	308.63	314.38	19.00	6,2	3		
PCB-118	A1	ng/ml	9,17	7.82	8.10	7.94	1.72	21,2	10	20	70
	M1	µg/kg	245	246.88	245.39	245.00	17.77	7,2	11	30	100
PCB-138	A1	ng/ml	27,74	25.00	24.99	24.80	4.44	17,8	10	20	80
	M1	µg/kg	271	275.61	271.41	270.00	57.46	21,2	11	30	91
PCB-153	A1	ng/ml	27,74	24.47	24.47	24.30	4.59	18,8	10	20	60
	M1	µg/kg	222	222.17	222.17	220.00	26.90	12,1	11	30	91
PCB-156	A1	ng/ml	9,17	8.98	8.98	9.08	1.86	20,7	5	20	60
	M1	µg/kg	27,1	27.14	27.14	27.21	5.29	19,5	6	30	83
PCB-170	A1	ng/ml	9,17	9.07	9.07	9.21	2.14	23,6	4	20	50
	M1	µg/kg	47,6	47.62	47.62	49.37	6.95	14,6	5		
PCB-180	A1	ng/ml	27,74	24.53	25.10	25.60	4.17	16,6	10	20	70
	M1	µg/kg	83,6	83.18	83.60	84.63	6.24	7,5	11	30	91
PCB-28	A1	ng/ml	27,74	25.79	25.79	23.80	5.74	22,3	9	20	67
	M1	µg/kg	16,6	16.75	16.62	15.20	3.98	23,9	10	30	78
PCB-28+31	A1	ng/ml	36,91	34.31	31.04	31.84	11.64	37,5	6	20	50
	M1	µg/kg	22,6	21.69	22.56	21.74	2.81	12,5	7	30	71
PCB-31	A1	ng/ml	9,17	6.79	8.39	7.35	3.83	45,7	4	20	25
	M1	µg/kg	8,52	8.52	8.52	7.69	1.94	22,8	5		
PCB-52	A1	ng/ml	27,74	23.27	24.82	23.90	4.85	19,5	10	20	80
	M1	µg/kg	162	163.97	162.25	169.00	20.50	12,6	11	30	73

Ass. Val.- the assigned value, Mean - the mean value, Mean rob - robust mean, Md - the median value, SD % - the standard deviation as percent, SD rob - the robust standard deviation, SD rob % - the robust standard deviation as percents, Num of Labs - the number of participants, 2·Targ. SD% - the total standard deviation for proficiency assessment at 95 % confidence level ($2 \cdot s_p$), Accepted z-val% - the satisfactory z scores: the results (%), where $|z| \leq 2$.

In this PT the participants were requested to report duplicate results for all measurements. The participants reported the replicates with the exception of one laboratory (Lab 6). The results of the replicate determinations based on the ANOVA statistical handling are presented in Table 2.

The within-laboratory standard deviation, s_w, describes the repeatability of measurements. While the between-laboratory standard deviation, s_b, describes the reproducibility of measurements. In this PT the reproducibility (s_b) was an average from 0.6 to 10 times higher than the repeatability (s_w). For the robust methods the ratio s_b / s_w, should not exceed 3.

Analyte	Sample	Unit	Ass. val.	Mean	Md	sw	sb	st	sw %	sb %	st %	2*Targ SD %	Num of labs	Ac-cepted. z-val %
PCB-101	A1	ng/ml	27,74	25,1	24,4	1,401	3,908	4,152	5,6	16	17	20	10	70
	M1	µg/kg	282	287,1	280	13,62	47,92	49,81	4,7	17	17	30	11	91
PCB-105	A1	ng/ml	9,17	8,438	8,035	0,6711	1,579	1,716	8	19	20	20	5	60
	M1	µg/kg	91,5	91,53	93,16	5,735	6,48	8,654	6,3	7,1	9,5	30	6	83
PCB-110	M1	µg/kg	309	308,6	314,4	17,76	11,09	20,94	5,8	3,6	6,8		3	
PCB-118	A1	ng/ml	9,17	7,802	7,8	0,1829	1,321	1,334	2,3	17	17	20	10	70
	M1	µg/kg	245	247,6	245	13,44	16,33	21,15	5,4	6,6	8,5	30	11	100
PCB-138	A1	ng/ml	27,74	25,06	24,8	1,714	3,833	4,199	6,8	15	17	20	10	80
	M1	µg/kg	271	274,7	270	15,65	59,92	61,93	5,7	22	23	30	11	91
PCB-153	A1	ng/ml	27,74	24,46	24,3	1,991	3,899	4,378	8,1	16	18	20	10	60
	M1	µg/kg	222	221	220	6,338	23,31	24,16	2,9	11	11	30	11	91
PCB-156	A1	ng/ml	9,17	8,979	9,08	0,7381	1,555	1,722	8,2	17	19	20	5	60
	M1	µg/kg	27,1	27,14	26,17	0,9313	4,621	4,714	3,4	17	17	30	6	83
PCB-170	A1	ng/ml	9,17	9,068	9,205	0,19	1,884	1,893	2,1	21	21	20	4	50
	M1	µg/kg	47,6	47,62	49,37	2,563	5,855	6,391	5,4	12	13		5	
PCB-180	A1	ng/ml	27,74	24,59	23,4	0,4216	3,475	3,5	1,7	14	14	20	10	60
	M1	µg/kg	83,6	82,72	84,6	2,462	5,949	6,438	3	7,2	7,8	30	11	91
PCB-28	A1	ng/ml	27,74	25,9	23,8	1,425	5,085	5,281	5,5	20	20	20	9	67
	M1	µg/kg	16,6	16,46	15,85	1,144	3,604	3,781	6,9	22	23	30	9	78
PCB-28+31	A1	ng/ml	36,91	34,31	32,35	1,918	7,029	7,286	5,6	20	21	20	6	50
	M1	µg/kg	22,6	21,69	22,73	1,514	4,275	4,535	7	20	21	30	7	71
PCB-31	A1	ng/ml	9,17	6,787	6,34	0,3509	1,257	1,305	5,2	19	19	20	4	25
	M1	µg/kg	8,52	8,515	8,15	0,5166	1,675	1,752	6,1	20	21		5	
PCB-52	A1	ng/ml	27,74	23,32	23,29	0,1862	3,151	3,157	0,8	14	14	20	10	60
	M1	µg/kg	162	164	164,5	8,769	20,81	22,58	5,3	13	14	30	11	73

Ass. val. - assigned value, Md - median, sw - repeatability standard error, sb - standard error between laboratories, st - reproducibility standard error

3.2 Analytical methods

The analytical methods used by the participants are presented in Appendix 10.1.

PCB compounds in soil

Determination of PCB compounds from soil samples based on several standard methods or EPAs methods [7–11]. Half of the methods were modifications of the standard methods. Extraction was mostly done by shaking. Most participants used splitless-injection technique. Six laboratories used GC-MS technique and five used GC-ECD technique.

Method comparison for the sample A1 was done between measurement techniques and for the soil sample M1 between extraction methods. Before method comparison the results were coded by coordinator as follows:

Sample A1

- Method 1: GC-MS
- Method 2: GC-ECD
- Method 3: GC-HRMS

Sample M1
- Method 4: Extraction with Shaking
- Method 5: Soxhlet extraction
- Method 6: Extraction with accelerated solvent extractor
- Method 7: Extraction with ultrasonic

No significant difference could be obtained between the methods. According to the graphical presentation the unsatisfactory results were not due to the applied technique (Appendix 10.2).

3.3 Uncertainties of the results

All laboratories reported the expanded uncertainties at least with some of their results (Appendix 8). Several procedures were used for estimating of measurement uncertainty (Appendix 11). The procedure based on data obtained in the method validation and the internal quality control (Meth 3) was the most common. The other procedures were used by only one or two laboratories. Due to low number of the participants it is difficult to draw conclusion from the relation of the uncertainty and the evaluation procedure.

Table 4. The range of the expanded uncertainties (%) reported by the participants

Analyte	Sample A1	Sample M1
PCB-28	20–40	20–80
PCB-31	20–30	20–40
PCB-28+31	20–91	20–91
PCB-52	10–40	18–54
PCB-101	10–57	18–57
PCB-105	15–30	20–35
PCB-110		10–35
PCB-118	15–42	18–49
PCB-138	15–40	18–60
PCB-153	10–40	15–69
PCB-156	15–30	20–35
PCB-170	15–30	20–35
PCB-180	15–45	18–63

4 EVALUATION OF PERFORMANCE

The evaluation of the participants was based on z scores, which were calculated using the estimated standard deviation for proficiency assessment. In the performance evaluation z scores were interpreted as follows:

$\lvert z \rvert \leq 2$	satisfactory results
$2 < \lvert z \rvert < 3$	questionable results
$\lvert z \rvert \geq 3$	unsatisfactory results

The calculated z scores are presented with the results of each participant (Appendix 8) and the summary of z scores is presented in Appendix 9.

When accepting deviations of 20 % from the assigned values for synthetic sample A1, 62 % of the results were satisfactory. In this PT the number of satisfactory results was slightly lower than in the previous PT 5/2007 [6]. The participants had most problems in correct analysis of PCB 31, which poorly separates from PCB-28.

Consequently, when accepting deviations of 30 % from the assigned values for the soil sample M1, 85 % of the results were satisfactory. The number of the satisfactory results in this PT was at the same level as in the PT 3/2005 [5], where the same soil sample was distributed and an average 84 % of the results were satisfactory.

In total, 76 % of the total result data in this PT were satisfactory. Most participants used accredited methods and 81 % of their results were satisfactory (Appendix 9).

5 SUMMARY

Proftest SYKE carried out the proficiency test for the determinations of PCB compounds in soil samples in the end of 2011. In total, 10 laboratories participated in the proficiency test. One artificial sample (A1) and one soil sample (M1) were delivered to the laboratories.

The calculated concentrations (sample A1) or the robust mean of the results reported by the participants (sample M1) were used as the assigned values for the measurands. The uncertainties of the calculated assigned values were less than 3 %. Respectively the uncertainties of the consensus assigned values (the robust mean) were from 5.3 % to 22 %.

In total, 76 % of the total result data in this PT were satisfactory when the deviations of 20–30 % from the assigned values were accepted.

Regardless of the several methods, extraction and instrument techniques used for the analysis of the soil sample M1 the results were surprisingly similar.

6 YHTEENVETO

Proftest SYKE järjesti pätevyyskokeen PCB yhdisteiden määrityksestä maanäytteestä (M1) loppuvuodesta 2011. Maanäytteen lisäksi osallistujille toimitettiin synteettinen näyte (A1). Pätevyyskokeeseen osallistui yhteensä 10 laboratoriota Ruotsista, Suomesta, Tanskasta ja Turkista.

Synteettisessä näytteessä (A1) PCB-pitoisuuden vertailuarvona käytettiin laskennallista pitoisuutta (teoreettinen arvo). PCB-yhdisteiden laskennallisten vertailuarvojen laajennetut epävarmuudet olivat alle 3 %. Maanäytteessä (M1) PCB-pitoisuuden vertailuarvona käytettiin osallistujien raportoimien tulosten robustiakeskiarvoa (sopimusarvo). Yhdisteestä ja raportoitujen tulosten hajonnasta riippuen vertailuarvon laajennettu epävarmuus 5,3−22 %, kun vertailuarvona käytettiin sopimusarvoa.

Synteettisten näytteiden tuloksissa sallittiin 20 %:n poikkeama vertailuarvosta, jolloin tuloksista oli hyväksyttäviä 62 %. Vuoden 2007 vastaavassa vertailussa PCB-yhdisteiden tuloksista oli hyväksyttäviä 84 %, mutta silloin määritettäviä PCB-yhdisteitä synteettisessä näytteessä oli enemmän [6].

Maanäytteen tulosten sallittiin poiketa vertailuarvosta 30 %, jolloin PCB-yhdisteiden tuloksista oli hyväksyttäviä 85 %. Vuoden 2005 vastaavassa vertailussa PCB-yhdisteiden tuloksista oli hyväksyttäviä 84 %.

Vaikka osallistujien käyttämät menetelmät, uutto- ja mittaustekniikat poikkesivat toisistaan huomattavasti, niin M1 näytteen tulokset olivat yllättävän vertailukelpoisia.

Tässä pätevyyskokeessa koko aineistossa hyväksyttäviä tuloksia oli yhteensä 76 %. Akkreditoituna ilmoitetuista tuloksista hyväksyttäviä oli 81 %.

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PARTICIPANTS

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Tubitak Butal, Bursa, Turkey

PREPARATION OF THE SAMPLES

Stock solution PCB Mix 20 (Certificated Dr. Ehrenstorfer PCB Mix 20): each PCB compounds 10 µg/mL in isooctane. Stock solution PCB Mix 1 (Certificated Dr. Ehrenstorfer PCB Mix 1); each PCB compounds 10 µg/mL in isooctane.

Preparing of sample A1: 0.4 mL (0.27628 g) of PCB Mix 20 and 0.8 mL (0.55954 g) of PCB Mix 1 were diluted with 33.51235 g of isooctane (Mix 20 + Mix 1). The sample A1 was prepared by diluting 3.94371 g of Mix 20 + 1 with 30.65627 g of isooctane.

Table 1. Concentrations of PCBs in the stock solutions and the sample A1

Compound	Concentration in stock solution PCB Mix 20, µg/mL	Concentration in stock solution PCB Mix 1, µg/mL	Concentration in Mix 20 + 1, µg/mL	Final PCB concentrations in the sample A1, ng/mL
PCB 28	10	10	0.2433	27.74
PCB 31	10		0.8044	9.17
PCB 52	10	10	0.2433	27.74
PCB 77	10		0.8044	9.17
PCB 101	10	10	0.2433	27.74
PCB 105	10		0.8044	9.17
PCB 118	10		0.8044	9.17
PCB 126	10		0.8044	9.17
PCB 128	10		0.8044	9.17
PCB 138	10	10	0.2433	27.74
PCB 153	10	10	0.2433	27.74
PCB 156	10		0.8044	9.17
PCB 169	10		0.8044	9.17
PCB 170	10		0.8044	9.17
PCB 180	10	10	0.2433	27.74

TESTING OF HOMOGENEITY

The homogeneity of the sample M1 was tested by analysing six sub samples as duplicate measurements.

Analyte / Sample	**Conc.** mg/kg	**s_p%**	**s_p**	**s_a**	**s_a / s_p**	**Was s_a / s_p < 0.5?**	**s_{bb}**	**s_{bb}^2**	**c**	**Was s_{bb}^2 < c?**
PCB 52 / M1	135	15	20.2	7.75	0.38	YES	5.48	30.0	183	YES
PCB 101 / M1	254	15	38.0	19.1	0.50	YES	13.51	182	904	YES
PCB 118 / M1	214	15	32.1	13.0	0.40	YES	9.17	84.1	489	YES
PCB 138 / M1	200	15	30.0	11.3	0.38	YES	7.99	63.8	395	YES
PCB 156 / M1	26.8	15	4.02	1.63	0.41	YES	1.15	1.33	7.70	YES
PCB 180 / M1	71.9	15	10.8	4.33	0.40	YES	3.06	9.36	54.8	YES

Conc. = Concentration
s_p = Target deviation, total target deviation / 2
s_p% = Target deviation as percent, total target deviation / 2
s_a = Analytical deviation, mean standard deviation of results in a sub sample
s_{bb} = Between-sample deviation, standard deviation of results between sub samples
c = $F1 \cdot s_{all}^2 + F2 \cdot s_a^2$
where:

$s_{all}^2 = (0.3 \cdot s_p)^2$
F1 = 2.21 when the number of sub samples is 6
F2 = 1.69 when the number of sub samples is 6

Conclusion: In each case $s_a / s_p < 0.5$ and $s_{bb}^2 < c$. The samples could be regarded as homogenous.

FEEDBACK FROM THE PROFICIENCY TEST

Lab	Comment	Action/SYKE
1	Method description sheet was not found on the electric result sheet.	The method sheet was completed and the corrected result sheet was sent to all participants by e-mail.
5, 7	The difference of the receiving weight of the sample A1 and the initial weight was more than 0.5 %.	A new sample A1 was sent to the participant.

FEEDBACK TO THE PARTICIPANTS

Lab	Comment
All	All participants sent their results on time.
6	Laboratory did not report duplicate results as it was requested.

ASSIGNED VALUES AND THEIR UNCERTAINTIES

Analyte Abbreviation	Sample	Assigned value	Unit	Evaluation of the assigned value	Uncertainty ($U = 2 u_c$) %
PCB-28	A1	27.74	ng/ml	Calculated	2.3
	M1	16.3	µg/kg	Robust mean	20
PCB-31	A1	9.17	ng/ml	Calculated	1.2
	M1	-	µg/kg	-	-
PCB-28+31	A1	36.91	ng/ml	Calculated	2.6
	M1	22.6	µg/kg	Robust mean	13
PCB-52	A1	27.74	ng/ml	Calculated	2.3
	M1	162	µg/kg	Robust mean	11
PCB-101	A1	27.74	ng/ml	Calculated	2.3
	M1	282	µg/kg	Robust mean	10
PCB-105	A1	9.17	ng/ml	Calculated	1.2
	M1	91.5	µg/kg	Robust mean	11
PCB-110	A1	-	-	-	-
	M1	-	-	-	-
PCB-118	A1	9.17	ng/ml	Calculated	1.2
	M1	245	µg/kg	Robust mean	5.3
PCB-138	A1	27.74	ng/ml	Calculated	2.3
	M1	271	µg/kg	Robust mean	16
PCB-153	A1	27.74	ng/ml	Calculated	2.3
	M1	222	µg/kg	Robust mean	9.6
PCB-156	A1	9.17	ng/ml	Calculated	1.2
	M1	27.1	µg/kg	Robust mean	22
PCB-170	A1	9.17	ng/ml	Calculated	1.2
	M1	-	µg/kg	Robust mean	-
PCB-180	A1	27.74	ng/ml	Calculated	2.3
	M1	83.6	µg/kg	Robust mean	5.9

TERMS IN THE RESULT TABLES

Results of each participants

Sample	the code of the sample
z-Graphics	z score - the graphical presentation
z value	calculated as follows: $z = (x_i - X)/s_p$, where x_i = the result of the individual laboratory X = the reference value (*the assigned value*) s_p = the target value of the standard deviation for proficiency assessment
Outl test OK	yes - the result passed the outlier test H = Hampel test (test for the mean value) C = Cochran test (replicate test)
Assigned value	the reference value
2* Targ SD %	the target value of total standard deviation for proficiency assessment (s_p) at the 95 % confidence level, equal $2 \cdot s_p$
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, %
Passed	The results passed the outlier test
Outl. failed	The results not passed the outlier test
Missing	i.e. < DL
Num of labs	the total number of the participants

Summary on the z scores

S – satisfactory ($-2 \leq z \leq 2$)
Q – questionable ($2 < z < 3$), positive error, the result deviates more than $2 \cdot s_p$ from the assigned value
q – questionable ($-3 > z > -2$), negative error, the result deviates more than $2 \cdot s_p$ from the assigned value
U – unsatisfactory ($z \geq 3$), positive error, the result deviates more than $3 \cdot s_p$ from the assigned value
u – unsatisfactory ($z \leq -3$), negative error, the result deviates more than $3 \cdot s_p$ from the assigned value

Robust analysis

X^* = median of x_i $(i = 1, 2, \ldots p)$
s^* = 1.483 median of $|x_i - x^*|$ $(i = 1, 2, \ldots p)$

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

$x_i^* = x^* - \varphi$ if $x_i < x^* - \varphi$
$x_i^* = x^* + \varphi$ if $x_i > x^* + \varphi$
$x_i^* = x_i$ otherwise

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.

Ref: Statistical methods for use in proficiency testing by inter laboratory comparisons, Annex C [3].

LIITE 7. RESULTS OF EACH PARTICIPANT

APPENDIX 7.

Analyte	Unit	Sample	z-Graphics -3 -2 -1 0 +1 +2 +3	Z- value	Outl test OK	Assig-ned value	2* Targ SD%	Lab's result	Md.	Mean	SD	SD%	Pas-sed	Outl. fai-led	Mis-sing	Num of labs
Laboratory 1																
PCB-101	ng/ml	A1		-1,820	yes	27,74	20	22,69	24,4	25,1	4,054	16,1	10	0	0	10
	µg/kg	M1		-0,216	yes	282	30	272,9	280	287,1	48,7	16,9	11	0	0	11
PCB-118	ng/ml	A1		-1,723	yes	9,17	20	7,59	7,8	7,802	1,295	16,5	9	1	0	10
	µg/kg	M1		1,182	yes	245	30	288,5	245	247,6	20,85	8,4	11	0	0	11
PCB-138	ng/ml	A1		-1,961	yes	27,74	20	22,3	24,8	25,06	4,106	16,3	10	0	0	10
	µg/kg	M1		1,220	yes	271	30	320,6	270	274,7	60,53	22,0	11	0	0	11
PCB-153	ng/ml	A1		-2,121	yes	27,74	20	21,85	24,3	24,46	4,285	17,5	10	0	0	10
	µg/kg	M1		5,947	H	222	30	420,1	220	221	23,56	10,6	10	1	0	11
PCB-180	ng/ml	A1		-2,053	yes	27,74	20	22,05	23,4	24,59	3,397	13,8	9	1	0	10
	µg/kg	M1		3,462	H	83,6	30	127	84,6	82,72	6,292	7,6	10	1	0	11
PCB-28+31	ng/ml	A1		-6,027	H	36,91	20	14,66	32,35	34,31	6,899	20,1	5	1	0	6
	µg/kg	M1		-2,789	yes	22,6	30	13,14	22,73	21,69	4,348	20,0	6	1	0	7
PCB-52	ng/ml	A1		-1,691	yes	27,74	20	23,05	23,29	23,32	3,05	13,0	8	2	0	10
	µg/kg	M1		0,133	yes	162	30	165,2	164,5	164	21,93	13,3	8	3	0	11
Laboratory 2																
PCB-101	ng/ml	A1		-0,267	yes	27,74	20	27	24,4	25,1	4,054	16,1	10	0	0	10
	µg/kg	M1		-0,047	yes	282	30	280	280	287,1	48,7	16,9	11	0	0	11
PCB-118	ng/ml	A1		0,851	yes	9,17	20	9,95	7,8	7,802	1,295	16,5	9	1	0	10
	µg/kg	M1		0,136	yes	245	30	250	245	247,6	20,85	8,4	11	0	0	11
PCB-138	ng/ml	A1		-0,447	yes	27,74	20	26,5	24,8	25,06	4,106	16,3	10	0	0	10
	µg/kg	M1		-0,025	yes	271	30	270	270	274,7	60,53	22,0	11	0	0	11
PCB-153	ng/ml	A1		-0,447	yes	27,74	20	26,5	24,3	24,46	4,285	17,5	10	0	0	10
	µg/kg	M1		-0,060	yes	222	30	220	220	221	23,56	10,6	10	1	0	11
PCB-180	ng/ml	A1		-0,429	yes	27,74	20	26,55	23,4	24,59	3,397	13,8	9	1	0	10
	µg/kg	M1		0,151	yes	83,6	30	85,5	84,6	82,72	6,292	7,6	10	1	0	11
PCB-28	ng/ml	A1		0,094	yes	27,74	20	28	23,8	25,9	5,135	19,8	9	0	0	9
	µg/kg	M1		0,161	yes	16,6	30	17	15,85	16,46	3,665	22,2	7	2	1	10
PCB-52	ng/ml	A1		-0,267	yes	27,74	20	27	23,29	23,32	3,05	13,0	8	2	0	10
	µg/kg	M1		0,535	yes	162	30	175	164,5	164	21,93	13,3	8	3	0	11
Laboratory 3																
PCB-101	µg/kg	M1		0,054	yes	282	30	284,3	280	287,1	48,7	16,9	11	0	0	11
PCB-105	µg/kg	M1		0,268	yes	91,5	30	95,17	93,16	91,53	8,38	9,2	5	1	0	6
PCB-110	µg/kg	M1			yes	309		327,7	314,4	308,6	20,34	6,6	3	0	0	3
PCB-118	µg/kg	M1		0,636	yes	245	30	268,4	245	247,6	20,85	8,4	11	0	0	11
PCB-138	µg/kg	M1		-0,460	yes	271	30	252,3	270	274,7	60,53	22,0	11	0	0	11
PCB-153	µg/kg	M1		0,505	yes	222	30	238,8	220	221	23,56	10,6	10	1	0	11
PCB-156	µg/kg	M1		-0,289	yes	27,1	30	25,92	26,17	27,14	4,456	16,4	5	1	0	6
PCB-170	µg/kg	M1			yes	47,6		41,14	49,37	47,62	6,086	12,7	5	0	0	5
PCB-180	µg/kg	M1		0,152	yes	83,6	30	85,5	84,6	82,72	6,292	7,6	10	1	0	11
PCB-28	µg/kg	M1		-0,115	yes	16,6	30	16,32	15,85	16,46	3,665	22,2	7	2	1	10
PCB-28+31	µg/kg	M1		0,707	yes	22,6	30	25	22,73	21,69	4,348	20,0	6	1	0	7
PCB-31	µg/kg	M1			yes	8,52		8,68	8,15	8,515	1,634	19,1	4	1	0	5
PCB-52	µg/kg	M1		0,134	yes	162	30	165,3	164,5	164	21,93	13,3	8	3	0	11

Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

Analyte	Unit	Sample	z-Graphics -3 -2 -1 0 +1 +2 +3	Z- value	Outl test OK	Assig-ned value	2* Targ SD%	Lab's result	Md.	Mean	SD	SD%	Pas-sed	Outl. fai-led	Mis-sing	Num of labs
Laboratory 4																
PCB-101	ng/ml	A1		-1,602	yes	27,74	20	23,3	24,4	25,1	4,054	16,1	10	0	0	10
	µg/kg	M1		-0,274	yes	282	30	270,4	280	287,1	48,7	16,9	11	0	0	11
PCB-105	ng/ml	A1		-2,339	yes	9,17	20	7,025	8,035	8,438	1,633	19,3	5	0	0	5
	µg/kg	M1		-0,182	yes	91,5	30	89	93,16	91,53	8,38	9,2	5	1	0	6
PCB-110	µg/kg	M1			yes	309		296,2	314,4	308,6	20,34	6,6	3	0	0	3
PCB-118	ng/ml	A1		-2,961	yes	9,17	20	6,455	7,8	7,802	1,295	16,5	9	1	0	10
	µg/kg	M1		0,308	yes	245	30	256,3	245	247,6	20,85	8,4	11	0	0	11
PCB-138	ng/ml	A1		-2,540	yes	27,74	20	20,7	24,8	25,06	4,106	16,3	10	0	0	10
	µg/kg	M1		-1,129	yes	271	30	225,1	270	274,7	60,53	22,0	11	0	0	11
PCB-153	ng/ml	A1		-2,536	yes	27,74	20	20,7	24,3	24,46	4,285	17,5	10	0	0	10
	µg/kg	M1		0,548	yes	222	30	240,2	220	221	23,56	10,6	10	1	0	11
PCB-156	ng/ml	A1		-2,874	yes	9,17	20	6,535	9,08	8,979	1,642	18,2	5	0	0	5
	µg/kg	M1		-0,422	yes	27,1	30	25,38	26,17	27,14	4,456	16,4	5	1	0	6
PCB-170	ng/ml	A1		-2,677	yes	9,17	20	6,715	9,205	9,068	1,754	19,3	4	0	0	4
	µg/kg	M1			yes	47,6		40,7	49,37	47,62	6,086	12,7	5	0	0	5
PCB-180	ng/ml	A1		-2,684	yes	27,74	20	20,3	23,4	24,59	3,397	13,8	9	1	0	10
	µg/kg	M1		0,092	yes	83,6	30	84,76	84,6	82,72	6,292	7,6	10	1	0	11
PCB-28	ng/ml	A1		-1,703	yes	27,74	20	23,02	23,8	25,9	5,135	19,8	9	0	0	9
	µg/kg	M1		-1,201	yes	16,6	30	13,61	15,85	16,46	3,665	22,2	7	2	1	10
PCB-28+31	ng/ml	A1		-1,533	yes	36,91	20	31,25	32,35	34,31	6,899	20,1	5	1	0	6
	µg/kg	M1		-0,389	yes	22,6	30	21,28	22,73	21,69	4,348	20,0	6	1	0	7
PCB-31	ng/ml	A1		-1,020	yes	9,17	20	8,235	6,34	6,787	1,178	17,3	3	1	0	4
	µg/kg	M1			yes	8,52		7,67	8,15	8,515	1,634	19,1	4	1	0	5
PCB-52	ng/ml	A1		-1,619	yes	27,74	20	23,25	23,29	23,32	3,05	13,0	8	2	0	10
	µg/kg	M1		-0,576	yes	162	30	148	164,5	164	21,93	13,3	8	3	0	11
Laboratory 5																
PCB-101	ng/ml	A1		-0,952	yes	27,74	20	25,1	24,4	25,1	4,054	16,1	10	0	0	10
	µg/kg	M1		-0,949	yes	282	30	241,8	280	287,1	48,7	16,9	11	0	0	11
PCB-105	ng/ml	A1		-1,985	yes	9,17	20	7,35	8,035	8,438	1,633	19,3	5	0	0	5
	µg/kg	M1		0,528	yes	91,5	30	98,75	93,16	91,53	8,38	9,2	5	1	0	6
PCB-118	ng/ml	A1		-1,276	yes	9,17	20	8	7,8	7,802	1,295	16,5	9	1	0	10
	µg/kg	M1		-0,203	yes	245	30	237,6	245	247,6	20,85	8,4	11	0	0	11
PCB-138	ng/ml	A1		-0,952	yes	27,74	20	25,1	24,8	25,06	4,106	16,3	10	0	0	10
	µg/kg	M1		0,857	yes	271	30	305,9	270	274,7	60,53	22,0	11	0	0	11
PCB-153	ng/ml	A1		-1,402	yes	27,74	20	23,85	24,3	24,46	4,285	17,5	10	0	0	10
	µg/kg	M1		-0,794	yes	222	30	195,6	220	221	23,56	10,6	10	1	0	11
PCB-156	ng/ml	A1		-0,622	yes	9,17	20	8,6	9,08	8,979	1,642	18,2	5	0	0	5
	µg/kg	M1		24,830	H	27,1	30	128,1	26,17	27,14	4,456	16,4	5	1	0	6
PCB-170	ng/ml	A1		-0,240	yes	9,17	20	8,95	9,205	9,068	1,754	19,3	4	0	0	4
	µg/kg	M1			yes	47,6		51,9	49,37	47,62	6,086	12,7	5	0	0	5
PCB-180	ng/ml	A1		-0,663	yes	27,74	20	25,9	23,4	24,59	3,397	13,8	9	1	0	10
	µg/kg	M1		-0,183	yes	83,6	30	81,3	84,6	82,72	6,292	7,6	10	1	0	11
PCB-28	ng/ml	A1		-1,853	yes	27,74	20	22,6	23,8	25,9	5,135	19,8	9	0	0	9
	µg/kg	M1		-5,301	H	16,6	30	3,4	15,85	16,46	3,665	22,2	7	2	1	10
PCB-28+31	ng/ml	A1		-2,306	yes	36,91	20	28,4	32,35	34,31	6,899	20,1	5	1	0	6
	µg/kg	M1		-5,295	H	22,6	30	4,65	22,73	21,69	4,348	20,0	6	1	0	7
PCB-31	ng/ml	A1		-3,675	yes	9,17	20	5,8	6,34	6,787	1,178	17,3	3	1	0	4
	µg/kg	M1			M	8,52		1,25	8,15	8,515	1,634	19,1	4	1	0	5
PCB-52	ng/ml	A1		-1,312	yes	27,74	20	24,1	23,29	23,32	3,05	13,0	8	2	0	10
	µg/kg	M1		11,000	H	162	30	429,3	164,5	164	21,93	13,3	8	3	0	11
Laboratory 6																
PCB-101	ng/ml	A1		-2,105	yes	27,74	20	21,9	24,4	25,1	4,054	16,1	10	0	0	10
	µg/kg	M1		0,686	yes	282	30	311	280	287,1	48,7	16,9	11	0	0	11
PCB-118	ng/ml	A1		-1,210	yes	9,17	20	8,06	7,8	7,802	1,295	16,5	9	1	0	10
	µg/kg	M1		-0,381	yes	245	30	231	245	247,6	20,85	8,4	11	0	0	11
PCB-138	ng/ml	A1		-1,420	yes	27,74	20	23,8	24,8	25,06	4,106	16,3	10	0	0	10
	µg/kg	M1		0,566	yes	271	30	294	270	274,7	60,53	22,0	11	0	0	11
PCB-153	ng/ml	A1		-1,132	yes	27,74	20	24,6	24,3	24,46	4,285	17,5	10	0	0	10
	µg/kg	M1		0,661	yes	222	30	244	220	221	23,56	10,6	10	1	0	11
PCB-180	ng/ml	A1		-1,565	yes	27,74	20	23,4	23,4	24,59	3,397	13,8	9	1	0	10
	µg/kg	M1		0,654	yes	83,6	30	91,8	84,6	82,72	6,292	7,6	10	1	0	11
PCB-28	ng/ml	A1		-1,420	yes	27,74	20	23,8	23,8	25,9	5,135	19,8	9	0	0	9
	µg/kg	M1		1,767	yes	16,6	30	21,0	15,85	16,46	3,665	22,2	7	2	1	10
PCB-52	ng/ml	A1		-1,889	yes	27,74	20	22,5	23,29	23,32	3,05	13,0	8	2	0	10
	µg/kg	M1		24,070	H	162	30	747	164,5	164	21,93	13,3	8	3	0	11

Analyte	Unit	Sample	z-Graphics -3 -2 -1 0 +1 +2 +3	Z- value	Outl test OK	Assig-ned value	2* Targ SD%	Lab's result	Md.	Mean	SD	SD%	Pas-sed	Outl. fai-led	Mis-sing	Num of labs
Laboratory 7																
PCB-101	ng/ml	A1		-2,880	yes	27,74	20	19,75	24,4	25,1	4,054	16,1	10	0	0	10
	µg/kg	M1		-0,485	yes	282	30	261,5	280	287,1	48,7	16,9	11	0	0	11
PCB-118	ng/ml	A1		-1,658	yes	9,17	20	7,65	7,8	7,802	1,295	16,5	9	1	0	10
	µg/kg	M1		-0,150	yes	245	30	239,5	245	247,6	20,85	8,4	11	0	0	11
PCB-138	ng/ml	A1		-1,943	yes	27,74	20	22,35	24,8	25,06	4,106	16,3	10	0	0	10
	µg/kg	M1		-1,747	yes	271	30	200	270	274,7	60,53	22,0	11	0	0	11
PCB-153	ng/ml	A1		-2,718	yes	27,74	20	20,2	24,3	24,46	4,285	17,5	10	0	0	10
	µg/kg	M1		0,090	yes	222	30	225	220	221	23,56	10,6	10	1	0	11
PCB-180	ng/ml	A1		-1,781	yes	27,74	20	22,8	23,4	24,59	3,397	13,8	9	1	0	10
	µg/kg	M1		-1,085	yes	83,6	30	70	84,6	82,72	6,292	7,6	10	1	0	11
PCB-28	ng/ml	A1		-1,979	yes	27,74	20	22,25	23,8	25,9	5,135	19,8	9	0	0	9
	µg/kg	M1		-1,446	yes	16,6	30	13	15,85	16,46	3,665	22,2	7	2	1	10
PCB-52	ng/ml	A1		-3,259	yes	27,74	20	18,7	23,29	23,32	3,05	13,0	8	2	0	10
	µg/kg	M1		-0,473	yes	162	30	150,5	164,5	164	21,93	13,3	8	3	0	11
Laboratory 8																
PCB-101	ng/ml	A1		-0,050	yes	27,74	20	27,6	24,4	25,1	4,054	16,1	10	0	0	10
	µg/kg	M1		-0,166	yes	282	30	275	280	287,1	48,7	16,9	11	0	0	11
PCB-105	ng/ml	A1		-0,632	yes	9,17	20	8,59	8,035	8,438	1,633	19,3	5	0	0	5
	µg/kg	M1		0,281	yes	91,5	30	95,35	93,16	91,53	8,38	9,2	5	1	0	6
PCB-110	ng/ml	A1						<1,5			1,7E308		0	0	1	1
	µg/kg	M1			yes	309		302	314,4	308,6	20,34	6,6	3	0	0	3
PCB-118	ng/ml	A1		-0,071	yes	9,17	20	9,105	7,8	7,802	1,295	16,5	9	1	0	10
	µg/kg	M1		0,245	yes	245	30	254	245	247,6	20,85	8,4	11	0	0	11
PCB-138	ng/ml	A1		-0,321	yes	27,74	20	26,85	24,8	25,06	4,106	16,3	10	0	0	10
	µg/kg	M1		-1,525	yes	271	30	209	270	274,7	60,53	22,0	11	0	0	11
PCB-153	ng/ml	A1		-0,789	yes	27,74	20	25,55	24,3	24,46	4,285	17,5	10	0	0	10
	µg/kg	M1		-0,676	yes	222	30	199,5	220	221	23,56	10,6	10	1	0	11
PCB-156	ng/ml	A1		-0,098	yes	9,17	20	9,08	9,08	8,979	1,642	18,2	5	0	0	5
	µg/kg	M1		0,320	yes	27,1	30	28,4	26,17	27,14	4,456	16,4	5	1	0	6
PCB-170	ng/ml	A1		0,120	yes	9,17	20	9,28	9,205	9,068	1,754	19,3	4	0	0	4
	µg/kg	M1			yes	47,6		52,75	49,37	47,62	6,086	12,7	5	0	0	5
PCB-180	ng/ml	A1		0,202	yes	27,74	20	28,3	23,4	24,59	3,397	13,8	9	1	0	10
	µg/kg	M1		0,359	yes	83,6	30	88,1	84,6	82,72	6,292	7,6	10	1	0	11
PCB-28	ng/ml	A1		-0,537	yes	27,74	20	26,25	23,8	25,9	5,135	19,8	9	0	0	9
	µg/kg	M1		-0,382	yes	16,6	30	15,65	15,85	16,46	3,665	22,2	7	2	1	10
PCB-28+31	ng/ml	A1		-1,168	yes	36,91	20	32,6	32,35	34,31	6,899	20,1	5	1	0	6
	µg/kg	M1		-0,029	yes	22,6	30	22,5	22,73	21,69	4,348	20,0	6	1	0	7
PCB-31	ng/ml	A1		-3,103	yes	9,17	20	6,325	6,34	6,787	1,178	17,3	3	1	0	4
	µg/kg	M1			yes	8,52		6,875	8,15	8,515	1,634	19,1	4	1	0	5
PCB-52	ng/ml	A1		-0,068	yes	27,74	20	27,55	23,29	23,32	3,05	13,0	8	2	0	10
	µg/kg	M1		0,206	yes	162	30	167	164,5	164	21,93	13,3	8	3	0	11

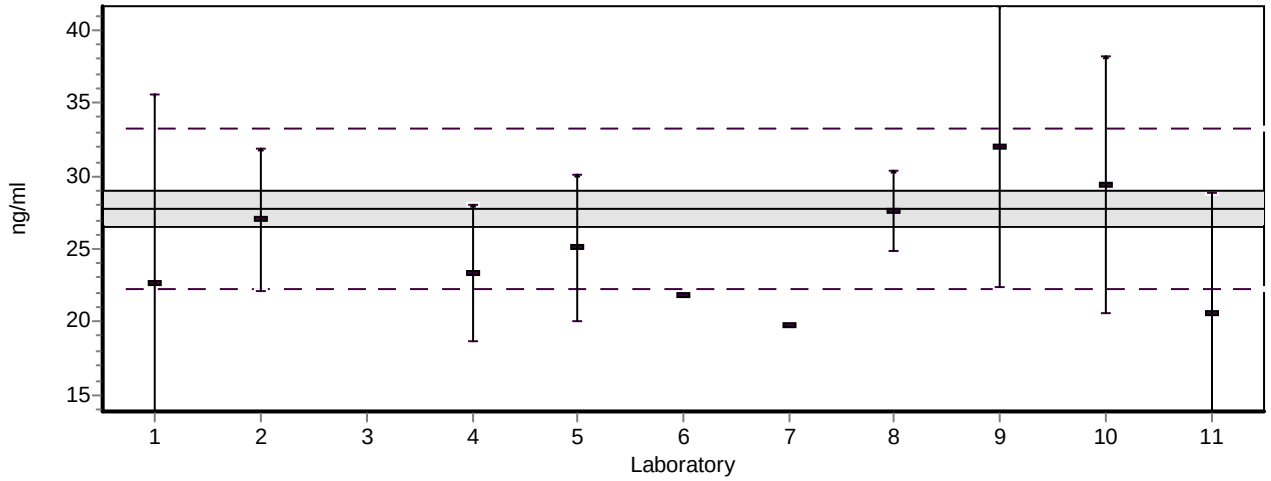
Analyte	Unit	Sample	z-Graphics -3 -2 -1 0 +1 +2 +3	Z- value	Outl test OK	Assig-ned value	2* Targ SD%	Lab's result	Md.	Mean	SD	SD%	Pas-sed	Outl. fai-led	Mis-sing	Num of labs
Laboratory 9																
PCB-101	ng/ml	A1		1,553	yes	27,74	20	32,05	24,4	25,1	4,054	16,1	10	0	0	10
	µg/kg	M1		2,912	yes	282	30	405,2	280	287,1	48,7	16,9	11	0	0	11
PCB-105	ng/ml	A1		2,191	yes	9,17	20	11,18	8,035	8,438	1,633	19,3	5	0	0	5
	µg/kg	M1		-0,882	yes	91,5	30	79,39	93,16	91,53	8,38	9,2	5	1	0	6
PCB-118	ng/ml	A1		2,220	C	9,17	20	11,21	7,8	7,802	1,295	16,5	9	1	0	10
	µg/kg	M1		-0,367	yes	245	30	231,5	245	247,6	20,85	8,4	11	0	0	11
PCB-138	ng/ml	A1		1,442	yes	27,74	20	31,74	24,8	25,06	4,106	16,3	10	0	0	10
	µg/kg	M1		3,268	yes	271	30	403,9	270	274,7	60,53	22,0	11	0	0	11
PCB-153	ng/ml	A1		1,006	yes	27,74	20	30,53	24,3	24,46	4,285	17,5	10	0	0	10
	µg/kg	M1		-0,614	yes	222	30	201,5	220	221	23,56	10,6	10	1	0	11
PCB-156	ng/ml	A1		2,022	yes	9,17	20	11,02	9,08	8,979	1,642	18,2	5	0	0	5
	µg/kg	M1		-1,332	yes	27,1	30	21,69	26,17	27,14	4,456	16,4	5	1	0	6
PCB-170	ng/ml	A1		2,352	yes	9,17	20	11,33	9,205	9,068	1,754	19,3	4	0	0	4
	µg/kg	M1			yes	47,6		51,59	49,37	47,62	6,086	12,7	5	0	0	5
PCB-180	ng/ml	A1		0,901	C	27,74	20	30,24	23,4	24,59	3,397	13,8	9	1	0	10
	µg/kg	M1		0,010	yes	83,6	30	83,72	84,6	82,72	6,292	7,6	10	1	0	11
PCB-28	ng/ml	A1		2,089	yes	27,74	20	33,54	23,8	25,9	5,135	19,8	9	0	0	9
	µg/kg	M1		-1,124	yes	16,6	30	13,8	15,85	16,46	3,665	22,2	7	2	1	10
PCB-28+31	ng/ml	A1		2,665	yes	36,91	20	46,75	32,35	34,31	6,899	20,1	5	1	0	6
	µg/kg	M1		0,600	yes	22,6	30	24,63	22,73	21,69	4,348	20,0	6	1	0	7
PCB-31	ng/ml	A1		4,408	H	9,17	20	13,21	6,34	6,787	1,178	17,3	3	1	0	4
	µg/kg	M1			yes	8,52		10,84	8,15	8,515	1,634	19,1	4	1	0	5
PCB-52	ng/ml	A1		1,635	C	27,74	20	32,28	23,29	23,32	3,05	13,0	8	2	0	10
	µg/kg	M1		1,842	yes	162	30	206,8	164,5	164	21,93	13,3	8	3	0	11
Laboratory 10																
PCB-101	ng/ml	A1		0,580	yes	27,74	20	29,35	24,4	25,1	4,054	16,1	10	0	0	10
	µg/kg	M1		-1,158	yes	282	30	233	280	287,1	48,7	16,9	11	0	0	11
PCB-105	ng/ml	A1		-1,227	yes	9,17	20	8,045	8,035	8,438	1,633	19,3	5	0	0	5
	µg/kg	M1		9,581	H	91,5	30	223	93,16	91,53	8,38	9,2	5	1	0	6
PCB-118	ng/ml	A1		-1,309	yes	9,17	20	7,97	7,8	7,802	1,295	16,5	9	1	0	10
	µg/kg	M1		-0,449	yes	245	30	228,5	245	247,6	20,85	8,4	11	0	0	11
PCB-138	ng/ml	A1		0,995	yes	27,74	20	30,5	24,8	25,06	4,106	16,3	10	0	0	10
	µg/kg	M1		0,996	yes	271	30	311,5	270	274,7	60,53	22,0	11	0	0	11
PCB-153	ng/ml	A1		1,211	yes	27,74	20	31,1	24,3	24,46	4,285	17,5	10	0	0	10
	µg/kg	M1		1,201	yes	222	30	262	220	221	23,56	10,6	10	1	0	11
PCB-156	ng/ml	A1		0,529	yes	9,17	20	9,655	9,08	8,979	1,642	18,2	5	0	0	5
	µg/kg	M1		1,771	yes	27,1	30	34,3	26,17	27,14	4,456	16,4	5	1	0	6
PCB-180	ng/ml	A1		0,905	yes	27,74	20	30,25	23,4	24,59	3,397	13,8	9	1	0	10
	µg/kg	M1		0,211	yes	83,6	30	86,25	84,6	82,72	6,292	7,6	10	1	0	11
PCB-28	ng/ml	A1		2,094	yes	27,74	20	33,55	23,8	25,9	5,135	19,8	9	0	0	9
	µg/kg	M1		2,811	yes	16,6	30	23,6	15,85	16,46	3,665	22,2	7	2	1	10
PCB-28+31	ng/ml	A1		-1,181	yes	36,91	20	32,55	32,35	34,31	6,899	20,1	5	1	0	6
	µg/kg	M1		0,295	yes	22,6	30	23,6	22,73	21,69	4,348	20,0	6	1	0	7
PCB-52	ng/ml	A1		0,815	C	27,74	20	30	23,29	23,32	3,05	13,0	8	2	0	10
	µg/kg	M1		11,130	H	162	30	432,5	164,5	164	21,93	13,3	8	3	0	11
Laboratory 11																
PCB-101	ng/ml	A1		-2,567	yes	27,74	20	20,62	24,4	25,1	4,054	16,1	10	0	0	10
	µg/kg	M1		1,241	yes	282	30	334,5	280	287,1	48,7	16,9	11	0	0	11
PCB-118	ng/ml	A1		-3,926	yes	9,17	20	5,57	7,8	7,802	1,295	16,5	9	1	0	10
	µg/kg	M1		-0,395	yes	245	30	230,5	245	247,6	20,85	8,4	11	0	0	11
PCB-138	ng/ml	A1		-2,740	yes	27,74	20	20,14	24,8	25,06	4,106	16,3	10	0	0	10
	µg/kg	M1		-0,775	yes	271	30	239,5	270	274,7	60,53	22,0	11	0	0	11
PCB-153	ng/ml	A1		-2,862	yes	27,74	20	19,8	24,3	24,46	4,285	17,5	10	0	0	10
	µg/kg	M1		-0,811	yes	222	30	195	220	221	23,56	10,6	10	1	0	11
PCB-180	ng/ml	A1		-2,354	yes	27,74	20	21,21	23,4	24,59	3,397	13,8	9	1	0	10
	µg/kg	M1		-0,698	yes	83,6	30	74,85	84,6	82,72	6,292	7,6	10	1	0	11
PCB-28	ng/ml	A1		-3,127	yes	27,74	20	19,06	23,8	25,9	5,135	19,8	9	0	0	9
	µg/kg	M1			H	16,6	30	<6,3	15,85	16,46	3,665	22,2	7	2	1	10
PCB-52	ng/ml	A1		-2,799	yes	27,74	20	19,98	23,29	23,32	3,05	13,0	8	2	0	10
	µg/kg	M1		-1,152	yes	162	30	134	164,5	164	21,93	13,3	8	3	0	11

Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

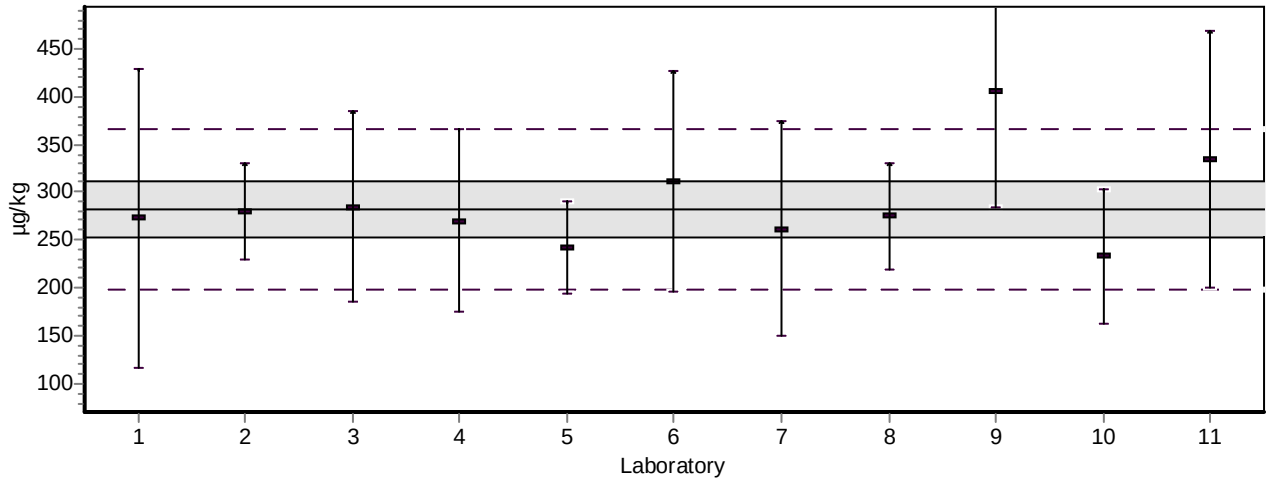
LIITE 8. RESULTS AND THEIR UNCERTAINTIES

APPENDIX 8.

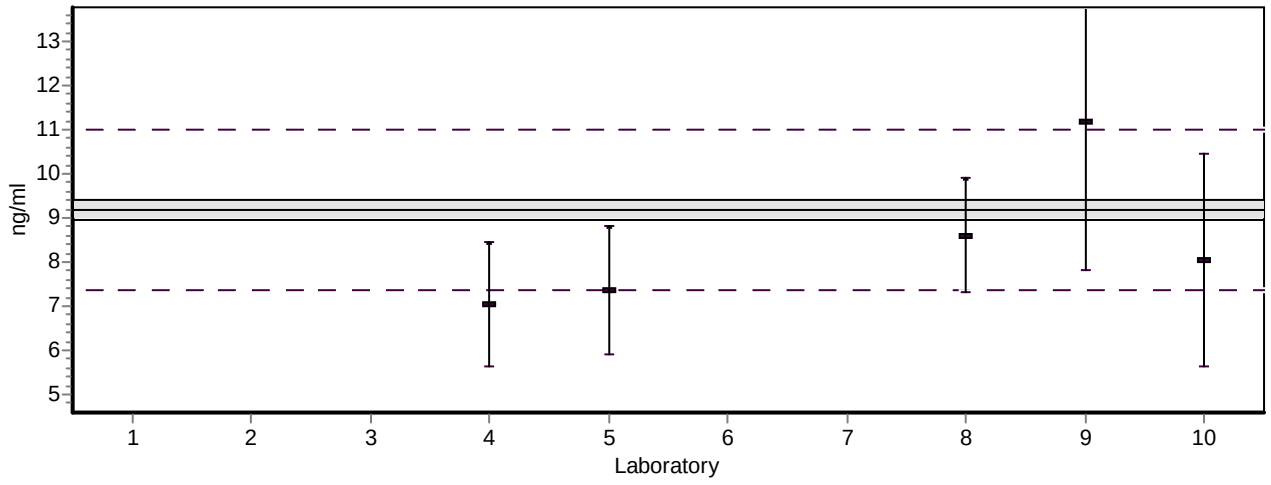
Analyytti *(Analyte)* **PCB-101** Näyte *(Sample)* A1



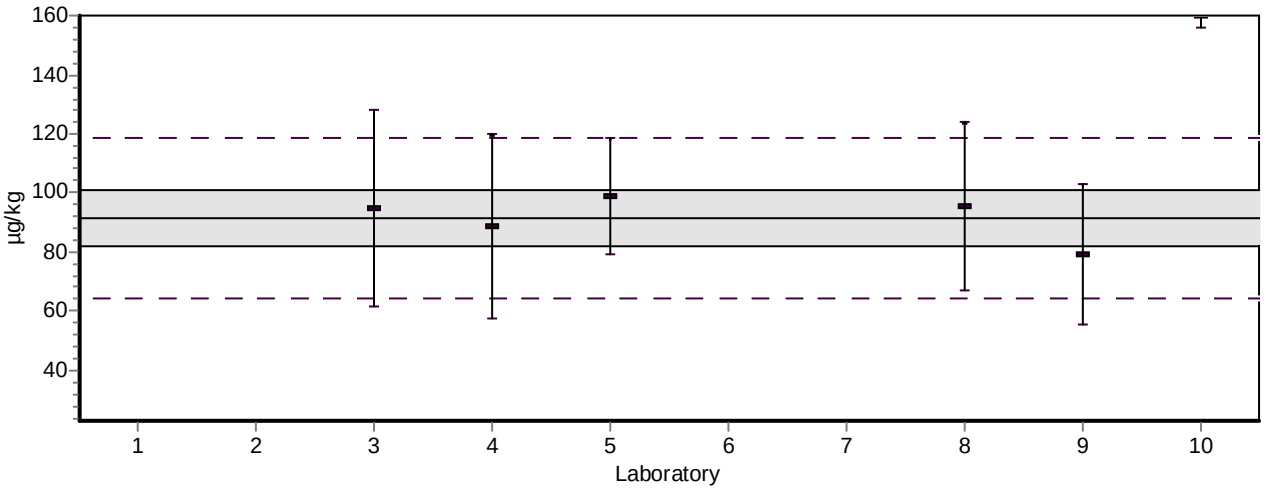
Analyytti *(Analyte)* **PCB-101** Näyte *(Sample)* M1



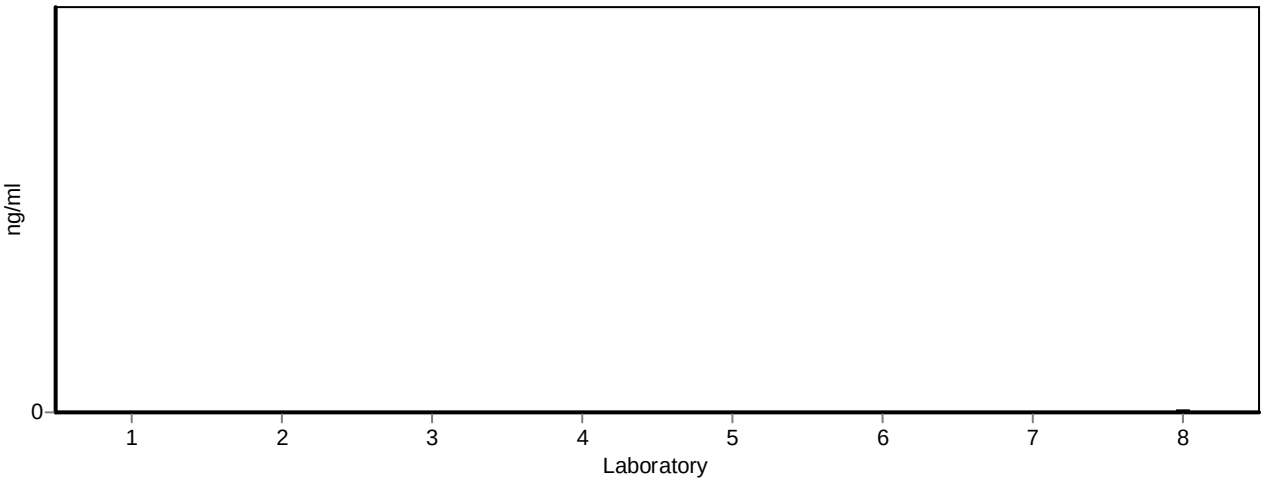
Analyytti *(Analyte)* **PCB-105** Näyte *(Sample)* A1



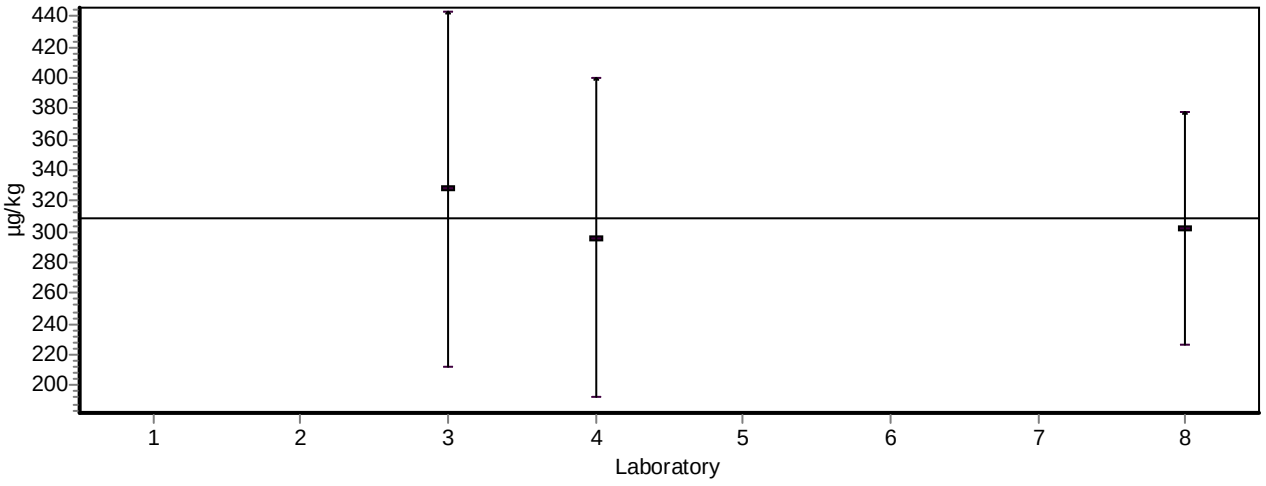
Analyytti *(Analyte)* **PCB-105** Näyte *(Sample)* M1



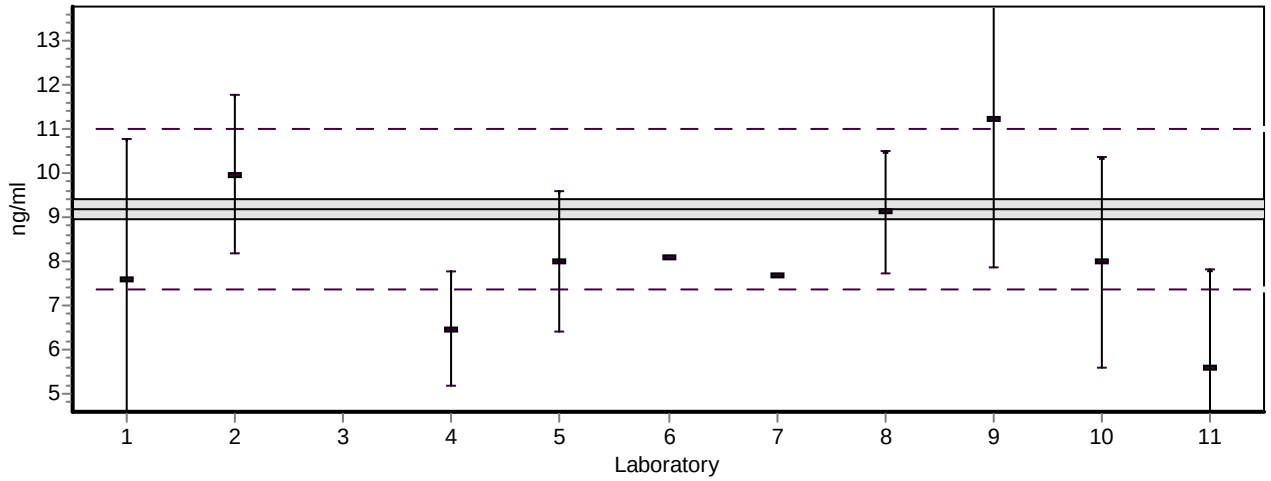
Analyytti *(Analyte)* **PCB-110** Näyte *(Sample)* A1



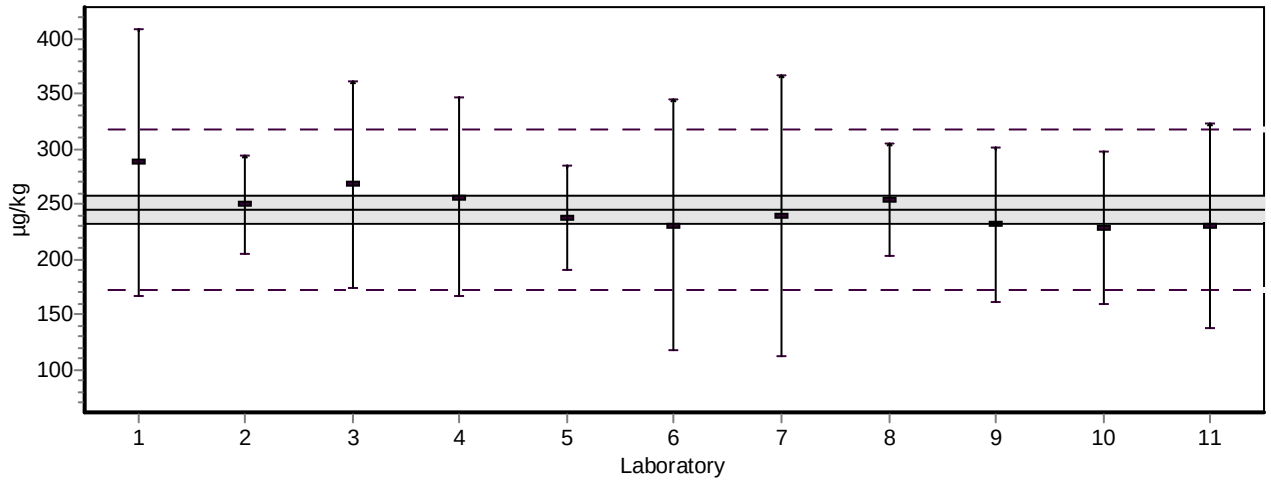
Analyytti *(Analyte)* **PCB-110** Näyte *(Sample)* M1



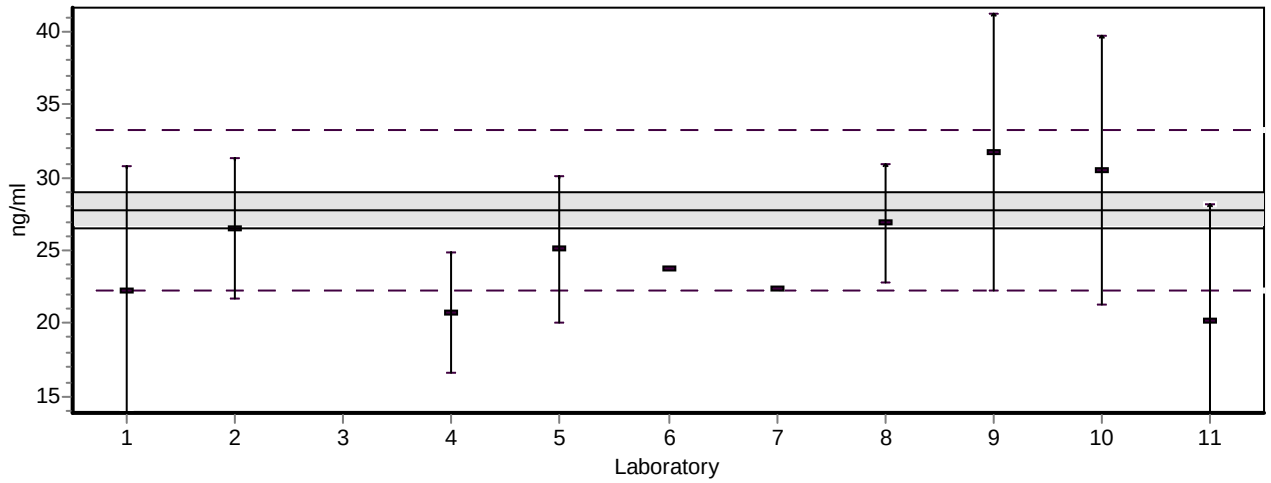
Analyytti *(Analyte)* **PCB-118** Näyte *(Sample)* A1



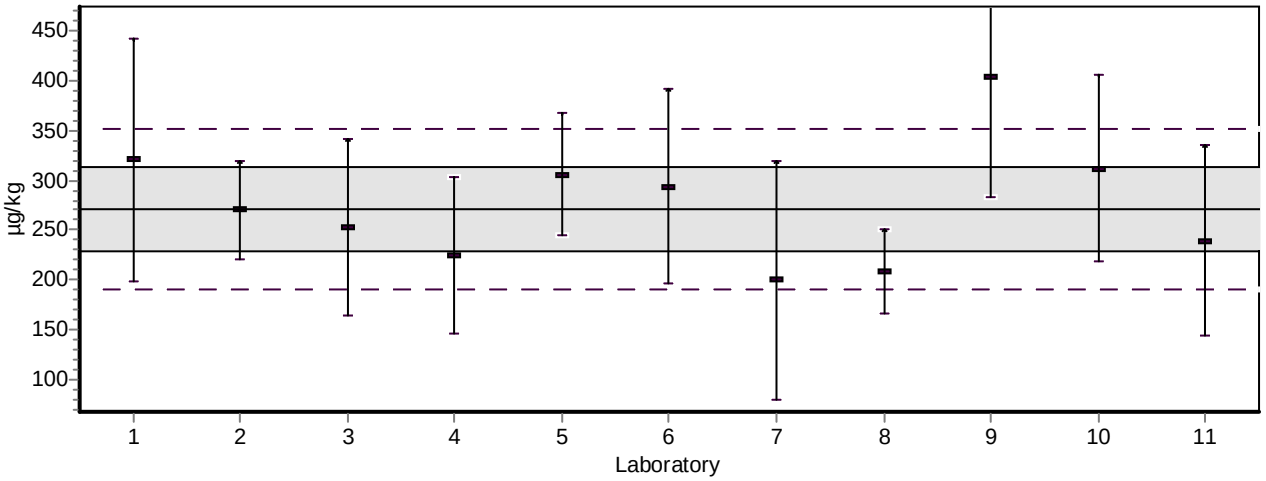
Analyytti *(Analyte)* **PCB-118** Näyte *(Sample)* M1



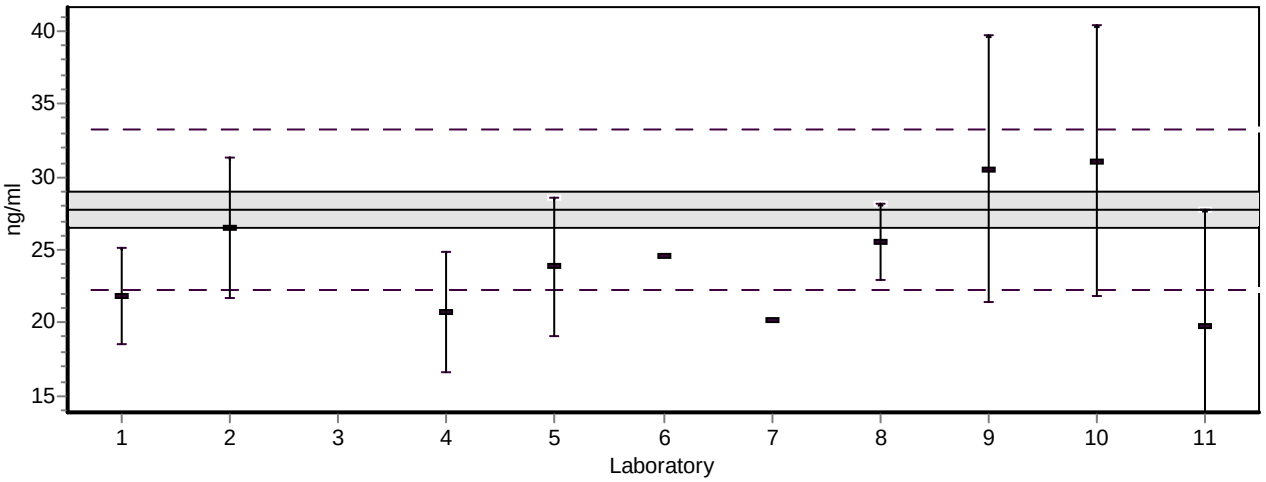
Analyytti *(Analyte)* **PCB-138** Näyte *(Sample)* A1



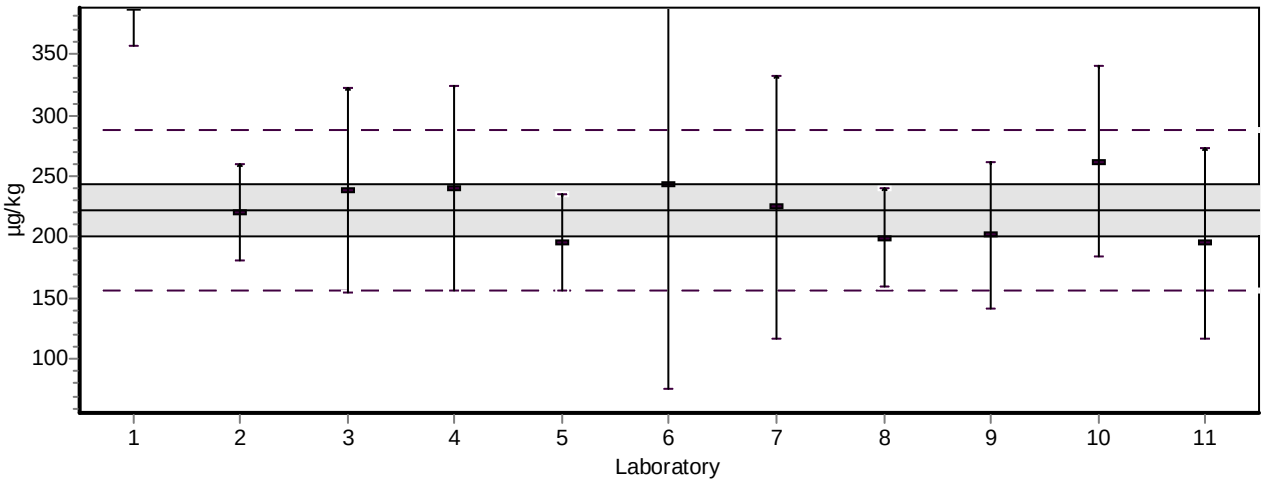
Analyytti *(Analyte)* **PCB-138** Näyte *(Sample)* M1



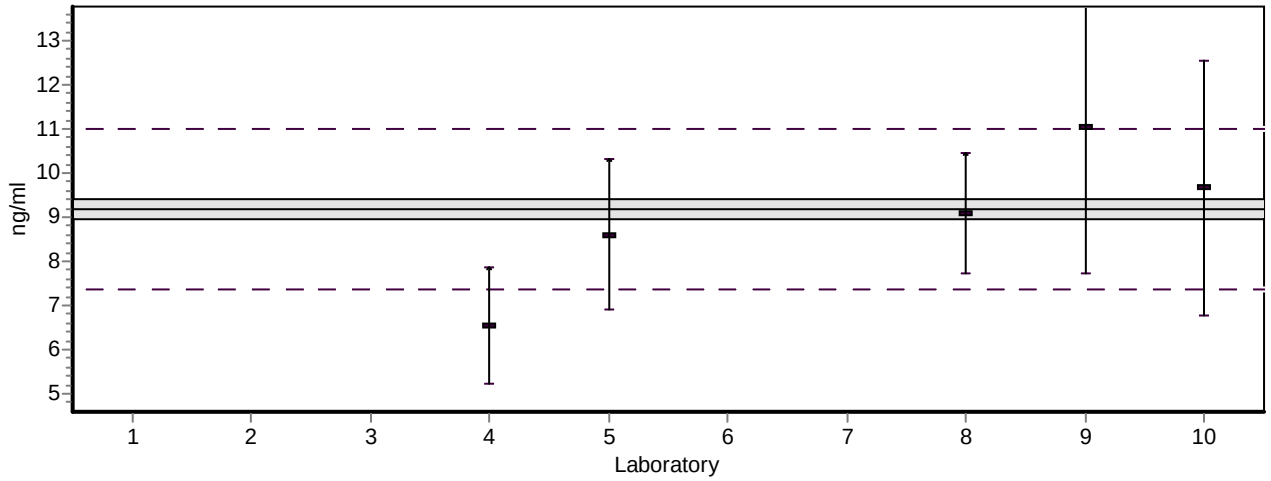
Analyytti *(Analyte)* **PCB-153** Näyte *(Sample)* A1



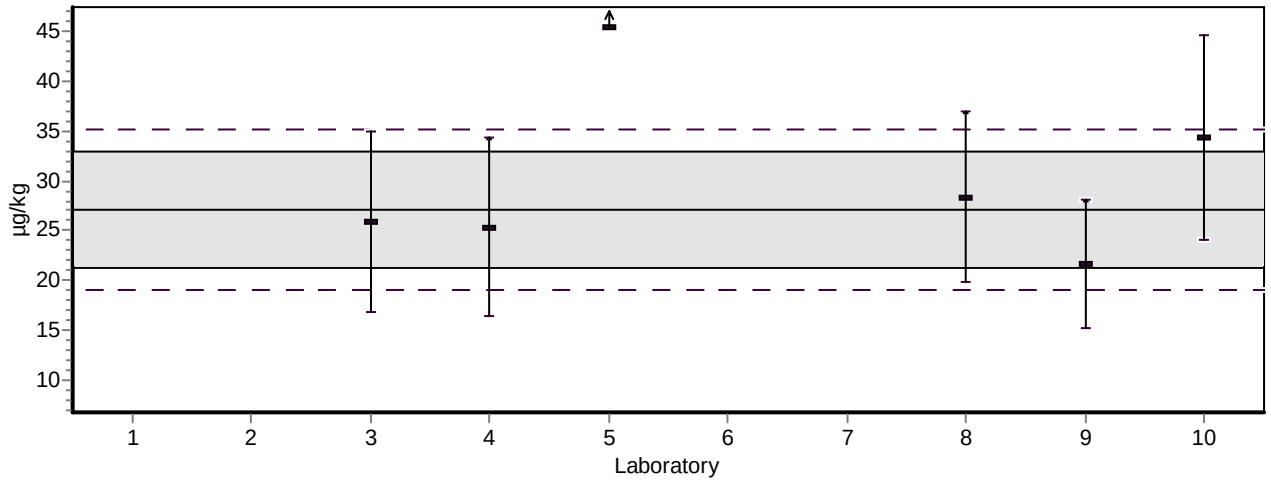
Analyytti *(Analyte)* **PCB-153** Näyte *(Sample)* M1



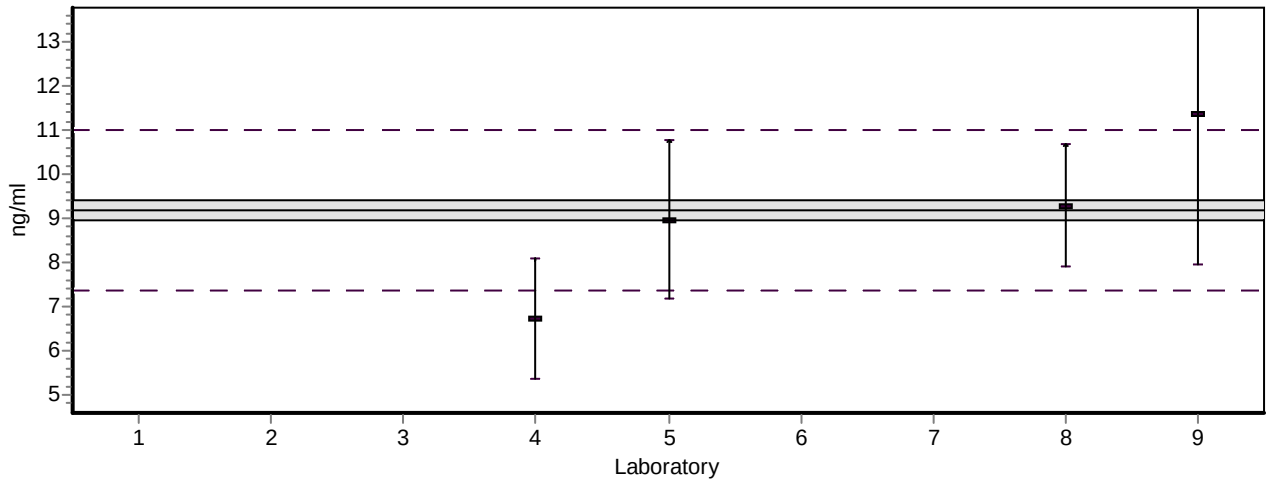
Analyytti *(Analyte)* **PCB-156** Näyte *(Sample)* A1



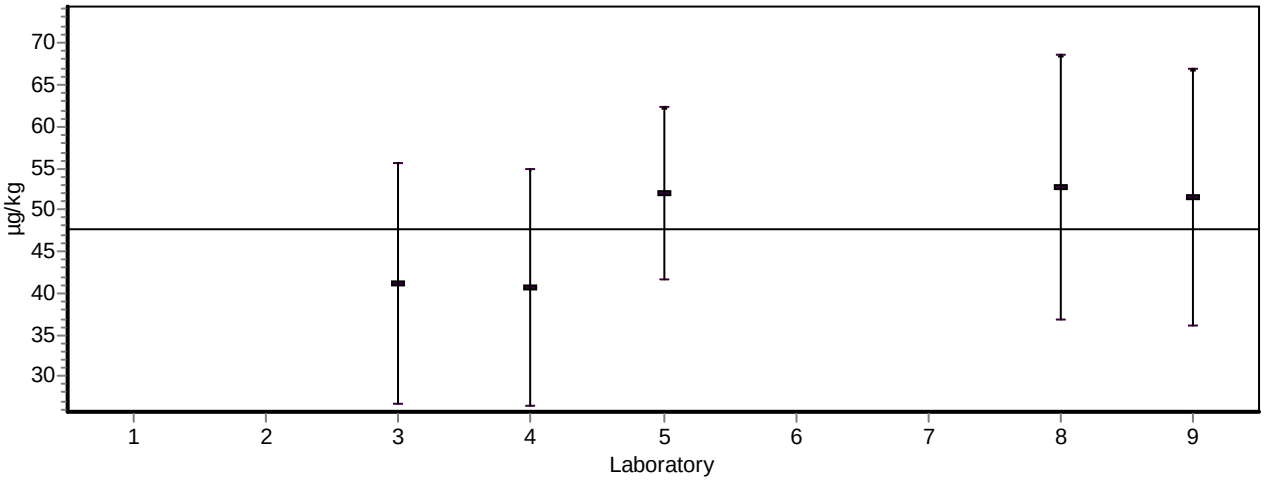
Analyytti *(Analyte)* **PCB-156** Näyte *(Sample)* M1



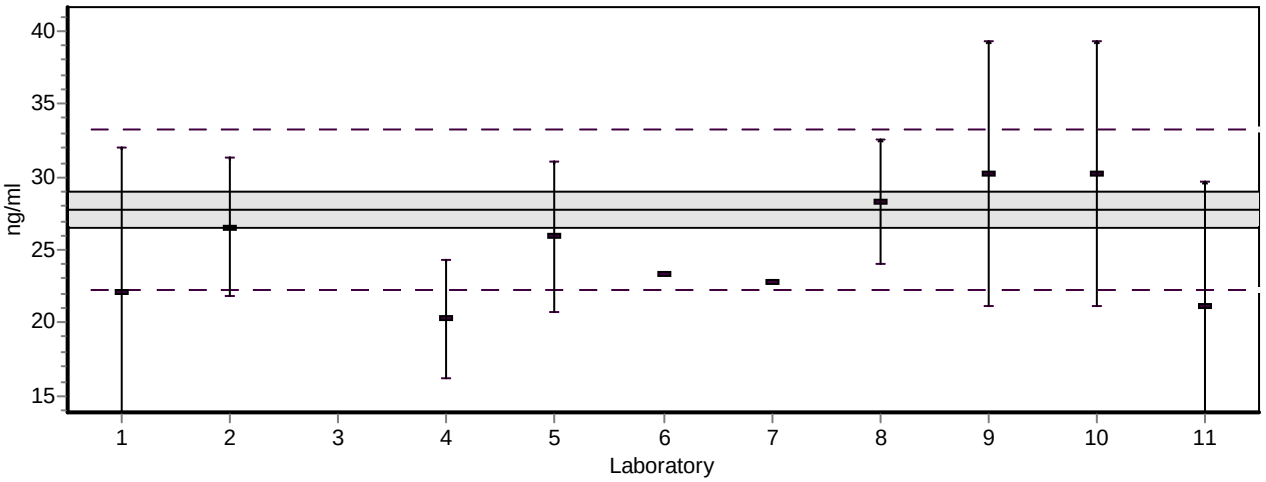
Analyytti *(Analyte)* **PCB-170** Näyte *(Sample)* A1



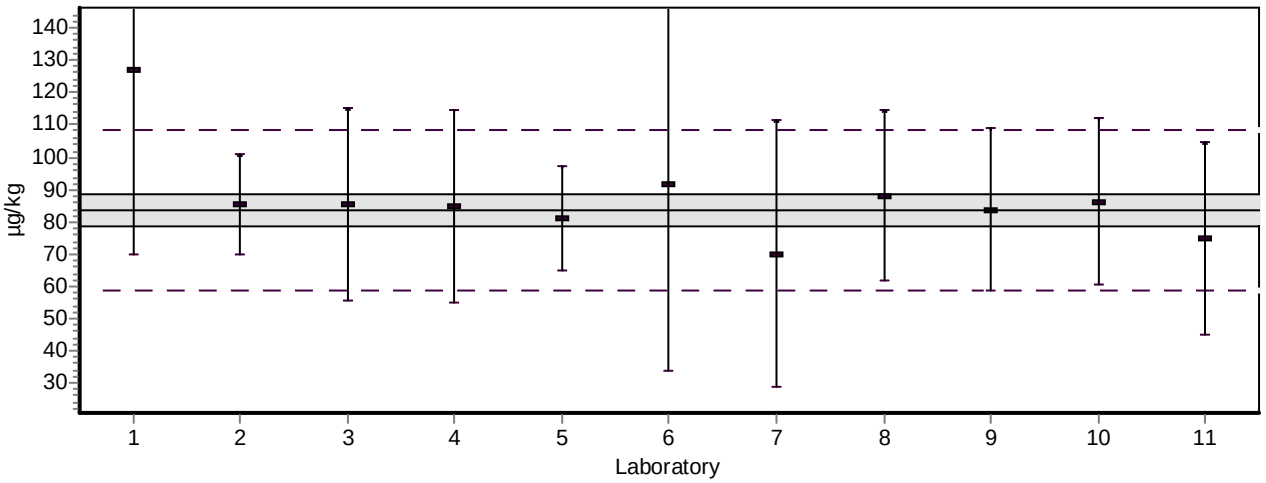
Analyytti *(Analyte)* **PCB-170** Näyte *(Sample)* M1



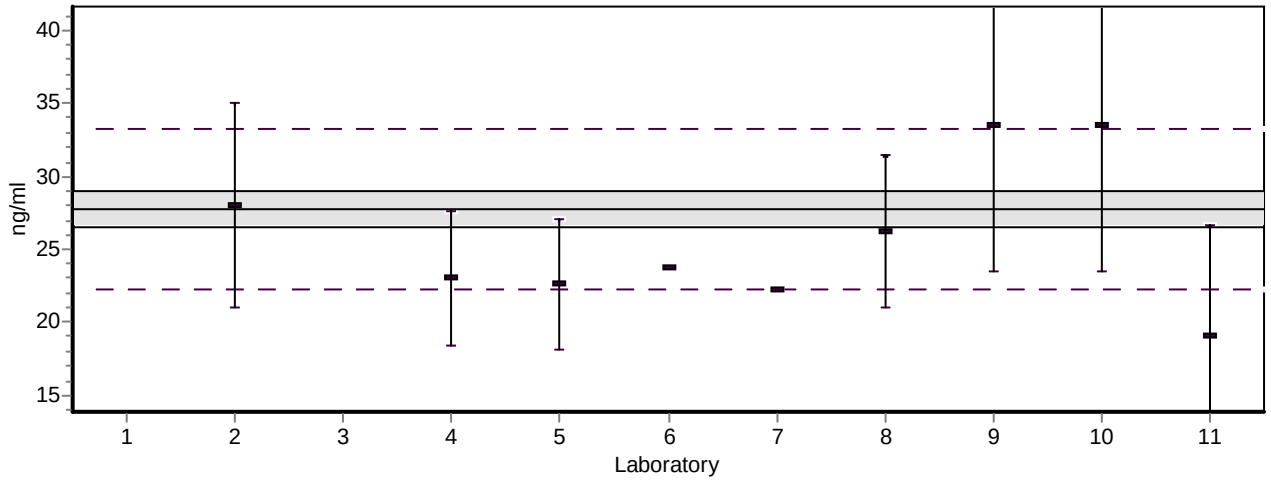
Analyytti *(Analyte)* **PCB-180** Näyte *(Sample)* A1



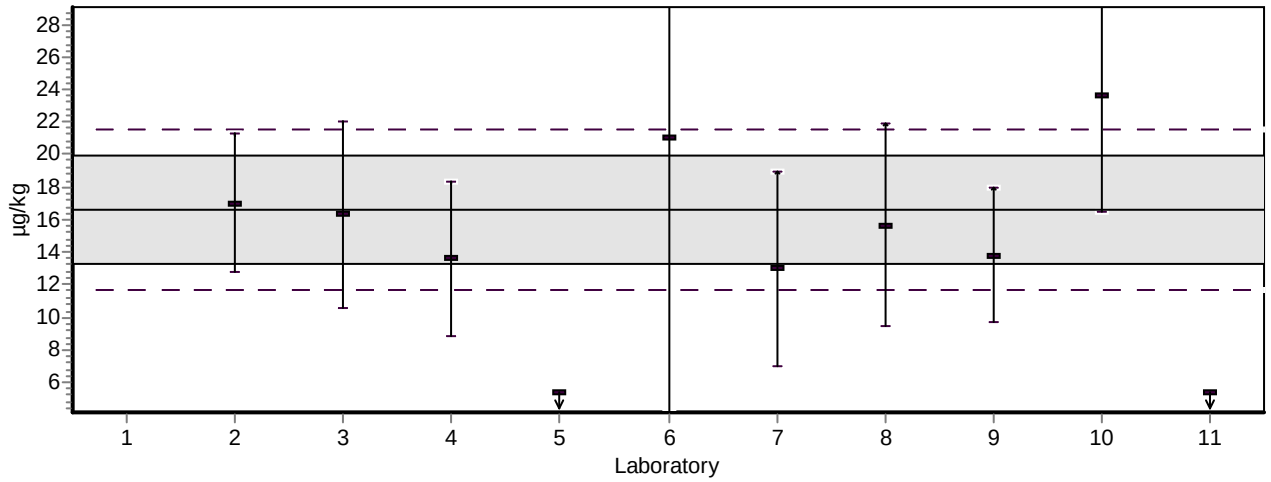
Analyytti *(Analyte)* **PCB-180** Näyte *(Sample)* M1



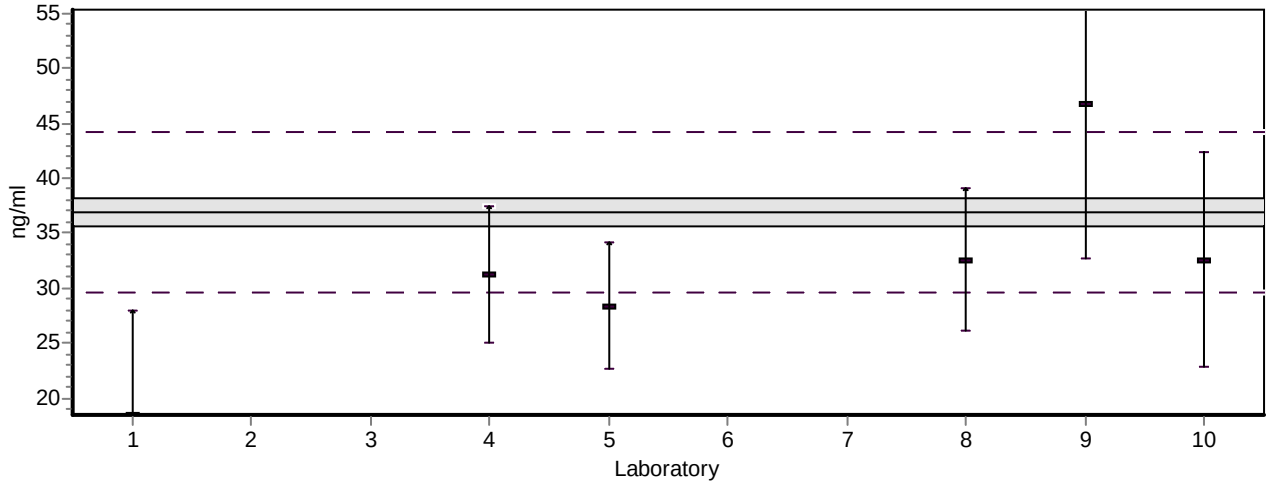
Analyytti *(Analyte)* **PCB-28** Näyte *(Sample)* A1



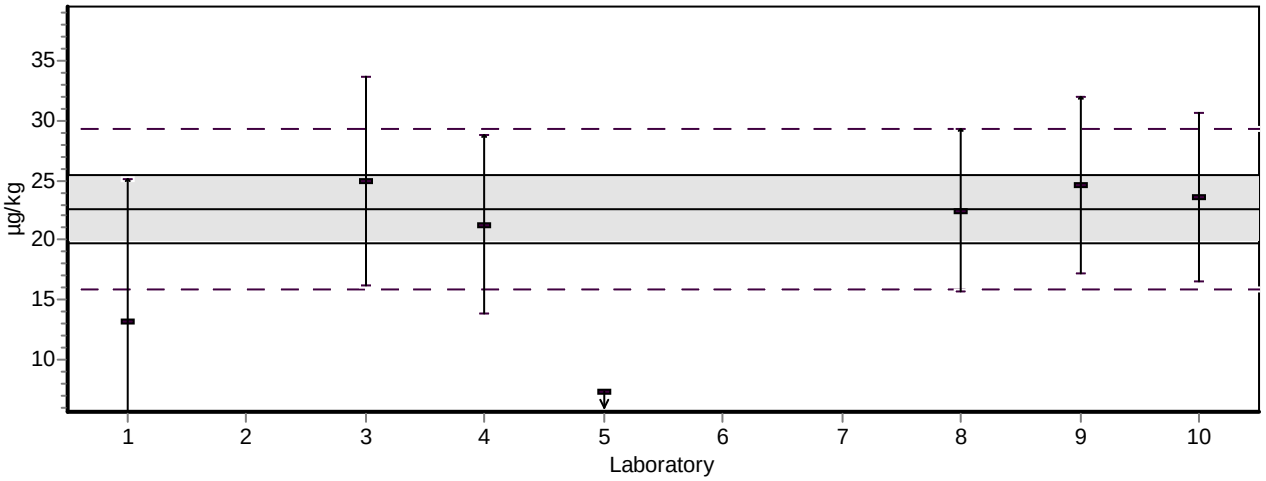
Analyytti *(Analyte)* **PCB-28** Näyte *(Sample)* M1



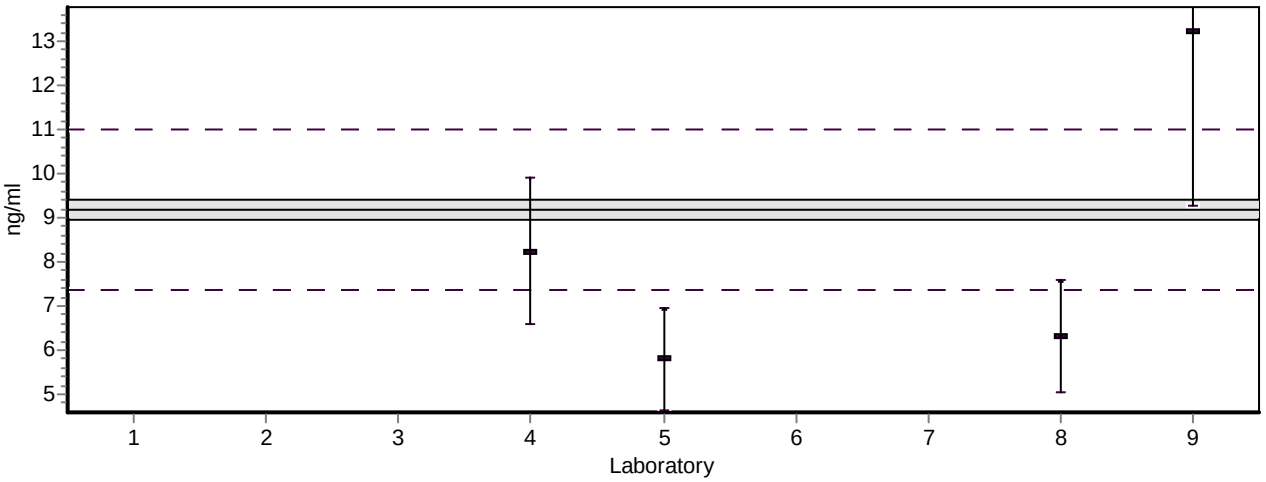
Analyytti *(Analyte)* **PCB-28+31** Näyte *(Sample)* A1



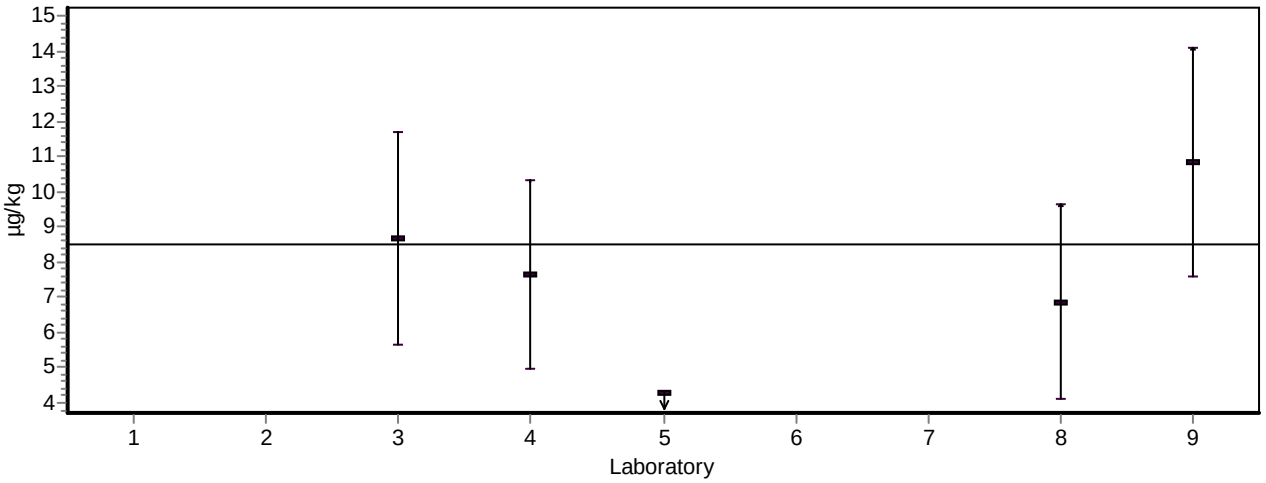
Analyytti *(Analyte)* **PCB-28+31** Näyte *(Sample)* M1



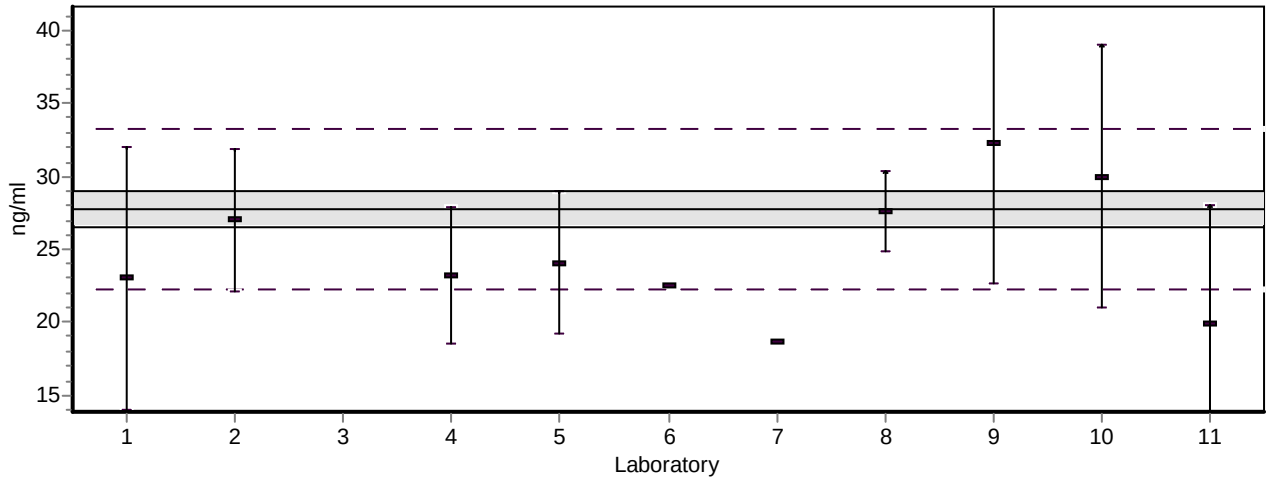
Analyytti *(Analyte)* **PCB-31** Näyte *(Sample)* A1



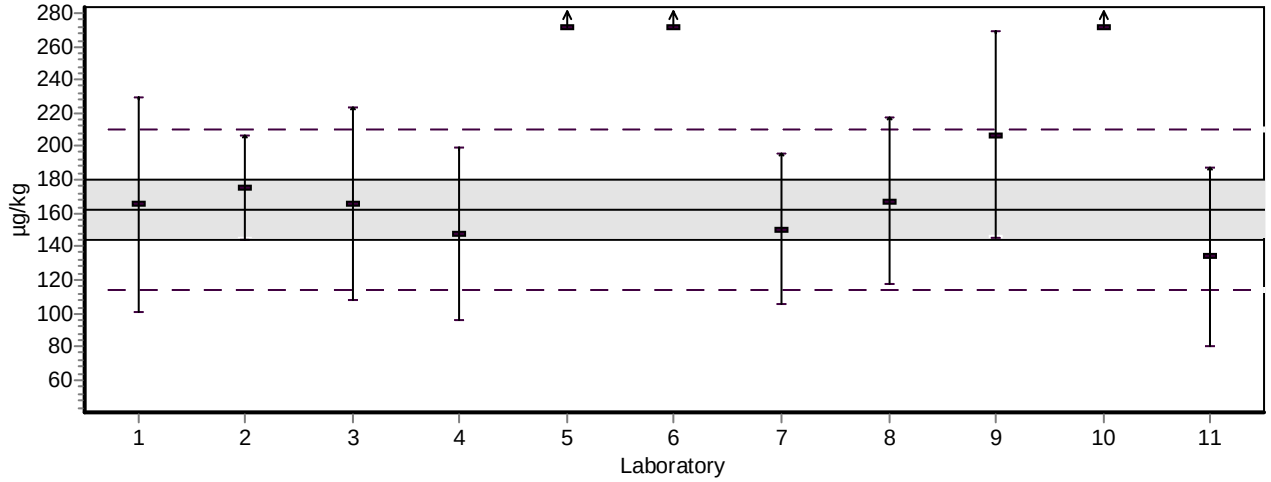
Analyytti *(Analyte)* **PCB-31** Näyte *(Sample)* M1



Analyytti *(Analyte)* **PCB-52** Näyte *(Sample)* A1



Analyytti *(Analyte)* **PCB-52** Näyte *(Sample)* M1



LIITE 9. SUMMARY OF THE z SCORES

APPENDIX 9.

Analyte	Sample\Lab	1	2	3	4	5	6	7	8	9	10	11	%
PCB-101	A1	S	S	.	S	S	q	q	S	S	S	q	70
	M1	S	S	S	S	S	S	S	S	Q	S	S	91
PCB-105	A1	.	.	.	q	S	.	.	S	Q	S	.	60
	M1	.	.	S	S	S	.	.	S	S	U	.	83
PCB-110	A1	.	.	.	.	.	.	.	.	.	.	.	
	M1	.	.	.	.	.	.	.	.	.	.	.	
PCB-118	A1	S	S	.	q	S	S	S	S	Q	S	u	70
	M1	S	S	S	S	S	S	S	S	S	S	S	100
PCB-138	A1	S	S	.	q	S	S	S	S	S	S	q	80
	M1	S	S	S	S	S	S	S	S	U	S	S	91
PCB-153	A1	q	S	.	q	S	S	q	S	S	S	q	60
	M1	U	S	S	S	S	S	S	S	S	S	S	91
PCB-156	A1	.	.	.	q	S	.	.	S	Q	S	.	60
	M1	.	.	S	S	U	.	.	S	S	S	.	83
PCB-170	A1	.	.	.	q	S	.	.	S	Q	.	.	50
	M1	.	.	.	.	.	.	.	.	.	.	.	
PCB-180	A1	q	S	.	q	S	S	S	S	S	S	q	70
	M1	U	S	S	S	S	S	S	S	S	S	S	91
PCB-28	A1	.	S	.	S	S	S	S	S	Q	Q	u	67
	M1	.	S	S	S	u	S	S	S	S	Q	.	78
PCB-28+31	A1	u	.	.	S	q	.	.	S	Q	S	.	50
	M1	q	.	S	S	u	.	.	S	S	S	.	71
PCB-31	A1	.	.	.	S	u	.	.	u	U	.	.	25
	M1	.	.	.	.	.	.	.	.	.	.	.	
PCB-52	A1	S	S	.	S	S	S	u	S	S	S	q	80
	M1	S	S	S	S	U	U	S	S	S	U	S	73
%		57	100	100	68	73	86	79	95	59	80	46	
Accredited		yes	yes	yes	yes	yes	yes	yes	yes		yes		

S - satisfactory ($-2 \leq z \leq 2$), Q - questionable ($2 < z < 3$), q - questionable ($-3 < z < -2$),
U - unsatisfactory ($z \geq 3$), u - unsatisfactory ($z \leq -3$)

%* - percentage of satisfactory results

Totally satisfactory, % In all: 76 In accredited: 81 In non-accredited: 54

ANALYTICAL METHODS

Soil – M1

Lab	Extraction	Internal standard	Sampling / Injection	Equipment	Reference
1	Shaking	PCB-155	Splitless	GC-MS	ISO 10382
2	Soxhlet	PCB-33, NCB	PTV	GC-ECD	?
3	ASE	PCB-53, TeCB	Splitless	GC-ECD	In house method
4	Ultrasonic	PCB-53, TeCB	Splitless	GC-ECD	In house method
5	Shaking	Deuterated biphenyl	Splitless	GC-MS	EPA 8082 modified
6	Shaking	PCB-53	Splitless	GC-MS	Nordtest TR 329
7	Ultrasonic	PCB-30, PCB-204	Splitless	GC-ECD	ISO 10382
8	Soxhlet	PCB-30, PCB-52[^{13}C], PCB-101[^{13}C], [PCB-118[^{13}C], [PCB-138[^{13}C], [PCB-153[^{13}C]	Spitless	GC-HRMS	In house method
9	Shaking	PCB-209	Splitless	GC-MS	ISO 16703, ISO 12766-2
10	Shaking	PCB-185	Pulsed splitless	GC-MS	ISO 10382 modified
11	Shaking	PCB-209	Splitless	GC-ECD	ISO 10382

ASE, Accelerated solvent extractor
PTV, Programmable Temperature Vaporization Injector

RESULTS GROUPED ACCORDING TO THE METHODS

No statistical comparison between the different techniques could be done because of the low number of the results. In this appendix the results of the participants are grouped according to the instrument techniques (A1) and extraction techniques (M1) as follows:

Sample A1

Meth 1. GC-MS

Meth 2. GC-ECD

Meth 3. GC-HRMS

Sample M1

Meth 4. Shaking

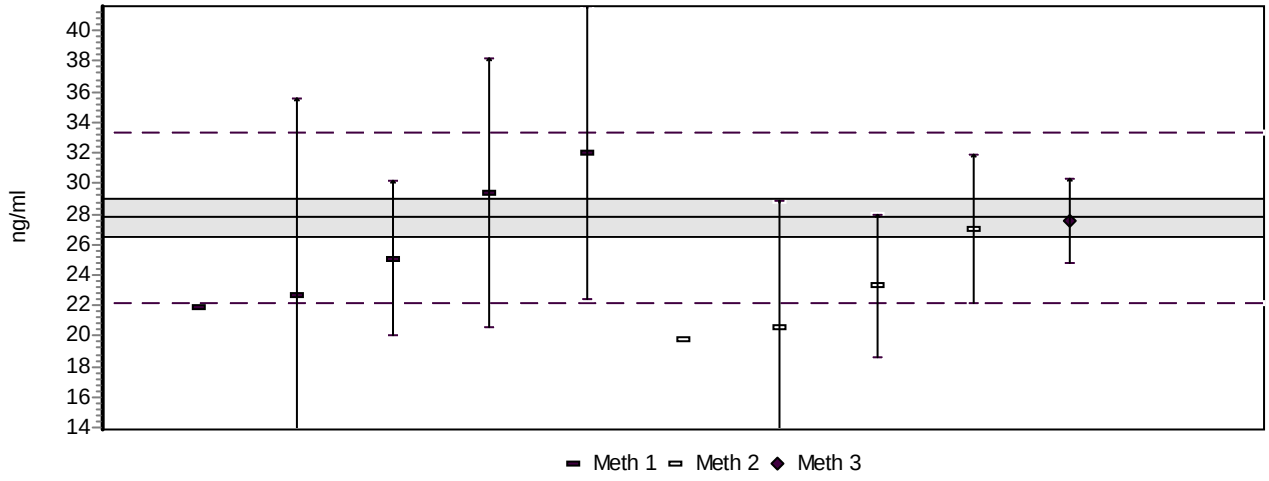
Meth 5. Soxhlet

Meth 6. ASE

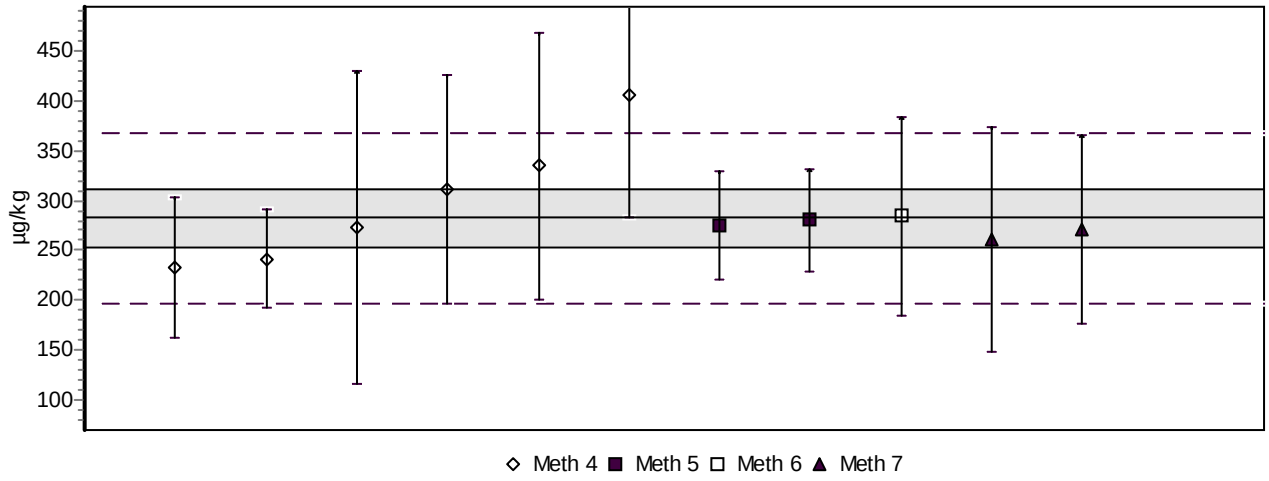
Meth 7. Ultrasonic

LIITE 10.2.
APPENDIX 10.2.

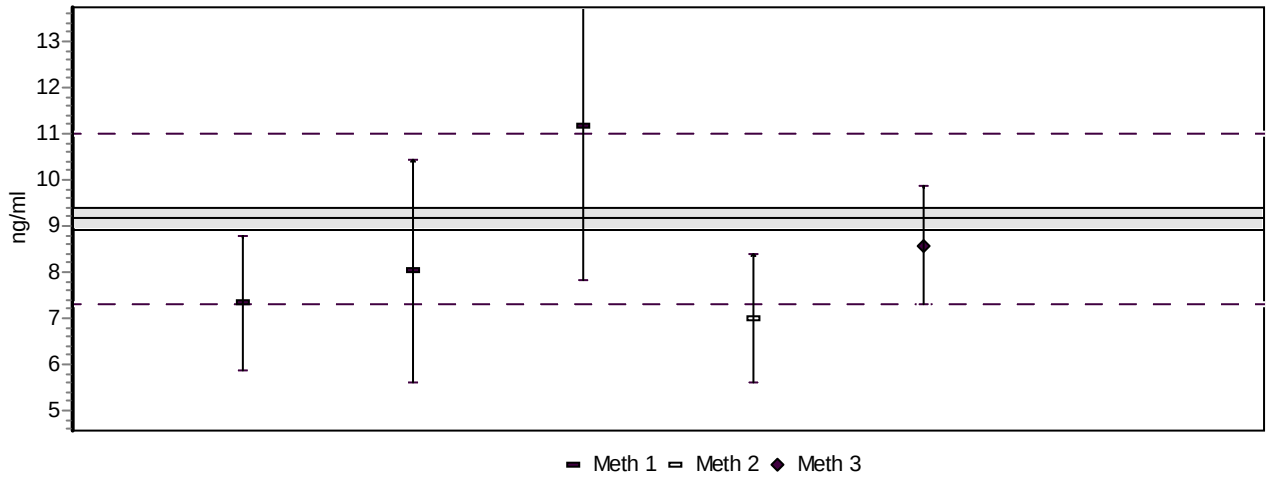
Analyytti *(Analyte)* **PCB-101** Näyte *(Sample)* A1



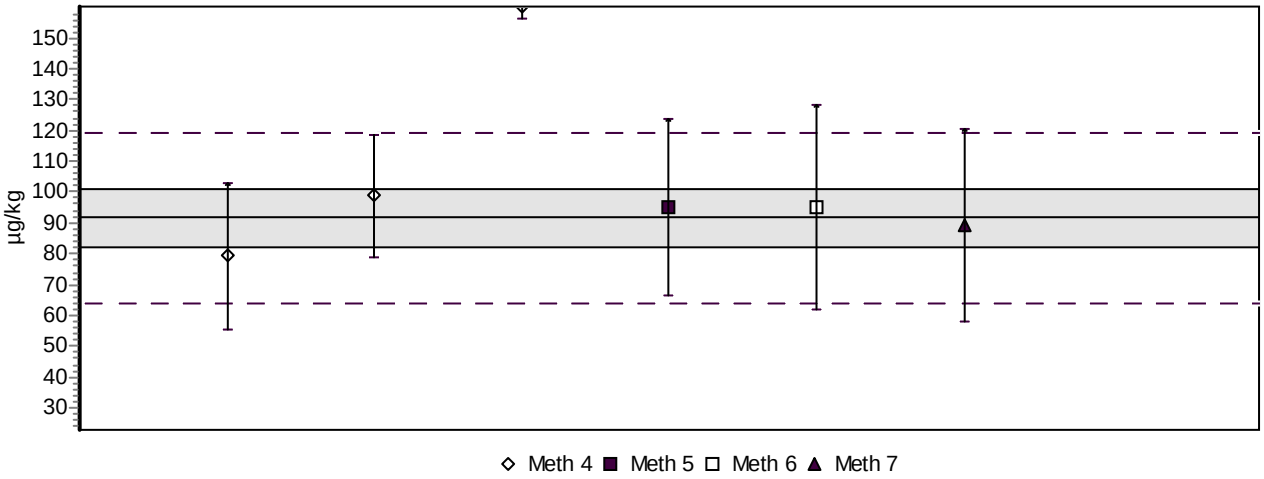
Analyytti *(Analyte)* **PCB-101** Näyte *(Sample)* M1



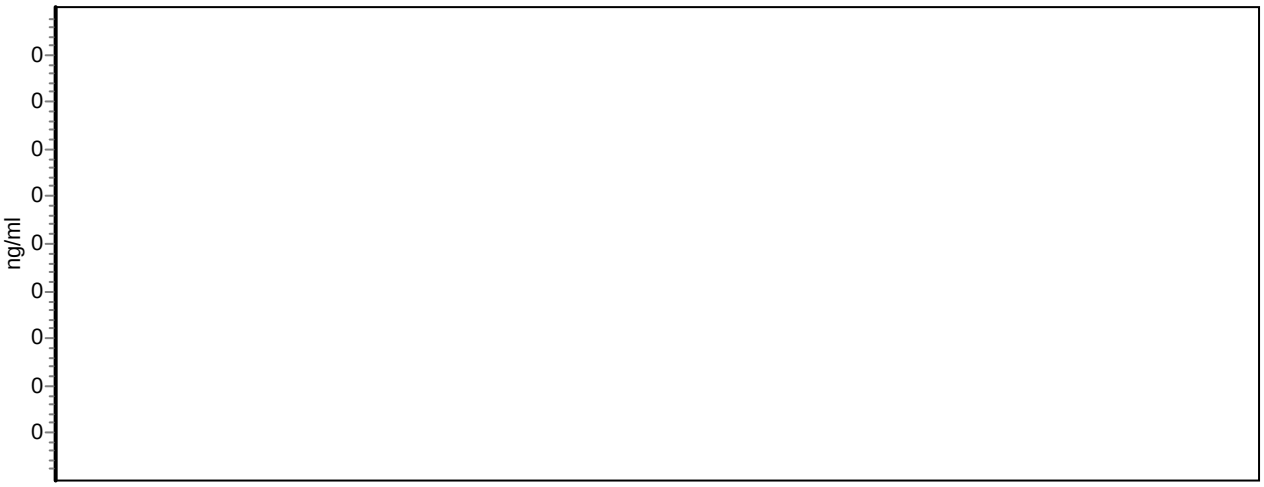
Analyytti *(Analyte)* **PCB-105** Näyte *(Sample)* A1



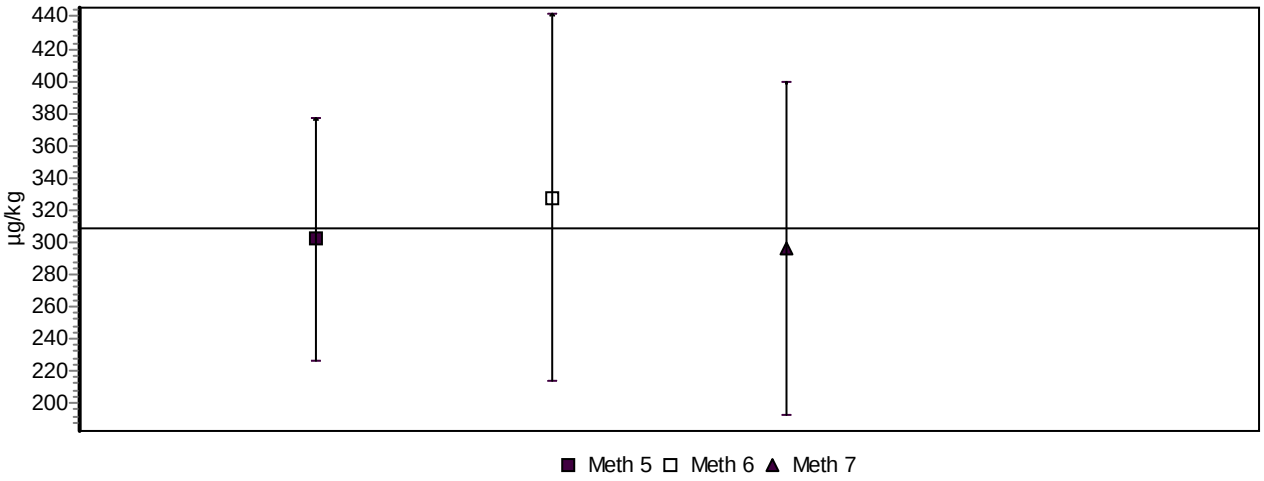
Analyytti *(Analyte)* **PCB-105** Näyte *(Sample)* M1



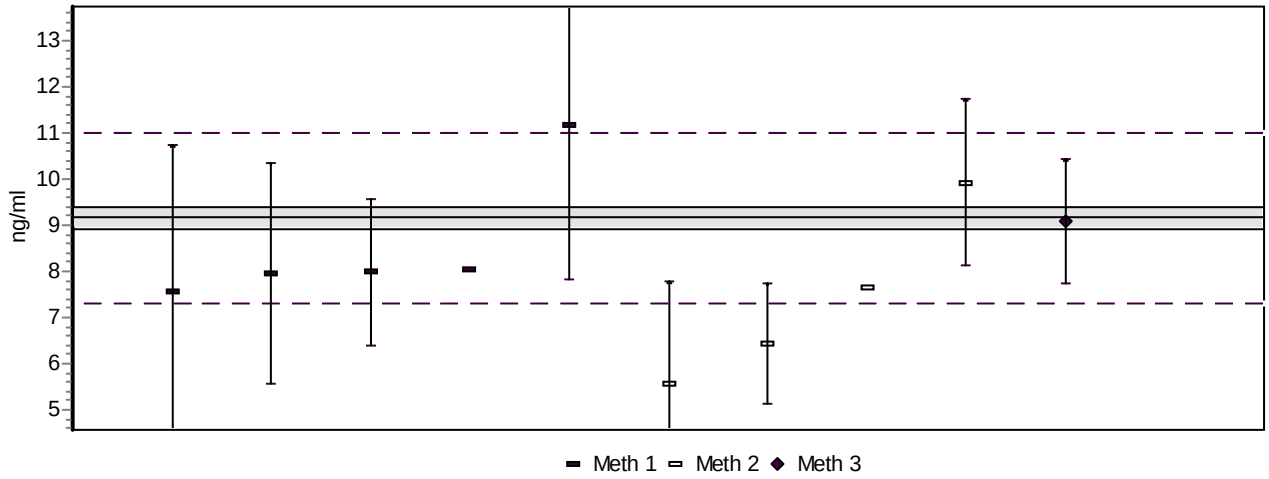
Analyytti *(Analyte)* **PCB-110** Näyte *(Sample)* A1



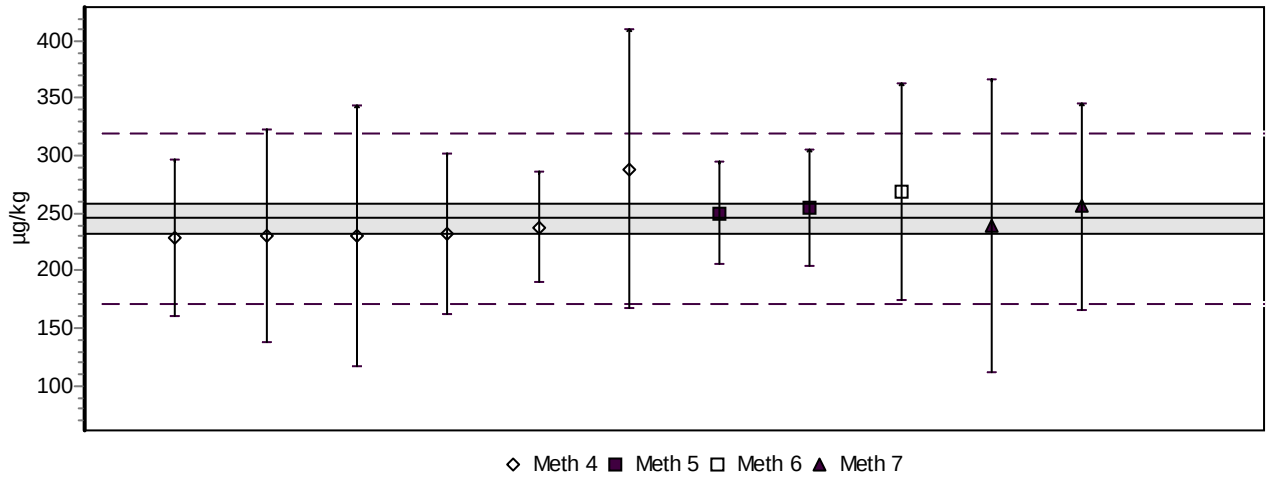
Analyytti *(Analyte)* **PCB-110** Näyte *(Sample)* M1



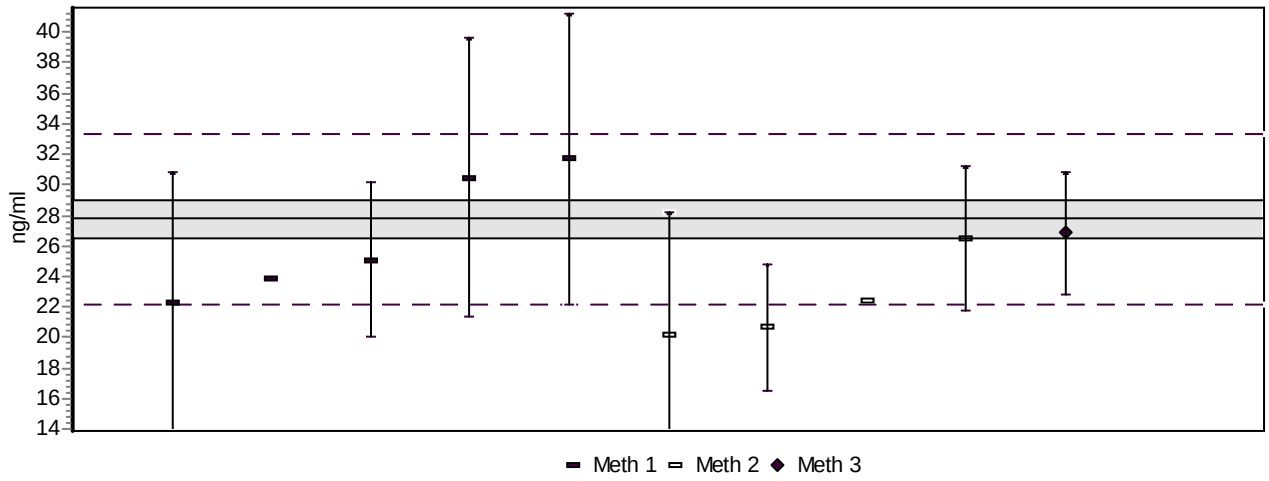
Analyytti *(Analyte)* **PCB-118** Näyte *(Sample)* A1



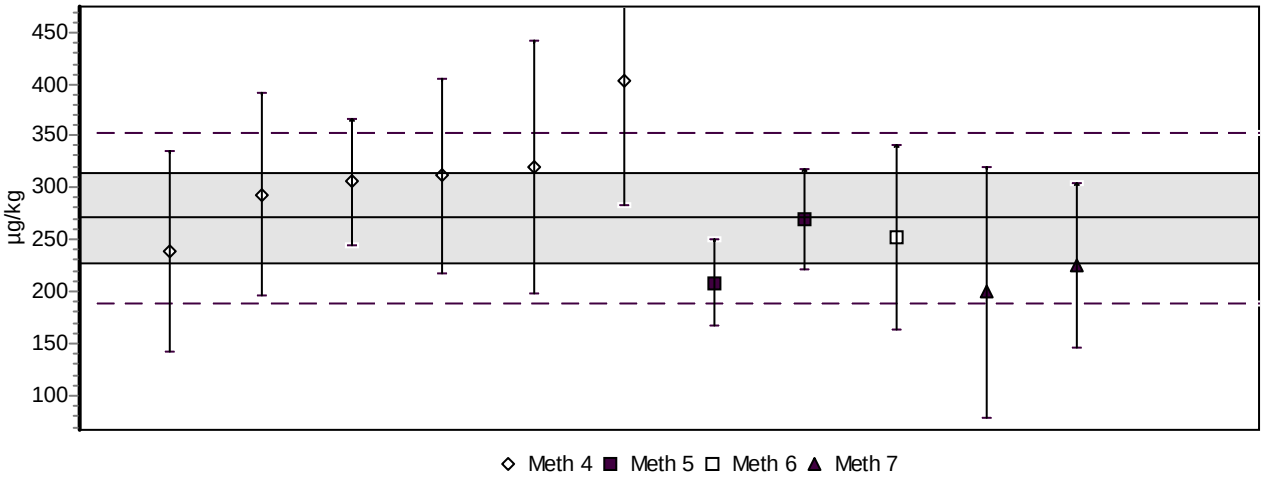
Analyytti *(Analyte)* **PCB-118** Näyte *(Sample)* M1



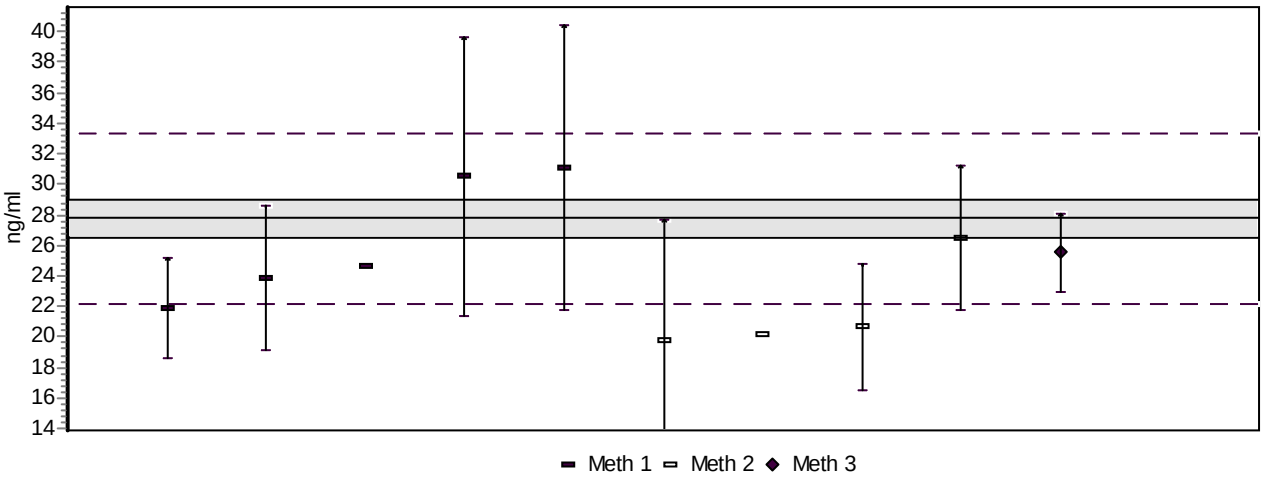
Analyytti *(Analyte)* **PCB-138** Näyte *(Sample)* A1



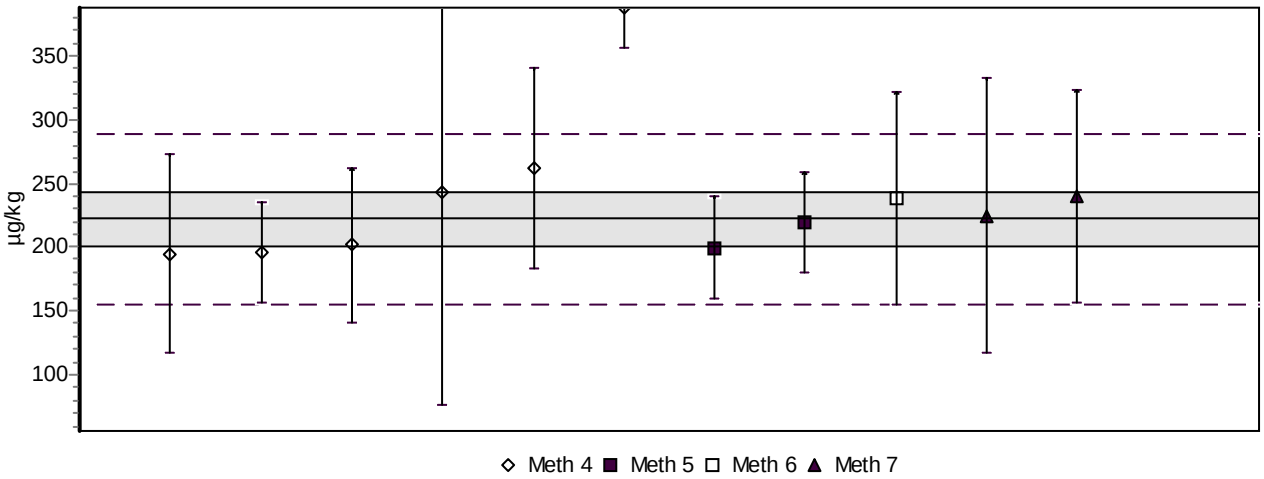
Analyytti *(Analyte)* **PCB-138** Näyte *(Sample)* M1



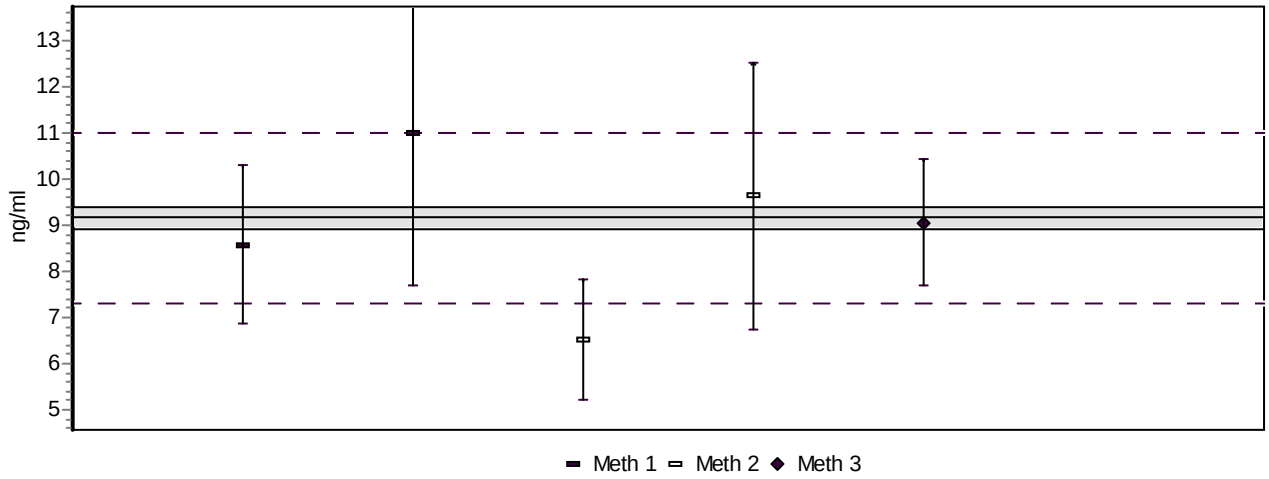
Analyytti *(Analyte)* **PCB-153** Näyte *(Sample)* A1



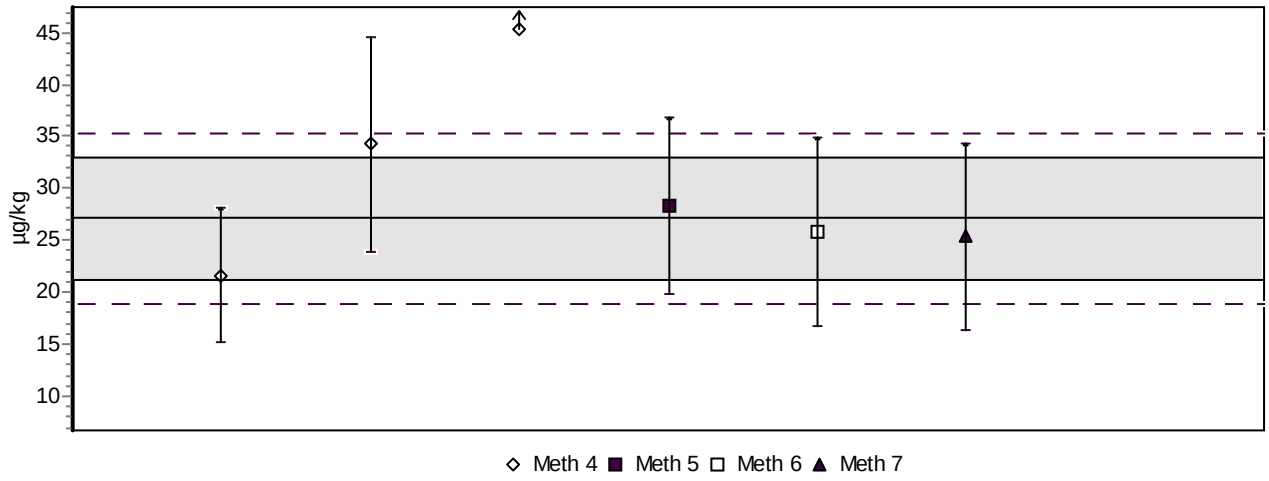
Analyytti *(Analyte)* **PCB-153** Näyte *(Sample)* M1



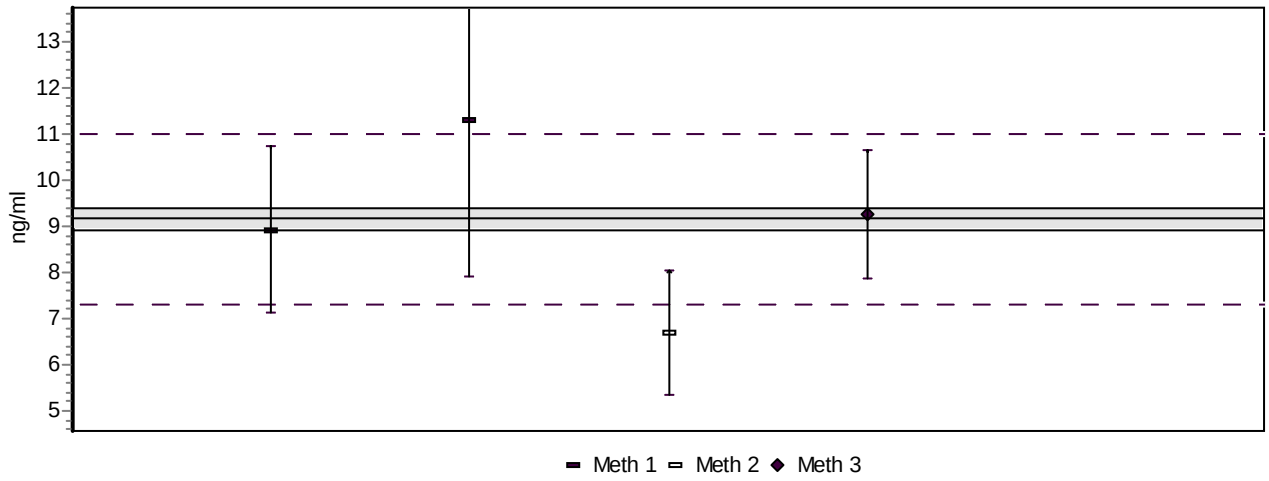
Analyytti *(Analyte)* **PCB-156** Näyte *(Sample)* A1



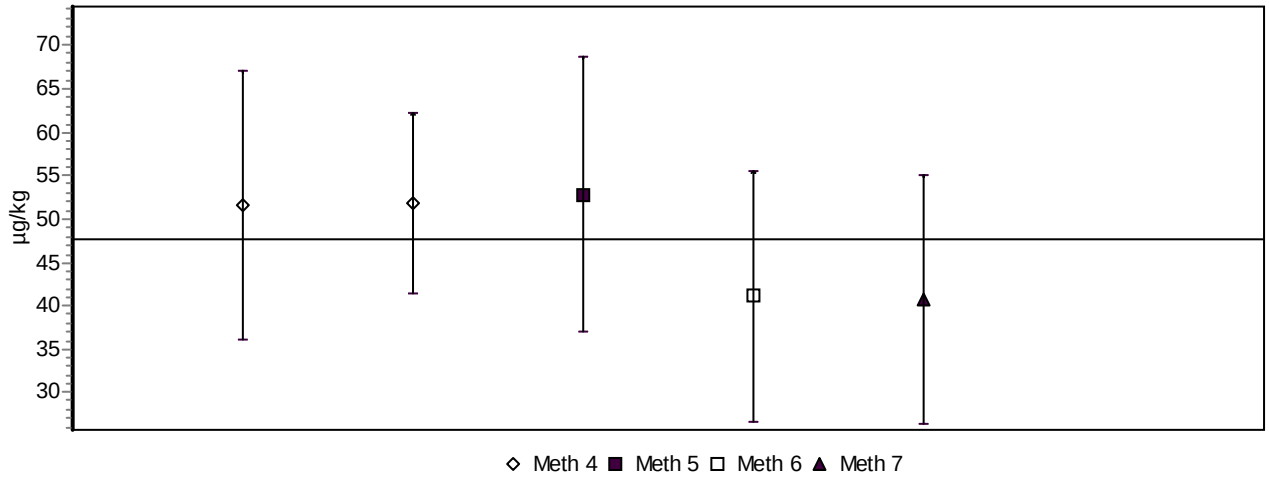
Analyytti *(Analyte)* **PCB-156** Näyte *(Sample)* M1



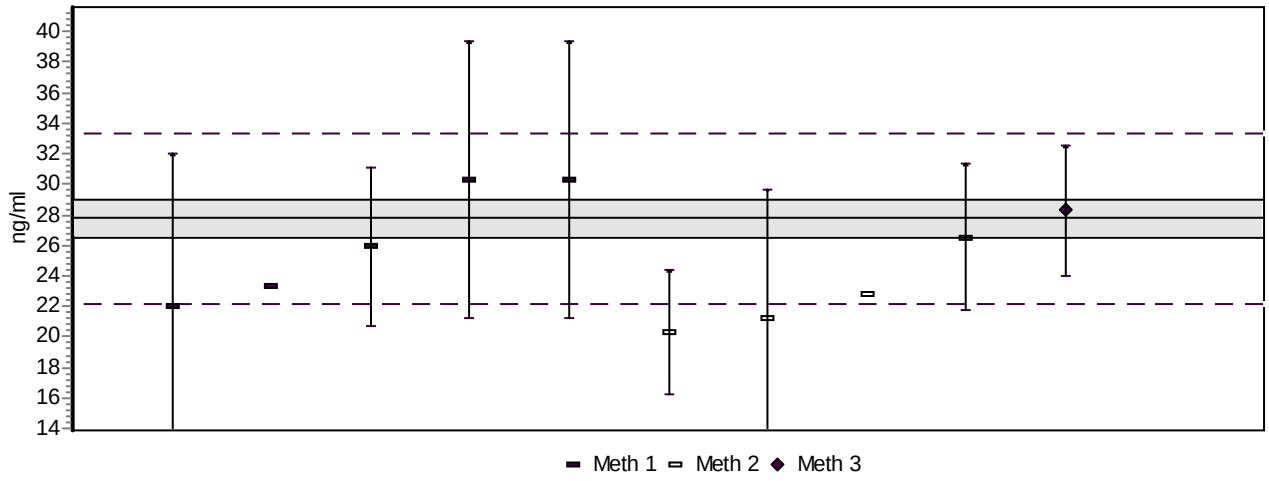
Analyytti *(Analyte)* **PCB-170** Näyte *(Sample)* A1



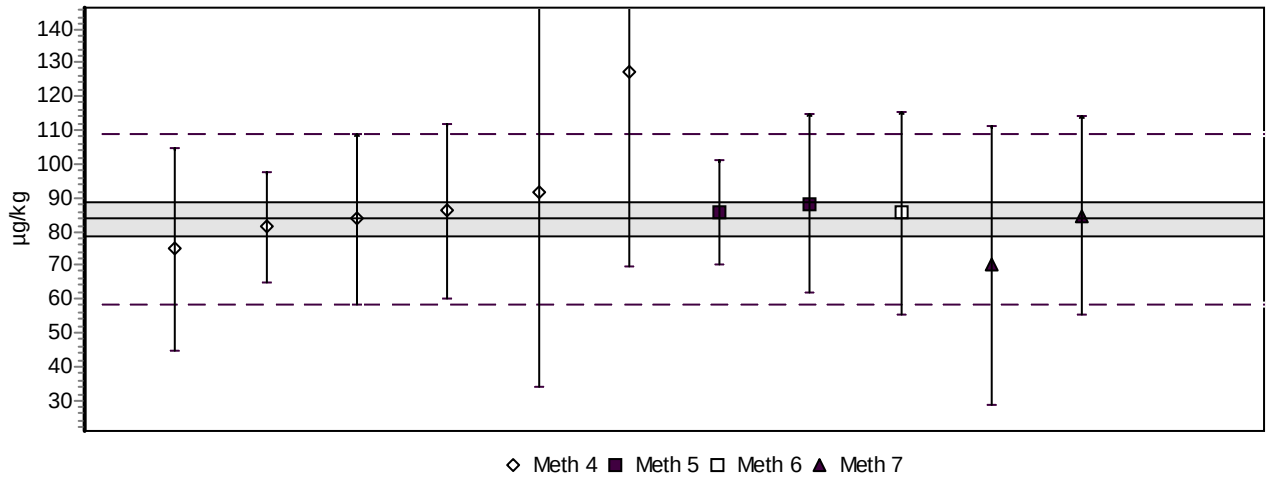
Analyytti *(Analyte)* **PCB-170** Näyte *(Sample)* M1



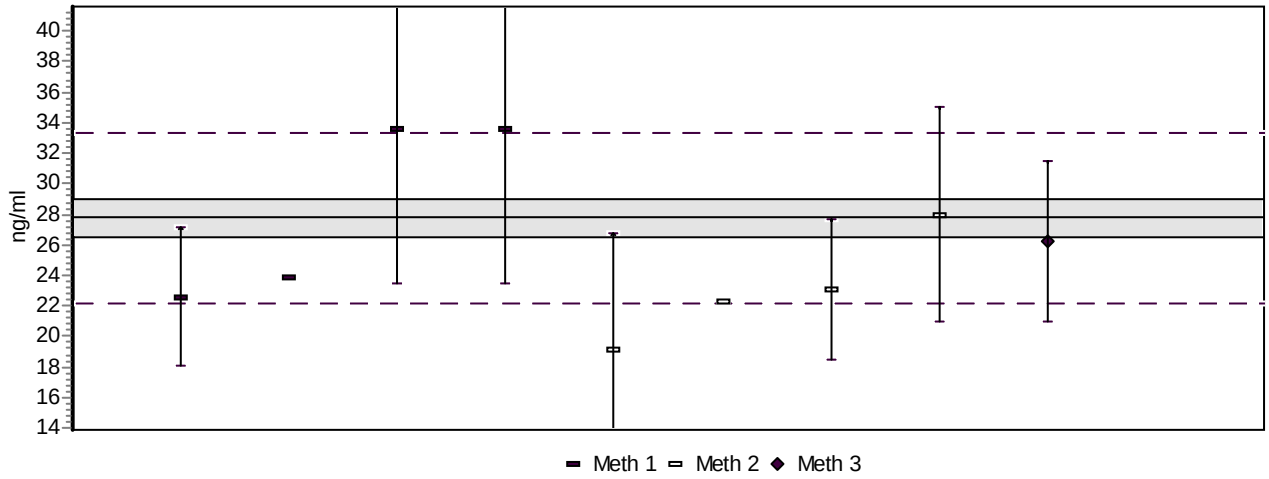
Analyytti *(Analyte)* **PCB-180** Näyte *(Sample)* A1



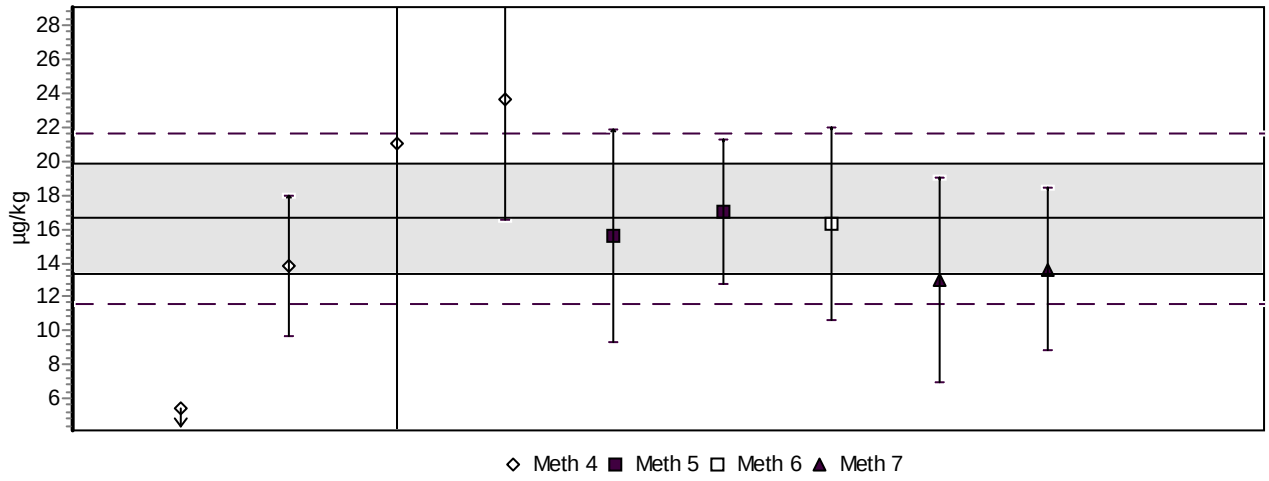
Analyytti *(Analyte)* **PCB-180** Näyte *(Sample)* M1



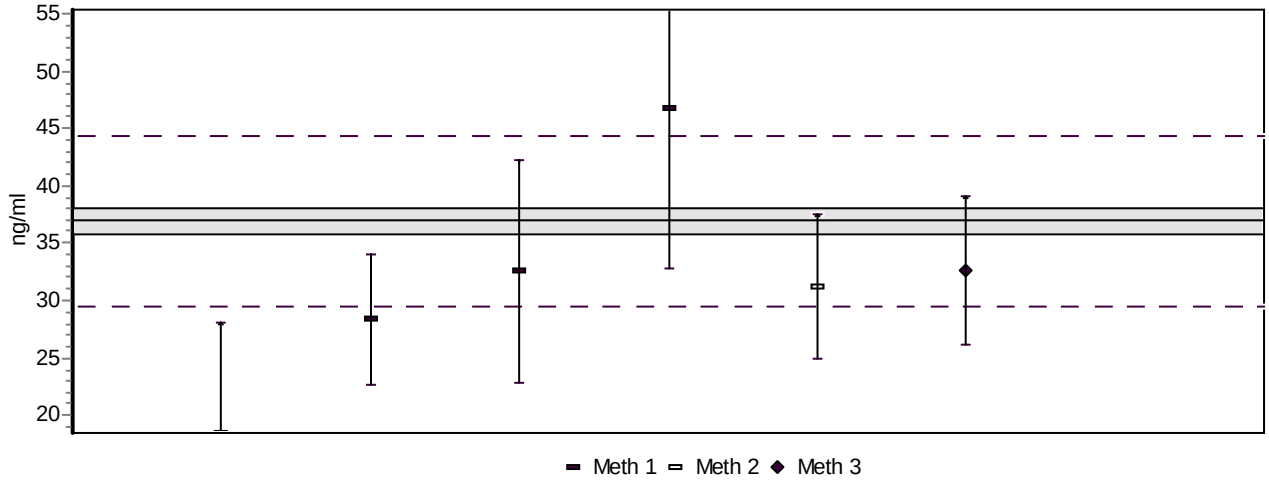
Analyytti *(Analyte)* **PCB-28** Näyte *(Sample)* A1



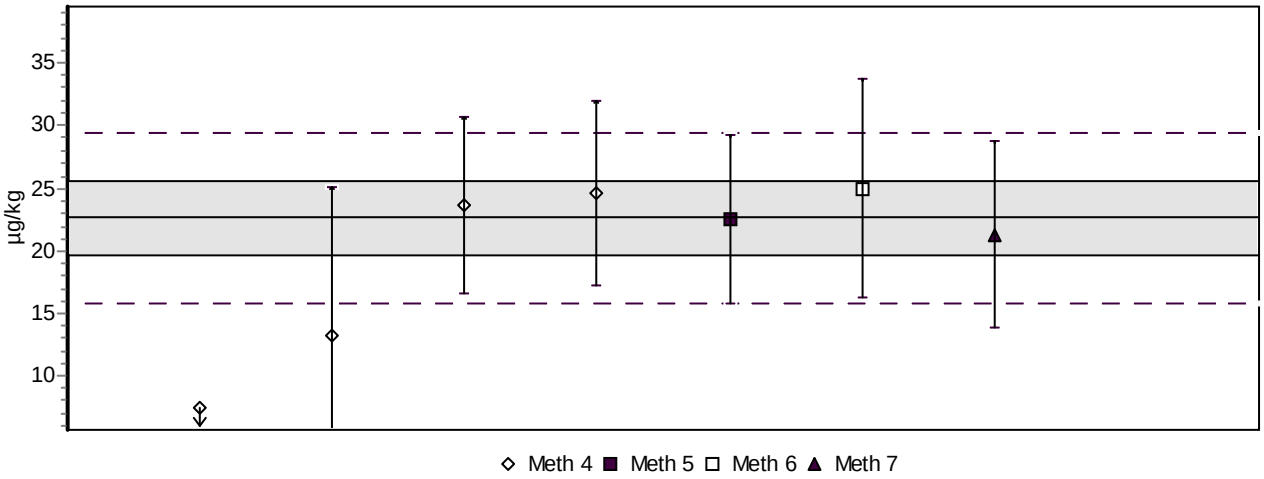
Analyytti *(Analyte)* **PCB-28** Näyte *(Sample)* M1



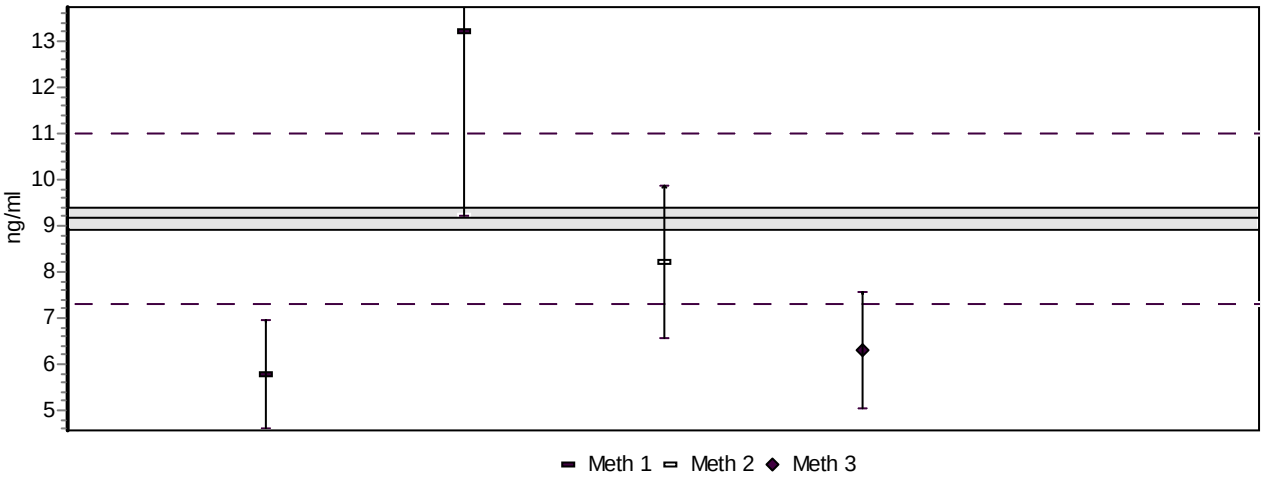
Analyytti *(Analyte)* **PCB-28+31** Näyte *(Sample)* A1



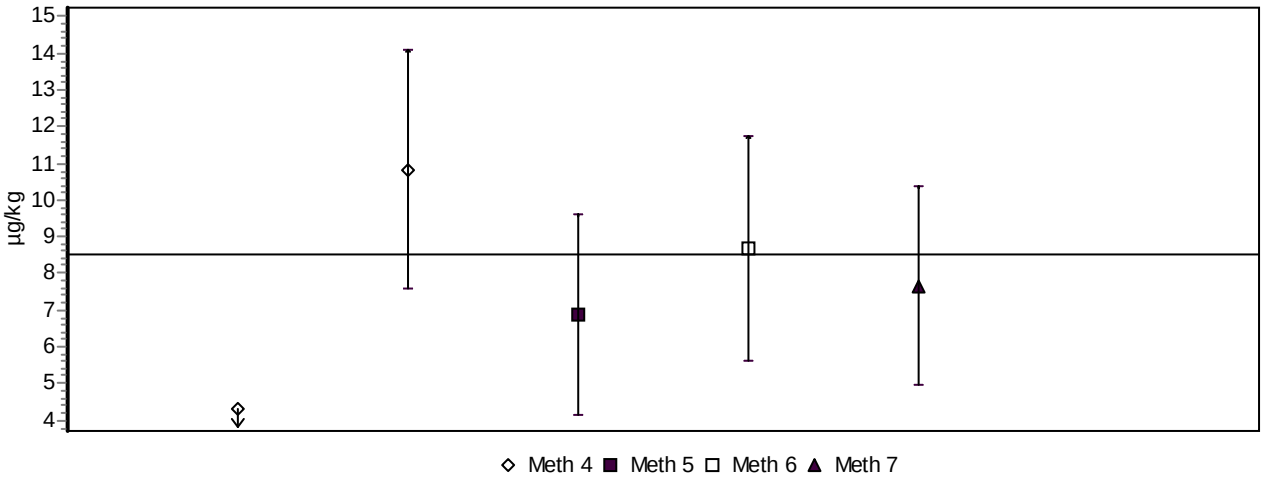
Analyytti *(Analyte)* **PCB-28+31** Näyte *(Sample)* M1



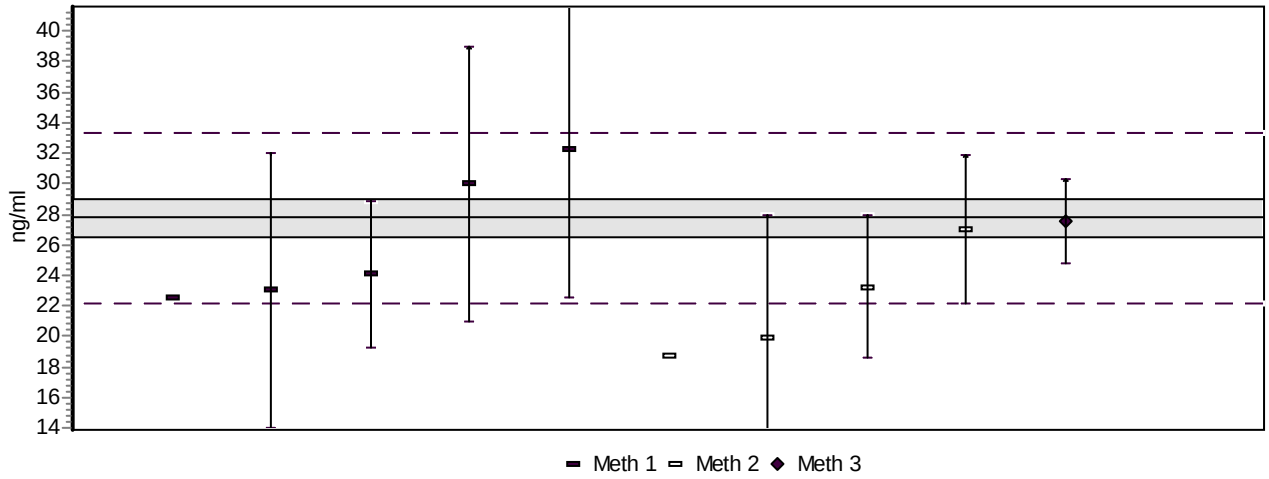
Analyytti *(Analyte)* **PCB-31** Näyte *(Sample)* A1



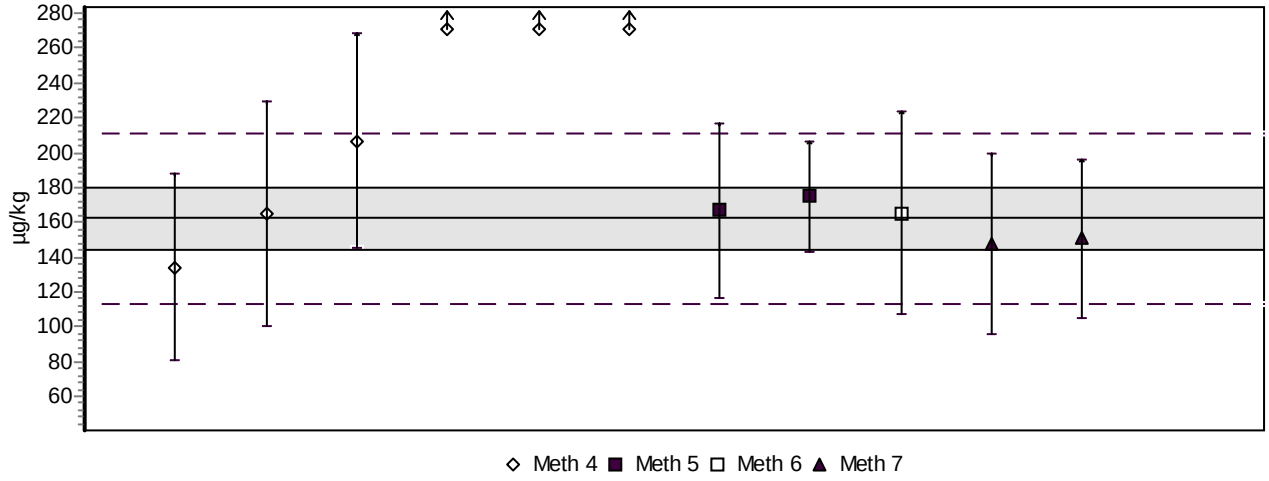
Analyytti *(Analyte)* **PCB-31** Näyte *(Sample)* M1



Analyytti *(Analyte)* **PCB-52** Näyte *(Sample)* A1



Analyytti *(Analyte)* **PCB-52** Näyte *(Sample)* M1



EXAMPLES OF MEASUREMENT UNCERTAINTIES REPORTED BY THE LABORATORIES

For evaluation of the measurement uncertainty the participants have used the procedures as follows:

1. Using the variation of the results in X chart (for the artificial samples)

2. Using the variation of the results in X chart and the variation of the replicates (r%- or R- chart for real samples)

3. Using the data obtained in method validation and IQC, see e.g. NORDTEST TR 537[1)]

4. Using the data obtained in the analysis of CRM (besides IQC data). see e.g. NORDTEST TR 537[1)]

5. Using the IQC data and the results obtained in proficiency tests. see e.g. NORDTEST TR 537[1)]

6. Using the "modelling approach" (GUM Guide or EURACHEM Guide Quantifying Uncertainty in Analytical Measurements[2)]

7. Other procedure

8. No uncertainty estimation

IQC = internal quality control

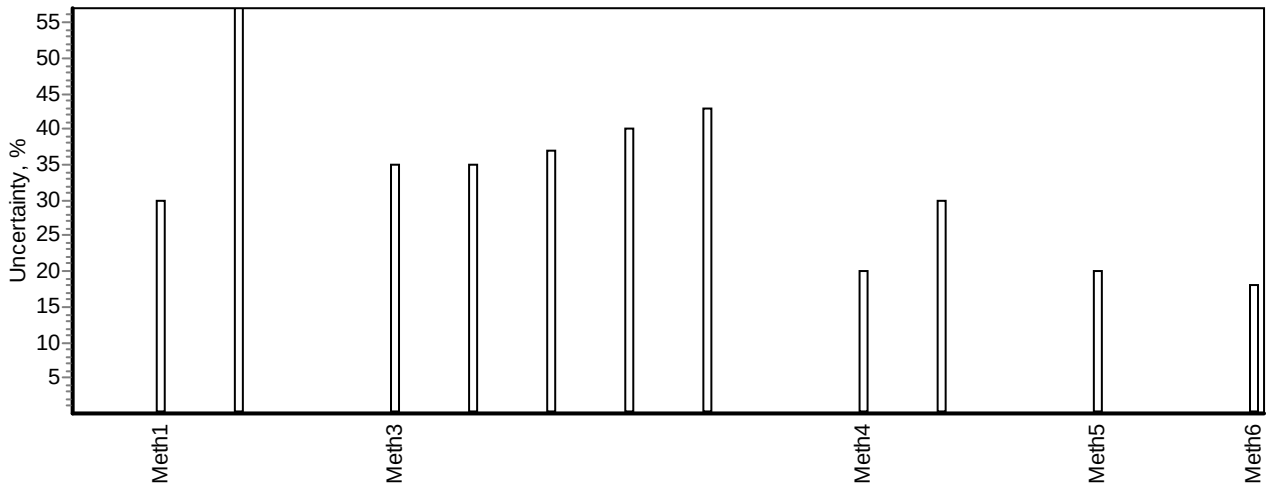
In the figures the procedures have been presented using the same code number.

[1)] http://www.nordtest.info

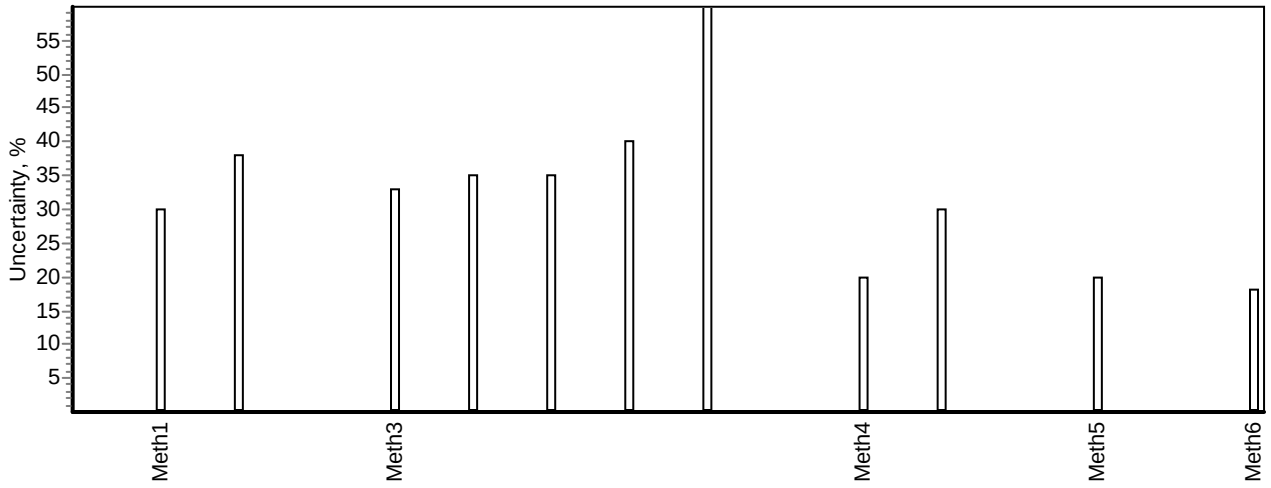
[2)] http://www.eurachem.org

LIITE 11.
APPENDIX 11.

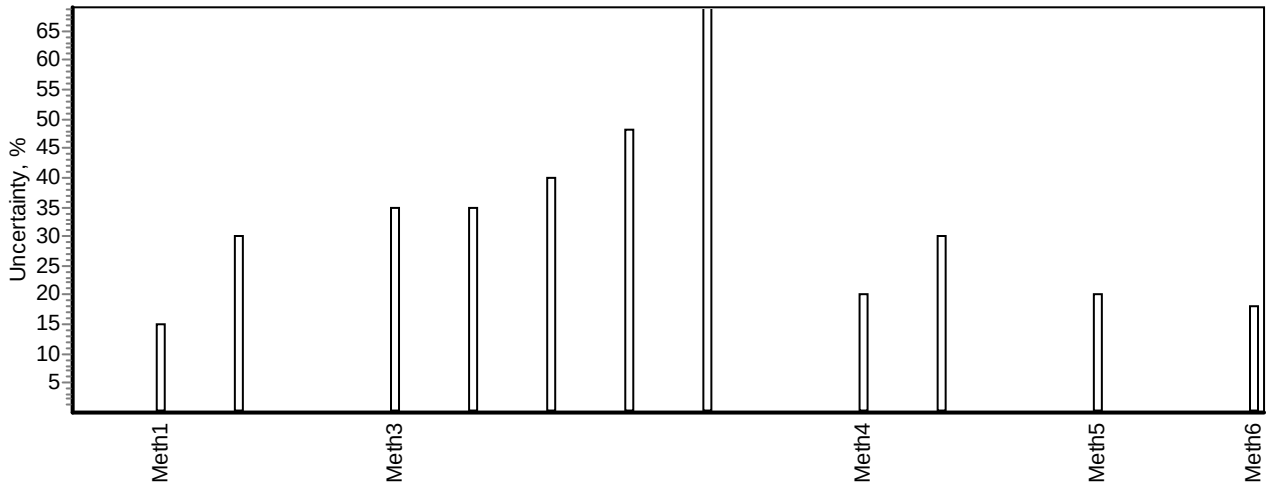
Analyytti *(Analyte)* **PCB-101** Näyte *(Sample)* M1



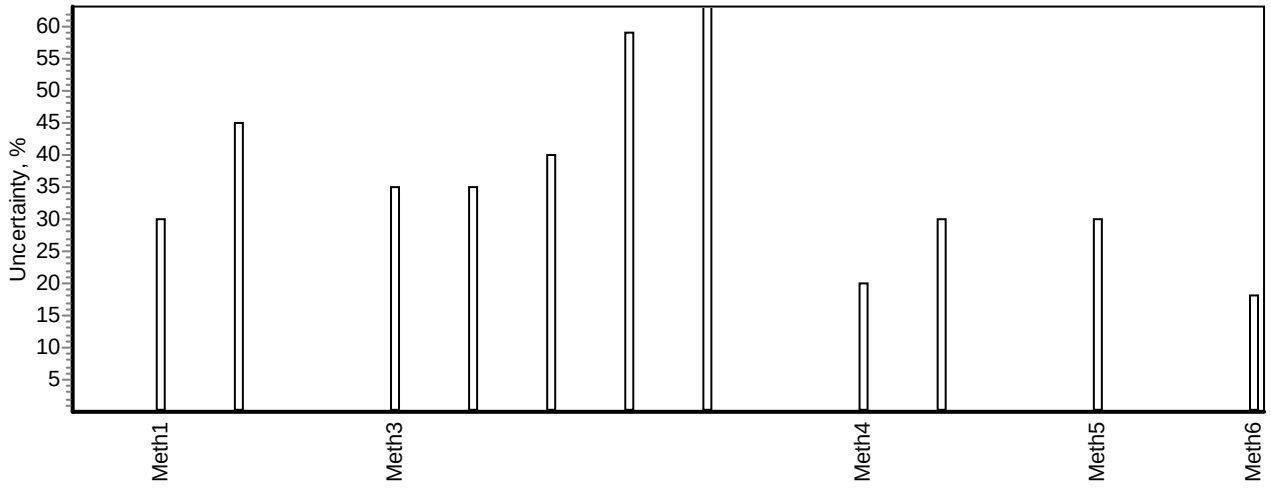
Analyytti *(Analyte)* **PCB-138** Näyte *(Sample)* M1



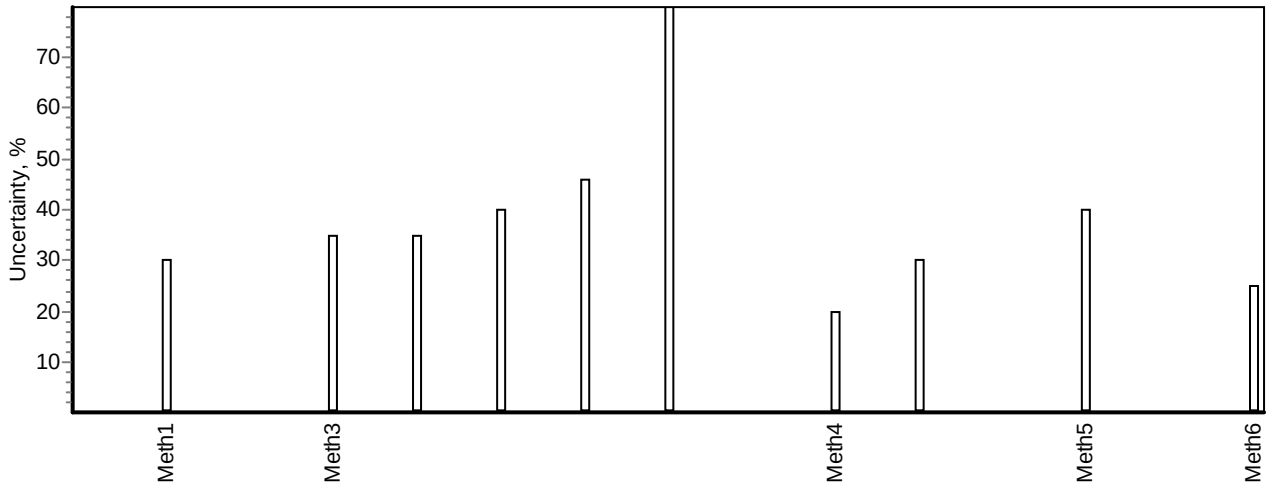
Analyytti *(Analyte)* **PCB-153** Näyte *(Sample)* M1



Analyytti *(Analyte)* **PCB-180** Näyte *(Sample)* M1



Analyytti *(Analyte)* **PCB-28** Näyte *(Sample)* M1



Documentation page

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Author(s)	Jari Nuutinen, Kaija Korhonen-Ylönen, Mirja Leivuori and Markku Ilmakunnas	
Title of publication	Proficiency Test SYKE 11/2011 PCB compounds in soil	
Parts of publication/ other project publications	The publication is available only on the internet www.ymparisto.fi/julkaisut.	
Abstract	Proftest SYKE carried out the proficiency test for analysis of PCB compounds in soil in end of 2011. In total 10 laboratories participated in the proficiency test. The sample types were: a reference PCB solution in isooctane and a contaminated soil sample. Basically, the calculated concentrations or the robust mean of the results reported by the participant were used as the assigned values for measurands. The evaluation of the performance of the participants was carried out using z score. In some cases the evaluation of the performance was not possible e.g. due to the low number of the participants. In total, 76 % of the total data in this proficiency test were satisfactory when the deviations of 20−30 % from the assigned values were accepted.	
Keywords	PCB compounds, soil, environmental laboratories, proficiency test, interlaboratory comparisons	
Publication series and number	Reports of Finnish Environment Institute 12/2012	
Theme of publication		
Project name and number, if any		
Financier/ commissioner		
Project organization		
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Julkaisun nimi	Proficiency Test SYKE 11/2011 PCB compounds in soil	
Julkaisun osat/ muut saman projektin tuottamat julkaisut	Julkaisu on saatavana vain internetistä. www.ymparisto.fi/julkaisut	
Tiivistelmä	Suomen ympäristökeskuksen laboratorio järjesti pätevyyskokeen ympäristönäytteitä analysoiville laboratorioille loppuvuonna 2011. Pätevyyskokeen näytteinä oli synteettinen PCB-liuos iso-oktaanissa ja kontaminoitunut maanäyte. Pätevyyskokeeseen osallistui yhteensä 10 laboratoriota, joista yksi raportoi kahdella eri menetelmällä analysoidut tulokset. Laboratorioiden pätevyyden arviointi tehtiin z-arvon avulla. Mittaussuureen vertailuarvona käytettiin synteettisessä näytteessä laskennallista pitoisuutta ja maanäytteessä osallistujien ilmoittamien tulosten robustia keskiarvoa. Koko tulosaineistossa hyväksyttäviä tuloksia oli 76 %, kun vertailuarvosta sallittiin 20–30 %:n poikkeama.	
Asiasanat	maa-analyysi, PCB-yhdisteet, vesi- ja ympäristölaboratoriot, pätevyyskoe, laboratorioiden välinen vertailumittaus	
Julkaisusarjan nimi ja numero	Reports of Finnish Environment Institute 12/2012	
Julkaisun teema		
Projektihankkeen nimi ja projektinumero		
Rahoittaja/ toimeksiantaja		
Projektiryhmään kuuluvat organisaatiot		
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	Sivuja 48	Kieli Englanti
	Luottamuksellisuus Julkinen	Hinta
Julkaisun myynti/ jakaja	Suomen ympäristökeskus, asiakaspalvelu Sähköpostiosoite: neuvonta.syke@ymparisto.fi puh. 020 610 183 faksi 09 5490 2190	
Julkaisun kustantaja	Suomen ympäristökeskus, PL 140, 00251 Helsinki	
Painopaikka ja -aika	Helsinki 2012	
Muut tiedot		

Presentationsblad

Utgivare	Finlands Miljöcentral (SYKE)	Datum April 2012
Författare	Jari Nuutinen, Kaija Korhonen-Ylönen, Mirja Leivuori och Markku Ilmakunnas	
Publikationens titel	Proficiency Test SYKE 11/2011 PCB compounds in soil	
Publikationens delar/ andra publikationer inom samma projekt	Publikationen finns tillgänglig på internet www.ymparisto.fi/julkaisut	
Sammandrag	Under slutåret 2011 genomförde Finlands Miljöcentral en provningsjämförelse, som omfattade bestämningen av PCB-föreningar in jord. Provet bestod av syntetiska och därtill ett jordprov. Tillsammans 10 laboratorier deltog i jämförelsen. Som referensvärde av analytens koncentration användes mest det teoretiska värdet eller robust medelvärdet av deltagarnas resultat. Resultaten värderades med hjälp av z-värden. I jämförelsen var 76 % av alla resultaten tillfredsställande, när total deviation på 20–30 % från referensvärdet accepterades.	
Nyckelord	jordanalyser, PCB-föreningar, provningsjämförelse, vatten- och miljölaboratorier	
Publikationsserie och nummer	Reports of Finnish Environment Institute 12/2012	
Publikationens tema		
Projektets namn och nummer		
Finansiär/ uppdragsgivare		
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