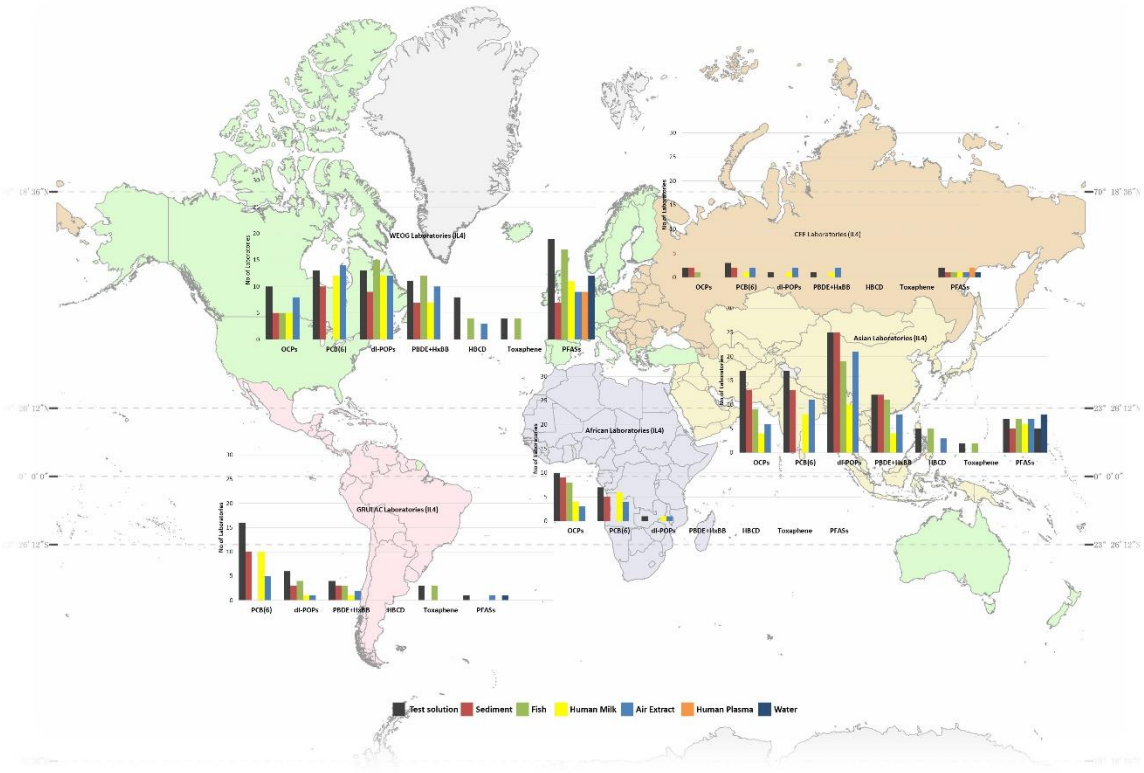
Bi-ennial Global Interlaboratory Assessment on Persistent Organic Pollutants – Fourth Round 2018/2019



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Sketch on title page: World map displaying regions and number of laboratories according to their capacity to analyse groups of POPs - PCB(6), dl-POPs, PBDE+HxBB, toxaphene or PFAS – and type of matrix – test solution, sediment, fish, human milk, air extract, human plasma, and water – as shown in the "Bi-ennial Global Interlaboratory Assessment on Persistent Organic Pollutants, 4th Round"; prepared by Haosong Jiao, Chemicals and Health Branch; Economy Division.

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ACRONYMS AND ABBREVIATIONS

AV	Assigned value
ASE	Accelerated solvent extraction
CEE	Central and Eastern Europe
COP	Conference of the Parties
CV	Coefficient of variation
DDT	Dichlorodiphenyltrichloroethane
dl-PCB	Dioxin-like polychlorinated biphenyls
dl-POPs	Dioxin-like persistent organic pollutants Includes: 29 congeners that were assigned a TEF by WHO/UNEP expert group (van den Berg et al., 2006)
EFSA	European Food Safety Authority
EPA	Environmental Protection Agency (USA)
EtFOSA	N-Ethyl perfluorooctane sulfonamide
EtFOSE	N-Ethyl perfluorooctane sulfonamidoethanol
EU	European Union
FOSA	Perfluorooctane sulfonamide(s)
FOSE	Perfluorooctane sulfonamidoethanol(s)
GC	Gas chromatograph(y)
GC/ECD	Gas chromatograph with electron capture detection
GC/MS	Gas chromatograph with mass spectrometric detection
GEF	Global Environment Facility
GMP	Global Monitoring Plan
GPC	Gel permeation chromatography
GRULAC	Group of Latin America and Caribbean
HCB	Hexachlorobenzene
HBCD	Hexabromocyclododecane
HCBD	Hexachlorobutadiene
HCH	Hexachlorocyclohexane
HDPE	High-density polyethylene
HPLC	High performance liquid chromatography
HRGC	High resolution gas chromatography
HRMS	High resolution mass spectrometry
HxBB	Hexabromobiphenyl
LB	Lower-bound

LC	Liquid chromatograph(y)
LCV	Left-censored values (values below detection limit)
LOD	Limit of detection
LRMS	Low resolution mass spectrometry
MeFOSA	*N*-Methyl perfluorooctane sulfonamides
MeFOSE	*N*-Methyl perfluorooctane sulfonamidoethanol
MS	Mass spectrometer or: mass spectrometry
MS/MS	Tandem mass spectrometry
MTM	Man-Technology-Environment
NA	Not applicable
NAV	No assigned value
NC	Not contained
ND	Not detected
OCP	Organochlorine pesticide
OECD	Organisation for Economic Co-operation and Development
PBB	Polybrominated biphenyl
PBDE	Polybrominated diphenyl ethers
PCB	Polychlorinated biphenyls
PCDD/PCDF	Polychlorinated dibenzo-*para*-dioxins/polychlorinated dibenzofurans
PFAS	Per- or polyfluoroalkyl substances
PFCA	Perfluoroalkyl carboxylic acids
PFOS	Perfluorooctane sulfonic acid (or sulfonate)
PFSA	Perfluoroalkane sulfonic acids
POPs	Persistent organic pollutants
QUASIMEME	Quality Assurance of Information for Marine Environmental Monitoring in Europe
QA/QC	Quality assurance/quality control
TeCDD	2,3,7,8-Tetrachloro-*p*-dibenzodioxin
TEF	Toxicity equivalency factor
TEQ	Toxicity equivalent
UB	Upper-bound
UN	United Nations
UPLC	Ultra performance liquid chromatography
WEOG	Western European and Other Groups
WEPAL	Wageningen Evaluating Programmes for Analytical Laboratories
WHO	World Health Organization

SUMMARY

The fourth Bi-ennial Global Interlaboratory Assessment on Persistent Organic Pollutants (POPs) was organized in 2018. After invitation to participate in this fourth round of the proficiency test, 148 laboratories from 62 countries had registered. In comparison to the 3rd round in which 175 laboratories had registered, this was somewhat lower. However, again several new laboratories (participating for the first time) joined this exercise. The test materials included test solutions of analytical standards, the abiotic matrices included sediment, air (extract) and water and the biotic matrices were fish, human milk and human plasma. The results for the 23 groups of POPs that were listed in the annexes of the Stockholm Convention until 2013 and in addition hexachlorobutadiene, pentachlorobenzene, α-HCH, β-HCH, γ-HCH, α-endosulfan, β-endosulfan, endosulfan sulfate were assessed. This resulted in a report with a wealth of information on POP analysis and a huge dataset from which the laboratories can evaluate their own methods and performance

The Global Monitoring Plan (GMP) of the Stockholm Convention requires that POP laboratories must be capable – at any time – to analyse samples for POPs within a variation of ±25%. Based on this target error of 25%, the statistical model used provided z-scores based on which the performance of each laboratory for each analyte in each matrix can be assessed.

The results show a scattered picture and in comparison, with previous rounds, the performance of many laboratories receded. For a number of analytes, in particularly for organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs), the performance was disappointing. In addition, several laboratories which had been trained within UNEP's or other's capacity building projects and have participated in this scheme for three or four times did not meet the expectations. Relatively low concentrations of OCPs in the test materials and a low fat content in the fish matrix could have played a role. However, these materials are realistic and non-spiked test materials.

A large number of laboratories only analysed a few matrices and especially the standard test solutions, where it was expected that after four rounds of this study, the capacity of the laboratories would have been extended to the analysis of most POPs and the performance would steadily improve. The standard test solution results were often disappointing as well.

More experienced laboratories showed a good to very good performance for chlorinated dibenzodioxins and dibenzofurans and dioxin-like (dl)-PCB, and for PBDE, PFASs and HBCD (α-HBCD in fish and γ-HBCD in sediment). The toxaphene results were encouraging for the test solutions but in a next round, test materials need higher concentrations of toxaphene to enable a realistic test.

This interlaboratory assessment on POPs remains to be among the largest ever organised. Given the overwhelming interest in this study and the need for a substantial increase in quality for many laboratories, it is strongly advised to continue with this study on a bi-ennial basis.

1 INTRODUCTION

This interlaboratory assessment is part of the United Nations Environment Programme's (UNEP) capacity building program for laboratories analysing persistent organic pollutants (POPs) that has started in 2005 with the Environment Facility (GEF) funding and implementing the recommendations by the Conference of the Parties to the Stockholm Convention as expressed in the Guidance on the global monitoring plan for POPs (hereinafter referred to as the guidance document) in article 16 of the Convention (for latest version, see UNEP, 2019c). In chapter 4, the guidance document states that "interlaboratory exercises are often used to assess the effectiveness of quality assurance/quality control (QA/QC) practices among several participating labs and to provide a measure of interlaboratory comparability. This usually involves the circulation and analysis of a common standard or reference sample, often at two or more concentration levels". In order to determine the 'true' concentration of chemicals (POPs) in a sample, a chemical laboratory must be able to prove that it is capable to identify and quantify chemicals (analytes) of interest at concentrations of interest. Such accuracy and precision in the determination of POPs is required by article 16 of the Convention and subsequent guidance developed for the Global Monitoring Plan (GMP). To provide reliable monitoring information for the Parties to the Stockholm Convention, the guidance document aims to "confirm a 50% decline in the levels of POPs within a 10-year period" (UNEP, 2019c). This means that POPs laboratories must be capable – at any time – to analyse samples for POPs within a margin of ±25% (Abalos et al., 2013). Participation in the UNEP-coordinated interlaboratory assessment is encouraged in the UNEP/GEF capacity building and data generation projects to support the Global Monitoring Plan (four regional projects during 2016-2020; for further information, see http://www.brsmeas.org/Decisionmaking/COPsandExCOPs/2019COPs/MeetingDocuments/tabid/7832/language/en-US/Default.aspx, COP and INF number UNEP/POPS/COP.9/INF/36). Some countries encourage laboratories reporting data to the GMP to participate in the interlaboratory assessments. Particularly for the fourth round, national food laboratories in Europe participated in this UNEP-coordinated study to assess their performance for the determination of perfluorooctane sulfonic acid (PFOS) and perfluorooctanoic acid (PFOA) following a human exposure assessment by the European Food Safety Authority (EFSA) (EFSA CONTAM Panel, 2018).

In an interlaboratory assessment, participating laboratories all analyse the same sample within a limited time frame for previously selected analytes and report the results to the coordinator of the study. All results are evaluated together according to international standards, thus allowing a performance classification. The current study gave more assistance than a typical proficiency test. For example, in contrast with a proficiency test, after a first inspection of the data by the coordinating institute, the participating laboratories were allowed to make small corrections for obvious errors, such as units, sum parameters, treatment of non-detects and use of decimals. Because many of the participating laboratories are relatively new in this field, an important objective of this assessment is to bring laboratories at a better level of performance. The results of this exercise and the z-scores obtained by the laboratories are very informative about the quality of the participating laboratories. However, more experienced laboratories that also participate should be careful when using these data for accreditation purposes, as in several cases the results may show some bias, due to the influence of a large group of underperforming laboratories. A careful interpretation is needed, in particular for the POP/matrix combinations, which appeared to be 'difficult' (e.g. concentration close to the detection limit, difficult chromatographic separation).

Within the framework of UNEP's capacity building project for training of laboratory staff on POPs analysis in developing countries, the Department of Environment & Health of the Vrije Universiteit Amsterdam, the Netherlands (VU E&H) and the Man-Technology-Environment (MTM) Research Centre, School of Science and Technology at the University of Örebro, Sweden, have organised the Bi-

ennial Global Interlaboratory Assessment on Persistent Organic Pollutants - Fourth Round 2018/2019; "IL4" for short. The results of the assessment are presented in this report and suggestions for improvement of the performance are given.

The POPs studied in this exercise were polychlorinated dibenzo-*p*-dioxins (PCDD), polychlorinated dibenzofurans (PCDF), polychlorinated biphenyls (PCB) and the organochlorine pesticides (OCP), *i.e.,* DDT and metabolites, aldrin, dieldrin, endrin, chlordanes, hexachlorobenzene (HCB), heptachlor and *cis*-heptachlorepoxide, and mirex. The 'new´ POPs included polybrominated diphenylethers (PBDE), hexachlorocyclohexanes (HCHs), chlordecone (kepone), pentachlorobenzene, α and β-endosulfan, endosulfan sulfate and perfluorinated alkane substances (PFAS) as well as hexachlorobutadiene (HCBD). Separate test solutions and assessments were prepared for toxaphene (three 'Parlar' congeners). In total, 16 matrices were offered for analysis: eight test solutions to cover all POPs, two air extracts (one in toluene for the chlorinated and brominated POPs and one in methanol for the fluorinated POPs), sediment, two fish samples (one naturally contaminated sample and the same sample spiked with toxaphene), human milk, human plasma and water (the latter two for PFAS only).

Hundred and forty-eight laboratories from 62 countries registered (see Appendix I: List of Participants for their names and addresses). However, about one fifth of the laboratories did not submit any result, so that, finally, 117 laboratories from 62 countries reported results for at least one POP and one test sample. All codes are confidential and kept with the organizers; they will only be revealed to third parties after permission of the participating laboratory.

2 MATERIALS AND METHODS

2.1 Identification and Preparation of the Test Samples

2.1.1 Naturally Contaminated Test Samples

All samples, apart from the air extracts, and the 'fish toxaphene' were naturally contaminated with the target analytes. The following samples were offered for POPs analysis:

1. The **sediment** test material was sediment originating from the harbour of Rotterdam (The Netherlands) which was dried at 40 °C and sieved (0.5 mm pore size). After homogenization, individual plastic containers were filled with the test matrix and stored at room temperature until shipment. These samples were obtained from the Wageningen Evaluating Programmes for Analytical Laboratories (WEPAL).

2. The '**fish A'** test material consisted of pike perch (*Stizostedion lucioperca*) originating from the river Amer (Rhine/Meuse delta) from the Netherlands. After cutting and homogenizing, glass jars were filled with ca. 40 g of the homogenate. The jars were sterilized by autoclaving, which made it possible to store and transport the samples at room temperature before opening of the jar.

3. The '**fish toxaphene'** test material consisted of pike perch (river Amer, the Netherlands) which was fortified with toxaphene congeners. After cutting and homogenizing, individual glass jars were filled with ca. 40 g of the homogenate. The jars were sterilized by autoclaving, which made it possible to store and transport the samples at room temperature before opening of the jar.

4. The **human milk** sample consisted of a pooled human milk sample from four milk banks in Sweden. It has been mixed with cows' milk from Sweden (approx. 25%; to reach the sample volume necessary for this interlaboratory assessment). Fifty mL milk was packed in polypropylene bottles and frozen (-20 °C) prior shipment. All results – except for perfluorinated compounds - should be reported on **a lipid weight basis.**

5. The **human plasma** sample consisted of a homogenized pooled human blood plasma of individuals in Sweden including some with potential exposures to PFASs. The samples were stored in HDPE vials and kept frozen (-20 °C). Results should be reported on **product basis (wet weight)** and **as an anion**. Results could be reported for **per- and polyfluoroalkyl substances including PFOS (linear and branched), PFOS precursors, sulfonic** and **carboxylic acids**.

6. The **air extract (TOL)** was an extract from PUFs and glassfiber filters in active samplers taken in Brno, Czech Republic and in Örebro, Sweden, in toluene, to which remaining spiked OCPs, PBDE and HBCD extracts from the 3rd round of the interlaboratory assessment were added. The extract was ampouled into 1.2 mL glass vials before shipment.

7. The **air extract for PFOS and precursor analyses (MeOH)** was a methanol extract of PUFs from active samplers, taken in Brno, Czech Republic and in Örebro, Sweden, mixed with remaining spiked extracts from the 3rd round of this study. The extract was ampouled into 1.2 mL glass vials before shipment.

8. The **water** test material was a combined surface water sample taken from different locations in the Netherlands. After bottling of the water in HDPE bottles, the material was sterilized by irradiation.

2.1.2 Test Solutions

1. The **test solution for OCP (Test solution Y)** consisted of a mixture of OCPs in iso-octane in a concentration range of 1 ng/g - 500 ng/g. This test solution was prepared by VU E&H out of individual stock solutions obtained from Cambridge Isotope Laboratories, Inc. (Tewksbury, USA). After preparation, the solution was ampouled, labelled and stored at room temperature. The OCPs present in the solution were aldrin, dieldrin, endrin, *cis*-chlordane (*alpha*), *trans*-chlordane (*gamma*), oxychlordane, *cis*-nonachlor, *trans*-nonachlor, heptachlor, *cis*-heptachloroepoxide, *trans*- heptachloroepoxide, *o,p'*-DDT, *p,p'*-DDT, *o,p'*-DDD, *p,p'*-DDD, *o,p'*-DDE, *p,p'*-DDE, α-HCH, β-HCH, γ-HCH, α-endosulfan, β-endosulfan, endosulfan sulfate, chlordecone, hexachlorobenzene, hexachlorobutadiene, mirex, and pentachlorobenzene.

2. The **test solution for PCB (Test solution Z)** consisted of a mixture of the indicator PCB (IUPAC nos. 28, 52, 101, 138, 153 and 180) in iso-octane in a concentration range of 1 ng/g - 20 ng/g. This test solution was prepared by VU E&H out of individual stock solutions obtained from Cambridge Isotope Laboratories, Inc. (Tewksbury, USA).

3. The **test solution for PCDD/PCDF** (**Test solution T**) consisted of a mixture of 17 2,3,7,8-substituted PCDD/PCDF congeners in nonane in the concentration range of 5 ng/g - 300 ng/g. This test solution was prepared and labelled by Wellington Laboratories (Guelph, Ontario, Canada).

4. The **test solution for dl-PCB (Test solution U)** consisted of a mixture of 12 dl-PCB in nonane in the concentration range of 5 ng/g - 300 ng/g. This test solution was prepared, ampouled and labelled by Wellington Laboratories (Guelph, Ontario, Canada).

5. The **test solution for PBDE/PBB (Test solution V)** consisted of a mixture of nine PBDE congeners (17, 28, 47, 99, 100, 153, 154, 183 and 209) and PBB 153 in iso-octane in the concentration range of 25 ng/g - 750 ng/g. This test solution was prepared, by VU E&H out of individual stock solutions obtained from Cambridge Isotope Laboratories, Inc. (Tewksbury, USA).

6. The **test solution for toxaphenes (Test solution AA)** consisted of a mixture of Parlar 26, 50, and 62 in nonane in the concentration range of 1 ng/g - 100 ng/g. This test solution was prepared by VU E&H out of individual stock solutions obtained from Cambridge Isotope Laboratories, Inc. (Tewksbury, USA).

7. The **test solution for HBCD (Test solution X)** consisted of a mixture of the α, β, and γ-isomers in toluene in the concentration range of 100 ng/g - 2,000 ng/g. This test solution was prepared, ampouled and labelled by Cambridge Isotope Laboratories, Inc. (Tewksbury, USA).

8. The **test solution for PFAS (Test solution W)** consisted of a mixture of **perfluoroalkyl substances** (perfluoroalkyl acids with perfluoroalkyl carboxylic acids (PFCAs) and perfluoroalkane sulfonic acids (PFSAs), perfluorooctane sulfonamides (FOSAs) and perfluorooctane sulfonamidoethanols (FOSEs)) in methanol in the concentration range of 10 ng/g - 300 ng/g. This test solution was prepared, ampouled and labelled by Wellington Laboratories (Guelph, Ontario, Canada).

2.2 Processing of Samples and Results

2.2.1 Distribution of Test Samples

The human milk, human plasma, and the air extracts as well as the test solutions for PCDD/PCDF, dl-PCB, HBCD, and PFAS were distributed by MTM Research Centre. The sediment, fish, and water test

materials and the test solutions for OCP, PCB, PBDE, and toxaphene were distributed by VU E&H. All shipments containing human milk or plasma samples were packed in polystyrene containers with frozen plastic ice blocks.

Each shipment was accompanied by (a) a letter listing the type of test samples contained in the shipment, (b) a customs letter stating the context of the interlaboratory assessment, especially the technical nature and non-commercial approach, (c) certificates on non-infectiousness of the materials, esp. for the human milk and the human plasma. Instructions on the nature of the test materials as well as a file (MS Excel) to report the results were sent by e-mail to all laboratories.

2.2.2 Reporting Results

All results were combined into one results database (MS Excel) according to laboratory (laboratory code), analyte and test material. In this assessment, these aggregated data were shared with the participating laboratories for a confirmation of their data and in addition, laboratories were allowed to make small corrections for obvious errors, such as units, sum parameters, treatment of non-detects and use of decimals.

2.3 Methods Used by Participants

All participating laboratories used in-house methods for sample preparation, clean-up, extraction and instrumental analysis. It shall be noted that not all laboratories provided information on their methods according to the reporting format.

The methods used for the dl-POP analysis by the participants included modified or adapted standard methods for example EPA 1613 and EU 1948. For PCDD/PCDF and dl-PCB, the vast majority of laboratories reported that high resolution GC/MS (HRGC/HRMS) sector-field systems were used. Few laboratories used MS/MS detection and only one laboratory reported use of a LR-MS detector. One laboratory used GC/ECD for the analysis of dioxin-like PCB and reported on toxic equivalents; they did not analyse PCDD/PCDF. None of the laboratories reported use of an Orbitrap for dl-POPs analysis.

For the non-dl POPs (apart from the PFASs) used methods were more diversified and GC/ECD, low resolution GC/MS (including GCxGC/MS), but also HRGC/HRMS was used.

A variety of techniques and methods was used for extraction and sample preparation. Soxhlet extraction was still the most popular extraction method, although more and more laboratories used pressurized liquid extraction (PLE).

Several organic solvents such as toluene, hexane, acetone, acetonitrile or dichloromethane were used in different combinations for extraction of especially the fish and sediment test materials. A mixture of hexane and acetone was the most preferred combination for the analyses of OCPs and PCBs. For PBDE this combination was also used for fish and sediment, but the most preferred solvent for PBDE in sediment was toluene. For the extraction of PFAS almost all participants used methanol, followed by acetonitrile.

Furthermore, a wide variety of sample clean up open column chromatography was used. Acid or base loaded silica was most often used followed by Florisil and alumina (especially for the OCPs). For the analysis of dl-POPs, some of the laboratories included a carbon column as the final separation step in agreement with the standard methods. Quick Easy Cheap Effective Rugged Safe (QuECHERS) was used by a few laboratories.

The sample extraction, clean-up and detection of the more polar PFAS compounds, the perfluoroalkyl carboxylic and sulfonic acids, including PFOS, is completely different from the traditional POPs. From the 39 laboratories that submitted information on instrumentation and methods used for PFAS analysis, all laboratories reported use of liquid chromatography (LC) approaches. The vast majority reported MS/MS detection; up to three laboratories used an Orbitrap instrument and one laboratory used a time-of-flight instrument.

2.4 Data Assessment

The data assessment was carried out according to the principles employed in the data assessment of the QUASIMEME proficiency testing organisation (www.quasimeme.org).

The assigned value, the between-lab coefficient of variation (CV) values and the laboratory assessment using z-scores are based on the Cofino Model (Cofino *et al.*, 2000, 2017, 2018), as was described in the report of the second round (UNEP, 2015).

The z-scores (Thompson and Wood, 1993; Thompson et al., 2006) are calculated for each participant's data for each matrix / analyte combination, which is given an assigned value.

The formula used is:

$$z\text{-score} = \frac{\text{Mean from Laboratory} - \text{Assigned Value}}{\text{Total Error}}$$

The z-scores can be interpreted as follows:

$|z| < 2$ Satisfactory performance

$2 < |z| < 3$ Questionable performance

$|z| > 3$ Unsatisfactory performance

$|z| > 6$ Extreme performance

Since it is not possible to calculate a z-score for values below the limit of detection (LOD), the so-called 'left censored values' (LCVs) are used. The quality criterion used for LCVs is:

LCV/2 < concentration corresponding to $|z|=3$: LCV consistent with assigned value (AV)

LCV/2 > concentration corresponding to $|z|=3$: LCV inconsistent with AV, *i.e.* LCV reported by laboratory is much higher than numerical values reported by other laboratories.

For the interpretation of the z-scores given, the following keys are used:

z score key:	S – Satisfactory	Color code in Appendix IV	S
	Q – Questionable		Q
	U – Unsatisfactory		U
LCV key:	C – Consistent		C
	I – Inconsistent		I
No data:	B – Blank		B

We consider an assigned value reliable and statistically valid when certain criteria are met. Four different categories are used:

Category 1: For data where the number of numerical observations is $\geqslant 7$:

– An AV is based on the mean when $\geqslant$ 25% of values have a z-score of $|z| < 2$.

- Where < 25% of the data have |z| < 2, the value is only indicative, i.e. at least 25% of the data must be in good agreement.

In this round there were a few cases where we considered it essential to deviate from this criterion. We will discuss this in the text.

Category 2: For data where the number of numerical observations is > 3 and < 7:

- An AV is based on the mean when ⩾ 70% of values have a z-score of |z| < 3 and a minimum of 4 observations have |z| < 2.
- Otherwise, the value is indicative, i.e. for small data sets, n > 3 and n < 7, there needs to be a very good agreement and a maximum of one extreme value before an assigned value can be given.

Category 3: For data where the number of numerical observations is < 4:

- No AV is given. Normally, the median value is given as an indicative value.

Category 4: For data where the high total error > 100% in combination with bad performance, no AV is given.

It is important to note that, in contrast with many other interlaboratory exercises, but in line with the three previous rounds, we have set a target error of 25% on which the z-scores are based. It was already explained in the Introduction that all laboratories producing results for the GMP of the Stockholm Convention should be able to distinguish between two values that differ 50% from each other. Consequently, this exercise may be stricter than most other interlaboratory studies that base the z-score on the standard deviation of the dataset, which is often substantially higher for this type of compounds than the desired ± 25%. This means that compared to other studies it is more difficult to obtain satisfactory z-scores here. It is important to be aware of this when comparing z-scores obtained here, with those from other studies.

In case of a dataset such as generated in an interlaboratory study, ideally one would like to receive duplicate or triplicate values. As this is a lot of work for laboratories, in most cases single values for each parameter are reported. This means that the probability density function can only be used when the dataset shows a normal distribution. In practice the data are often not normal distributed. The choice for the Cofino statistics as a model for evaluation of the data in this study is based on extensive comparisons of evaluations of datasets with different statistical models (Cofino et al., 2017). Of all models tested, the Cofino statistics is the least sensitive for deviations in a dataset from a normal distribution. Many other models using robust statistics suffer from down weighting procedures that insufficiently correct for the outlying data.

3 RESULTS

All results of the 117 laboratories that reported results are given in Appendix II. The z-scores are given in Appendix III. The assessment of the z-scores is given in Appendix IV. Appendix V shows the four plots that characterize the results for each matrix-determinant combination. The submitted results have been evaluated statistically and whenever the data met the requirements as shown in section 2.2, an assigned value was established. Summaries of the assigned values and the percentage of satisfactory to unsatisfactory z-scores are presented below. Whenever numerical LCVs were reported, their consistency with the assigned value was assessed. All Appendices are available online from https://www.oru.se/english/research/research-environments/ent/mtm/research-projects/global-monitoring-plan/Downloads18-19/.

3.1 Participation *per* United Nations Region

In total, 148 laboratories from all five UN regions Africa, Asia-Pacific, Central and Eastern Europe (CEE), Latin America and Caribbean (GRULAC), as well as West European and other groups (WEOG) registered for the interlaboratory assessment. They represented a total of 62 countries. Of these, 117 laboratories submitted data for the test solutions, the sediment, fish, human milk, human plasma, air extracts, or water samples.

The laboratories that submitted results can be assigned to the five UN regions as follows: Africa (n=14), Asia (n=44), CEE (n=5), GRULAC (n=25), and WEOG (n=29). From Table 77 to Table 83 the number of laboratories submitting results *per* region, *per* compound group and *per* matrix is given.

Table 1 shows the degree of participation *per* compound class and matrix. Clearly, the analysis of HxBB, toxaphene and HBCD is still low for many participants. Dioxin laboratories were fewer than in previous rounds (41 participated). For PFASs the number of participants is similar to the number of laboratories for PBDE, although the number of laboratories for PFASs increased slightly whereas the number of laboratories for PBDE decreased. The number of basic POPs laboratories (analyzing OCPs or indicator PCB) is almost 60.

For all other groups, ca. 30-40 laboratories (PCB/OCP) participated, which is somewhat lower than last round. Quite a few of them only analysed the test solutions and a limited number of other matrices.

Table 1: Participating degree per compound class (maximum number of labs is given).

Group	Test solutions	Sediment	Fish	Human milk	Human plasma	Air extract	Water
OCP	58	39	32	22	-	22	-
PCB	56	40	47	37	-	36	-
PCDD/PCDF	41	35	38	23	-	31	-
dl-PCB	41	28	37	25	-		-
PBDE	28	22	26	13	-	25	-
HxBB	10	12	13	6	-	9	-
Toxaphene	10	7	9	5	-	7	-
HBCD	13	8	9	4	-	6	-
PFAS	29	13	25	18	16	18	21

3.2 Compound Group-Specific Results

3.2.1 Organochlorine Pesticides (OCPs)

Table 2: Summary results OCPs, test solution Y (ng/g)

Test Solution Y	n									Between	Inclusion
				Theoretical		Media				lab CV	rate
Analyte	Total	Numerical	LCV	conc.	AV	n	Mean	Min	Max	(%)	(%)
Aldrin	50	48	2	71.5	58.1	61.8	58.1	0.11	154	39	68
Dieldrin	51	51	0	55.4	45.7	51.2	45.7	0.01	176	54	75
Endrin	50	48	2	64.3	47.0	48.2	47.0	0.45	229	53	68
Sum Drins LB (ND = 0)	**56**	**56**	**0**	**191.1**	**NAV**	**131.2**	**135.5**	**0.008**	**498**	**61**	**78**
Sum Drins UB (ND = LOD)	**44**	**44**	**0**	**191.1**	**156**	**173**	**156**	**0.010**	**498**	**41**	**71**
α-Chlordane	45	44	1	69.1	49.4	54.8	49.4	1.00	177	55	73
γ-Chlordane	44	43	1	43.2	37.4	39.6	37.4	1.00	106	33	62
Oxychlordane	27	26	1	47.6	45.3	44.6	45.3	1.00	68	24	62
cis-Nonachlor	21	20	1	60.4	64.3	62.1	64.3	1.00	105	23	62
trans-Nonachlor	24	23	1	57.4	53.1	52.9	53.1	1.00	67	18	67
Sum Chlordanes LB (ND = 0)	**45**	**44**	**1**	**277.7**	**NAV**	**163**	**157**	**0.00**	**370**	**80**	**83**
Sum Chlordanes UB (ND = LOD)	**18**	**18**	**0**	**277.7**	**269**	**259**	**269**	**5.00**	**370**	**19**	**61**
Heptachlor	56	54	2	53.5	40.7	46.5	40.7	0.04	114	59	72
cis-Heptachlorepoxide	45	44	1	88.6	82.6	87.3	82.6	0.68	977	42	68
trans-Heptachlorepoxide	34	29	5	26.8	24.3	26.0	24.3	0.68	1340	37	60
Sum Heptachlors LB (ND = 0)	**57**	**56**	**1**	**168.9**	**NAV**	**120**	**111**	**0.00**	**1485**	**71**	**76**
Sum Heptachlors UB (ND = LOD)	**27**	**27**	**0**	**168.9**	**141**	**167**	**141**	**1.59**	**1485**	**44**	**66**
o,p'-DDT	38	38	0	91.8	73.4	83.0	73.4	0.13	236	59	74
p,p'-DDT	52	49	3	84.9	66.5	77.1	66.5	1.00	240	53	69
o,p'-DDD	40	38	2	40.0	35.9	37.4	35.9	0.15	400	40	66
p,p'-DDD	50	47	3	35.0	31.3	34.4	31.3	0.15	475	71	71
o,p'-DDE	39	39	0	47.1	42.9	44.8	42.9	0.11	150	37	70
p,p'-DDE	56	54	2	29.5	24.2	26.7	24.2	0.11	741	45	67
Sum DDTs LB (ND = 0)	**57**	**56**	**1**	**328.2**	**NAV**	**239**	**220**	**0.00**	**1393**	**72**	**77**
Sum DDTs UB (ND = LOD)	**31**	**31**	**0**	**328.2**	**293**	**288**	**293**	**33.0**	**770**	**34**	**68**
α-HCH	54	52	2	19.7	17.6	18.5	17.6	0.02	72	49	70
β -HCH	49	47	2	212.3	63.2	68.7	63.2	0.07	230	57	72
γ-HCH	57	55	2	48.0	38.3	42.2	38.3	0.98	340	52	69
Sum HCHs LB (ND = 0)	**58**	**57**	**1**	**279.9**	**106**	**125**	**106**	**0.00**	**412**	**67**	**75**
Sum HCHs UB (ND = LOD)	**49**	**49**	**0**	**279.9**	**121**	**132**	**121**	**0.09**	**414**	**57**	**76**
α-Endosulfan	51	48	3	114.1	NAV	81.9	76.2	0.04	531	66	73
β-Endosulfan	47	42	5	68.9	55.8	58.4	55.8	12.0	143	44	69
Endosulfan sulfate	36	32	4	59.2	43.5	44.6	43.5	1.00	92	48	67
Sum Endosulfans LB (ND = 0)	**51**	**49**	**2**	**242.2**	**158**	**159**	**158**	**0.00**	**531**	**56**	**76**
Sum Endosulfans UB (ND = LOD)	**36**	**36**	**0**	**242.2**	**161**	**158**	**161**	**0.04**	**320**	**53**	**79**
Chlordecone	5	4	1	208.6	NAV	34.4	34.1	1.00	95	119	62
Hexachlorobenzene	44	42	2	18.7	17.9	18.6	17.9	0.98	45	39	62
Hexachlorobutadiene	10	9	1	98.2	NAV	78.0	73.5	1.00	134	51	63
Mirex	33	33	0	124.3	103	102	103	1.00	331	40	66
Pentachlorobenzene	24	23	1	13.8	13.4	13.2	13.4	0.21	25	12	56

Table 3: Summary of laboratory performance OCPs, test solution Y

Test Solution Y **Analyte**	**% of the data received**	**% of z-scores |z|<2 Satisfactory**	**% of z-scores 3>|z|>2 Questionable**	**% of z-scores 6>|z|>3 Unsatisfactory**	**% of z-scores |z|>6 Extreme**
Aldrin	34	38	24	24	10
Dieldrin	34	37	16	29	18
Endrin	34	28	20	26	22
Sum Drins LB (ND = 0)	**38**	**0**	**0**	**0**	**0**
Sum Drins UB (ND = LOD)	**30**	**36**	**30**	**23**	**11**
α-Chlordane	30	29	13	38	18
γ-Chlordane	30	50	9	23	16
Oxychlordane	18	56	11	15	15
cis-Nonachlor	14	62	5	10	19
trans-Nonachlor	16	71	13	0	13
Sum Chlordanes LB (ND = 0)	**30**	**0**	**0**	**0**	**0**
Sum Chlordanes UB (ND = LOD)	**12**	**61**	**6**	**17**	**17**
Heptachlor	38	32	18	27	20
cis-Heptachlorepoxide	30	44	11	20	22
trans-Heptachlorepoxide	23	47	9	12	18
Sum Heptachlors LB (ND = 0)	**39**	**0**	**0**	**0**	**0**
Sum Heptachlors UB (ND = LOD)	**18**	**33**	**22**	**26**	**19**
o,p'-DDT	26	29	18	29	24
p,p'-DDT	35	31	17	29	17
o,p'-DDD	27	45	13	20	18
p,p'-DDD	34	28	16	20	30
o,p'-DDE	26	51	15	10	23
p,p'-DDE	38	46	13	14	23
Sum DDTs LB (ND = 0)	**39**	**0**	**0**	**0**	**0**
Sum DDTs UB (ND = LOD)	**21**	**52**	**16**	**19**	**13**
α-HCH	36	43	9	24	20
β -HCH	33	27	18	31	20
γ-HCH	39	33	16	25	23
Sum HCHs LB (ND = 0)	**39**	**26**	**10**	**33**	**29**
Sum HCHs UB (ND = LOD)	**33**	**29**	**24**	**27**	**20**
α-Endosulfan	34	0	0	0	0
β-Endosulfan	32	36	21	23	9
Endosulfan sulfate	24	36	17	28	8
Sum Endosulfans LB (ND = 0)	**34**	**27**	**20**	**29**	**20**
Sum Endosulfans UB (ND = LOD)	**24**	**33**	**17**	**36**	**14**
Chlordecone	3	0	0	0	0
Hexachlorobenzene	30	48	9	11	27
Hexachlorobutadiene	7	0	0	0	0
Mirex	22	45	15	18	21
Pentachlorobenzene	16	58	4	17	17

Table 4: Summary results OCPs, sediment (ng/g)

Sediment	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
Aldrin	35	22	13	NAV	5.0	3.8	0.11	39.1	87	55
Dieldrin	33	21	12	NAV	3.7	3.0	0.08	9.3	67	54
Endrin	32	20	12	NAV	1.9	1.6	0.17	932	130	54
Sum Drins LB (ND = 0)	**37**	**26**	**11**	**8.6**	**9.2**	**8.6**	**0.000**	**932**	**76**	**73**
Sum Drins UB (ND = LOD)	**29**	**29**	**0**	**NAV**	**8.3**	**8.2**	**0.003**	**341**	**90**	**75**
α-Chlordane	27	11	16	NAV	0.58	0.35	0.004	7.3	201	49
γ-Chlordane	27	12	15	NAV	0.56	0.35	0.03	5.8	189	50
Oxychlordane	13	5	8	NAV	1.22	0.35	0.22	11.5	242	37
cis-Nonachlor	12	8	4	NAV	0.33	0.18	0.001	4.0	216	55
trans-Nonachlor	12	8	4	0.1	0.12	0.07	0.04	12.0	126	58
Sum Chlordanes LB (ND = 0)	**28**	**17**	**11**	**NAV**	**1.9**	**1.5**	**0.00**	**25.0**	**163**	**71**
Sum Chlordanes UB (ND = LOD)	**9**	**9**	**0**	**NAV**	**0.82**	**0.44**	**0.19**	**25.0**	**184**	**55**
Heptachlor	38	18	20	NAV	2.3	1.6	0.005	3633	142	43
cis-Heptachlorepoxide	30	14	16	NAV	0.98	0.73	0.005	560	174	47
trans-Heptachlorepoxide	23	9	14	NAV	1.20	0.50	0.002	12.0	252	37
Sum Heptachlors LB (ND = 0)	**39**	**21**	**18**	**NAV**	**2.7**	**1.7**	**0.000**	**3633**	**191**	**64**
Sum Heptachlors UB (ND = LOD)	**17**	**17**	**0**	**NAV**	**0.95**	**0.44**	**0.003**	**349**	**271**	**53**
o,p'-DDT	23	14	9	0.1	0.18	0.10	0.06	25.4	140	54
p,p'-DDT	33	19	14	0.6	0.74	0.55	0.33	23.5	87	51
o,p'-DDD	24	15	9	0.5	0.68	0.47	0.15	8.9	73	51
p,p'-DDD	37	25	12	1.1	1.4	1.1	0.15	246	98	52
o,p'-DDE	24	14	10	0.1	0.13	0.12	0.08	19.0	32	55
p,p'-DDE	37	26	11	1.9	2.2	1.9	0.08	38.6	52	53
Sum DDTs LB (ND = 0)	**38**	**28**	**10**	**5.4**	**6.0**	**5.4**	**0.00**	**256**	**77**	**61**
Sum DDTs UB (ND = LOD)	**19**	**19**	**0**	**5.2**	**6.4**	**5.2**	**3.1**	**87.3**	**72**	**58**
α-HCH	37	23	14	NAV	1.23	0.60	0.01	992	242	50
β -HCH	35	19	16	0.3	0.34	0.27	0.03	381	114	49
γ-HCH	39	24	15	NAV	0.33	0.16	0.002	57.0	241	50
Sum HCHs LB (ND = 0)	**39**	**26**	**13**	**NAV**	**1.47**	**0.67**	**0.000**	**1374**	**256**	**56**
Sum HCHs UB (ND = LOD)	**33**	**33**	**0**	**NAV**	**3.2**	**2.2**	**0.003**	**1374**	**187**	**62**
α-Endosulfan	35	15	20	NAV	2.1	1.0	0.06	650	206	42
β-Endosulfan	31	13	18	NAV	4.2	1.9	0.07	498	149	42
Endosulfan sulfate	24	8	16	NAV	2.8	1.4	0.03	24.7	186	41
Sum Endosulfans LB (ND = 0)	**35**	**20**	**15**	**NAV**	**5.6**	**4.1**	**0.000**	**1148**	**169**	**71**
Sum Endosulfans UB (ND = LOD)	**24**	**24**	**0**	**NAV**	**7.1**	**6.3**	**0.003**	**30.0**	**120**	**78**
Chlordecone	2	1	1	NAV	NAV	NAV	0.66	0.66	NAV	NAV
Hexachlorobenzene	27	21	6	3.2	3.4	3.2	0.00	1675	45	64
Hexachlorobutadiene	4	2	2	NAV	NAV	NAV	0.49	0.61	NAV	NAV
Mirex	17	10	7	NAV	2.6	1.8	0.71	4.6	71	51
Pentachlorobenzene	15	13	2	1.8	1.9	1.8	0.61	43	26	59

Table 5: Summary of laboratory performance OCPs, sediment

Sediment Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
Aldrin	24	0	0	0	0
Dieldrin	22	0	0	0	0
Endrin	22	0	0	0	0
Sum Drins LB (ND = 0)	**25**	**22**	**5**	**22**	**22**
Sum Drins UB (ND = LOD)	**20**	**0**	**0**	**0**	**0**
α-Chlordane	18	0	0	0	0
γ-Chlordane	18	0	0	0	0
Oxychlordane	9	0	0	0	0
cis-Nonachlor	8	0	0	0	0
trans-Nonachlor	8	33	0	8	25
Sum Chlordanes LB (ND = 0)	**19**	**0**	**0**	**0**	**0**
Sum Chlordanes UB (ND = LOD)	**6**	**0**	**0**	**0**	**0**
Heptachlor	26	0	0	0	0
cis-Heptachlorepoxide	20	0	0	0	0
trans-Heptachlorepoxide	16	0	0	0	0
Sum Heptachlors LB (ND = 0)	**26**	**0**	**0**	**0**	**0**
Sum Heptachlors UB (ND = LOD)	**11**	**0**	**0**	**0**	**0**
o,p'-DDT	16	30	0	4	26
p,p'-DDT	22	18	12	0	27
o,p'-DDD	16	25	0	17	21
p,p'-DDD	25	19	5	11	32
o,p'-DDE	16	38	0	0	21
p,p'-DDE	25	32	5	8	24
Sum DDTs LB (ND = 0)	**26**	**21**	**8**	**13**	**32**
Sum DDTs UB (ND = LOD)	**13**	**42**	**5**	**11**	**42**
α-HCH	25	0	0	0	0
β -HCH	24	20	6	3	26
γ-HCH	26	0	0	0	0
Sum HCHs LB (ND = 0)	**26**	**0**	**0**	**0**	**0**
Sum HCHs UB (ND = LOD)	**22**	**0**	**0**	**0**	**0**
α-Endosulfan	24	0	0	0	0
β-Endosulfan	21	0	0	0	0
Endosulfan sulfate	16	0	0	0	0
Sum Endosulfans LB (ND = 0)	**24**	**0**	**0**	**0**	**0**
Sum Endosulfans UB (ND = LOD)	**16**	**0**	**0**	**0**	**0**
Chlordecone	1	0	0	0	0
Hexachlorobenzene	18	30	19	19	11
Hexachlorobutadiene	3	0	0	0	0
Mirex	11	0	0	0	0
Pentachlorobenzene	10	47	7	27	7

Table 6: Summary results OCPs, fish (product basis) (ng/g)

Fish A	n								Between lab CV	Inclusion rate
Analyte	**Total**	**Numerical**	**LCV**	**AV**	**Median**	**Mean**	**Min**	**Max**	**(%)**	**(%)**
Aldrin	28	7	21	NAV	2.4	0.3	0.007	7063	260	34
Dieldrin	27	7	20	NAV	0.45	0.07	0.04	1801	373	37
Endrin	29	10	19	NAV	4.3	0.27	0.004	1325	768	33
Sum Drins LB (ND = 0)	**30**	**11**	**19**	**NAV**	**4.6**	**1.9**	**0.000**	**9477**	**292**	**55**
Sum Drins UB (ND = LOD)	**27**	**27**	**0**	**NAV**	**1.5**	**1.0**	**0.003**	**9477**	**186**	**66**
α-Chlordane	23	4	19	NAV	0.06	0.02	0.02	3.2	163	47
γ-Chlordane	22	3	19	NAV	0.01	0.01	0.01	5.6	10	48
Oxychlordane	14	4	10	NAV	2.5	0.17	0.01	6.2	441	45
cis-Nonachlor	13	4	9	NAV	0.03	0.02	0.02	11.0	81	61
trans-Nonachlor	13	5	8	0.0	0.04	0.04	0.03	9.0	41	64
Sum Chlordanes LB (ND = 0)	**24**	**8**	**16**	**NAV**	**1.7**	**1.4**	**0.00**	**25.0**	**184**	**63**
Sum Chlordanes UB (ND = LOD)	**10**	**10**	**0**	**NAV**	**0.40**	**0.28**	**0.10**	**7.6**	**144**	**61**
Heptachlor	31	10	21	NAV	1.9	0.33	0.0000007	4029	381	33
cis-Heptachlorepoxide	26	9	17	NAV	0.03	0.01	0.000004	700	255	49
trans-Heptachlorepoxide	20	5	15	NAV	0.50	0.06	0.0000005	57.8	510	31
Sum Heptachlors LB (ND = 0)	**30**	**13**	**17**	**NAV**	**1.3**	**0.67**	**0.000000**	**4029**	**243**	**64**
Sum Heptachlors UB (ND = LOD)	**16**	**16**	**0**	**NAV**	**0.65**	**0.41**	**0.000006**	**222**	**196**	**66**
o,p'-DDT	20	8	12	NAV	0.54	0.24	0.006	4831	219	44
p,p'-DDT	27	10	17	NAV	0.48	0.14	0.01	120	378	39
o,p'-DDD	19	10	9	0.1	0.16	0.12	0.08	1975	60	61
p,p'-DDD	28	18	10	0.4	0.54	0.42	0.26	923	58	61
o,p'-DDE	20	11	9	0.0	0.06	0.05	0.04	11.0	47	54
p,p'-DDE	28	21	7	2.3	2.3	2.3	0.06	918	51	62
Sum DDTs LB (ND = 0)	**29**	**23**	**6**	**3.3**	**3.9**	**3.3**	**0.00**	**8653**	**62**	**63**
Sum DDTs UB (ND = LOD)	**17**	**17**	**0**	**3.0**	**3.5**	**3.0**	**2.1**	**60**	**41**	**57**
α-HCH	32	11	21	NAV	1.2	0.37	0.002	3736	321	35
β -HCH	28	15	13	0.1	0.09	0.06	0.004	2332	171	54
γ-HCH	32	14	18	NAV	0.75	0.41	0.002	31.0	225	41
Sum HCHs LB (ND = 0)	**32**	**18**	**14**	**NAV**	**0.74**	**0.33**	**0.000**	**6069**	**278**	**61**
Sum HCHs UB (ND = LOD)	**28**	**28**	**0**	**NAV**	**0.90**	**0.44**	**0.003**	**6069**	**270**	**57**
α-Endosulfan	30	7	23	NAV	5.0	0.15	0.009	248	484	39
β-Endosulfan	28	7	21	NAV	2.6	0.17	0.006	36.0	531	34
Endosulfan sulfate	20	3	17	NAV	0.90	0.06	0.13	1.3	248	44
Sum Endosulfans LB (ND = 0)	**30**	**9**	**21**	**NAV**	**7.1**	**4.6**	**0.000**	**248**	**194**	**60**
Sum Endosulfans UB (ND = LOD)	**20**	**20**	**0**	**NAV**	**1.2**	**0.83**	**0.003**	**30**	**202**	**59**
Chlordecone	2	0	2	NAV	NAV	NAV			NAV	NAV
Hexachlorobenzene	25	18	7	NAV	0.70	0.55	0.08	2087	112	57
Hexachlorobutadiene	4	2	2	NAV	NAV	NAV	0.03	0.32	NAV	NAV
Mirex	17	4	13	NAV	0.01	0.01	0.002	1.3	82	67
Pentachlorobenzene	14	7	7	0.1	0.07	0.05	0.01	15.0	79	72

Table 7: Summary of laboratory performance OCPs, fish

Fish A Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
Aldrin	19	0	0	0	0
Dieldrin	18	0	0	0	0
Endrin	20	0	0	0	0
Sum Drins LB (ND = 0)	**20**	**0**	**0**	**0**	**0**
Sum Drins UB (ND = LOD)	**18**	**0**	**0**	**0**	**0**
α-Chlordane	16	0	0	0	0
γ-Chlordane	15	0	0	0	0
Oxychlordane	9	0	0	0	0
cis-Nonachlor	9	0	0	0	0
trans-Nonachlor	9	31	0	0	8
Sum Chlordanes LB (ND = 0)	**16**	**0**	**0**	**0**	**0**
Sum Chlordanes UB (ND = LOD)	**7**	**0**	**0**	**0**	**0**
Heptachlor	21	0	0	0	0
cis-Heptachlorepoxide	18	0	0	0	0
trans-Heptachlorepoxide	14	0	0	0	0
Sum Heptachlors LB (ND = 0)	**20**	**0**	**0**	**0**	**0**
Sum Heptachlors UB (ND = LOD)	**11**	**0**	**0**	**0**	**0**
o,p'-DDT	14	0	0	0	0
p,p'-DDT	18	0	0	0	0
o,p'-DDD	13	32	5	0	16
p,p'-DDD	19	25	14	7	18
o,p'-DDE	14	30	0	5	20
p,p'-DDE	19	32	7	25	11
Sum DDTs LB (ND = 0)	**20**	**34**	**3**	**14**	**28**
Sum DDTs UB (ND = LOD)	**11**	**47**	**12**	**0**	**41**
α-HCH	22	0	0	0	0
β -HCH	19	21	7	4	21
γ-HCH	22	0	0	0	0
Sum HCHs LB (ND = 0)	**22**	**0**	**0**	**0**	**0**
Sum HCHs UB (ND = LOD)	**19**	**0**	**0**	**0**	**0**
α-Endosulfan	20	0	0	0	0
β-Endosulfan	19	0	0	0	0
Endosulfan sulfate	14	0	0	0	0
Sum Endosulfans LB (ND = 0)	**20**	**0**	**0**	**0**	**0**
Sum Endosulfans UB (ND = LOD)	**14**	**0**	**0**	**0**	**0**
Chlordecone	1	0	0	0	0
Hexachlorobenzene	17	0	0	0	0
Hexachlorobutadiene	3	0	0	0	0
Mirex	11	0	0	0	0
Pentachlorobenzene	9	29	14	0	7

Table 8: Summary results OCPs, human milk (lipid weight basis) (ng/g)

Human milk	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
Aldrin	19	8	11	NAV	4.0	2.5	0.11	137	171	38
Dieldrin	18	9	9	NAV	1.8	0.94	0.63	847	132	45
Endrin	18	7	11	NAV	51.0	18.0	0.20	4432	290	31
Sum Drins LB (ND = 0)	**20**	**13**	**7**	**NAV**	**4.9**	**2.6**	**0.000**	**5279**	**198**	**53**
Sum Drins UB (ND = LOD)	**17**	**17**	**0**	**NAV**	**13.2**	**8.2**	**0.003**	**5279**	**184**	**70**
α-Chlordane	16	3	13	NAV	100	1.5	9.3	117	219	36
γ-Chlordane	15	4	11	NAV	0.44	0.22	0.06	90	193	47
Oxychlordane	9	4	5	0.9	0.89	0.88	0.84	1.0	5	46
cis-Nonachlor	7	4	3	NAV	0.27	0.24	0.17	1.4	33	67
trans-Nonachlor	8	6	2	NAV	1.2	1.1	0.65	1.7	32	65
Sum Chlordanes LB (ND = 0)	**16**	**9**	**7**	**NAV**	**2.1**	**1.6**	**0.00**	**208**	**79**	**69**
Sum Chlordanes UB (ND = LOD)	**6**	**6**	**0**	**NAV**	**2.6**	**2.4**	**2.0**	**22.0**	**16**	**61**
Heptachlor	20	6	14	NAV	5.1	2.1	0.23	155	249	30
cis-Heptachlorepoxide	16	10	6	NAV	1.7	0.79	0.39	472	195	53
trans-Heptachlorepoxide	16	3	13	NAV	1.3	0.05	0.52	666	420	40
Sum Heptachlors LB (ND = 0)	**20**	**12**	**8**	**NAV**	**3.5**	**1.9**	**0.00**	**1138**	**210**	**61**
Sum Heptachlors UB (ND = LOD)	**12**	**12**	**0**	**NAV**	**6.0**	**4.5**	**0.52**	**1138**	**173**	**65**
o,p'-DDT	15	8	7	NAV	0.53	0.35	0.20	793	107	60
p,p'-DDT	18	9	9	2.1	2.6	2.1	1.5	54.0	50	48
o,p'-DDD	15	3	12	NAV	29.1	1.5	0.37	794	370	36
p,p'-DDD	19	7	12	NAV	1.7	0.57	0.03	2164	299	36
o,p'-DDE	15	5	10	NAV	0.82	0.40	0.03	352	223	43
p,p'-DDE	21	15	6	25.9	30.7	25.9	0.91	1597	56	52
Sum DDTs LB (ND = 0)	**21**	**16**	**5**	**31.6**	**33.8**	**31.6**	**0.00**	**5700**	**93**	**67**
Sum DDTs UB (ND = LOD)	**11**	**11**	**0**	**26.7**	**30.0**	**26.7**	**2.5**	**234**	**44**	**67**
α-HCH	22	9	13	NAV	7.0	2.1	0.07	1915	360	36
β -HCH	20	12	8	2.2	2.4	2.2	1.2	7017	30	51
γ-HCH	22	8	14	NAV	2.2	0.90	0.08	171	228	43
Sum HCHs LB (ND = 0)	**22**	**15**	**7**	**2.5**	**2.8**	**2.5**	**0.000**	**9103**	**66**	**58**
Sum HCHs UB (ND = LOD)	**20**	**20**	**0**	**NAV**	**7.2**	**5.2**	**0.003**	**9103**	**155**	**63**
α-Endosulfan	19	6	13	NAV	5.1	1.7	0.55	1679	233	39
β-Endosulfan	16	4	12	NAV	12.8	1.8	1.4	45.7	244	39
Endosulfan sulfate	14	2	12	NAV	NAV	NAV	8.0	95.6	NAV	NAV
Sum Endosulfans LB (ND = 0)	**19**	**6**	**13**	**NAV**	**20.27**	**10.92**	**0.000**	**1679**	**196**	**62**
Sum Endosulfans UB (ND = LOD)	**14**	**14**	**0**	**NAV**	**10.57**	**7.06**	**0.003**	**1071**	**147**	**67**
Chlordecone	2	0	2	NAV	NAV	NAV			NAV	NAV
Hexachlorobenzene	17	13	4	3.9	4.5	3.9	0.47	88.4	65	70
Hexachlorobutadiene	3	2	1	NAV	NAV	NAV	0.16	0.24	NAV	NAV
Mirex	12	3	9	NAV	0.15	0.07	0.09	0.68	85	58
Pentachlorobenzene	9	5	4	NAV	0.19	0.15	0.04	1.00	135	58

Table 9: Summary of laboratory performance OCPs, human milk

Human milk Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
Aldrin	13	0	0	0	0
Dieldrin	12	0	0	0	0
Endrin	12	0	0	0	0
Sum Drins LB (ND = 0)	**14**	**0**	**0**	**0**	**0**
Sum Drins UB (ND = LOD)	**11**	**0**	**0**	**0**	**0**
α-Chlordane	11	0	0	0	0
γ-Chlordane	10	0	0	0	0
Oxychlordane	6	44	0	0	0
cis-Nonachlor	5	0	0	0	0
trans-Nonachlor	5	0	0	0	0
Sum Chlordanes LB (ND = 0)	**11**	**0**	**0**	**0**	**0**
Sum Chlordanes UB (ND = LOD)	**4**	**0**	**0**	**0**	**0**
Heptachlor	14	0	0	0	0
cis-Heptachlorepoxide	11	0	0	0	0
trans-Heptachlorepoxide	11	0	0	0	0
Sum Heptachlors LB (ND = 0)	**14**	**0**	**0**	**0**	**0**
Sum Heptachlors UB (ND = LOD)	**8**	**0**	**0**	**0**	**0**
o,p'-DDT	10	0	0	0	0
p,p'-DDT	12	22	11	0	17
o,p'-DDD	10	0	0	0	0
p,p'-DDD	13	0	0	0	0
o,p'-DDE	10	0	0	0	0
p,p'-DDE	14	33	5	10	24
Sum DDTs LB (ND = 0)	**14**	**24**	**5**	**10**	**38**
Sum DDTs UB (ND = LOD)	**7**	**45**	**18**	**9**	**27**
α-HCH	15	0	0	0	0
β -HCH	14	35	0	5	20
γ-HCH	15	0	0	0	0
Sum HCHs LB (ND = 0)	**15**	**32**	**0**	**9**	**27**
Sum HCHs UB (ND = LOD)	**14**	**0**	**0**	**0**	**0**
α-Endosulfan	13	0	0	0	0
β-Endosulfan	11	0	0	0	0
Endosulfan sulfate	9	0	0	0	0
Sum Endosulfans LB (ND = 0)	**13**	**0**	**0**	**0**	**0**
Sum Endosulfans UB (ND = LOD)	**9**	**0**	**0**	**0**	**0**
Chlordecone	1	0	0	0	0
Hexachlorobenzene	11	29	0	29	18
Hexachlorobutadiene	2	0	0	0	0
Mirex	8	0	0	0	0
Pentachlorobenzene	6	0	0	0	0

Table 10: Summary results OCPs, air extract (TOL) (ng/g)

Air extract (TOL)	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
Aldrin	18	15	3	NAV	3.0	2.5	0.34	2770	113	64
Dieldrin	18	17	1	2.4	3.0	2.4	0.86	1186	77	62
Endrin	19	13	6	NAV	1.5	1.1	0.18	8718	109	60
Sum Drins LB (ND = 0)	**19**	**19**	**0**	**NAV**	**6.4**	**5.1**	**1.1**	**12674**	**135**	**57**
Sum Drins UB (ND = LOD)	**17**	**17**	**0**	**8.2**	**9.5**	**8.2**	**2.3**	**12674**	**118**	**57**
α-Chlordane	20	18	2	1.5	1.9	1.5	0.37	223	65	60
γ-Chlordane	20	17	3	1.5	1.6	1.5	0.02	234	76	69
Oxychlordane	14	11	3	1.3	1.4	1.3	0.22	5.9	25	60
cis-Nonachlor	11	7	4	0.2	0.19	0.18	0.12	18.1	49	61
trans-Nonachlor	13	9	4	NAV	0.32	0.23	0.08	14.7	121	50
Sum Chlordanes LB (ND = 0)	**20**	**18**	**2**	**4.6**	**5.5**	**4.6**	**0.00**	**457**	**89**	**61**
Sum Chlordanes UB (ND = LOD)	**8**	**8**	**0**	**NAV**	**8.4**	**6.9**	**4.5**	**66.6**	**70**	**67**
Heptachlor	20	14	6	1.4	1.6	1.4	0.18	9375	56	61
cis-Heptachlorepoxide	18	15	3	1.6	2.0	1.6	0.29	2117	94	62
trans-Heptachlorepoxide	13	9	4	NAV	8.4	2.2	1.2	3487	340	41
Sum Heptachlors LB (ND = 0)	**20**	**16**	**4**	**NAV**	**4.8**	**3.9**	**0.00**	**14979**	**130**	**59**
Sum Heptachlors UB (ND = LOD)	**11**	**11**	**0**	**4.8**	**4.9**	**4.8**	**3.4**	**14979**	**40**	**60**
o,p'-DDT	18	16	2	2.2	2.3	2.2	0.13	5.6	15	55
p,p'-DDT	19	17	2	2.9	3.1	2.9	0.55	204	22	53
o,p'-DDD	18	15	3	1.4	1.5	1.4	0.45	1136	31	57
p,p'-DDD	20	17	3	1.6	1.7	1.6	0.31	2970	50	62
o,p'-DDE	19	17	2	1.6	1.6	1.6	0.44	448	22	61
p,p'-DDE	21	20	1	6.4	6.3	6.4	0.11	12.4	22	60
Sum DDTs LB (ND = 0)	**21**	**20**	**1**	**16.0**	**17.1**	**16.0**	**0.00**	**4553**	**34**	**65**
Sum DDTs UB (ND = LOD)	**15**	**15**	**0**	**17.4**	**17.2**	**17.4**	**6.5**	**58**	**23**	**64**
α-HCH	22	20	2	2.1	2.3	2.1	0.16	46840	49	62
β -HCH	20	17	3	2.2	2.6	2.2	0.14	1591	76	61
γ-HCH	22	21	1	5.8	5.9	5.8	0.51	17079	20	57
Sum HCHs LB (ND = 0)	**22**	**21**	**1**	**10.0**	**11.1**	**10.0**	**0.00**	**65510**	**41**	**61**
Sum HCHs UB (ND = LOD)	**20**	**20**	**0**	**10.2**	**10.9**	**10.2**	**0.81**	**65510**	**35**	**60**
α-Endosulfan	19	14	5	NAV	3.2	2.4	0.24	8323	131	60
β-Endosulfan	16	13	3	NAV	3.1	2.5	0.44	98.1	156	54
Endosulfan sulfate	12	8	4	NAV	7.3	2.8	0.04	395	309	44
Sum Endosulfans LB (ND = 0)	**19**	**16**	**3**	**NAV**	**9.1**	**6.3**	**0.00**	**8323**	**172**	**60**
Sum Endosulfans UB (ND = LOD)	**11**	**11**	**0**	**NAV**	**10.0**	**7.0**	**1.2**	**526**	**154**	**63**
Chlordecone	1	1	0	NAV	NAV	NAV	3.2	3.2	NAV	NAV
Hexachlorobenzene	19	18	1	5.0	5.0	5.0	0.88	6214	20	61
Hexachlorobutadiene	2	2	0	NAV	NAV	NAV	0.70	2.1	NAV	NAV
Mirex	16	12	4	1.9	2.1	1.9	0.13	12.4	102	58
Pentachlorobenzene	14	10	4	0.8	0.74	0.75	0.33	1.0	21	69

Table 11: Summary of laboratory performance OCPs, air extract (TOL)

Air extract (TOL) Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
Aldrin	12	0	0	0	0
Dieldrin	12	39	11	11	33
Endrin	13	0	0	0	0
Sum Drins LB (ND = 0)	**13**	**0**	**0**	**0**	**0**
Sum Drins UB (ND = LOD)	**11**	**29**	**12**	**12**	**47**
α-Chlordane	14	35	10	10	35
γ-Chlordane	14	35	5	15	30
Oxychlordane	9	50	0	7	21
cis-Nonachlor	7	36	0	9	18
trans-Nonachlor	9	0	0	0	0
Sum Chlordanes LB (ND = 0)	**14**	**35**	**10**	**5**	**40**
Sum Chlordanes UB (ND = LOD)	**5**	**0**	**0**	**0**	**0**
Heptachlor	14	35	5	5	25
cis-Heptachlorepoxide	12	28	6	11	39
trans-Heptachlorepoxide	9	0	0	0	0
Sum Heptachlors LB (ND = 0)	**14**	**0**	**0**	**0**	**0**
Sum Heptachlors UB (ND = LOD)	**7**	**45**	**9**	**9**	**36**
o,p'-DDT	12	56	6	6	22
p,p'-DDT	13	47	11	5	26
o,p'-DDD	12	44	6	17	17
p,p'-DDD	14	30	20	5	30
o,p'-DDE	13	53	11	11	16
p,p'-DDE	14	57	10	19	10
Sum DDTs LB (ND = 0)	**14**	**48**	**5**	**24**	**19**
Sum DDTs UB (ND = LOD)	**10**	**60**	**7**	**20**	**13**
α-HCH	15	41	5	18	27
β -HCH	14	25	15	10	35
γ-HCH	15	64	0	0	32
Sum HCHs LB (ND = 0)	**15**	**45**	**0**	**18**	**32**
Sum HCHs UB (ND = LOD)	**14**	**50**	**5**	**10**	**35**
α-Endosulfan	13	0	0	0	0
β-Endosulfan	11	0	0	0	0
Endosulfan sulfate	8	0	0	0	0
Sum Endosulfans LB (ND = 0)	**13**	**0**	**0**	**0**	**0**
Sum Endosulfans UB (ND = LOD)	**7**	**0**	**0**	**0**	**0**
Chlordecone	1	0	0	0	0
Hexachlorobenzene	13	58	5	11	21
Hexachlorobutadiene	1	0	0	0	0
Mirex	11	31	6	0	38
Pentachlorobenzene	9	50	14	7	0

3.2.2 Polychlorinated Biphenyls (PCB)

Table 12: Summary results indicator PCB, test solution Z (ng/g)

Test Solution Z	n									Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	Theoretical conc.	AV	Median	Mean	Min	Max	(%)	(%)
PCB 28	53	52	1	4.4	3.9	4.2	3.9	0.34	41.1	42	70
PCB 52	56	54	2	9.6	10.5	11.4	10.5	1.2	121	38	67
PCB 101	56	55	1	5.3	5.0	5.4	5.0	0.48	50.6	43	69
PCB 138	55	53	2	5.7	6.5	6.9	6.5	0.70	48.4	34	66
PCB 153	56	54	2	12.3	11.6	12.1	11.6	0.66	235	38	70
PCB 180	55	53	2	11.4	10.4	11.2	10.4	1.2	48.4	36	69
Sum Indicator PCB LB (ND = 0)	**56**	**55**	**1**	**48.7**	**48.8**	**50.8**	**48.8**	**0.00**	**486**	**35**	**71**
Sum Indicator PCB UB (ND = LOD)	**53**	**53**	**0**	**48.7**	**48.2**	**50.8**	**48.2**	**5.4**	**486**	**36**	**73**

Table 13: Summary of laboratory performance indicator PCB, test solution Z

Test Solution Z	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
PCB 28	36	42	21	19	17
PCB 52	38	48	11	21	16
PCB 101	38	43	16	21	18
PCB 138	37	49	15	18	15
PCB 153	38	50	16	16	14
PCB 180	37	49	16	20	11
Sum Indicator PCB LB (ND = 0)	**38**	**52**	**14**	**18**	**14**
Sum Indicator PCB UB (ND = LOD)	**36**	**51**	**17**	**19**	**13**

Table 14: Summary results indicator PCB, sediment (ng/g)

Sediment	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
PCB 28	39	36	3	4.4	4.5	4.4	0.007	157	58	73
PCB 52	40	38	2	3.9	4.0	3.9	0.005	209	28	61
PCB 101	40	36	4	6.1	6.2	6.1	0.01	348	24	60
PCB 138	40	36	4	8.1	8.2	8.1	0.02	84.0	31	64
PCB 153	40	38	2	10.7	11.0	10.7	0.02	36.0	29	63
PCB 180	40	34	6	6.2	5.9	6.2	0.01	134	33	62
Sum Indicator PCB LB (ND = 0)	**40**	**38**	**2**	**39.9**	**40.7**	**39.9**	**0.00**	**698**	**25**	**69**
Sum Indicator PCB UB (ND = LOD)	**39**	**39**	**0**	**40.1**	**40.8**	**40.1**	**0.07**	**698**	**26**	**68**

Table 15: Summary of laboratory performance indicator PCB, sediment

Sediment	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
PCB 28	26	31	18	21	23
PCB 52	27	55	10	8	23
PCB 101	27	55	13	10	13
PCB 138	27	55	13	5	18
PCB 153	27	60	5	10	20
PCB 180	27	45	15	15	10
Sum Indicator PCB LB (ND = 0)	**27**	**63**	**8**	**15**	**10**
Sum Indicator PCB UB (ND = LOD)	**26**	**62**	**10**	**15**	**13**

Table 16: Summary results indicator PCB, fish (product basis) (ng/g)

Fish A	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
PCB 28	46	42	4	NAV	0.52	0.43	0.01	26.8	126	69
PCB 52	46	42	4	NAV	2.8	2.2	0.03	521	97	73
PCB 101	46	42	4	NAV	7.6	5.8	0.07	450	107	70
PCB 138	45	43	2	NAV	7.3	7.4	0.12	641	111	74
PCB 153	46	44	2	NAV	12.2	10.5	0.12	210	117	75
PCB 180	46	42	4	NAV	4.1	3.5	0.03	2138	109	71
Sum Indicator PCB LB (ND = 0)	**47**	**45**	**2**	**NAV**	**34.0**	**30.2**	**0.00**	**3706**	**108**	**78**
Sum Indicator PCB UB (ND = LOD)	**44**	**44**	**0**	**NAV**	**35.1**	**29.4**	**0.38**	**3706**	**108**	**78**

Table 17: Summary of laboratory performance indicator PCB, fish

Fish A	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
PCB 28	31	0	0	0	0
PCB 52	31	0	0	0	0
PCB 101	31	0	0	0	0
PCB 138	30	0	0	0	0
PCB 153	31	0	0	0	0
PCB 180	31	0	0	0	0
Sum Indicator PCB LB (ND = 0)	**32**	**0**	**0**	**0**	**0**
Sum Indicator PCB UB (ND = LOD)	**30**	**0**	**0**	**0**	**0**

Table 18: Summary results indicator PCB, human milk (lipid weight basis) (ng/g)

Human milk	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
PCB 28	35	28	7	0.77	0.87	0.77	0.02	66.0	58	64
PCB 52	37	29	8	0.36	0.46	0.36	0.02	116	97	67
PCB 101	35	25	10	0.30	0.35	0.30	0.01	644	63	65
PCB 138	36	29	7	7.5	8.1	7.5	0.18	483	41	58
PCB 153	37	34	3	12.6	14.1	12.6	0.37	337	51	59
PCB 180	35	28	7	6.9	7.4	6.9	0.06	179	40	55
Sum Indicator PCB LB (ND = 0)	**37**	**35**	**2**	**26.8**	**30.1**	**26.8**	**0.00**	**1342**	**49**	**61**
Sum Indicator PCB UB (ND = LOD)	**34**	**34**	**0**	**27.8**	**30.3**	**27.8**	**0.81**	**1342**	**51**	**62**

Table 19: Summary of laboratory performance indicator PCB, human milk

Human milk	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
PCB 28	24	34	9	14	23
PCB 52	25	22	16	14	27
PCB 101	24	31	9	14	17
PCB 138	24	42	6	8	25
PCB 153	25	41	8	11	32
PCB 180	24	37	9	11	23
Sum Indicator PCB LB (ND = 0)	**25**	**41**	**3**	**19**	**32**
Sum Indicator PCB UB (ND = LOD)	**23**	**41**	**6**	**15**	**38**

Table 20: Summary results indicator PCB, air extract (TOL) (ng/g)

Air extract (TOL)	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
PCB 28	35	34	1	4.3	4.2	4.3	0.54	3108	32	66
PCB 52	36	36	0	4.0	4.2	4.0	0.41	2183	27	64
PCB 101	36	36	0	5.2	5.5	5.2	0.54	3787	20	61
PCB 138	35	35	0	3.8	3.9	3.8	0.47	2704	20	59
PCB 153	36	36	0	4.6	4.8	4.6	0.45	929	26	60
PCB 180	36	35	1	2.2	2.3	2.2	0.23	9984	34	65
Sum Indicator PCB LB (ND = 0)	**36**	**36**	**0**	**24.8**	**25.3**	**24.8**	**2.64**	**22695**	**21**	**62**
Sum Indicator PCB UB (ND = LOD)	**35**	**35**	**0**	**25.0**	**25.4**	**25.0**	**2.64**	**22695**	**20**	**64**

Table 21: Summary of laboratory performance indicator PCB, air extract (TOL)

Air extract (TOL)	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
PCB 28	24	51	11	23	11
PCB 52	24	53	14	14	19
PCB 101	24	64	6	14	17
PCB 138	24	60	6	11	23
PCB 153	24	56	6	19	19
PCB 180	24	50	8	19	19
Sum Indicator PCB LB (ND = 0)	**24**	**64**	**3**	**14**	**19**
Sum Indicator PCB UB (ND = LOD)	**24**	**66**	**3**	**11**	**20**

3.2.3 Dioxin-like POPs (PCDD/PCDF and dl-PCB)

Table 22: Summary results dl-POPs, test solutions T and U (ng/g)

Test Solution T and T	n									Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	Theoretical conc.	AV	Median	Mean	Min	Max	(%)	(%)
2,3,7,8-TeCDD	41	41	0	80.8	84.8	86.0	84.8	1.2	202	13	64
1,2,3,7,8-PnCDD	41	40	1	55.7	54.1	53.4	54.1	0.67	112	8	64
1,2,3,4,7,8-HxCDD	40	40	0	55.7	56.2	55.4	56.2	0.75	120	14	68
1,2,3,6,7,8-HxCDD	40	39	1	55.7	57.5	56.3	57.5	0.79	112	14	68
1,2,3,7,8,9-HxCDD	40	39	1	195	194	191	194	2.7	516	19	69
1,2,3,4,6,7,8-HpCDD	41	40	1	125	126	125	126	1.4	361	12	65
OCDD	41	40	1	111	111	111	111	0.06	287	13	65
2,3,7,8-TeCDF	41	40	1	11.1	11.1	10.9	11.1	0.16	16.4	14	67
1,2,3,7,8-PnCDF	41	41	0	55.7	53.5	53.3	53.5	0.70	106	11	70
2,3,4,7,8-PnCDF	41	40	1	195	202	198	202	2.7	478	13	66
1,2,3,4,7,8-HxCDF	40	39	1	55.7	55.9	54.5	55.9	0.74	113	14	69
1,2,3,6,7,8-HxCDF	40	39	1	55.7	55.4	55.0	55.4	0.74	112	13	70
1,2,3,7,8,9-HxCDF	41	40	1	195	181	181	181	2.4	404	28	60
2,3,4,6,7,8-HxCDF	41	41	0	55.7	54.5	55.1	54.5	0.74	250	18	59
1,2,3,4,6,7,8-HpCDF	41	40	1	55.7	53.3	52.2	53.3	0.58	89.2	11	62
1,2,3,4,7,8,9-HpCDF	41	40	1	125	125	122	125	1.5	284	12	68
OCDF	41	41	0	181	174	175	174	0.01	379	14	61
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	**41**	**41**	**0**	**268**	**276**	**274**	**276**	**3.6**	**618**	**10**	**63**
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	**40**	**40**	**0**	**268**	**276**	**274**	**276**	**3.6**	**618**	**9**	**64**
PCB 77	40	40	0	87.1	81.3	80.9	81.3	0.007	41700	27	67
PCB 81	40	39	1	17.4	16.0	16.1	16.0	0.002	6300	25	64
PCB 126	40	40	0	17.4	17.1	17.4	17.1	0.20	12400	22	63
PCB 169	40	39	1	87.1	87.2	85.4	87.2	0.97	78700	12	59
PCB 105	40	39	1	17.4	16.5	16.6	16.5	0.0005	10840	19	62
PCB 114	40	39	1	157	151	150	151	0.004	80000	11	57
PCB 118	40	39	1	105	99.7	99.5	99.7	0.003	25200	18	65
PCB 123	39	39	0	17.4	16.4	16.8	16.4	0.0005	80200	23	68
PCB 156	40	40	0	17.4	16.6	16.8	16.6	0.0005	109300	15	59
PCB 157	39	39	0	157	153	150	153	0.004	16100	17	61
PCB 167	39	38	1	17.4	16.1	16.3	16.1	0.0002	14000	18	61
PCB 189	38	38	0	17.4	16.0	15.8	16.0	0.0005	17600	18	66
WHO2005-TEQ (dl-PCB) LB (ND = 0)	**41**	**41**	**0**	**4.38**	**4.4**	**4.3**	**4.4**	**0.02**	**3618**	**16**	**61**
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	**38**	**38**	**0**	**4.38**	**4.3**	**4.3**	**4.3**	**0.05**	**3618**	**18**	**65**
WHO2005-TEQ (total) LB (ND = 0)	**36**	**36**	**0**	**272**	**282**	**279**	**282**	**3.7**	**623**	**9**	**63**
WHO2005-TEQ (total) UB (ND = LOD)	**35**	**35**	**0**	**272**	**281**	**279**	**281**	**3.7**	**623**	**9**	**63**

Table 23: Summary of laboratory performance dl-POPs, test solutions T and U

Test Solution T and U Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
2,3,7,8-TeCDD	28	76	2	10	12
1,2,3,7,8-PnCDD	28	83	2	2	10
1,2,3,4,7,8-HxCDD	27	78	10	0	13
1,2,3,6,7,8-HxCDD	27	78	8	0	13
1,2,3,7,8,9-HxCDD	27	73	8	5	13
1,2,3,4,6,7,8-HpCDD	28	76	7	0	15
OCDD	28	73	10	0	15
2,3,7,8-TeCDF	28	78	7	2	10
1,2,3,7,8-PnCDF	28	83	5	0	12
2,3,4,7,8-PnCDF	28	78	5	5	10
1,2,3,4,7,8-HxCDF	27	80	5	0	13
1,2,3,6,7,8-HxCDF	27	80	5	0	13
1,2,3,7,8,9-HxCDF	28	56	7	22	12
2,3,4,6,7,8-HxCDF	28	61	7	2	29
1,2,3,4,6,7,8-HpCDF	28	78	5	2	12
1,2,3,4,7,8,9-HpCDF	28	76	10	0	12
OCDF	28	66	12	5	17
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	**28**	**80**	**5**	**5**	**10**
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	**27**	**80**	**5**	**5**	**10**
PCB 77	27	58	18	10	15
PCB 81	27	60	10	10	18
PCB 126	27	63	10	13	15
PCB 169	27	68	5	13	13
PCB 105	27	68	5	10	15
PCB 114	27	70	8	5	15
PCB 118	27	70	8	5	15
PCB 123	26	67	10	10	13
PCB 156	27	63	8	10	20
PCB 157	26	62	10	8	21
PCB 167	26	62	10	8	18
PCB 189	26	66	11	8	16
WHO2005-TEQ (dl-PCB) LB (ND = 0)	**28**	**66**	**10**	**10**	**15**
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	**26**	**71**	**5**	**11**	**13**
WHO2005-TEQ (total) LB (ND = 0)	**24**	**81**	**3**	**6**	**11**
WHO2005-TEQ (total) UB (ND = LOD)	**24**	**80**	**3**	**6**	**11**

Table 24: Summary results dl-POPs, sediment (pg/g)

Sediment	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
2,3,7,8-TeCDD	35	35	0	4.0	4.1	4.0	1.6	19.6	25	74
1,2,3,7,8-PnCDD	35	33	2	1.1	1.2	1.1	0.56	15.5	35	68
1,2,3,4,7,8-HxCDD	34	32	2	1.3	1.4	1.3	0.11	10.8	41	72
1,2,3,6,7,8-HxCDD	34	34	0	3.3	3.4	3.3	0.28	10.5	25	64
1,2,3,7,8,9-HxCDD	34	34	0	2.5	2.5	2.5	0.21	8.7	32	71
1,2,3,4,6,7,8-HpCDD	35	34	1	51.8	52.3	51.8	0.40	199	27	66
OCDD	35	35	0	509	506	509	0.12	2181	29	69
2,3,7,8-TeCDF	35	35	0	8.5	8.2	8.5	0.85	128	17	71
1,2,3,7,8-PnCDF	35	35	0	7.0	7.1	7.0	0.24	15.8	22	73
2,3,4,7,8-PnCDF	35	34	1	7.3	7.5	7.3	2.4	281	26	72
1,2,3,4,7,8-HxCDF	34	34	0	24.5	24.8	24.5	2.3	47.3	20	70
1,2,3,6,7,8-HxCDF	34	34	0	9.3	9.3	9.3	0.83	17.8	25	68
1,2,3,7,8,9-HxCDF	35	33	2	NAV	2.1	2.0	0.08	57.2	110	68
2,3,4,6,7,8-HxCDF	35	35	0	5.3	5.7	5.3	0.36	18.6	56	78
1,2,3,4,6,7,8-HpCDF	35	35	0	73.8	74.6	73.8	0.59	159	25	61
1,2,3,4,7,8,9-HpCDF	35	35	0	10.4	10.6	10.4	0.09	216	20	63
OCDF	35	35	0	345	337	345	0.04	936	35	75
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	**36**	**35**	**1**	**14.9**	**15.1**	**14.9**	**0.00**	**145**	**21**	**69**
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	**35**	**35**	**0**	**15.1**	**15.6**	**15.1**	**6.3**	**15803**	**20**	**67**
PCB 77	26	25	1	410	399	410	0.04	535	17	60
PCB 81	26	23	3	6.2	6.7	6.2	0.0007	457	40	55
PCB 126	26	25	1	19.7	19.8	19.7	1.6	1012	25	60
PCB 169	26	22	4	3.5	3.5	3.5	0.04	56.2	31	58
PCB 105	27	27	0	905	877	905	0.03	1040	14	70
PCB 114	27	25	2	37.1	37.4	37.1	0.001	4055	62	67
PCB 118	27	27	0	3712	3566	3712	0.14	4384	13	63
PCB 123	26	23	3	43.8	48.2	43.8	0.002	520	60	56
PCB 156	27	27	0	755	737	755	0.02	1260	15	63
PCB 157	27	27	0	117	113	117	0.004	776	16	63
PCB 167	27	27	0	374	369	374	0.005	735	22	64
PCB 189	27	26	1	155	152	155	0.004	218	24	66
WHO2005-TEQ (dl-PCB) LB (ND = 0)	**28**	**27**	**1**	**2.2**	**2.2**	**2.2**	**0.00**	**102**	**32**	**61**
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	**27**	**27**	**0**	**2.3**	**2.3**	**2.3**	**0.17**	**653**	**28**	**58**
WHO2005-TEQ (total) LB (ND = 0)	**27**	**26**	**1**	**16.7**	**16.6**	**16.7**	**0.00**	**107**	**31**	**75**
WHO2005-TEQ (total) UB (ND = LOD)	**26**	**26**	**0**	**16.8**	**16.9**	**16.8**	**6.51**	**16456**	**31**	**72**

Table 25: Summary of laboratory performance dl-POPs, sediment

Sediment Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
2,3,7,8-TeCDD	24	67	17	11	3
1,2,3,7,8-PnCDD	24	56	11	14	11
1,2,3,4,7,8-HxCDD	24	46	14	20	11
1,2,3,6,7,8-HxCDD	24	54	11	23	9
1,2,3,7,8,9-HxCDD	24	57	11	20	9
1,2,3,4,6,7,8-HpCDD	24	56	14	11	14
OCDD	24	53	17	17	11
2,3,7,8-TeCDF	24	75	8	6	8
1,2,3,7,8-PnCDF	24	81	3	3	11
2,3,4,7,8-PnCDF	24	61	14	17	3
1,2,3,4,7,8-HxCDF	24	71	9	9	9
1,2,3,6,7,8-HxCDF	24	60	14	14	9
1,2,3,7,8,9-HxCDF	24	0	0	0	0
2,3,4,6,7,8-HxCDF	24	28	22	33	14
1,2,3,4,6,7,8-HpCDF	24	50	17	17	14
1,2,3,4,7,8,9-HpCDF	24	58	19	11	8
OCDF	24	50	19	17	11
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	**24**	**67**	**11**	**17**	**3**
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	**24**	**69**	**9**	**20**	**3**
PCB 77	18	63	7	11	11
PCB 81	18	37	7	15	26
PCB 126	18	56	7	11	19
PCB 169	18	44	4	15	19
PCB 105	19	75	4	11	7
PCB 114	19	39	4	18	29
PCB 118	19	68	7	7	14
PCB 123	18	30	11	11	33
PCB 156	19	68	0	14	14
PCB 157	19	68	0	11	18
PCB 167	19	61	7	14	14
PCB 189	19	57	7	21	7
WHO2005-TEQ (dl-PCB) LB (ND = 0)	**19**	**50**	**7**	**18**	**21**
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	**18**	**52**	**7**	**22**	**19**
WHO2005-TEQ (total) LB (ND = 0)	**18**	**67**	**0**	**26**	**4**
WHO2005-TEQ (total) UB (ND = LOD)	**18**	**65**	**4**	**23**	**8**

Table 26: Summary results dl-POPs, fish (product basis) (pg/g)

Fish A	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
2,3,7,8-TeCDD	38	31	7	NAV	0.24	0.27	0.004	0.56	96	73
1,2,3,7,8-PnCDD	38	16	22	0.02	0.03	0.02	0.003	0.75	89	48
1,2,3,4,7,8-HxCDD	37	8	29	NAV	0.02	0.01	0.000	0.10	219	45
1,2,3,6,7,8-HxCDD	37	11	26	NAV	0.02	0.01	0.000	0.11	131	48
1,2,3,7,8,9-HxCDD	38	10	28	NAV	0.03	0.01	0.000	0.40	259	43
1,2,3,4,6,7,8-HpCDD	38	21	17	0.02	0.03	0.02	0.0025	0.81	182	47
OCDD	38	27	11	0.07	0.09	0.07	0.0003	3.6	102	57
2,3,7,8-TeCDF	38	34	4	NAV	0.34	0.41	0.007	0.90	100	79
1,2,3,7,8-PnCDF	38	32	6	0.09	0.08	0.09	0.002	0.46	111	77
2,3,4,7,8-PnCDF	38	33	5	0.10	0.09	0.10	0.002	0.53	108	74
1,2,3,4,7,8-HxCDF	38	24	14	0.03	0.05	0.03	0.005	0.28	133	58
1,2,3,6,7,8-HxCDF	37	20	17	NAV	0.02	0.01	0.002	0.40	160	50
1,2,3,7,8,9-HxCDF	37	6	31	NAV	0.06	0.01	0.0000	0.31	119	45
2,3,4,6,7,8-HxCDF	37	15	22	0.01	0.03	0.01	0.0006	0.13	178	48
1,2,3,4,6,7,8-HpCDF	38	17	21	NAV	0.01	0.01	0.0000	1.1	197	47
1,2,3,4,7,8,9-HpCDF	37	9	28	NAV	0.04	0.01	0.0000	0.12	228	47
OCDF	38	16	22	0.02	0.05	0.02	0.0000	4.5	197	44
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	**38**	**36**	**2**	**NAV**	**0.30**	**0.35**	**0.000**	**0.99**	**110**	**83**
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	**36**	**36**	**0**	**NAV**	**0.37**	**0.41**	**0.007**	**1.4**	**99**	**82**
PCB 77	37	36	1	NAV	20.3	20.1	0.006	197	111	81
PCB 81	37	30	7	NAV	1.3	0.9	0.002	445	171	64
PCB 126	37	34	3	NAV	4.2	4.3	0.06	1739	110	73
PCB 169	37	27	10	NAV	0.59	0.56	0.008	64.1	113	69
PCB 105	37	36	1	NAV	369	431	0.04	1369	114	74
PCB 114	37	30	7	NAV	29.1	34.8	0.002	5832	115	63
PCB 118	37	36	1	NAV	1817	2035	0.21	10393	115	73
PCB 123	37	30	7	NAV	32.0	30.3	0.003	5056	125	63
PCB 156	37	36	1	NAV	258	238	0.03	1687	131	67
PCB 157	37	34	3	NAV	52.7	51.4	0.006	808	123	67
PCB 167	37	36	1	NAV	164	165	0.06	626	108	72
PCB 189	37	34	3	NAV	32.9	34.3	0.003	152	112	69
WHO2005-TEQ (dl-PCB) LB (ND = 0)	**37**	**37**	**0**	**NAV**	**0.41**	**0.46**	**0.009**	**174**	**116**	**70**
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	**37**	**37**	**0**	**NAV**	**0.54**	**0.55**	**0.009**	**177**	**115**	**74**
WHO2005-TEQ (total) LB (ND = 0)	**35**	**35**	**0**	**NAV**	**0.77**	**0.92**	**0.01**	**78.8**	**105**	**78**
WHO2005-TEQ (total) UB (ND = LOD)	**35**	**35**	**0**	**NAV**	**0.79**	**1.00**	**0.02**	**78.8**	**98**	**77**

Table 27: Summary of laboratory performance dl-POPs, fish

Fish A Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
2,3,7,8-TeCDD	26	0	0	0	0
1,2,3,7,8-PnCDD	26	32	0	5	5
1,2,3,4,7,8-HxCDD	25	0	0	0	0
1,2,3,6,7,8-HxCDD	25	0	0	0	0
1,2,3,7,8,9-HxCDD	26	0	0	0	0
1,2,3,4,6,7,8-HpCDD	26	29	3	3	21
OCDD	26	37	5	5	24
2,3,7,8-TeCDF	26	0	0	0	0
1,2,3,7,8-PnCDF	26	29	11	37	8
2,3,4,7,8-PnCDF	26	26	8	42	11
1,2,3,4,7,8-HxCDF	26	42	5	3	13
1,2,3,6,7,8-HxCDF	25	0	0	0	0
1,2,3,7,8,9-HxCDF	25	0	0	0	0
2,3,4,6,7,8-HxCDF	25	27	3	8	3
1,2,3,4,6,7,8-HpCDF	26	0	0	0	0
1,2,3,4,7,8,9-HpCDF	25	0	0	0	0
OCDF	26	21	3	5	13
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	**26**	**0**	**0**	**0**	**0**
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	**24**	**0**	**0**	**0**	**0**
PCB 77	25	0	0	0	0
PCB 81	25	0	0	0	0
PCB 126	25	0	0	0	0
PCB 169	25	0	0	0	0
PCB 105	25	0	0	0	0
PCB 114	25	0	0	0	0
PCB 118	25	0	0	0	0
PCB 123	25	0	0	0	0
PCB 156	25	0	0	0	0
PCB 157	25	0	0	0	0
PCB 167	25	0	0	0	0
PCB 189	25	0	0	0	0
WHO2005-TEQ (dl-PCB) LB (ND = 0)	**25**	**0**	**0**	**0**	**0**
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	**25**	**0**	**0**	**0**	**0**
WHO2005-TEQ (total) LB (ND = 0)	**24**	**0**	**0**	**0**	**0**
WHO2005-TEQ (total) UB (ND = LOD)	**24**	**0**	**0**	**0**	**0**

Table 28: Summary results dl-POPs, human milk (lipid weight basis) (pg/g)

Human milk	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
2,3,7,8-TeCDD	23	17	6	0.22	0.21	0.22	0.005	5.0	31	60
1,2,3,7,8-PnCDD	23	18	5	0.56	0.62	0.56	0.009	13.6	44	65
1,2,3,4,7,8-HxCDD	23	17	6	0.28	0.30	0.28	0.04	0.77	30	58
1,2,3,6,7,8-HxCDD	23	22	1	1.9	2.0	1.9	0.05	46.4	34	69
1,2,3,7,8,9-HxCDD	23	19	4	0.51	0.56	0.51	0.002	12.0	27	64
1,2,3,4,6,7,8-HpCDD	23	23	0	3.2	3.4	3.2	0.10	76.7	31	65
OCDD	23	23	0	20.4	21.6	20.4	0.45	594	28	65
2,3,7,8-TeCDF	23	18	5	0.37	0.41	0.37	0.02	11.2	38	56
1,2,3,7,8-PnCDF	23	18	5	0.23	0.27	0.23	0.005	3.9	75	64
2,3,4,7,8-PnCDF	23	22	1	1.7	1.7	1.7	0.05	42.0	26	64
1,2,3,4,7,8-HxCDF	23	22	1	0.68	0.69	0.68	0.01	15.3	24	70
1,2,3,6,7,8-HxCDF	23	21	2	0.62	0.65	0.62	0.01	14.1	23	65
1,2,3,7,8,9-HxCDF	23	10	13	NAV	0.19	0.12	0.001	1.0	149	58
2,3,4,6,7,8-HxCDF	22	18	4	0.44	0.44	0.44	0.001	9.5	30	63
1,2,3,4,6,7,8-HpCDF	23	22	1	1.1	1.0	1.1	0.02	23.5	31	67
1,2,3,4,7,8,9-HpCDF	22	12	10	0.07	0.09	0.07	0.005	1.1	115	64
OCDF	23	12	11	NAV	0.47	0.36	0.01	2.2	124	61
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	**23**	**23**	**0**	**1.7**	**1.7**	**1.7**	**0.03**	**43.4**	**41**	**72**
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	**22**	**22**	**0**	**2.0**	**1.9**	**2.0**	**0.04**	**44.0**	**35**	**65**
PCB 77	23	20	3	NAV	10.4	9.7	0.26	159	94	73
PCB 81	23	16	7	1.2	1.3	1.2	0.005	5.0	36	54
PCB 126	24	21	3	10.6	10.3	10.6	0.17	274	27	68
PCB 169	24	20	4	6.5	6.6	6.5	0.13	171	34	58
PCB 105	24	23	1	412	419	412	9.6	10590	15	65
PCB 114	24	22	2	97.3	93.4	97.3	2.4	2600	20	66
PCB 118	24	23	1	1826	1808	1826	49.7	50480	11	61
PCB 123	23	19	4	23.5	25.4	23.5	0.38	490	34	62
PCB 156	24	23	1	1299	1300	1299	33.7	33190	17	65
PCB 157	24	23	1	205	206	205	5.7	5410	18	62
PCB 167	24	23	1	286	290	286	7.5	7800	16	58
PCB 189	23	22	1	127	126	127	3.6	185	18	66
WHO2005-TEQ (dl-PCB) LB (ND = 0)	**25**	**23**	**2**	**1.4**	**1.4**	**1.4**	**0.00**	**35.9**	**26**	**62**
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	**23**	**23**	**0**	**1.4**	**1.4**	**1.4**	**0.07**	**36.2**	**25**	**63**
WHO2005-TEQ (total) LB (ND = 0)	**23**	**22**	**1**	**3.0**	**3.2**	**3.0**	**0.0**	**79.3**	**32**	**71**
WHO2005-TEQ (total) UB (ND = LOD)	**22**	**22**	**0**	**3.5**	**3.5**	**3.5**	**0.1**	**80.2**	**33**	**72**

Table 29: Summary of laboratory performance dl-POPs, human milk

Human milk Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
2,3,7,8-TeCDD	16	52	9	4	9
1,2,3,7,8-PnCDD	16	48	4	13	13
1,2,3,4,7,8-HxCDD	16	52	0	9	13
1,2,3,6,7,8-HxCDD	16	52	22	9	13
1,2,3,7,8,9-HxCDD	16	61	0	0	22
1,2,3,4,6,7,8-HpCDD	16	57	13	13	17
OCDD	16	61	13	0	26
2,3,7,8-TeCDF	16	43	13	4	17
1,2,3,7,8-PnCDF	16	26	17	13	22
2,3,4,7,8-PnCDF	16	61	13	9	13
1,2,3,4,7,8-HxCDF	16	78	4	4	9
1,2,3,6,7,8-HxCDF	16	70	4	9	9
1,2,3,7,8,9-HxCDF	16	0	0	0	0
2,3,4,6,7,8-HxCDF	15	50	9	9	14
1,2,3,4,6,7,8-HpCDF	16	57	17	9	13
1,2,3,4,7,8,9-HpCDF	15	23	5	9	18
OCDF	16	0	0	0	0
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	**16**	**48**	**17**	**17**	**17**
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	**15**	**50**	**14**	**18**	**18**
PCB 77	16	0	0	0	0
PCB 81	16	29	13	8	17
PCB 126	17	56	8	8	12
PCB 169	17	44	4	12	20
PCB 105	17	72	0	8	12
PCB 114	17	64	8	4	12
PCB 118	17	72	0	8	12
PCB 123	16	50	8	0	21
PCB 156	17	64	16	0	12
PCB 157	17	64	12	4	12
PCB 167	17	60	8	12	12
PCB 189	16	67	13	4	8
WHO2005-TEQ (dl-PCB) LB (ND = 0)	**17**	**56**	**8**	**4**	**24**
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	**16**	**61**	**9**	**4**	**26**
WHO2005-TEQ (total) LB (ND = 0)	**16**	**48**	**22**	**13**	**13**
WHO2005-TEQ (total) UB (ND = LOD)	**15**	**59**	**9**	**18**	**14**

Table 30: Summary results dl-POPs, air extract (TOL) (pg/g)

Air extract (TOL)	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
2,3,7,8-TeCDD	33	32	1	7.0	7.1	7.0	3.6	20.0	18	63
1,2,3,7,8-PnCDD	34	34	0	14.1	14.4	14.1	3.5	31.5	17	66
1,2,3,4,7,8-HxCDD	34	34	0	12.8	12.6	12.8	1.1	36.8	17	66
1,2,3,6,7,8-HxCDD	34	34	0	14.6	14.5	14.6	1.2	37.8	12	68
1,2,3,7,8,9-HxCDD	34	33	1	14.1	14.3	14.1	0.90	123	21	62
1,2,3,4,6,7,8-HpCDD	34	34	0	41.1	41.1	41.1	0.36	118	16	68
OCDD	34	33	1	63.0	63.4	63.0	0.02	161	16	61
2,3,7,8-TeCDF	34	34	0	9.3	9.1	9.3	0.74	33.8	21	67
1,2,3,7,8-PnCDF	34	34	0	16.3	16.0	16.3	0.40	82.9	16	61
2,3,4,7,8-PnCDF	34	34	0	15.9	16.0	15.9	4.0	62.3	18	66
1,2,3,4,7,8-HxCDF	34	34	0	15.4	15.2	15.4	1.3	44.1	16	67
1,2,3,6,7,8-HxCDF	34	34	0	15.7	15.3	15.7	1.3	43.3	15	70
1,2,3,7,8,9-HxCDF	34	33	1	13.2	13.8	13.2	0.62	43.6	35	69
2,3,4,6,7,8-HxCDF	34	34	0	15.0	15.1	15.0	1.3	96.2	17	68
1,2,3,4,6,7,8-HpCDF	34	33	1	32.4	32.8	32.4	0.25	109	14	61
1,2,3,4,7,8,9-HpCDF	34	34	0	27.1	27.6	27.1	0.22	104	15	66
OCDF	34	34	0	31.4	31.4	31.4	0.003	186	28	64
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	**34**	**34**	**0**	**39.1**	**38.7**	**39.1**	**17.1**	**113**	**16**	**62**
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	**33**	**33**	**0**	**39.2**	**39.0**	**39.2**	**17.4**	**113**	**16**	**61**
PCB 77	31	28	3	99.5	98.1	99.5	0.01	460	19	62
PCB 81	30	23	7	13.4	13.3	13.4	0.004	163	53	60
PCB 126	30	24	6	19.9	20.8	19.9	2.1	136	29	59
PCB 169	30	21	9	8.6	9.5	8.6	0.42	17.1	49	62
PCB 105	31	29	2	664	678	664	0.03	996	22	65
PCB 114	31	26	5	51.4	51.8	51.4	0.002	1493	30	58
PCB 118	30	28	2	1922	1900	1922	0.10	2475	12	61
PCB 123	30	25	5	38.8	40.5	38.8	0.002	264	35	56
PCB 156	31	28	3	171	170	171	0.01	245	19	61
PCB 157	30	25	5	38.0	38.0	38.0	0.002	193	18	51
PCB 167	30	28	2	85.8	84.9	85.8	0.01	340	27	66
PCB 189	30	24	6	18.5	17.8	18.5	0.001	22.8	16	56
WHO2005-TEQ (dl-PCB) LB (ND = 0)	**31**	**29**	**2**	**2.1**	**2.3**	**2.1**	**0.00**	**13.7**	**52**	**63**
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	**30**	**30**	**0**	**2.3**	**2.5**	**2.3**	**0.22**	**79.7**	**38**	**62**
WHO2005-TEQ (total) LB (ND = 0)	**29**	**29**	**0**	**39.6**	**39.0**	**39.6**	**0.11**	**63.5**	**24**	**71**
WHO2005-TEQ (total) UB (ND = LOD)	**28**	**28**	**0**	**40.3**	**39.1**	**40.3**	**0.11**	**63.5**	**24**	**70**

Table 31: Summary of laboratory performance dl-POPs, air extract (TOL)

Air extract (TOL) Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
2,3,7,8-TeCDD	22	67	9	15	6
1,2,3,7,8-PnCDD	23	76	3	15	6
1,2,3,4,7,8-HxCDD	23	74	3	15	9
1,2,3,6,7,8-HxCDD	23	82	0	9	9
1,2,3,7,8,9-HxCDD	23	59	15	12	12
1,2,3,4,6,7,8-HpCDD	23	74	6	9	12
OCDD	23	65	15	9	9
2,3,7,8-TeCDF	23	68	9	15	9
1,2,3,7,8-PnCDF	23	68	9	9	15
2,3,4,7,8-PnCDF	23	74	3	18	6
1,2,3,4,7,8-HxCDF	23	74	6	9	12
1,2,3,6,7,8-HxCDF	23	79	3	9	9
1,2,3,7,8,9-HxCDF	23	53	12	21	12
2,3,4,6,7,8-HxCDF	23	76	3	9	12
1,2,3,4,6,7,8-HpCDF	23	74	3	3	18
1,2,3,4,7,8,9-HpCDF	23	76	3	9	12
OCDF	23	59	6	18	18
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	**23**	**71**	**9**	**18**	**3**
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	**22**	**70**	**6**	**21**	**3**
PCB 77	21	61	6	10	13
PCB 81	20	30	10	10	27
PCB 126	20	43	10	10	17
PCB 169	20	27	7	20	17
PCB 105	21	65	3	16	10
PCB 114	21	45	10	16	13
PCB 118	21	68	3	13	6
PCB 123	20	33	17	10	23
PCB 156	21	55	10	16	10
PCB 157	20	47	10	13	13
PCB 167	20	57	10	7	20
PCB 189	20	60	3	10	7
WHO2005-TEQ (dl-PCB) LB (ND = 0)	**21**	**29**	**16**	**16**	**32**
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	**20**	**50**	**3**	**23**	**23**
WHO2005-TEQ (total) LB (ND = 0)	**20**	**66**	**7**	**24**	**3**
WHO2005-TEQ (total) UB (ND = LOD)	**19**	**64**	**7**	**25**	**4**

3.2.4 Polybrominated Diphenyl Ethers (PBDE)

Table 32: Summary results PBDE, test solution V (ng/g)

Test Solution V	n									Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	Theoretical conc.	AV	Median	Mean	Min	Max	(%)	(%)
BDE 17	19	19	0	200	210	208	210	60.3	260	7	56
BDE 28	26	25	1	37.9	37.7	37.3	37.7	3.7	155	13	64
BDE 47	27	27	0	129	124	123	124	14.1	166	19	69
BDE 99	28	28	0	233	224	224	224	43.1	290	23	65
BDE 100	28	28	0	57.0	56.9	56.0	56.9	7.6	225	11	61
BDE 153	27	27	0	109	105	106	105	24.4	275	17	55
BDE 154	26	26	0	168	140	142	140	33.2	184	25	75
BDE 183	27	27	0	43.4	39.2	40.0	39.2	19.2	62	25	74
BDE 209	20	20	0	592	464	516	464	18.7	709	50	79
Sum PBDE LB (ND = 0)	**28**	**28**	**0**	**1569**	**NAV**	**1164**	**1125**	**148**	**1862**	**50**	**79**
Sum PBDE UB (ND = LOD)	**17**	**17**	**0**	**1569**	**1492**	**1495**	**1492**	**695**	**1862**	**18**	**69**
PBB 153	10	10	0	73.8	68.5	71.1	68.5	44.0	101	26	77

Table 33: Summary of laboratory performance PBDE, test solution V

Test Solution V	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
BDE 17	13	89	0	11	0
BDE 28	18	69	4	15	8
BDE 47	18	70	7	19	4
BDE 99	19	64	7	25	4
BDE 100	19	71	4	14	11
BDE 153	18	59	7	22	11
BDE 154	18	62	23	12	4
BDE 183	18	63	15	22	0
BDE 209	14	40	15	35	10
Sum PBDE LB (ND = 0)	**19**	**0**	**0**	**0**	**0**
Sum PBDE UB (ND = LOD)	**11**	**76**	**6**	**18**	**0**
PBB 153	7	80	10	10	0

Table 34: Summary results PBDE, sediment (ng/g)

Sediment	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
BDE 17	16	14	2	0.11	0.11	0.11	0.02	0.32	12	65
BDE 28	20	19	1	0.14	0.15	0.14	0.011	236	12	62
BDE 47	21	20	1	1.6	1.7	1.6	0.12	3650	18	64
BDE 99	22	21	1	1.6	1.7	1.6	0.12	3508	44	63
BDE 100	22	21	1	0.42	0.44	0.42	0.03	797	32	58
BDE 153	21	20	1	0.31	0.32	0.31	0.03	574	18	58
BDE 154	20	18	2	0.21	0.22	0.21	0.01	367	31	64
BDE 183	21	18	3	0.15	0.17	0.15	0.04	414	28	60
BDE 209	15	15	0	65.4	63.3	65.4	3.03	134	15	69
Sum PBDE LB (ND = 0)	**22**	**22**	**0**	**56.7**	**64.4**	**56.7**	**0.36**	**9546**	**53**	**61**
Sum PBDE UB (ND = LOD)	**14**	**14**	**0**	**70.8**	**68.9**	**70.8**	**6.5**	**297**	**15**	**68**
PBB 153	12	8	4	0.03	0.03	0.03	0.02	63.7	20	55

Table 35: Summary of laboratory performance PBDE, sediment

Sediment Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
BDE 17	11	69	0	13	6
BDE 28	14	70	5	10	10
BDE 47	14	67	5	10	14
BDE 99	15	45	9	14	27
BDE 100	15	55	5	5	32
BDE 153	14	62	5	14	14
BDE 154	14	60	5	5	20
BDE 183	14	57	0	10	19
BDE 209	10	80	0	7	13
Sum PBDE LB (ND = 0)	**15**	**32**	**9**	**18**	**41**
Sum PBDE UB (ND = LOD)	**9**	**79**	**0**	**7**	**14**
PBB 153	8	58	0	0	8

Table 36: Summary results PBDE, fish (product basis) (ng/g)

Fish A Analyte	n Total	Numerical	LCV	AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
BDE 17	20	14	6	NAV	0.005	0.004	0.0007	0.04	98	62
BDE 28	24	20	4	0.02	0.02	0.02	0.002	0.09	97	77
BDE 47	25	24	1	NAV	0.64	0.68	0.08	157	104	81
BDE 99	26	24	2	NAV	0.26	0.26	0.03	48	109	76
BDE 100	26	25	1	NAV	0.24	0.25	0.03	56	109	77
BDE 153	25	23	2	0.06	0.05	0.06	0.008	10.7	99	73
BDE 154	24	21	3	0.06	0.06	0.06	0.007	12.8	108	74
BDE 183	24	11	13	NAV	0.0010	0.0008	0.0002	0.10	86	69
BDE 209	19	10	9	0.03	0.04	0.03	0.004	0.45	155	57
Sum PBDE LB (ND = 0)	**26**	**25**	**1**	**NAV**	**1.4**	**1.5**	**0.00**	**284**	**103**	**81**
Sum PBDE UB (ND = LOD)	**17**	**17**	**0**	**NAV**	**1.3**	**1.6**	**0.24**	**3.5**	**85**	**82**
PBB 153	13	7	6	0.02	0.02	0.02	0.008	0.03	63	68

Table 37: Summary of laboratory performance PBDE, fish

Fish A Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
BDE 17	14	0	0	0	0
BDE 28	16	79	0	4	0
BDE 47	17	0	0	0	0
BDE 99	18	0	0	0	0
BDE 100	18	0	0	0	0
BDE 153	17	44	24	16	8
BDE 154	16	38	21	25	4
BDE 183	16	0	0	0	0
BDE 209	13	32	0	5	16
Sum PBDE LB (ND = 0)	**18**	**0**	**0**	**0**	**0**
Sum PBDE UB (ND = LOD)	**11**	**0**	**0**	**0**	**0**
PBB 153	9	54	0	0	0

Table 38: Summary results PBDE, human milk (lipid weight basis) (ng/g)

Human milk	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
BDE 17	6	2	4	NAV	NAV	NAV	0.01	0.05	NAV	NAV
BDE 28	13	10	3	0.02	0.03	0.02	0.01	13.3	74	60
BDE 47	13	11	2	0.30	0.32	0.30	0.25	7.5	18	58
BDE 99	13	11	2	0.08	0.08	0.08	0.06	1.7	27	75
BDE 100	13	12	1	0.06	0.06	0.06	0.05	1.0	18	66
BDE 153	13	11	2	0.25	0.25	0.25	0.03	4.5	17	58
BDE 154	13	6	7	NAV	0.007	0.007	0.005	0.04	8	60
BDE 183	13	7	6	NAV	0.014	0.010	0.002	0.26	75	76
BDE 209	10	6	4	NAV	0.93	0.58	0.005	10.7	146	61
Sum PBDE LB (ND = 0)	**13**	**13**	**0**	**0.9**	**1.0**	**0.9**	**0.48**	**26**	**52**	**64**
Sum PBDE UB (ND = LOD)	**6**	**6**	**0**	**1.5**	**1.6**	**1.5**	**1.2**	**26**	**33**	**75**
PBB 153	6	2	4	NAV	NAV	NAV	0.03	0.49	NAV	NAV

Table 39: Summary of laboratory performance PBDE, human milk

Human milk	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
BDE 17	4	0	0	0	0
BDE 28	9	46	15	0	15
BDE 47	9	62	0	15	8
BDE 99	9	69	0	8	8
BDE 100	9	69	0	15	8
BDE 153	9	62	8	8	8
BDE 154	9	0	0	0	0
BDE 183	9	0	0	0	0
BDE 209	7	0	0	0	0
Sum PBDE LB (ND = 0)	**9**	**46**	**0**	**23**	**31**
Sum PBDE UB (ND = LOD)	**4**	**67**	**17**	**0**	**17**
PBB 153	4	0	0	0	0

Table 40: Summary results PBDE, air extract (TOL) (ng/g)

Air extract (TOL)	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
BDE 17	14	14	0	0.43	0.43	0.43	0.25	0.53	18	74
BDE 28	19	19	0	0.87	0.86	0.87	0.07	1.4	17	71
BDE 47	21	19	2	2.5	2.5	2.5	0.20	4.8	9	59
BDE 99	22	22	0	3.5	3.4	3.5	0.28	5.8	16	68
BDE 100	22	22	0	1.3	1.4	1.3	0.09	4.3	19	66
BDE 153	20	20	0	0.84	0.83	0.84	0.07	1.8	17	68
BDE 154	20	19	1	0.82	0.81	0.82	0.07	1.4	14	70
BDE 183	21	18	3	0.62	0.62	0.62	0.05	0.85	17	62
BDE 209	16	12	4	0.81	0.90	0.81	0.05	6.0	52	58
Sum PBDE LB (ND = 0)	**22**	**22**	**0**	**11.1**	**11.0**	**11.1**	**0.83**	**18.8**	**20**	**69**
Sum PBDE UB (ND = LOD)	**14**	**14**	**0**	**12.2**	**12.3**	**12.2**	**7.8**	**124**	**19**	**69**
PBB 153	9	9	0	1.2	1.2	1.2	0.44	1.3	4	55

Table 41: Summary of laboratory performance PBDE, air extract (TOL)

Air extract (Tol)	**% of the data received**	**% of z-scores |z|<2**	**% of z-scores 3>|z|>2**	**% of z-scores 6>|z|>3**	**% of z-scores |z|>6**
Analyte		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
BDE 17	9	86	14	0	0
BDE 28	13	79	5	11	5
BDE 47	14	67	10	5	10
BDE 99	15	73	9	14	5
BDE 100	15	73	0	9	18
BDE 153	14	75	5	10	10
BDE 154	14	75	10	5	5
BDE 183	14	67	10	5	5
BDE 209	11	38	6	0	31
Sum PBDE LB (ND = 0)	**15**	**68**	**14**	**14**	**5**
Sum PBDE UB (ND = LOD)	**9**	**71**	**7**	**14**	**7**
PBB 153	6	89	0	11	0

3.2.5 Toxaphenes

Table 42: Summary results toxaphenes, test solution AA (ng/g)

Test Solution AA	n									Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	Theoretical conc.	AV	Median	Mean	Min	Max	(%)	(%)
Parlar 26	10	10	0	40.7	39.7	38.8	39.7	31.9	898	13	75
Parlar 50	10	10	0	56.4	53.8	53.7	53.8	11.1	1163	10	67
Parlar 62	10	10	0	101	101	101	101	8.3	1906	12	68
Sum toxaphenes LB (ND = 0)	**10**	**10**	**0**	**198**	**195**	**195**	**195**	**56.8**	**3967**	**11**	**70**
Sum toxaphenes UB (ND = LOD)	**10**	**10**	**0**	**198**	**195**	**195**	**195**	**56.8**	**3967**	**11**	**70**

Table 43: Summary of laboratory performance toxaphenes, test solution AA

Test Solution AA	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
Parlar 26	7	90	0	0	10
Parlar 50	7	80	0	0	20
Parlar 62	7	80	0	0	20
Sum toxaphenes LB (ND = 0)	**7**	**80**	**0**	**10**	**10**
Sum toxaphenes UB (ND = LOD)	**7**	**80**	**0**	**10**	**10**

Table 44: Summary results toxaphenes, sediment (ng/g)

Sediment	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
Parlar 26	7	2	5	NAV	NAV	NAV	0.61	1.4	NAV	NAV
Parlar 50	7	1	6	NAV	NAV	NAV	1.0	1.0	NAV	NAV
Parlar 62	7	1	6	NAV	NAV	NAV	0.08	0.08	NAV	NAV
Sum toxaphenes LB (ND = 0)	**7**	**2**	**5**	**NAV**	**NAV**	**NAV**	**0.000**	**2.4**	**NAV**	**NAV**
Sum toxaphenes UB (ND = LOD)	**7**	**7**	**0**	**NAV**	**0.70**	**0.55**	**0.002**	**3.4**	**148**	**70**

Table 45: Summary of laboratory performance toxaphenes, sediment

Sediment	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
Parlar 26	5	0	0	0	0
Parlar 50	5	0	0	0	0
Parlar 62	5	0	0	0	0
Sum toxaphenes LB (ND = 0)	**5**	**0**	**0**	**0**	**0**
Sum toxaphenes UB (ND = LOD)	**5**	**0**	**0**	**0**	**0**

Table 46: Summary results toxaphenes, fish (product basis) (ng/g)

Fish (toxaphene)	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
Parlar 26	9	6	3	NAV	0.72	0.62	0.27	13.2	38	50
Parlar 50	9	6	3	NAV	0.86	0.63	0.12	15.0	71	53
Parlar 62	9	5	4	NAV	0.79	0.50	0.06	11.6	64	46
Sum toxaphenes LB (ND = 0)	**9**	**6**	**3**	**NAV**	**2.2**	**2.1**	**0.00**	**39.9**	**23**	**61**
Sum toxaphenes UB (ND = LOD)	**9**	**9**	**0**	**1.9**	**2.3**	**1.9**	**0.05**	**39.9**	**44**	**66**

Table 47: Summary of laboratory performance toxaphenes, fish

Fish (toxaphene) Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
Parlar 26	6	0	0	0	0
Parlar 50	6	0	0	0	0
Parlar 62	6	0	0	0	0
Sum toxaphenes LB (ND = 0)	**6**	**0**	**0**	**0**	**0**
Sum toxaphenes UB (ND = LOD)	**6**	**44**	**22**	**11**	**22**

Table 48: Summary results toxaphenes, human milk (lipid weight basis) (ng/g)

Human milk	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
Parlar 26	5	4	1	NAV	0.25	0.25	0.10	0.45	49	73
Parlar 50	5	4	1	NAV	0.49	0.52	0.10	0.56	13	67
Parlar 62	5	0	5	NAV	NAV	NAV	0.00	0.00	NAV	NAV
Sum toxaphenes LB (ND = 0)	**5**	**4**	**1**	**NAV**	**0.76**	**0.81**	**0.00**	**0.98**	**25**	**68**
Sum toxaphenes UB (ND = LOD)	**5**	**5**	**0**	**NAV**	**1.5**	**1.7**	**0.30**	**5.7**	**103**	**75**

Table 49: Summary of laboratory performance toxaphenes, human milk

Human milk Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
Parlar 26	3	0	0	0	0
Parlar 50	3	0	0	0	0
Parlar 62	3	0	0	0	0
Sum toxaphenes LB (ND = 0)	**3**	**0**	**0**	**0**	**0**
Sum toxaphenes UB (ND = LOD)	**3**	**0**	**0**	**0**	**0**

Table 50: Summary results toxaphenes, air extract (TOL) (ng/g)

Air extract (TOL)	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
Parlar 26	7	2	5	NAV	NAV	NAV	0.21	2.1	NAV	NAV
Parlar 50	7	1	6	NAV	NAV	NAV	0.40	0.40	NAV	NAV
Parlar 62	7	0	7	NAV	NAV	NAV	0.00	0.00	NAV	NAV
Sum toxaphenes LB (ND = 0)	**7**	**2**	**5**	**NAV**	**NAV**	**NAV**	**0.00**	**2.5**	**NAV**	**NAV**
Sum toxaphenes UB (ND = LOD)	**7**	**7**	**0**	**NAV**	**2.6**	**2.3**	**1.09**	**79.2**	**48**	**64**

Table 51: Summary of laboratory performance toxaphenes, air extract (TOL)

Air extract (TOL) Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
Parlar 26	5	0	0	0	0
Parlar 50	5	0	0	0	0
Parlar 62	5	0	0	0	0
Sum toxaphenes LB (ND = 0)	**5**	**0**	**0**	**0**	**0**
Sum toxaphenes UB (ND = LOD)	**5**	**0**	**0**	**0**	**0**

3.2.6 Hexabromocylcododecane (HBCD)

Table 52: Summary results HBCD, test solution X (ng/g)

Test Solution X	n									Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	Theoretical conc.	AV	Median	Mean	Min	Max	(%)	(%)
α-HBCD	13	13	0	865	775	750	775	587	923	13	77
β-HBCD	13	13	0	1153	1053	1069	1053	618	1280	18	78
γ-HBCD	13	13	0	288	293	290	293	181	437	8	53
Sum HBCD LB (ND = 0)	13	13	0	2307	2102	2120	2102	1642	2450	16	81
Sum HBCD UB (ND = LOD)	13	13	0	2307	2102	2120	2102	1642	2450	16	81

Table 53: Summary of laboratory performance HBCD, test solution X

Test Solution X	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
α-HBCD	9	100	0	0	0
β-HBCD	9	92	0	8	0
γ-HBCD	9	69	8	23	0
Sum HBCD LB (ND = 0)	9	100	0	0	0
Sum HBCD UB (ND = LOD)	9	100	0	0	0

Table 54: Summary results HBCD, sediment (ng/g)

Sediment	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
α-HBCD	8	6	2	NAV	11.3	10.8	7.9	31.4	37	54
β-HBCD	8	6	2	NAV	3.7	3.6	3.2	11.4	11	46
γ-HBCD	8	6	2	NAV	39.4	38.6	27.8	246	25	52
Sum HBCD LB (ND = 0)	8	6	2	NAV	54.2	52.6	0.000	289	26	68
Sum HBCD UB (ND = LOD)	8	8	0	NAV	50.2	44.9	0.006	289	59	65

Table 55: Summary of laboratory performance HBCD, sediment

Sediment	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
α-HBCD	5	0	0	0	0
β-HBCD	5	0	0	0	0
γ-HBCD	5	0	0	0	0
Sum HBCD LB (ND = 0)	5	0	0	0	0
Sum HBCD UB (ND = LOD)	5	0	0	0	0

Table 56: Summary results HBCD, fish (product basis) (ng/g)

Fish A	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
α-HBCD	9	7	2	0.03	0.03	0.03	0.02	0.10	58	74
β-HBCD	9	2	7	NAV	NAV	NAV	0.004	0.05	NAV	NAV
γ-HBCD	9	3	6	NAV	0.06	0.009	0.008	0.06	84	44
Sum HBCD LB (ND = 0)	9	7	2	0.05	0.05	0.05	0.00	0.15	37	72
Sum HBCD UB (ND = LOD)	9	9	0	0.08	0.09	0.08	0.05	0.47	69	67

Table 57: Summary of laboratory performance HBCD, fish

Fish A Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
α-HBCD	6	67	0	11	0
β-HBCD	6	0	0	0	0
γ-HBCD	6	0	0	0	0
Sum HBCD LB (ND = 0)	6	67	0	11	0
Sum HBCD UB (ND = LOD)	6	67	0	11	22

Table 58: Summary results HBCD, human milk (lipid weight basis) (ng/g)

Human milk	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
α-HBCD	4	1	3	NAV	NAV	NAV	0.76	0.76	NAV	NAV
β-HBCD	3	0	3	NAV	NAV	NAV	0.00	0.00	NAV	NAV
γ-HBCD	3	0	3	NAV	NAV	NAV	0.00	0.00	NAV	NAV
Sum HBCD LB (ND = 0)	**4**	**1**	**3**	**NAV**	**NAV**	**NAV**	**0.00**	**0.76**	**NAV**	**NAV**
Sum HBCD UB (ND = LOD)	**3**	**3**	**0**	**NAV**	**0.52**	**0.55**	**0.30**	**0.90**	**57**	**82**

Table 59: Summary of laboratory performance HBCD, human milk

Human milk Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
α-HBCD	3	0	0	0	0
β-HBCD	2	0	0	0	0
γ-HBCD	2	0	0	0	0
Sum HBCD LB (ND = 0)	**3**	**0**	**0**	**0**	**0**
Sum HBCD UB (ND = LOD)	**2**	**0**	**0**	**0**	**0**

Table 60: Summary results HBCD, air extract (TOL) (ng/g)

Air extract (TOL)	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
α-HBCD	6	4	2	82.7	82.4	82.7	74.0	93.1	11	54
β-HBCD	6	4	2	NAV	25.3	24.1	22.5	59.0	14	44
γ-HBCD	6	4	2	103	104	103	95.0	128	12	47
Sum HBCD LB (ND = 0)	**6**	**4**	**2**	**223**	**222**	**223**	**0.00**	**243**	**8**	**80**
Sum HBCD UB (ND = LOD)	**6**	**6**	**0**	**NAV**	**209**	**223**	**0.60**	**243**	**15**	**63**

Table 61: Summary of laboratory performance HBCD, air extract (TOL)

Air extract (TOL) Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
α-HBCD	4	67	0	0	0
β-HBCD	4	0	0	0	0
γ-HBCD	4	67	0	0	0
Sum HBCD LB (ND = 0)	**4**	**67**	**0**	**0**	**0**
Sum HBCD UB (ND = LOD)	**4**	**0**	**0**	**0**	**0**

3.2.7 Perfluoroalkyl Substances (PFAS)

Table 62: Summary results PFAS, test solution W (ng/g)

Test Solution W	n									Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	Theoretical conc.	AV	Median	Mean	Min	Max	(%)	(%)
L-PFOS anion	28	28	0	58.7	61.4	60.8	61.4	31.8	103	16	69
br-PFOS anion	19	18	1	15.8	12.3	12.7	12.3	7.4	23.4	32	71
tot-PFOS LB (ND = 0)	**29**	**29**	**0**	**74.5**	**69.7**	**70.0**	**69.7**	**40.9**	**103**	**18**	**72**
tot-PFOS UB (ND = LOD)	**20**	**20**	**0**	**74.5**	**68.8**	**68.8**	**68.8**	**40.9**	**99.0**	**18**	**71**
FOSA	20	20	0	63.2	59.2	62.5	59.2	38.0	203	17	73
MeFOSA	14	14	0	126	126	126	126	51.5	179	8	66
EtFOSA	14	14	0	190	183	189	183	98.0	261	16	71
MeFOSE	13	13	0	126	128	128	128	72.0	154	7	54
EtFOSE	13	13	0	126	132	130	132	69.8	156	11	61
PFOS precursors LB (ND = 0)	**20**	**20**	**0**	**631.2**	**NAV**	**497**	**492**	**49.0**	**732**	**49**	**74**
PFOS precursors UB (ND = LOD)	**12**	**12**	**0**	**631.2**	**650**	**644**	**650**	**368**	**732**	**4**	**61**
PFBA	24	24	0	63.2	61.4	62.5	61.4	33.7	94.0	11	69
PFPeA	25	25	0	63.2	60.7	61.0	60.7	43.9	87.6	12	69
PFHxA	28	28	0	94.8	92.9	92.3	92.9	62.7	114	15	78
PFHpA	28	28	0	63.2	61.8	61.0	61.8	33.8	81.5	15	77
PFOA	29	29	0	63.2	60.6	61.0	60.6	32.0	111	12	67
PFNA	28	28	0	126	118	116	118	60.4	150	15	81
PFDA	28	28	0	63.2	62.4	61.1	62.4	38.0	74.2	10	72
PFUnDA	28	28	0	63.2	61.4	61.1	61.4	38.8	101	16	76
PFDoDA	28	28	0	190	177	178	177	95.5	249	13	66
PFTrDA	27	27	0	63.2	64.8	65.0	64.8	24.1	102	19	70
PFTeDA	27	27	0	63.2	63.4	64.2	63.4	40.0	85.0	12	72
L-PFBS	27	27	0	83.9	84.8	83.9	84.8	51.5	118	17	79
L-PFHxS	28	28	0	59.8	60.3	61.4	60.3	33.1	99.7	12	62
L-PFDS	26	26	0	60.9	63.7	62.9	63.7	40.3	83.5	15	70
6:2 FTSA	14	14	0	63.2	53.3	56.9	53.3	39.4	115.5	22	76
PFCAs + PFSAs LB (ND = 0)	**29**	**29**	**0**	**1184**	**1113**	**1126**	**1113**	**98.0**	**1422**	**11**	**67**
PFCAs + PFSAs UB (ND = LOD)	**12**	**12**	**0**	**1184**	**1145**	**1138**	**1145**	**830**	**1375**	**11**	**73**

Table 63: Summary of laboratory performance PFAS, test solution W

Test Solution W **Analyte**	**% of the data received**	**% of z-scores |z|<2 Satisfactory**	**% of z-scores 3>|z|>2 Questionable**	**% of z-scores 6>|z|>3 Unsatisfactory**	**% of z-scores |z|>6 Extreme**
L-PFOS anion	19	75	11	14	0
br-PFOS anion	13	58	16	11	11
tot-PFOS LB (ND = 0)	**20**	**79**	**3**	**17**	**0**
tot-PFOS UB (ND = LOD)	**14**	**80**	**5**	**15**	**0**
FOSA	14	80	10	0	10
MeFOSA	9	86	0	14	0
EtFOSA	9	79	7	14	0
MeFOSE	9	77	15	8	0
EtFOSE	9	85	8	8	0
PFOS precursors LB (ND = 0)	**14**	**0**	**0**	**0**	**0**
PFOS precursors UB (ND = LOD)	**8**	**83**	**8**	**8**	**0**
PFBA	16	83	8	8	0
PFPeA	17	80	16	4	0
PFHxA	19	93	7	0	0
PFHpA	19	86	11	4	0
PFOA	20	83	7	7	3
PFNA	19	89	7	4	0
PFDA	19	93	4	4	0
PFUnDA	19	86	11	4	0
PFDoDA	19	75	18	7	0
PFTrDA	18	74	11	15	0
PFTeDA	18	89	11	0	0
L-PFBS	18	89	4	7	0
L-PFHxS	19	79	11	11	0
L-PFDS	18	81	19	0	0
6:2 FTSA	9	79	14	0	7
PFCAs + PFSAs LB (ND = 0)	**20**	**86**	**10**	**0**	**3**
PFCAs + PFSAs UB (ND = LOD)	**8**	**92**	**8**	**0**	**0**

Table 64: Summary results PFAS, sediment (ng/g)

Sediment	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
L-PFOS anion	12	12	0	3.8	3.8	3.8	2.3	8.0	23	71
br-PFOS anion	7	5	2	0.51	0.55	0.51	0.39	0.93	45	68
tot-PFOS LB (ND = 0)	**13**	**13**	**0**	**4.0**	**4.3**	**4.0**	**2.3**	**8.0**	**32**	**80**
tot-PFOS UB (ND = LOD)	**9**	**9**	**0**	**4.1**	**4.3**	**4.1**	**2.3**	**5.9**	**30**	**81**
PFBA	10	3	7	NAV	0.18	0.04	0.11	0.28	157	51
PFPeA	11	2	9	NAV	NAV	NAV	0.09	0.13	NAV	NAV
PFHxA	11	8	3	0.20	0.21	0.20	0.17	1.5	26	65
PFHpA	11	4	7	0.05	0.05	0.05	0.04	0.07	17	58
PFOA	12	11	1	0.47	0.52	0.47	0.27	4.3	23	59
PFNA	11	6	5	0.08	0.08	0.08	0.06	0.12	34	77
PFDA	11	8	3	0.30	0.32	0.30	0.17	6.6	53	68
PFUnDA	11	8	3	0.30	0.27	0.30	0.18	0.47	41	79
PFDoDA	11	7	4	0.32	0.32	0.32	0.27	0.38	17	73
PFTrDA	11	6	5	0.10	0.10	0.10	0.08	0.15	21	59
PFTeDA	11	4	7	0.07	0.09	0.07	0.04	0.11	45	54
L-PFBS	11	7	4	0.13	0.17	0.13	0.07	2.4	51	66
L-PFHxS	11	7	4	0.08	0.09	0.08	0.05	0.94	20	51
L-PFDS	10	3	7	NAV	0.09	0.09	0.09	0.26	5	38
6:2 FTSA	8	5	3	0.69	0.69	0.69	0.44	0.87	2	39
PFCAs + PFSAs LB (ND = 0)	**12**	**11**	**1**	**2.1**	**2.2**	**2.1**	**0.00**	**15.8**	**57**	**74**
PFCAs + PFSAs UB (ND = LOD)	**7**	**7**	**0**	**3.4**	**3.9**	**3.4**	**2.3**	**19.0**	**40**	**67**

Table 65: Summary of laboratory performance PFAS, sediment

Sediment	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
L-PFOS anion	8	67	17	8	8
br-PFOS anion	5	57	0	14	0
tot-PFOS LB (ND = 0)	**9**	**77**	**0**	**15**	**8**
tot-PFOS UB (ND = LOD)	**6**	**67**	**11**	**22**	**0**
PFBA	7	0	0	0	0
PFPeA	7	0	0	0	0
PFHxA	7	45	9	9	9
PFHpA	7	36	0	0	0
PFOA	8	50	17	17	8
PFNA	7	55	0	0	0
PFDA	7	36	9	18	9
PFUnDA	7	36	27	9	0
PFDoDA	7	64	0	0	0
PFTrDA	7	45	9	0	0
PFTeDA	7	36	0	0	0
L-PFBS	7	36	18	0	9
L-PFHxS	7	55	0	0	9
L-PFDS	7	0	0	0	0
6:2 FTSA	5	50	13	0	0
PFCAs + PFSAs LB (ND = 0)	**8**	**42**	**8**	**25**	**17**
PFCAs + PFSAs UB (ND = LOD)	**5**	**57**	**14**	**0**	**29**

Table 66: Summary results PFAS, fish (product basis) (ng/g)

Fish A	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
L-PFOS anion	21	21	0	8.5	8.6	8.5	6.6	48.6	11	71
br-PFOS anion	13	12	1	0.52	0.53	0.52	0.00	0.68	32	78
tot-PFOS LB (ND = 0)	**25**	**25**	**0**	**8.7**	**8.9**	**8.7**	**6.8**	**48.6**	**14**	**70**
tot-PFOS UB (ND = LOD)	**16**	**16**	**0**	**8.7**	**8.8**	**8.7**	**6.8**	**24.5**	**16**	**76**
PFBA	15	1	14	NAV	NAV	NAV	1.4	1.4	NAV	NAV
PFPeA	16	0	16	NAV	NAV	NAV	0.00	0.00	NAV	NAV
PFHxA	19	1	18	NAV	NAV	NAV	0.27	0.27	NAV	NAV
PFHpA	19	1	18	NAV	NAV	NAV	0.30	0.30	NAV	NAV
PFOA	22	6	16	NAV	0.06	0.02	0.01	0.23	174	49
PFNA	21	9	12	0.04	0.05	0.04	0.03	0.51	38	52
PFDA	21	20	1	0.80	0.83	0.80	0.57	5.5	13	58
PFUnDA	21	18	3	0.44	0.50	0.44	0.07	3.3	34	54
PFDoDA	21	21	0	0.88	0.89	0.88	0.14	3.6	16	63
PFTrDA	18	17	1	0.51	0.54	0.51	0.30	1.8	41	70
PFTeDA	18	15	3	0.52	0.61	0.52	0.22	1.8	52	63
L-PFBS	20	5	15	NAV	0.05	0.01	0.02	0.48	167	42
L-PFHxS	20	7	13	0.05	0.06	0.05	0.04	0.19	81	53
L-PFDS	17	4	13	NAV	0.11	0.04	0.03	0.17	121	58
6:2 FTSA	10	3	7	NAV	0.14	0.01	0.05	0.14	166	37
PFCAs + PFSAs LB (ND = 0)	**22**	**21**	**1**	**3.1**	**3.2**	**3.1**	**0.00**	**16.4**	**23**	**68**
PFCAs + PFSAs UB (ND = LOD)	**8**	**8**	**0**	**NAV**	**8.1**	**9.3**	**2.5**	**17.0**	**68**	**78**

Table 67: Summary of laboratory performance PFAS, fish

Fish A	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
L-PFOS anion	14	90	0	0	10
br-PFOS anion	9	69	15	8	0
tot-PFOS LB (ND = 0)	**17**	**84**	**4**	**0**	**12**
tot-PFOS UB (ND = LOD)	**11**	**88**	**6**	**0**	**6**
PFBA	10	0	0	0	0
PFPeA	11	0	0	0	0
PFHxA	13	0	0	0	0
PFHpA	13	0	0	0	0
PFOA	15	0	0	0	0
PFNA	14	29	0	0	14
PFDA	14	62	10	0	24
PFUnDA	14	48	5	14	19
PFDoDA	14	71	5	5	19
PFTrDA	12	50	22	11	11
PFTeDA	12	33	11	28	11
L-PFBS	14	0	0	0	0
L-PFHxS	14	20	0	5	10
L-PFDS	11	0	0	0	0
6:2 FTSA	7	0	0	0	0
PFCAs + PFSAs LB (ND = 0)	**15**	**68**	**9**	**5**	**14**
PFCAs + PFSAs UB (ND = LOD)	**5**	**0**	**0**	**0**	**0**

Table 68: Summary results PFAS, human milk (product basis) (ng/g)

Human milk	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
L-PFOS anion	15	9	6	0.02	0.03	0.02	0.02	7.5	40	61
br-PFOS anion	12	8	4	0.02	0.02	0.02	0.005	0.58	105	60
tot-PFOS LB (ND = 0)	**18**	**12**	**6**	**0.03**	**0.04**	**0.03**	**0.00**	**8.0**	**59**	**56**
tot-PFOS UB (ND = LOD)	**15**	**15**	**0**	**0.07**	**0.08**	**0.07**	**0.03**	**8.0**	**103**	**64**
PFBA	8	2	6	NAV	NAV	NAV	2.8	20.4	NAV	NAV
PFPeA	8	0	8	NAV	NAV	NAV	0.00	0.00	NAV	NAV
PFHxA	9	1	8	NAV	NAV	NAV	0.13	0.13	NAV	NAV
PFHpA	10	0	10	NAV	NAV	NAV	0.00	0.00	NAV	NAV
PFOA	14	9	5	0.03	0.03	0.03	0.02	22.3	38	51
PFNA	13	3	10	NAV	0.7	0.01	0.003	3.0	611	48
PFDA	12	2	10	NAV	NAV	NAV	0.007	1.1	NAV	NAV
PFUnDA	12	3	9	NAV	1.0	0.0	0.70	2.6	231	51
PFDoDA	10	1	9	NAV	NAV	NAV	1.4	1.4	NAV	NAV
PFTrDA	9	0	9	NAV	NAV	NAV	0.00	0.00	NAV	NAV
PFTeDA	9	0	9	NAV	NAV	NAV	0.00	0.00	NAV	NAV
L-PFBS	10	1	9	NAV	NAV	NAV	0.90	0.90	NAV	NAV
L-PFHxS	12	4	8	NAV	0.03	0.01	0.01	0.22	113	56
L-PFDS	9	0	9	NAV	NAV	NAV	0.00	0.00	NAV	NAV
6:2 FTSA	4	2	2	NAV	NAV	NAV	0.007	2.7	NAV	NAV
PFCAs + PFSAs LB (ND = 0)	**13**	**11**	**2**	**0.05**	**0.07**	**0.05**	**0.00**	**43.3**	**97**	**58**
PFCAs + PFSAs UB (ND = LOD)	**1**	**1**	**0**	**NAV**	**NAV**	**NAV**	**1.5**	**1.5**	**NAV**	**NAV**

Table 69: Summary of laboratory performance PFAS, human milk

Human milk	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
L-PFOS anion	10	40	0	7	13
br-PFOS anion	8	42	0	8	17
tot-PFOS LB (ND = 0)	**12**	**39**	**0**	**0**	**28**
tot-PFOS UB (ND = LOD)	**10**	**47**	**7**	**27**	**20**
PFBA	5	0	0	0	0
PFPeA	5	0	0	0	0
PFHxA	6	0	0	0	0
PFHpA	7	0	0	0	0
PFOA	9	36	7	0	21
PFNA	9	0	0	0	0
PFDA	8	0	0	0	0
PFUnDA	8	0	0	0	0
PFDoDA	7	0	0	0	0
PFTrDA	6	0	0	0	0
PFTeDA	6	0	0	0	0
L-PFBS	7	0	0	0	0
L-PFHxS	8	0	0	0	0
L-PFDS	6	0	0	0	0
6:2 FTSA	3	0	0	0	0
PFCAs + PFSAs LB (ND = 0)	**9**	**46**	**0**	**8**	**31**
PFCAs + PFSAs UB (ND = LOD)	**1**	**0**	**0**	**0**	**0**

Table 70: Summary results PFAS, human plasma (product basis) (ng/g)

Human plasma	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
L-PFOS anion	14	14	0	14.7	14.4	14.7	0.50	24.5	9	59
br-PFOS anion	10	10	0	5.4	5.1	5.4	0.60	10.3	38	65
tot-PFOS LB (ND = 0)	**16**	**16**	**0**	**19.8**	**19.5**	**19.8**	**1.1**	**24.5**	**22**	**77**
tot-PFOS UB (ND = LOD)	**12**	**12**	**0**	**20.1**	**19.9**	**20.1**	**1.1**	**23.8**	**9**	**62**
FOSA	7	0	7	NAV	NAV	NAV	0.00	0.00	NAV	NAV
MeFOSA	3	0	3	NAV	NAV	NAV	0.00	0.00	NAV	NAV
EtFOSA	3	0	3	NAV	NAV	NAV	0.00	0.00	NAV	NAV
MeFOSE	3	0	3	NAV	NAV	NAV	0.00	0.00	NAV	NAV
EtFOSE	3	0	3	NAV	NAV	NAV	0.00	0.00	NAV	NAV
PFOS precursors LB (ND = 0)	**6**	**0**	**6**	**NAV**	**NAV**	**NAV**	**0.00**	**0.00**	**NAV**	**NAV**
PFOS precursors UB (ND = LOD)	**3**	**3**	**0**	**NAV**	**0.50**	**0.95**	**0.50**	**2.5**	**117**	**80**
PFBA	12	3	9	NAV	0.14	0.05	0.11	14.5	83	50
PFPeA	12	1	11	NAV	NAV	NAV	0.64	0.64	NAV	NAV
PFHxA	13	0	13	NAV	NAV	NAV	0.00	0.00	NAV	NAV
PFHpA	13	2	11	NAV	NAV	NAV	0.03	0.18	NAV	NAV
PFOA	16	15	1	2.1	2.1	2.1	1.0	11.8	9	60
PFNA	15	14	1	0.95	0.95	0.95	0.41	3.2	12	63
PFDA	14	11	3	0.53	0.52	0.53	0.29	0.70	10	47
PFUnDA	15	11	4	0.48	0.48	0.48	0.34	2.6	16	55
PFDoDA	14	7	7	0.07	0.08	0.07	0.05	0.72	47	74
PFTrDA	13	2	11	NAV	NAV	NAV	0.03	0.15	NAV	NAV
PFTeDA	13	0	13	NAV	NAV	NAV	0.00	0.00	NAV	NAV
L-PFBS	13	1	12	NAV	NAV	NAV	0.47	0.47	NAV	NAV
L-PFHxS	15	13	2	6.3	6.2	6.3	3.0	6.7	7	61
L-PFDS	12	3	9	NAV	5.9	0.08	0.01	8.2	351	49
6:2 FTSA	7	1	6	NAV	NAV	NAV	2.0	2.0	NAV	NAV
PFCAs + PFSAs LB (ND = 0)	**16**	**16**	**0**	**9.9**	**10.2**	**9.9**	**2.8**	**34.1**	**19**	**64**
PFCAs + PFSAs UB (ND = LOD)	**5**	**5**	**0**	**12.0**	**12.1**	**12.0**	**10.3**	**14.2**	**13**	**80**

Table 71: Summary of laboratory performance PFAS, human plasma

Human plasma Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
L-PFOS anion	9	71	7	14	7
br-PFOS anion	7	50	10	20	20
tot-PFOS LB (ND = 0)	**11**	**88**	**0**	**6**	**6**
tot-PFOS UB (ND = LOD)	**8**	**92**	**0**	**0**	**8**
FOSA	5	0	0	0	0
MeFOSA	2	0	0	0	0
EtFOSA	2	0	0	0	0
MeFOSE	2	0	0	0	0
EtFOSE	2	0	0	0	0
PFOS precursors LB (ND = 0)	**4**	**0**	**0**	**0**	**0**
PFOS precursors UB (ND = LOD)	**2**	**0**	**0**	**0**	**0**
PFBA	8	0	0	0	0
PFPeA	8	0	0	0	0
PFHxA	9	0	0	0	0
PFHpA	9	0	0	0	0
PFOA	11	75	6	6	6
PFNA	10	67	13	7	7
PFDA	9	64	7	7	0
PFUnDA	10	60	7	0	7
PFDoDA	9	36	7	0	7
PFTrDA	9	0	0	0	0
PFTeDA	9	0	0	0	0
L-PFBS	9	0	0	0	0
L-PFHxS	10	73	7	7	0
L-PFDS	8	0	0	0	0
6:2 FTSA	5	0	0	0	0
PFCAs + PFSAs LB (ND = 0)	**11**	**69**	**6**	**13**	**13**
PFCAs + PFSAs UB (ND = LOD)	**3**	**100**	**0**	**0**	**0**

Table 72: Summary results PFAS, air extract (MeOH) (ng/g)

Air extract (MeOH)	n								Between lab CV	Inclusion rate
	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
L-PFOS anion	17	17	0	4.2	4.5	4.2	2.5	14.2	21	67
br-PFOS anion	10	5	5	NAV	0.16	0.09	0.07	1.7	99	56
tot-PFOS LB (ND = 0)	**18**	**18**	**0**	**4.1**	**4.4**	**4.1**	**2.5**	**14.2**	**21**	**63**
tot-PFOS UB (ND = LOD)	**11**	**11**	**0**	**4.1**	**4.1**	**4.1**	**2.7**	**9.4**	**25**	**74**
FOSA	10	10	0	48.9	48.5	48.9	19.0	71.0	23	64
MeFOSA	9	9	0	175	174	175	59.0	233	6	59
EtFOSA	9	9	0	180	176	180	46.0	325	10	63
MeFOSE	10	10	0	84.9	91.5	84.9	39.0	258	28	64
EtFOSE	10	10	0	91.1	91.5	91.1	33.0	204	4	56
PFOS precursors LB (ND = 0)	**10**	**10**	**0**	**596**	**580**	**596**	**200**	**889**	**41**	**77**
PFOS precursors UB (ND = LOD)	**9**	**9**	**0**	**627**	**583**	**627**	**200**	**889**	**27**	**67**
PFBA	13	13	0	6.4	6.5	6.4	3.5	9.5	41	84
PFPeA	13	12	1	3.1	3.1	3.1	1.8	4.4	34	81
PFHxA	15	15	0	6.2	6.5	6.2	3.6	12.0	22	66
PFHpA	14	14	0	3.2	3.4	3.2	1.7	6.0	27	71
PFOA	16	16	0	3.4	3.5	3.4	1.8	5.5	25	67
PFNA	14	14	0	3.1	3.1	3.1	1.7	4.7	21	69
PFDA	15	15	0	6.3	6.6	6.3	3.9	10.3	21	68
PFUnDA	14	14	0	2.9	2.9	2.9	1.6	5.6	30	67
PFDoDA	13	13	0	2.9	3.0	2.9	1.7	6.0	30	68
PFTrDA	14	14	0	3.0	3.3	3.0	1.1	5.7	55	82
PFTeDA	13	12	1	3.0	3.4	3.0	1.2	5.8	56	76
L-PFBS	15	15	0	7.1	7.5	7.1	3.7	12.0	26	70
L-PFHxS	15	15	0	4.1	4.3	4.1	2.7	10.5	19	64
L-PFDS	11	11	0	3.9	3.5	3.9	0.83	5.3	35	76
6:2 FTSA	9	8	1	0.26	0.30	0.26	0.20	2.0	51	66
PFCAs + PFSAs LB (ND = 0)	**17**	**17**	**0**	**54.4**	**55.2**	**54.4**	**3.6**	**92.0**	**33**	**67**
PFCAs + PFSAs UB (ND = LOD)	**7**	**7**	**0**	**NAV**	**67.1**	**66.3**	**3.6**	**92.0**	**49**	**83**

Table 73: Summary of laboratory performance PFAS, air extract (MeOH)

Air extract (MeOH) Analyte	% of the data received	% of z-scores |z|<2 Satisfactory	% of z-scores 3>|z|>2 Questionable	% of z-scores 6>|z|>3 Unsatisfactory	% of z-scores |z|>6 Extreme
L-PFOS anion	11	65	12	6	18
br-PFOS anion	7	0	0	0	0
tot-PFOS LB (ND = 0)	**12**	**67**	**6**	**11**	**17**
tot-PFOS UB (ND = LOD)	**7**	**73**	**9**	**9**	**9**
FOSA	7	60	10	30	0
MeFOSA	6	78	11	11	0
EtFOSA	6	67	0	22	11
MeFOSE	7	50	20	10	20
EtFOSE	7	70	0	10	20
PFOS precursors LB (ND = 0)	**7**	**50**	**10**	**40**	**0**
PFOS precursors UB (ND = LOD)	**6**	**56**	**11**	**33**	**0**
PFBA	9	38	31	31	0
PFPeA	9	46	23	23	0
PFHxA	10	60	13	13	13
PFHpA	9	57	21	7	14
PFOA	11	63	13	25	0
PFNA	9	64	21	14	0
PFDA	10	67	13	20	0
PFUnDA	9	57	14	14	14
PFDoDA	9	62	8	15	15
PFTrDA	9	43	7	43	7
PFTeDA	9	31	23	31	8
L-PFBS	10	60	13	27	0
L-PFHxS	10	60	20	0	20
L-PFDS	7	64	18	9	9
6:2 FTSA	6	44	11	11	22
PFCAs + PFSAs LB (ND = 0)	**11**	**59**	**12**	**18**	**12**
PFCAs + PFSAs UB (ND = LOD)	**5**	**0**	**0**	**0**	**0**

Table 74: Summary results PFAS, water (pg/g)

Water	n								Between lab CV	Inclusion rate
Analyte	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	(%)
L-PFOS anion	19	18	1	2.4	2.5	2.4	0.001	2294	33	70
br-PFOS anion	14	14	0	2.0	1.9	2.0	0.37	2162	40	67
tot-PFOS LB (ND = 0)	**21**	**20**	**1**	**3.9**	**4.2**	**3.9**	**0.00**	**4456**	**42**	**66**
tot-PFOS UB (ND = LOD)	**16**	**16**	**0**	**4.3**	**4.5**	**4.3**	**1.72**	**4456**	**33**	**67**
PFBA	16	16	0	6.9	7.6	6.9	0.006	64430	34	66
PFPeA	15	13	2	5.7	5.8	5.7	0.009	8.6	36	71
PFHxA	17	17	0	7.9	7.7	7.9	0.007	9.4	17	76
PFHpA	17	17	0	3.7	3.8	3.7	0.003	8.7	19	68
PFOA	19	18	1	10.1	10.1	10.1	0.01	17928	23	68
PFNA	18	13	5	0.53	0.54	0.53	0.0003	3217	16	58
PFDA	17	10	7	0.34	0.35	0.34	0.0002	751	14	52
PFUnDA	18	3	15	NAV	0.27	0.05	0.07	2514	224	43
PFDoDA	16	3	13	NAV	0.20	0.06	0.07	1.8	115	53
PFTrDA	14	0	14	NAV	NAV	NAV	0.00	0.00	NAV	NAV
PFTeDA	14	2	12	NAV	NAV	NAV	0.06	0.11	NAV	NAV
L-PFBS	18	17	1	7.1	6.8	7.1	0.010	2351	24	68
L-PFHxS	17	16	1	1.4	1.4	1.4	0.002	464	16	63
L-PFDS	15	0	15	NAV	NAV	NAV	0.000	0.000	NAV	NAV
6:2 FTSA	9	9	0	15.7	16.5	15.7	0.02	21490	31	54
PFCAs + PFSAs LB (ND = 0)	**20**	**20**	**0**	**45.0**	**45.9**	**45.0**	**0.06**	**111930**	**40**	**66**
PFCAs + PFSAs UB (ND = LOD)	**7**	**7**	**0**	**NAV**	**66.0**	**67.4**	**0.06**	**108**	**44**	**76**

Table 75: Summary of laboratory performance PFAS, water

Water	% of the data received	% of z-scores |z|<2	% of z-scores 3>|z|>2	% of z-scores 6>|z|>3	% of z-scores |z|>6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
L-PFOS anion	13	53	11	16	16
br-PFOS anion	9	43	14	21	21
tot-PFOS LB (ND = 0)	14	48	5	24	19
tot-PFOS UB (ND = LOD)	11	56	6	19	19
PFBA	11	63	6	6	25
PFPeA	11	38	25	13	6
PFHxA	12	78	6	6	6
-PFHpA	12	67	6	11	11
PFOA	13	58	21	5	11
PFNA	12	44	11	0	17
PFDA	11	41	0	0	18
PFUnDA	12	0	0	0	0
PFDoDA	11	0	0	0	0
PFTrDA	10	0	0	0	0
PFTeDA	10	0	0	0	0
L-PFBS	12	67	6	11	11
L-PFHxS	12	61	6	6	17
L-PFDS	10	0	0	0	0
6:2 FTSA	6	56	0	0	44
PFCAs + PFSAs LB (ND = 0)	14	45	15	20	20
PFCAs + PFSAs UB (ND = LOD)	5	0	0	0	0

3.3 Regional Participation

The following Table 76 shows the distribution of laboratories that submitted results for at least one POP in one of the test samples and where a z-score could be assigned. It can be seen that most laboratories obtained z-scores for OCPs, namely 86 laboratories, followed by laboratories analyzing PCB(6) with 71 laboratories. For dioxin-like POPs, where analysis is considered costly and demanding, 67 laboratories did participate. A conclusion from this IL4 is that the African region does not have capacity for the analysis of brominated flame retardants (here: PBDE with HxBB combined), HBCD or PFAS. CEE and GRULAC do not have proven capacity for HBCD. GRULAC and CEE are emerging and start to build up capacity for PFAS with one and two laboratories, respectively.

Table 76: Number of reporting laboratories per POP group and region

Region/POP Group	OCPs	PCB(6)	dl-POPs	PBDE+HxBB	HBCD	PFAS
Africa	16	9	1			
Asia	23	20	32	15	7	11
CEE	3	4	2	2		2
GRULAC	26	17	8	5		1
WEOG	18	21	21	18	8	25
Grand Total	**86**	**71**	**64**	**40**	**15**	**39**

The following Tables (Table 77 to Table 83) show the number of laboratories reporting results for each matrix and per region. The total number of laboratories that reported results for a given POP in any of the test samples is summarized in column “Total Labs”. For some POP/matrix combinations, no z-scores could be assigned and therefore, there is no qualified laboratory. These are: PCB(6) in fish, HBCD in fish and human milk, and toxaphene in sediment, human milk and air extract.

The lowest number of laboratories reported results for toxaphene (9 laboratories; Table 82) and HBCD (15 laboratories; Table 81). A quite impressive number of laboratories reported results for the more advanced POPs such as for dl-POPs (64 laboratories; Table 79), or PFAS (39 laboratories; Table 83). However, it shall be noted that the vast majority of these laboratories are found in the Asia-Pacific and the WEOG regions.

From all test samples, the test solution for POPs standards had the highest reporting rate in general. Exceptions often can be found in the WEOG region where laboratories prefer “real samples” such as for PCB(6) in air extract, dl-POPs in fish, or PFAS in water in the Asia-Pacific region. The air extract, a core matrix in the GMP, in general, is quite frequently analyzed. High interest in all regions and for all group of POPs is for fish (not a core matrix in the GMP).

Table 77: Number of reporting laboratories for OCPs per region

Region	Total Labs	Test Solution	Sediment	Fish	Human milk	Air Extract
Africa	16	10	9	9	4	3
Asia	23	17	13	9	4	6
CEE	3	2	2	1		
GRULAC	26	21	11	9	9	5
WEOG	18	10	5	5	5	8
Grand Total	86	60	40	33	22	22

Table 78: Number of reporting laboratories for PCB per region

Region	Total Labs	Test Solution	Sediment	Fish	Human milk	Air Extract
Africa	9	7	5		6	4
Asia	20	17	13		8	11
CEE	4	3	2		1	2
GRULAC	17	16	10		10	5
WEOG	21	13	10		12	14
Grand Total	71	56	40		37	36

Table 79: Number of reporting laboratories for dl-POPs per region

Region	Total Labs	Test Solution	Sediment	Fish	Human milk	Air Extract
Africa	1	1			1	1
Asia	32	25	25	19	10	21
CEE	2	1			1	2
GRULAC	8	6	3	4	1	1
WEOG	21	13	9	15	12	12
Grand Total	64	46	37	38	25	37

Table 80: Number of reporting laboratories for PBDE and HxBB per region

Region	Total Labs	Test Solution	Sediment	Fish	Human milk	Air Extract
Africa						
Asia	15	12	12	11	4	8
CEE	2	1			1	2
GRULAC	5	4	3	3	1	2
WEOG	18	11	7	12	7	10
Grand Total	40	28	22	26	13	22

Table 81: Number of reporting laboratories for HBCDs per region

Region	Total Labs	Test Solution	Sediment	Fish	Human Milk	Air Extract
Africa						
Asia	7	5		5		3
CEE						
GRULAC						
WEOG	8	8		4		3
Grand Total	15	13		9		6

Table 82: Number of reporting laboratories for toxaphenes per region

Region	Total Labs	Test Solution	Sediment	Fish	Human Milk	Air Extract
Africa						
Asia	2	2		2		
CEE						
GRULAC	3	3		3		
WEOG	4	4		4		
Grand Total	9	9		9		

Table 83: Number of reporting laboratories for PFAS per region

Region	Total Labs	Test Solution	Sediment	Fish	Human milk	Air	Human plasma	Water
Africa								
Asia	11	7	5	7	6	7	5	8
CEE	2	2	1	1	1	1	2	1
GRULAC	1	1				1		1
WEOG	25	19	7	17	11	9	9	12
Grand Total	39	29	13	25	18	18	16	22

3.4 Methodological Considerations

The number of laboratories submitting results for each group of analytes, the concentrations of the target compounds in the test materials, and variations in the analytical methods used by the participants are factors that may influence the interpretation and the outcome (Wells and De Boer, 2006). Calculation and dilution errors are other factors that may impede the understanding of the data. Nonetheless, based on the results and previous experience with interlaboratory studies, several problems could again be elucidated.

The POPs concentrations in all matrices except human milk are presented on a wet weight (w/w) basis. Participants were asked, however, to report the lipid content of human milk, so it could be used when needed for interpretation of the data.

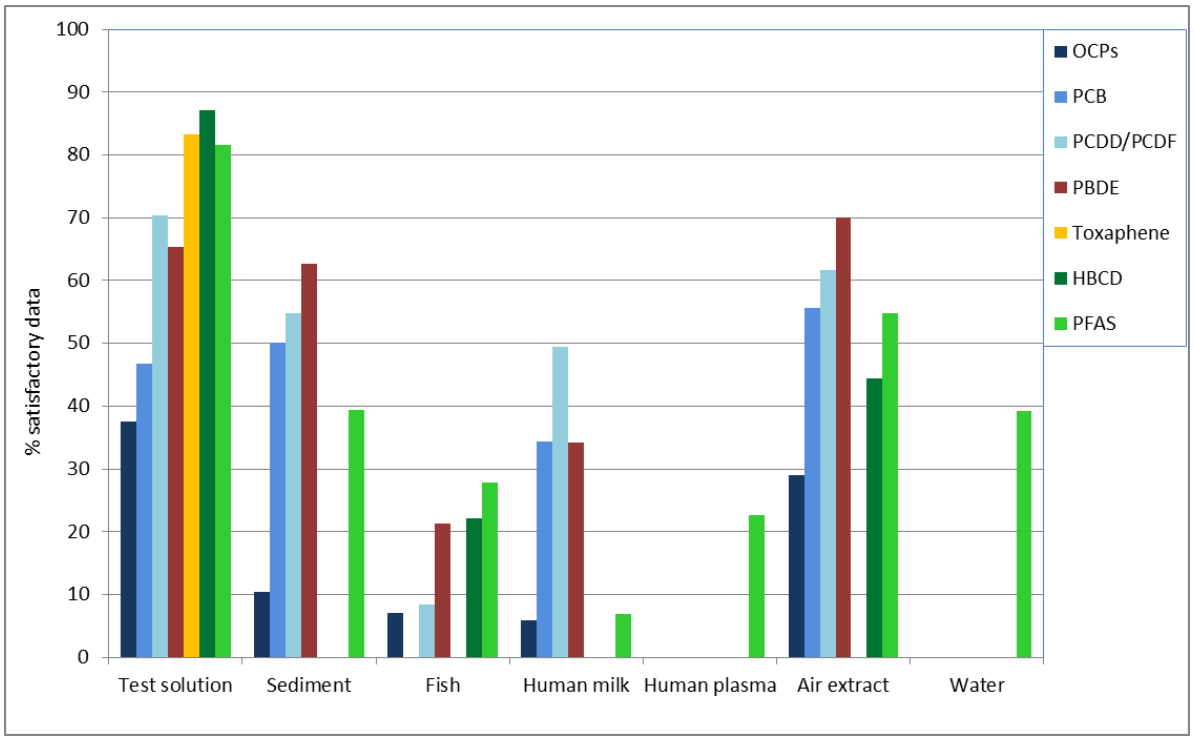

Figure 1 Percentage of laboratories with satisfactory z-scores in the analysis of OCPs, PCB, PCDD/PCDF, PBDE, toxaphene, HBCD and PFAS, with the compounds included, which did not receive an assigned value.

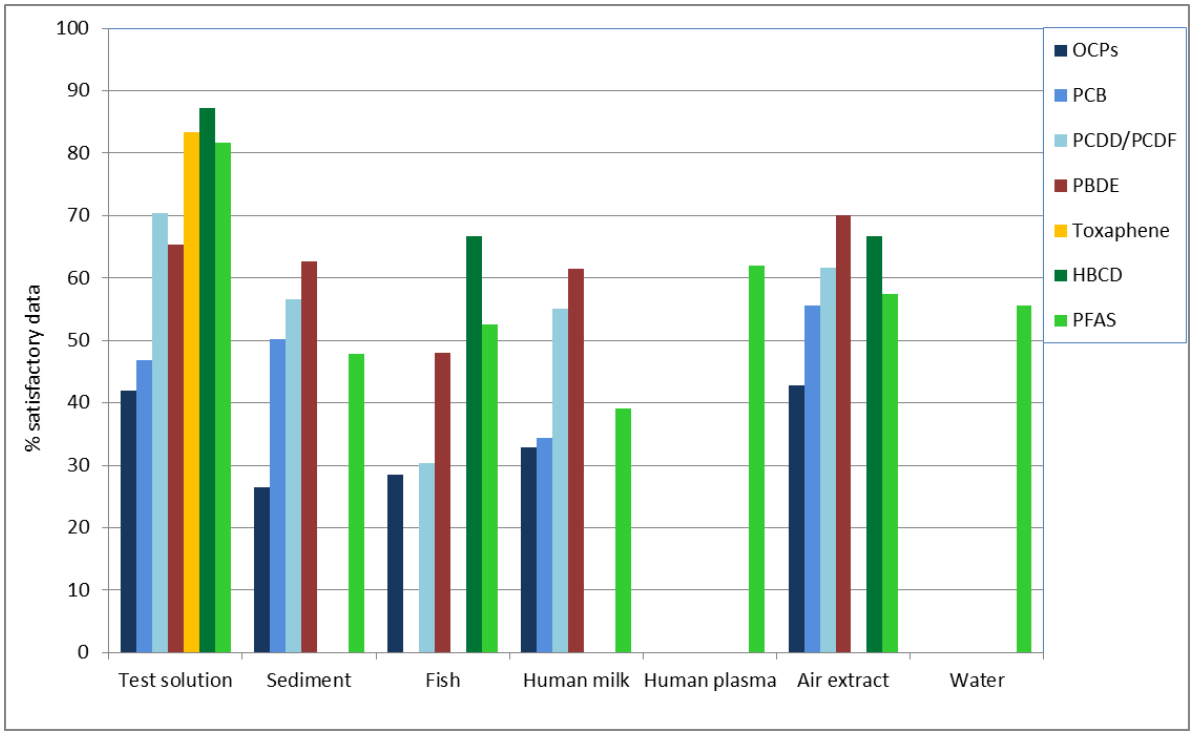

Figure 2 Percentage of laboratories with satisfactory z-scores in the analysis of OCPs, PCB, PCDD/PCDF, PBDE, toxaphene, HBCD and PFAS, for all the compounds, which received an assigned value.

The overall performance of labs measuring the test solution (certified test solutions) was not satisfactory. Laboratories should be able to analyse a test solution. A standard solution contains no matrix and in fact the only variables tested in this way are ability to dilute, to add internal standard and the instrumental method. Possibly some of the laboratories have not stored their stock solutions in a proper way.

Figure 1 and Figure 2 show that less than 50% had satisfactory z-scores for OCPs and less than 60% for PCB and HxBB. Failure to analyse a test solution properly, makes all efforts for matrix test materials more or less in vain. It is a clear signal to go back to the basics and check instrumentation, calibration and basic techniques.

Some of the compounds, such as the PCDD/PCDF, PBDE, PFAS and toxaphene, showed a better performance, although in fact with the target of 25% CV the performance should be closer to 100%.

During the evaluation of these results the question came up if some laboratories had reported results for the test solutions on a weight per volume (w/v) basis as is done in other studies. Therefore, we asked all participants to check if they had reported according to the instructions (w/w basis) or if they had reported on w/v basis. Forty-five out of 95 laboratories answered. Forty-two of the these had reported on a w/w basis, according to the instructions. Two laboratories had reported on a w/v basis and indeed those can be found on the lower side of all results. One laboratory reported an error. Many labs that reported on a w/w basis had used density corrections, which might have introduced small deviations. This outcome does not help in explaining the discrepancies between theoretical values and assigned values. Apart from the two laboratories, no serious mistakes or misinterpretation of the guidelines were made. It means that laboratories should pay much more attention to the storage and preparation of their calibration solutions.

As expected, the between-lab CV values were larger for the matrix-based test materials. Fewer satisfactory z-scores were obtained using the same criteria (z = 2, so 25% CV for the group performance). In particular, the OCP and PCB results were rather disappointing. The pike perch test material had a low fat percentage (ca. 0.7%) which made it possibly difficult for laboratories to determine the correct concentrations. It may be assumed that a number of laboratories took in too little matrix for their determination, possibly partly caused by the quantity of material provided; 35 g is not so much for the analysis of a large suite of parameters. Other laboratories may first have calculated the results on a lipid weight basis and then used the wrong fat content to calculate back to the total concentration on a wet weight basis. The fat of the pike perch consists mainly of phospholipids with only a small amount of triglycerides. If a laboratory does not use a more polar solvent for extraction in combination with a non-polar solvent, mistakes can easily be made, both in the fat content as well as in the POP concentration. For a proper determination of the fat content a method according to Bligh and Dyer (1959) or Smedes (1999) is strongly recommended. The air extract results show somewhat better results, which is probably due to the absence of matrix and the fortification of POP concentrations. These results are hopeful as air is an important matrix in the GMP. The results for PFAS in the water and human plasma sample were promising.

Overall, there are still too few laboratories submitting satisfactory results. Also, more laboratories should report a more complete set of data.

3.5 Analyte Group - Specific Performance

3.5.1 Organochlorine Pesticides

The individual results for the OCPs for the test solution show between-lab model CV values of 39%-54% for the drins, 18%-55% for the chlordanes and 37%-71% for the DDTs (Table 2). Since the test solution is without any matrix, no extraction or cleaning is required, and the results represent the performance on the instrumental analyses only. To be able to analyse more complex matrices laboratories should be able to have a good performance on the instrumental analyses, meaning that the overall performance on the test solution should be much better than ±25% (|z|= 2). In the third round the results were already disappointing with only 44% of the laboratories receiving a satisfactory z-score (|z| < 2), but in this round even less (average 38%) participants were able to analyse OCPs satisfactory (Table 3). In Figure 3 the percentage of laboratories with satisfactory z-scores for the test solution is given per OCP group.

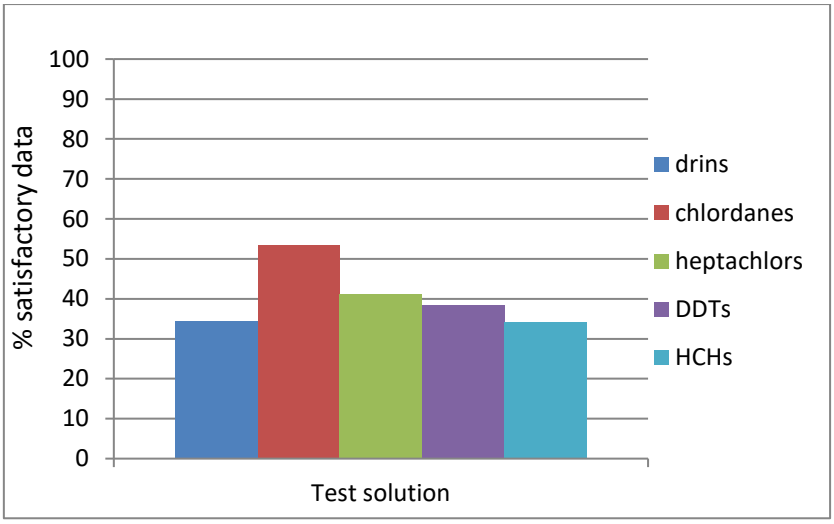

Figure 3 Percentage of laboratories with satisfactory z-scores for analysis of OCPs in the test solution.

As an example, the variation of reported results for the test solution is shown in Figure 4 for dieldrin (54%), in which the individual results from each laboratory are given in addition to the consensus value as calculated by the Cofino statistics and the UNEP criteria of 12.5% (z = 1) and 25% (z = 2) (UNEP, 2012). The WEOG and Asian laboratories do generally a better job than laboratories from the other regions. However, also in the WEOG group an extreme (high) outlier was found. GRULAC and African laboratories tend to report too low values.

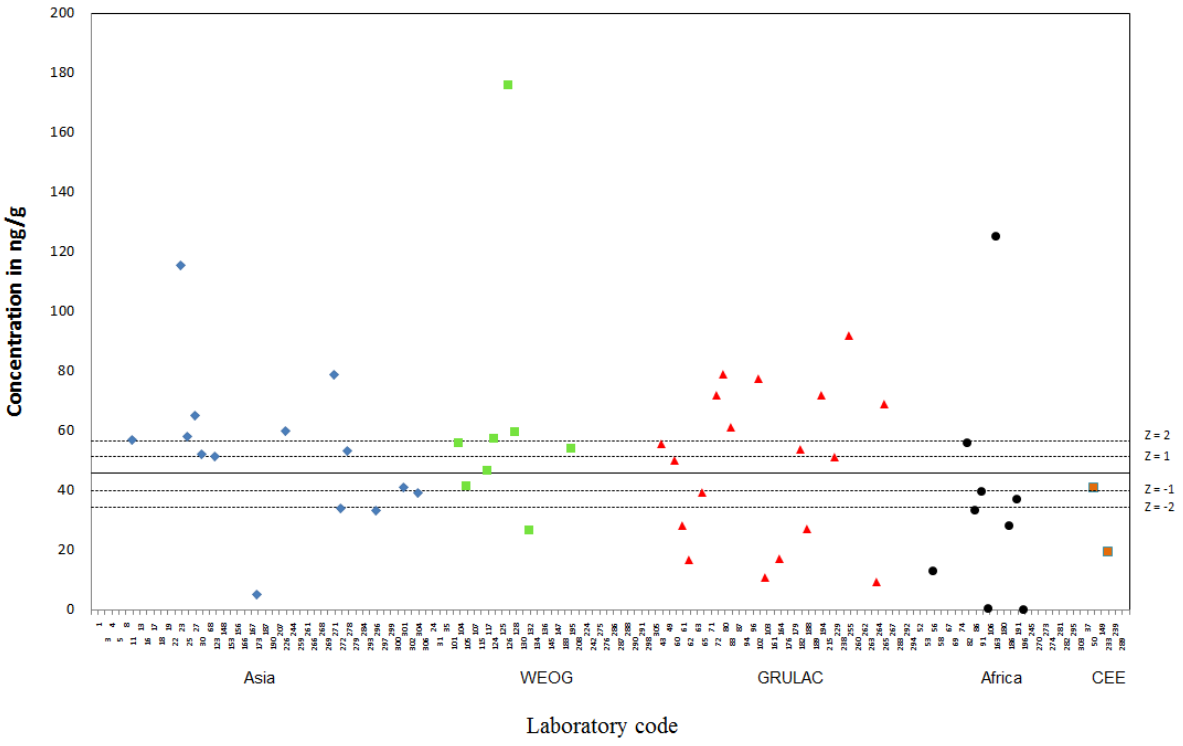

Figure 4 Results for dieldrin in the test solution.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

The performance of OCPs in the air extract was also less good compared to the third round, with an increase in CV from 27% in the third round to 76% in this round. This result is partially caused by the performance for trans-heptachlorepoxide (CV= 340%) (Figure 5). Although only nine results of trans-heptachlorepoxide > LCV were reported, arranging those results by detection method shows a clear difference per method (Figure 6). Using ECD resulted in higher concentrations and more deviation between laboratories, than using HR-MS. This clearly points to an overlap of the trans-heptachloro epoxide peak with an interference. Mass spectrometry is able to correct for that. If not available, a second GC column in GC-ECD would be essential here, keeping in mind that not all phases would be able to separate this interference and trans-hepo. Also, for other OCPs like the chlordanes, the DDTs and the HCHs smaller CVs are observed for HR-MS data (Figure 7).

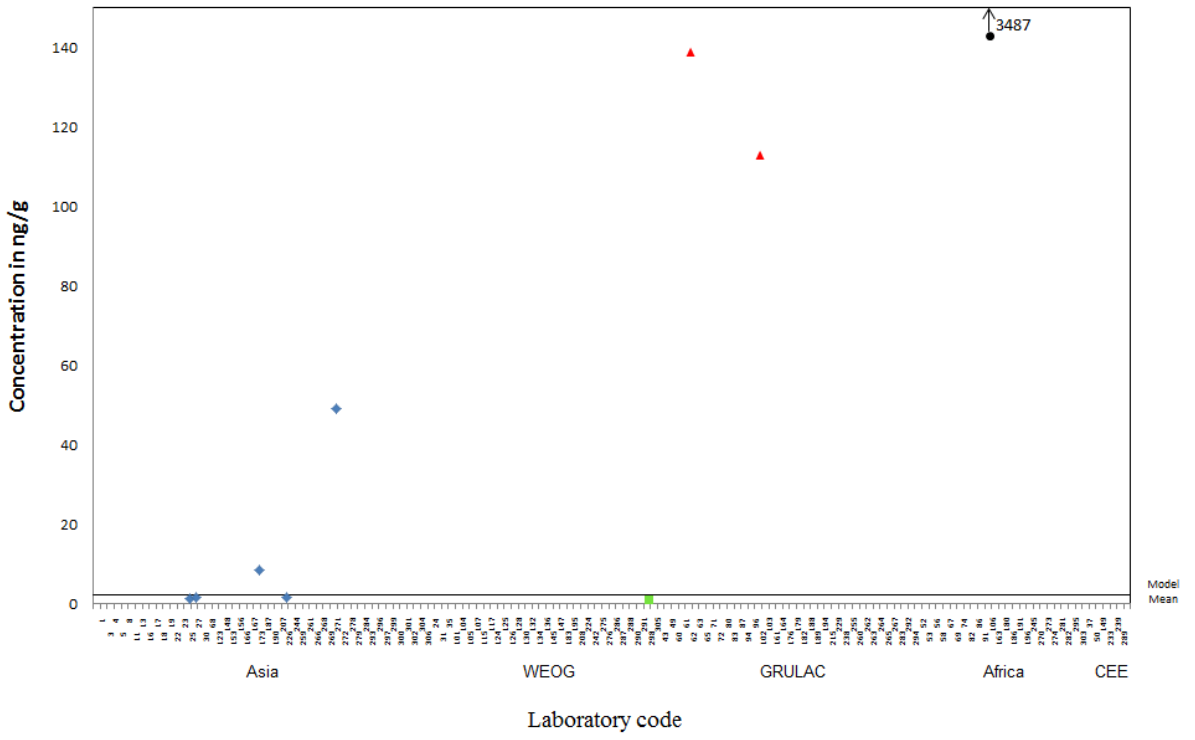

Figure 5 Results for trans-heptachlorepoxide in the air extract.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The model mean value is given by the straight line. Laboratory code on the x-axis, concentration in ng/g on the y-axis. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

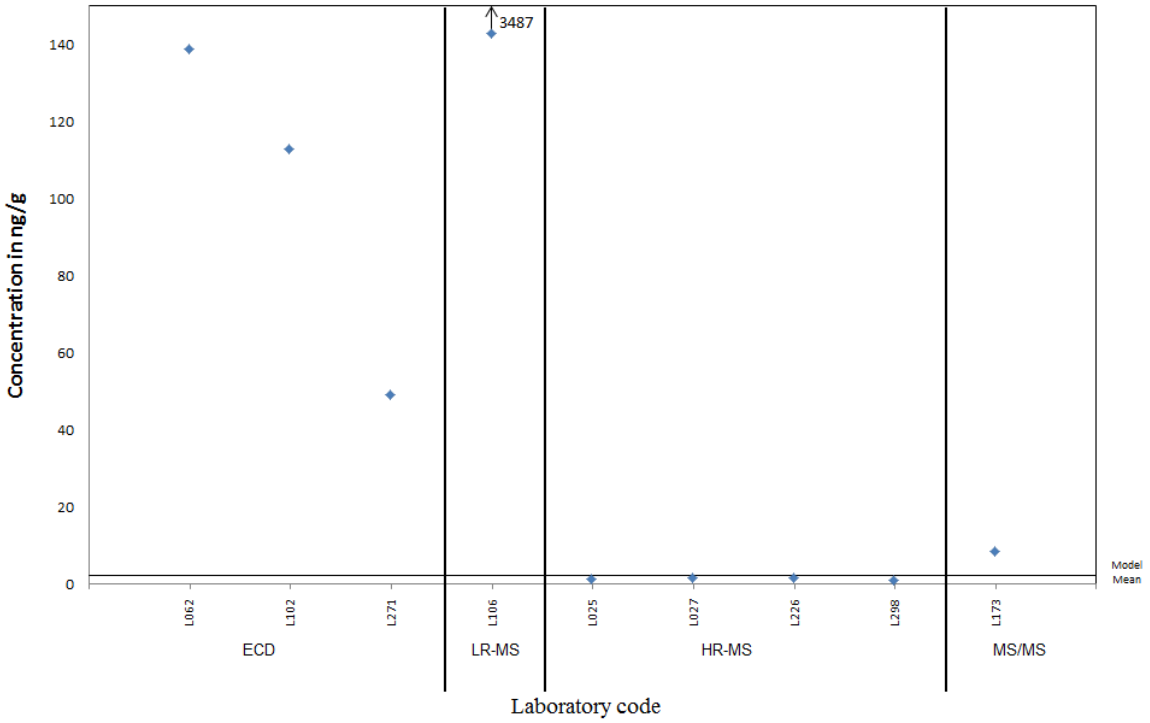

Figure 6 Results for trans-heptachlorepoxide in the air extract arranged by detection method.
Laboratory code on the x-axis, concentration in ng/g on the y-axis, concentration in ng/g on the y-axis. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

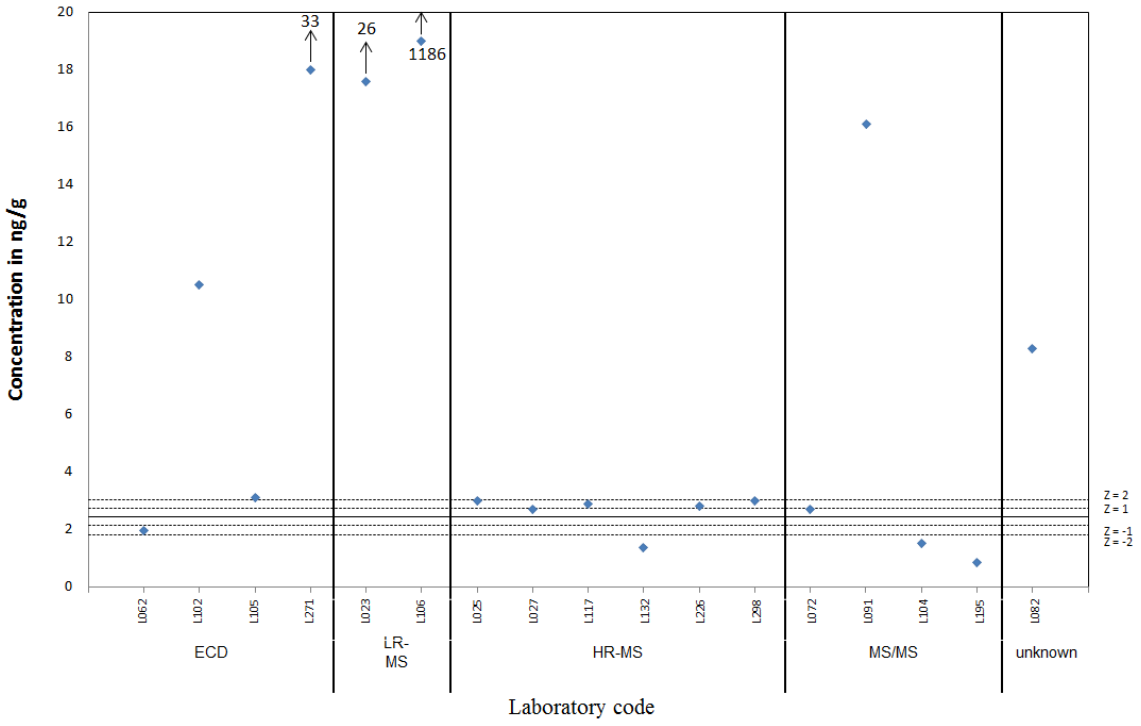

Figure 7: Results for dieldrin in the air extract arranged by detection method.
Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

Based on the criteria described in section 2.2, an assigned value could be calculated for the milk sample only for five OCPs (oxychlordane, p,p'-DDT, p,p'-DDE, β-HCH, hexachlorobenzene) of which the mean concentrations were between 0.9 and 26 ng/g (Table 8). For the other OCPs, less than four numerical results were submitted for seven OCPs (category 3 (section 2.2)), between 3 and 7 numerical values were reported with too much variation for eight other OCPs (category 2), and seven or more numerical values were reported for eight compounds, with less than 25% of the z-score $|z| < 2$ (category 1). Mean concentrations of those OCPs were low (0.05-18 ng/g), which might have led to these results.

In the third round, the average CV% of OCPs in the sediment test material was extremely high (196%), which might have been caused by a high background contamination, since the sediment originated from a highly polluted river. The sediment in this round, originating from a different location (Rotterdam harbour, the Netherlands) contained mean OCP concentrations of 0.07 ng/g -4 ng/g (average 1.1 ng/g), and the average CV% decreased to 138%. For the drins in the sediment sample, the average CV% decreased from 307% in the third round to 95% in this round. In the report of the third round (Fiedler et al., 2017), it has been discussed that submitted results on the drins could be higher when analysed with other detection methods than MS, due to interferences in the chromatogram, which could not be removed during cleaning, since sulphuric acid treatment is not allowed, because of degradation of the drins. This was clearly shown by the results reported for dieldrin in sediment in that round where nine participants out of 28 reported to have used an MS method. Although only one participant (L191) who used ECD in the third round for dieldrin in sediment, switched to an MS method in this round, a higher percentage of participants used an MS method (10 out of 21 participants), which might explain the lower CV% for the drins (Figure 8). Of course, also a CV of 95% is still much too high.

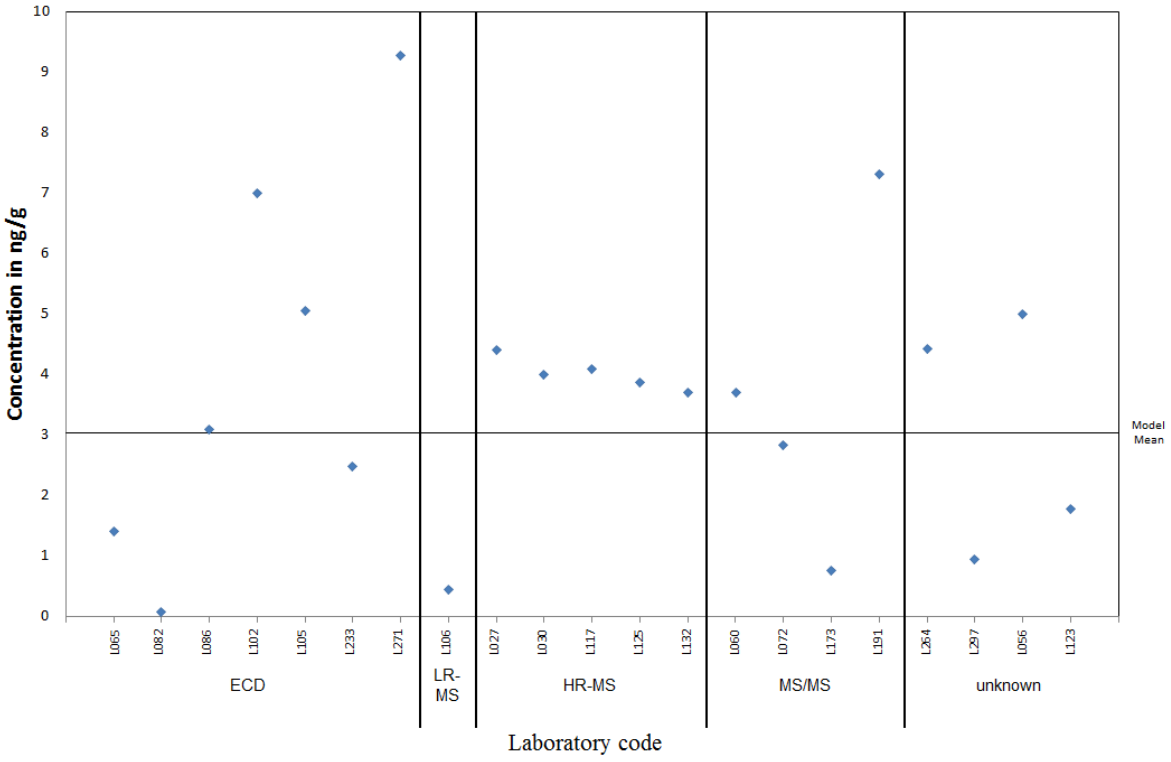

Figure 8 Results for dieldrin in the sediment sample arranged by detection method.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

An even larger variation was seen for OCPs in the fish test material. Only for seven OCPs an assigned value could be calculated, and CV values were extremely high (10% (γ-chlordane) -768% (endrin), average 244%) (Table 6). Mean OCP concentrations were on the low side (0.01-2.3 ng/g, average 0.25 ng/g), and for most compounds (22 out of 28) more than 50% of the participants reported a value < LCV (Table 6).

The results on OCP analyses were disappointing for all matrices in this round. For only six OCPs in the test solution (Table 3) and seven in the air extract (Table 11) more than 50% of the data showed satisfactory z-scores, while for none of the compounds in the sediment sample, the fish sample, and the human milk more than 50% of the data were satisfactory (see Table 4, Table 6, and Table 8). In Figure 9 the performance per matrix is given for the analyses of drins, chlordanes, heptachlors, DDTs, and HCHs, showing that the high average CV values are caused by the results of all OCPs groups.

Although an MS is not always available in a laboratory, and especially HR-MS is too costly for some laboratories, results on OCPs in this interlaboratory study show that MS, and especially HR-MS is giving more consistent results for OCP analyses.

On one hand spiking of test materials should be considered, which would help the laboratories in their performance as higher concentrations are less prone to errors. On the other hand, OCP concentrations at most places in the world are not very high anymore and laboratories should also be able to measure these low concentrations.

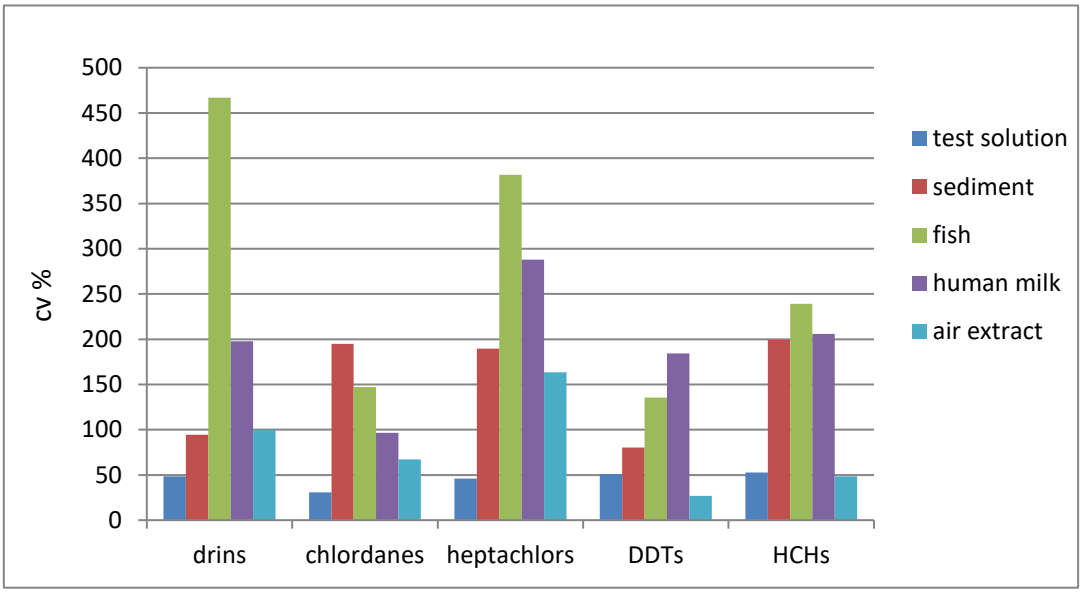

Figure 9 Performances per matrix for the analyses of drins, chlordanes, heptachlors, DDTs, and HCHs.

3.5.2 Polychlorinated Biphenyls

Although for the indicator PCB results were more satisfactory than for the OCPs, only for the air extract more than 50% of the results on average were satisfactory (|z| < 2) (Figure 1). In comparison with the previous studies, the percentage of satisfactory z-scores received for the test solution decreased from 86% in the first study, to 66% in the second study, to 57% in the third study to only 47% in the present study (Figure 1, Table 13), and average model between lab CV values increased from 13% in the first study, to 22% in the second study, to 27% in the third study, to 38% in the present study, while all concentrations were in the same order of magnitude.

The increasing CVs and the decreasing number of satisfactory data might be related to experience of the laboratories. Of the 148 participants in this round, 49 laboratories participated for the first time, and 10 laboratories participated for the first time in the third round. For the sum of PCB (UB) the CV was 36% (Table 12). Performing the statistical evaluation only on the results of the laboratories who already participated in the first or second round results in a much lower CV (26%), while calculating the model CV over the results of the participant who participated for the first time in the third round or in the present round, resulted in a much higher CV (59%). The difference of reported results of the more experienced participants (CV=29%), and of the first time participants in the third, or fourth round (CV=90%) are shown for PCB 28 in the test solution in Figure 10.

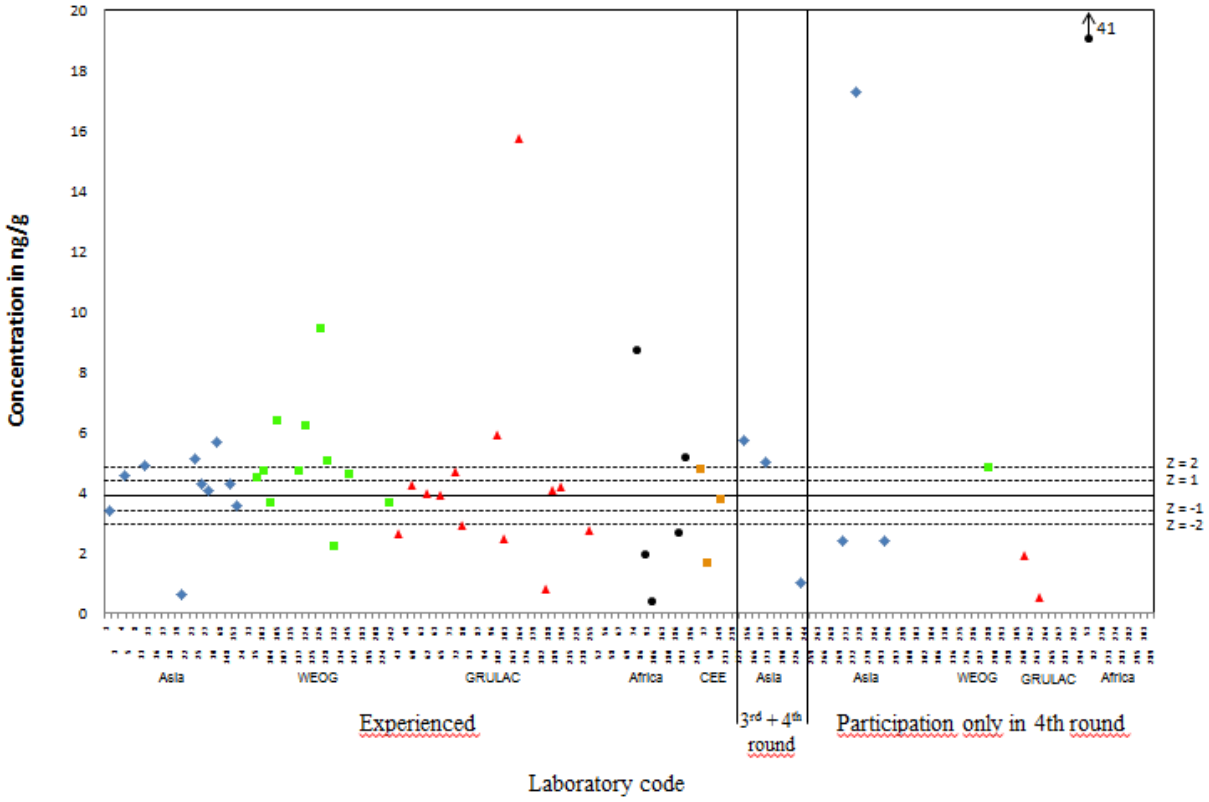

Figure 10 Results for PCB 28 in the test solution.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

For the air extract, the results were slightly better than for the test solution. Between-lab CV values were 20%-34% (average 26%) (Table 20, Figure 11). Sediment is a dirtier matrix than an air extract, and as expected the results for the sediment sample show a larger variation (24%-58%, average 34%). However, in comparison with the previous ILS (CV= 53%-75%, average 63%) the results improved, with 50% satisfactory results compared to 31% in the third round.

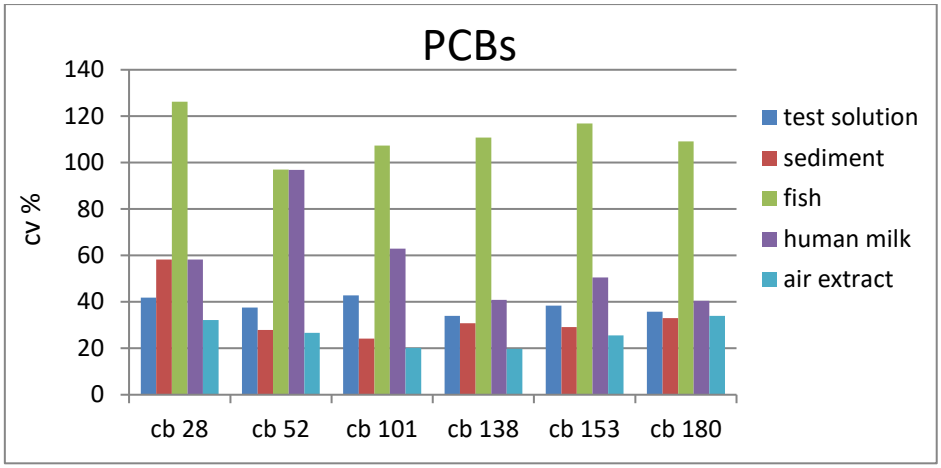

Figure 11 Performances per matrix for the analyses of PCB.

In Figure 12 the reported results for PCB 153 in the sediment are plotted per detection method. With HR-MS most participants obtained a satisfactory z-score (87%). With ECD 44% of the results were satisfactory, with LR-MS 63%, and with MS/MS 40%.

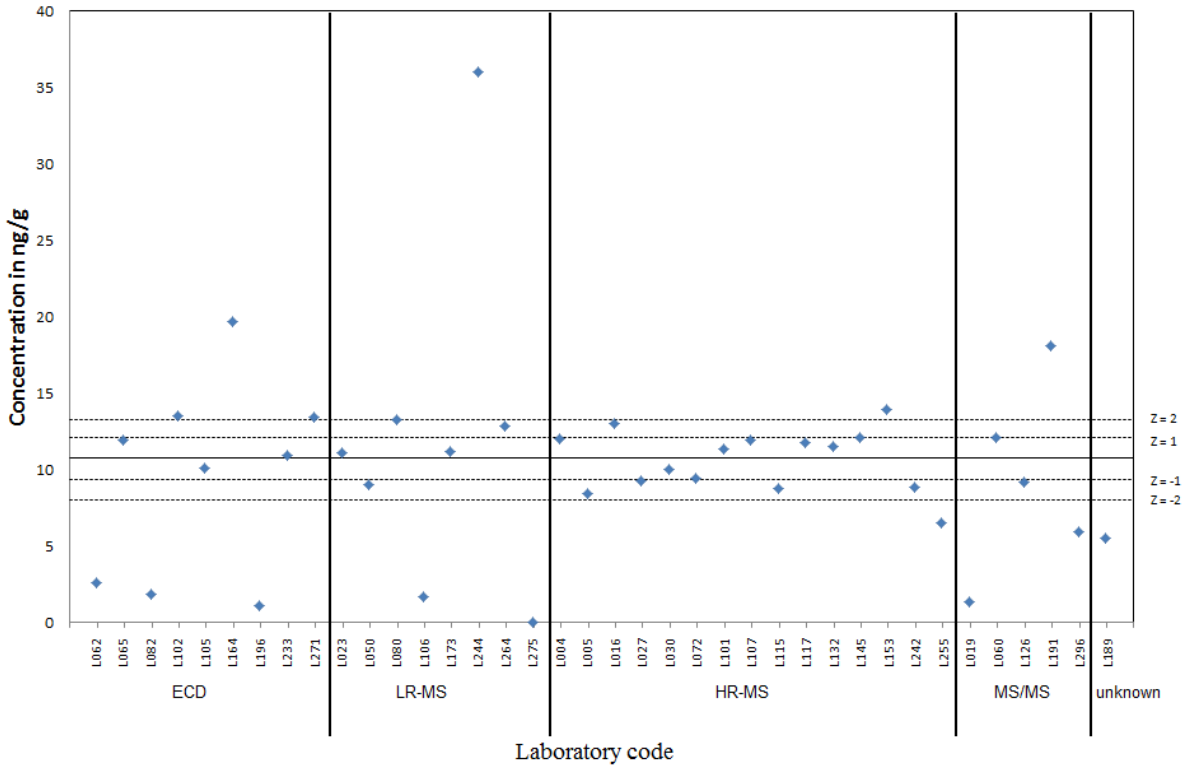

Figure 12 Results for PCB 153 in the sediment sample arranged by detection method.
Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

PCB concentrations in the human milk (0.3-13 ng/g) were in the same range as in the third round. The performance was a little better with 34% satisfactory z-scores in the present round (Table 19) compared to 30% in the third round, and a model between lab CV of 58% (present) (Table 18, Figure 11) compared to 63% (third round).

The largest variation for PCBs was found for the fish sample (CV=111%). For none of the PCBs an assigned value could be calculated (Table 16), because <25% of the data was satisfactory (category 1, section 2.2). Median PCB concentrations (0.43-11 ng/g) were in the same range as in the test solution, the air extract, and milk sample, but since fish is a more complex, it could be more difficult to analyse low concentrations. Compared to the third round the model between lab CV increased substantially, from 63% to 111%. Interestingly, the PCB concentrations in the fish test material were not very low. Laboratories, even when not very experienced should not have any problem with analysing these levels, even when using ECD. The only possible explanation of these disappointing results is the low fat content and the composition of the fat. Pike perch contains mainly phospholipids. PCBs are also present in phospholipids (de Boer, 1988). But to extract the phosphoplids a polar solvent like chloroform and methanol is needed (Bligh and Dyer, 1959, de Boer, 1988). To extract the PCBs from the phospholipids a mixture of a non-polar and polar solvent such as pentane and dichloromethane or hexane and acetone is needed. The first one is preferred because it results in less co-extraction and cleaner chromatograms.

3.5.3 Dioxin-like POPs

A total of 64 laboratories reported at least one result for a dl-POP in one of the test samples (and was assigned a z-score). For the individual matrices, the number of reporting laboratories was smaller since very often, the laboratories are specialized on either abiotic or biotic matrices. For the dioxin-like POPs, almost 3,000 satisfactory performance results have been generated in this interlaboratory assessment (see right column of Table 84). However, the regional distribution varies highly as can be seen in Table 84. The majority of the laboratories is located in the Asia and the WEOG regions. In these two regions, also the good performances can be found. It should be mentioned that especially in the GRULAC region the number of dioxin laboratories has increased and in this IL4 have achieved more than 250 satisfactory results. In the African region, the analytical capacity is still restricted to one laboratory. The performance of this African dioxin laboratory continues to be quite satisfactory.

Table 84: Regional distribution of laboratories submitting results for dl-POPs and number of satisfactory results for the dl-POPs

Region	# of Labs	# of S results (dl-POPs)
Africa	1	64
Asia	32	1411
CEE	2	111
GRULAC	8	251
WEOG	21	986
Grand Total	**64**	**2823**

All dl-POPs analysis was done with gas chromatographic systems; one laboratory used ECD as the detector. Among the mass spectrometric instrumentation, HRMS as sector-field instrument, is detector; corresponding to 78% to 87% of all detectors named. MS/MS systems were mentioned by 3-4 laboratories and one or two used LRMS instruments.

The most common extraction procedure was Soxhlet extraction; manual systems seemed to be used more frequently than automatic systems. A wide range of clean-up approaches was used with a majority for alumina and/or silica columns. Florisil clean-up was listed only once. The vast majority of the laboratories used internal labeled standards but two of them also used another native standard substance and two mentioned that they do not use a (labeled) internal standard.

The global picture across all test samples for PCDD/PCDF is shown in Figure 13 and for dl-PCB in Figure 14.

With respect to the PCDD/PCDF, the CV values were satisfactory for the test solution (CV = 10 for lower bound (LB) and CV=9 for upper bound (UB) (Table 22) and for the sediment sample (CV=21 (LB) and CV=20 (UB) (Table 24), both on WHO_{2005}-TEQ basis. For individual congeners, the CV values ranged from 8 to 28 for the test solution and from 17 to 110 for the sediment (Figure 13). The very high CV of 110 was obtained for 1,2,3,7,8,9-HxCDF. Also, for the air extract, satisfactory performance was achieved with CVs=16 for LB and UB for the WHO_{2005}-TEQ. The CVs for the congeners ranged 15-35 (Table 30).

The CVs expressed as WHO_{2005}-TEQ of the human milk (Table 28) and especially of the fish sample (Table 26), were unsatisfactory. For the WHO_{2005}-TEQs the CVs were CV=41 (LB) and CV=35 (UB) for human milk and CV=110 (LB) and CV=99 (UB) for the fish sample. The individual congeners had CVs in the ranges 23-149 for human milk and 89-259 for fish. The high CV of 259 was for 1,2,3,7,8,9-HxCDD; in principle, none of the CVs was in an acceptable range (lowest was CV=89 for 1,2,3,7,8-PnCDD.

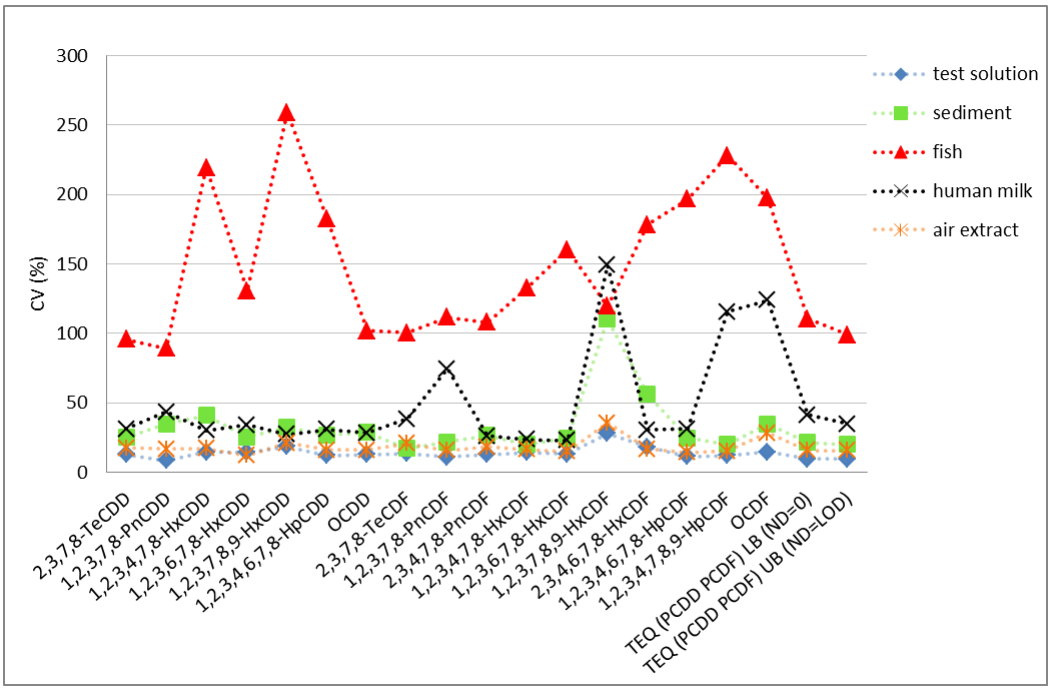

Figure 13: Performance of laboratories for analysis of PCDD/PCDF per congener and TEQ (as %CV).

With respect to the dl-PCB, the CV values on WHO_{2005}-TEQ basis were satisfactory for the test solution (CV for LB and CV=18 for UB) (Figure 14, Table 22). For the individual twelve congeners, the CV values ranged from 11 to 27 for the test solution. For the sediment (Table 24) and the human milk samples (Table 28), the CV values for the WHO_{2005}-TEQ were questionable with CV=32 for LB and CV=28 for UB for the sediment and CV=26 (LB) and CV=25 (UB) for the human milk sample. The CVs for individual congeners ranged from 13 to 62 for sediment and from 11 to 94 for human milk.

The CVs for for the WHO_{2005}-TEQ of the fish (Table 26) and the air extract (Table 20) were unsatisfactory with CV=116 (LB) and CV=115 (UB) for fish and CV= 52 (LB) and CV=38 (UB) for the air extract. The ranges for the individual congeners were 108-171 for fish and 12-53 for air extract.

In general, the higher CV values for the WHO_{2005}-TEQ are due to the higher weight of the non-ortho-PCB in the TEQ calculation.

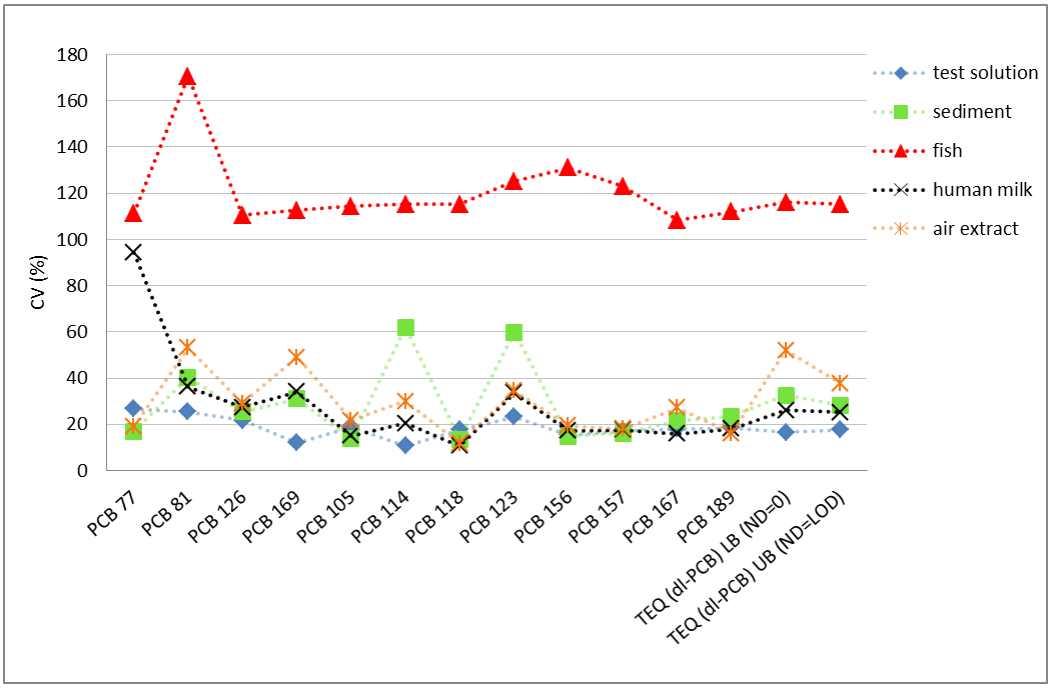

Figure 14: Performance of laboratories for analysis of dl-POPs per congener and TEQ (as %CV).

3.5.4 Polybrominated Diphenyl Ethers

The performance for the PBDE analyses was better than for the OCP and PCB analyses (Figure 1). For the test solution, 65% of the results were satisfactory with a model between lab CV of 22% on average (Table 32, Table 33). In this round PBDE 209 was included for the first time. Analysing this compound is more challenging than analysing the other PBDE. For PBDE 209 in the test solution 40% of the results were satisfactory with a CV of 50% (Figure 15, Table 32, Table 33).

The results for the PBDE in the air extract were relatively good, and comparable with the results of the test solution with between lab CV values of 9%-19%. The analyses of PBDE 209 was more challenging, with 38% of satisfactory results, and a between lab CV of 52% (Table 40, Table 41). For the sediment the PBDE concentrations were in the same range (0.1-1.6 ng/g) as in the third round, but the performance improved substantially from 19% satisfactory results in the third round to 63% in the present study (Table 35). Apart from a better performance of the laboratories, the sediment quality may have played a role as in the sediment of the previous round many interferences were present. Although sediment is a more difficult matrix to analyse than a test solution or an air extract, an acceptable 80% of the participants were able to obtain a satisfactory results on PBDE 209 in sediment (Figure 16).

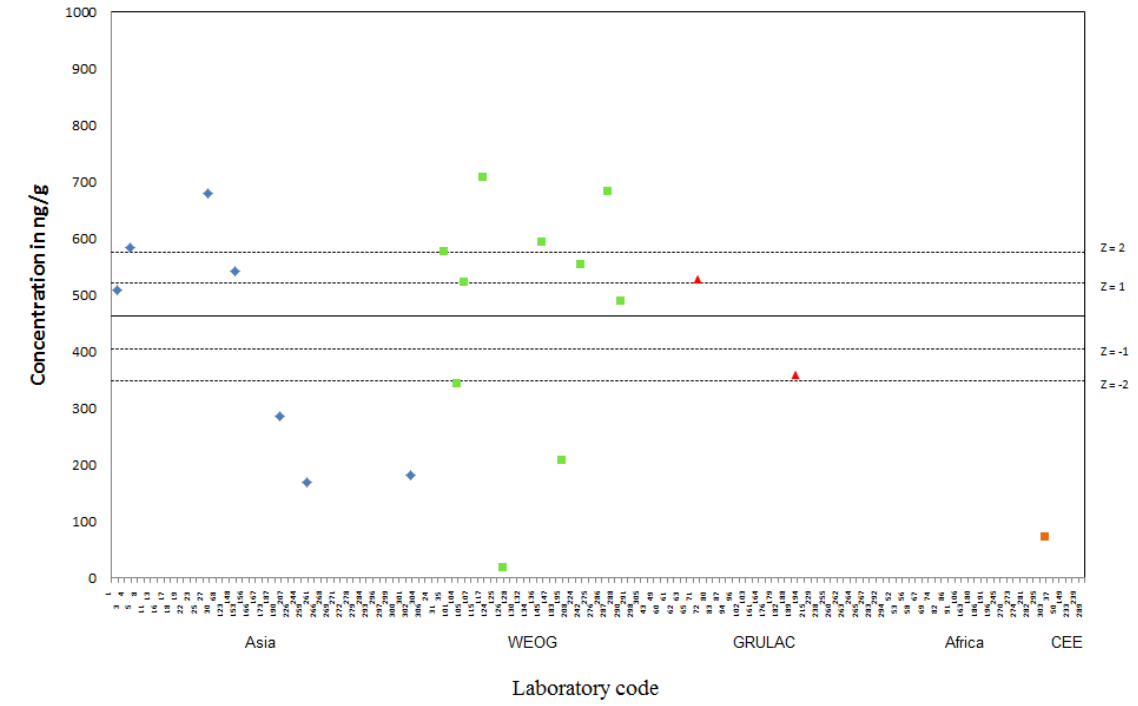

Figure 15 Results for PBDE 209 in the test solution.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

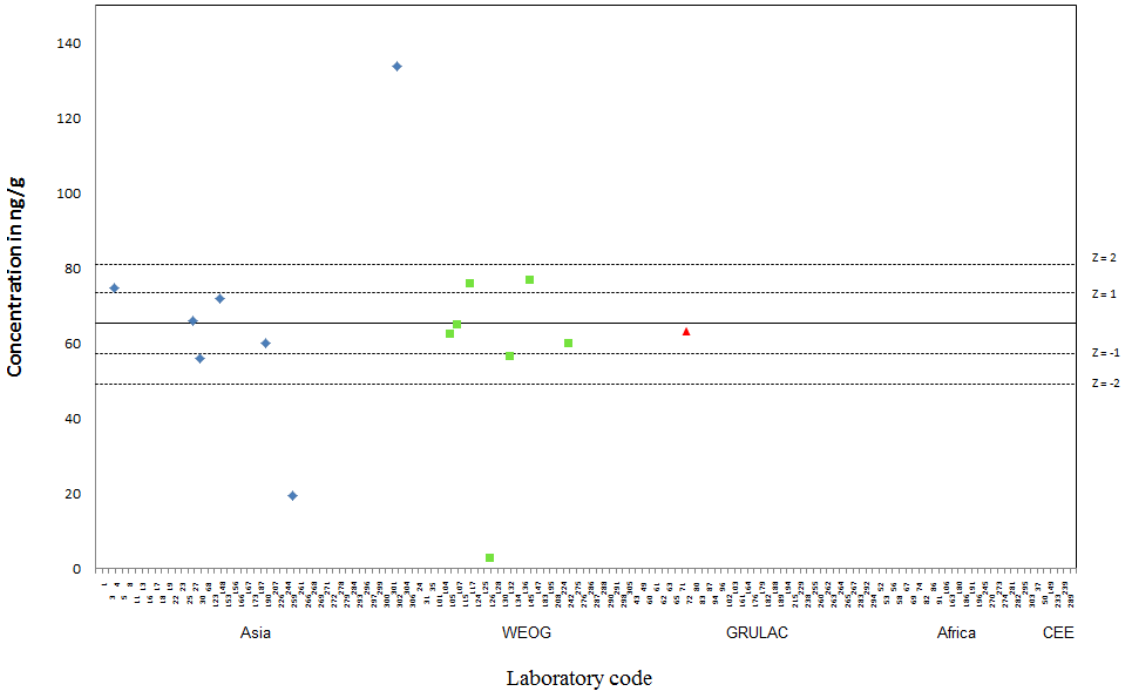

Figure 16 Results for PBDE 209 in the sediment sample.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

Concentrations of PBDE in the human milk test material, and in the fish sample were all on the low side. For the human milk, the model mean concentrations were 0.01-0.3 ng/g, which resulted in no assigned values for PBDE 17, 154 and 183. For PBDE 209 only six numerical results were reported with a mean concentration of 0.58 ng/g, and a model between lab CV of 146% (Table 48). For the other PBDE 62% of the data was satisfactory (Table 39).

For the fish sample, it was only possible to calculate an assigned value for PBDE 28, 153,154, and 209. Although most participants reported a numerical value, the concentrations (model means 0.0008-0.68 ng/g) were too low to get a good agreement between the reported results. For PBDE 28, 153, 154, and 209 an assigned value could be calculated even though the concentrations for those compounds were also low (0.02-0.06 ng/g), but this resulted in extreme high model between lab CVs of 97-155% (Figure 17, Table 36).

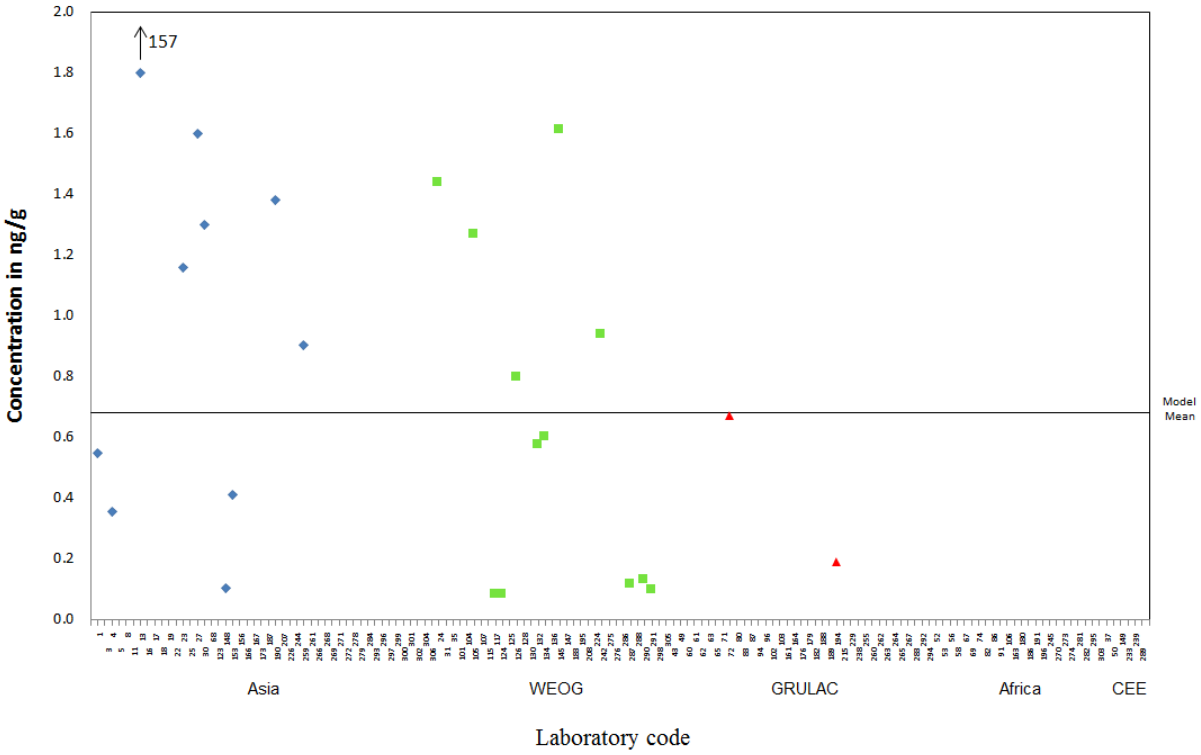

Figure 17 Results for PBDE 47 in the fish sample.

Laboratory code on the x-axis, concentration in ng/kg on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

Overall, the results on the PBDE were relatively good (Figure 18), except for the fish sample in which the concentrations were too low to get a good agreement, and for some of the PBDE with very low concentrations in the human milk sample. Unfortunately, no data at all were reported by African and CEE laboratories, although some of them have been trained in this type of analysis.

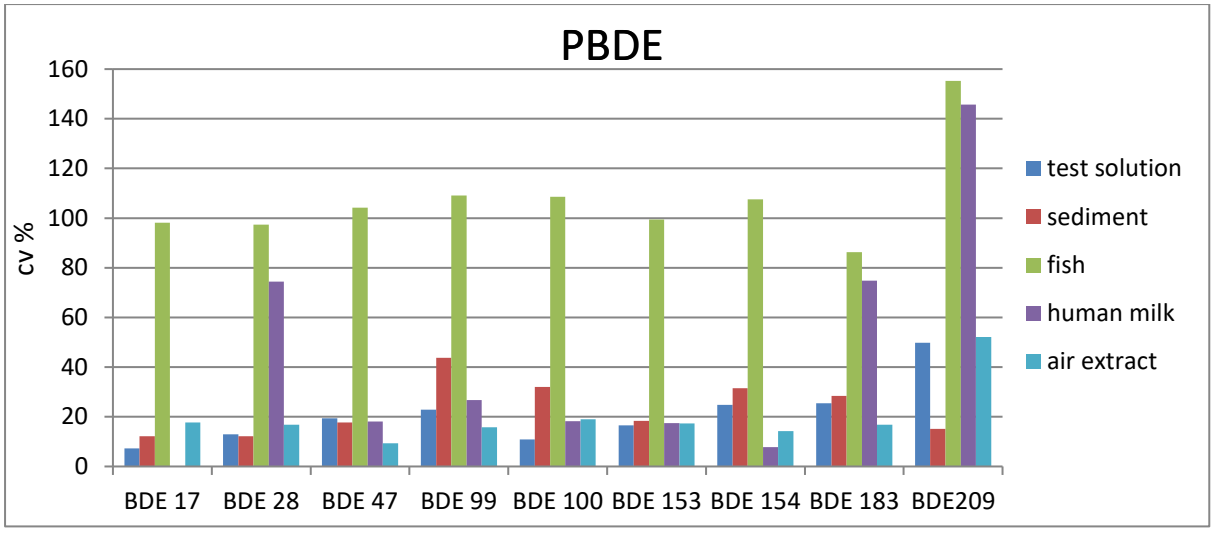

Figure 18 Performances per matrix for the analyses of PBDE.

3.5.5 Hexabromobiphenyl

In the third round, PBB 153 could be analysed in two different solutions. The first solution contained PBB 153 (696 ng/g) together with the PBDE. In the other solution, PBB 153 was provided as the sole compound (11.3 ng/g). In the present study one solution (V) was provided, containing PBB 153 (73.8 ng/g) together with the PBDE (Table 32). Ten participants submitted results, of which eight obtained a satisfactory score (Figure 19, Table 33).

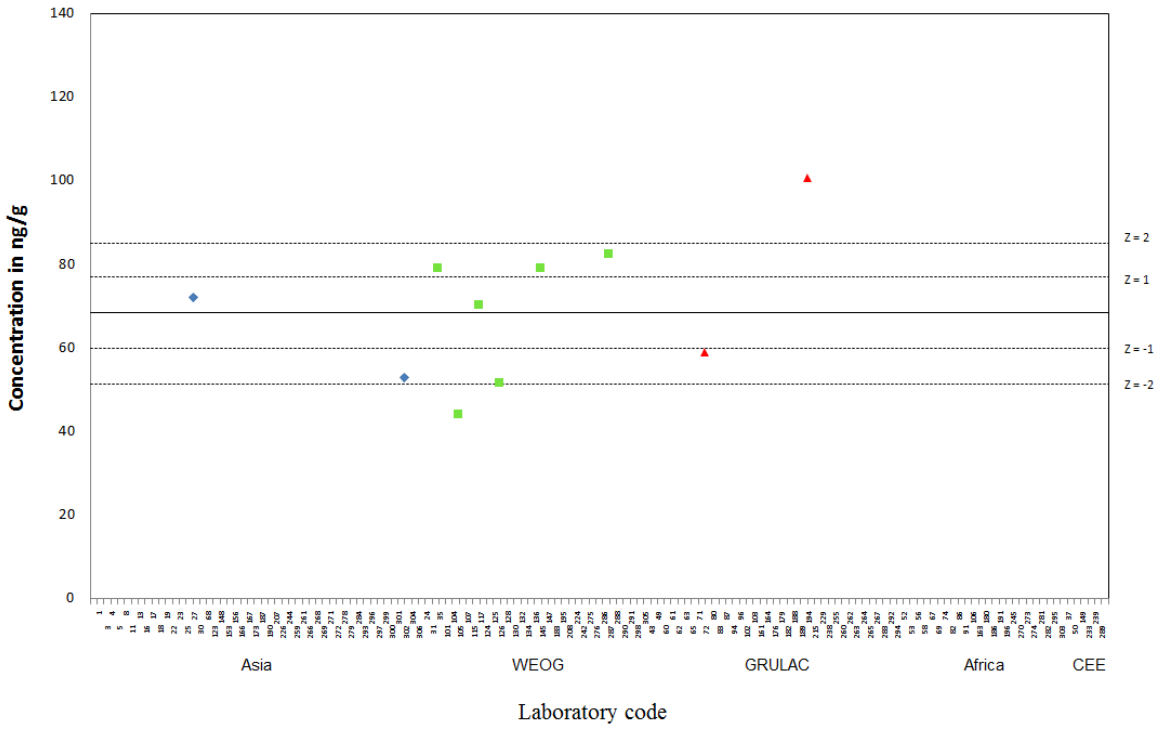

Figure 19 Results for PBB 153 in test solution V.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value is given by a straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

Even though the concentrations of PBB 153 in the air extract are more than 50 fold lower than in the test solution, the performance was better, with a model between-lab CV of 3.5%, and 89% of the results being satisfactory (Figure 20, Table 40, Table 41).

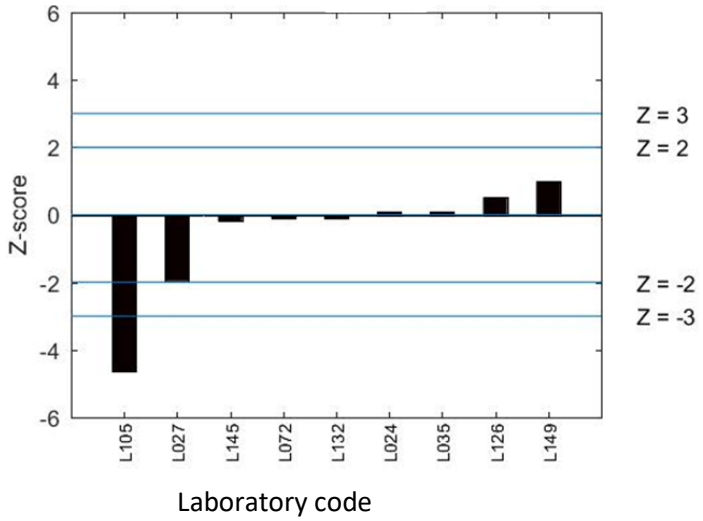

Figure 20 Z-scores obtained for PBB 153 in the air extract.
Laboratory code on the x-axis, Z-score on the y-axis.

The concentration of PBB 153 in the sediment sample was low (model mean 0.03 ng/g), but still 8 out of 12 submitted results were > LCV (Table 34). Despite the low concentration and the difficult matrix, all laboratories except one were able to obtain a satisfactory z-score. The concentration of PBB 153 in the fish was even lower (0.016 ng/g), which resulted in six laboratories reporting a value < LCV, and seven participants submitting a numerical value (Table 36). All of those numerical results were satisfactory (Figure 21).

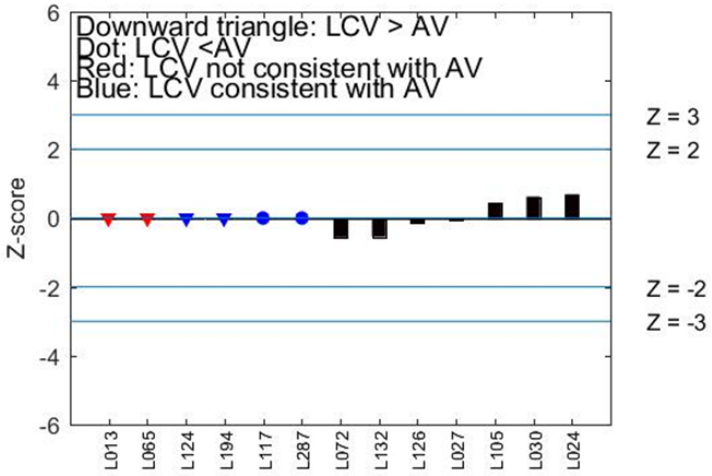

Figure 21 Z-scores obtained for PBB 153 in the fish sample.
Laboratory code on the x-axis, Z-score on the y-axis.

Unfortunately, the concentration of PBB 153 in the human milk sample was so low that only two participants were able to report a numerical value (Table 38).

Overall, the results on PBB 153 in this round were satisfactory. In this round all of the participants analysing PBB 153 reported to have used an MS method (LR-MS: n=4, HR-MS: n= 11, MS/MS: n=3), which might have resulted in a better agreement between the laboratories.

3.5.6 Toxaphenes

In the third round of the study toxaphenes were included for the first time. In that round 14 laboratories analysed the test solution, with a good agreement. The individual results for the toxaphenes showed between-lab CV values of 11%-26%, and 83% of the participants received satisfactory z-scores. Theoretical concentrations in the test solution were relatively high compared to environmental concentrations (Parlar 26, 97.7 ng/g; Parlar 50, 139 ng/g; Parlar 62, 100 ng/g), which might have contributed to the good agreement. In the present round the concentrations were a little lower for Parlar 26 (41 ng/g), and Parlar 50 (56 ng/g), and equal for Parlar 62 (101 ng/g) (Table 42). Ten participants submitted results on the test solution. The results of one of the participants was unsatisfactory for the analyses of all three toxaphenes, and the results of one other participant was unsatisfactory for the analyses of Parlar 50 and 62 (Figure 22). Also, in this round the results are all in good agreement, with low model between lab CV% (Parlar 26, 13%; Parlar 50, 9.6%; Parlar 62, 12%).

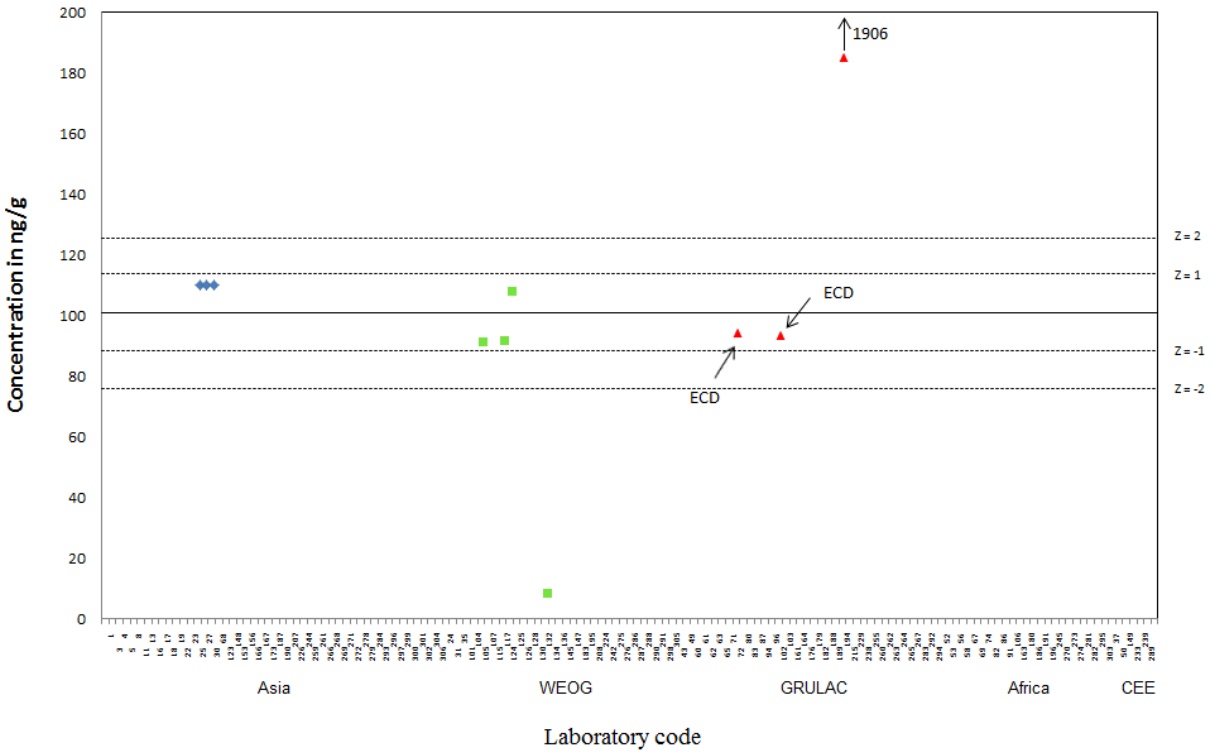

Figure 22 Results for Parlar 62 in the test solution.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

In the third round, only one participant reported to have used ECD for the analyses of toxaphenes. Concentrations of toxaphene reported by that participant were much lower than concentrations reported by other participants. Since this was only one result, it was not possible to draw any conclusions in that round. In the present round all 10 participants reported which detection system they used. An MS method was used by the majority (LR-MS (n=2), TOF-MS (n=2), HR-MS (n=2), MS/MS

(n=2) ECD was used by two of the participants (Figure 22). This time no difference in results is observed between the use of an ECD and MS detection systems.

Although it is preferred to use naturally contaminated samples for an intercomparison study, it is of no use to send a material which is so low in contamination that no participant will be able to analyse it above LCV. For this reason, the fish sample in the present study was fortified with Parlar 26, 50 and 62 (Table 46). Nine participants analysed this fish test material, of which six were able to report a numerical value for Parlar 26 and Parlar 50, and five were able to analyse Parlar 62 > LCV (Table 46). Mean concentrations reported were 0.50- 0.63 ng/g, which is 60-200 fold lower than concentrations in the test solution. This resulted in CV values of 38-71 (Figure 23, Table 46). Unfortunately, it was not possible to calculate an assigned value (category 2, see section 2.2). Yet, the results are really promising.

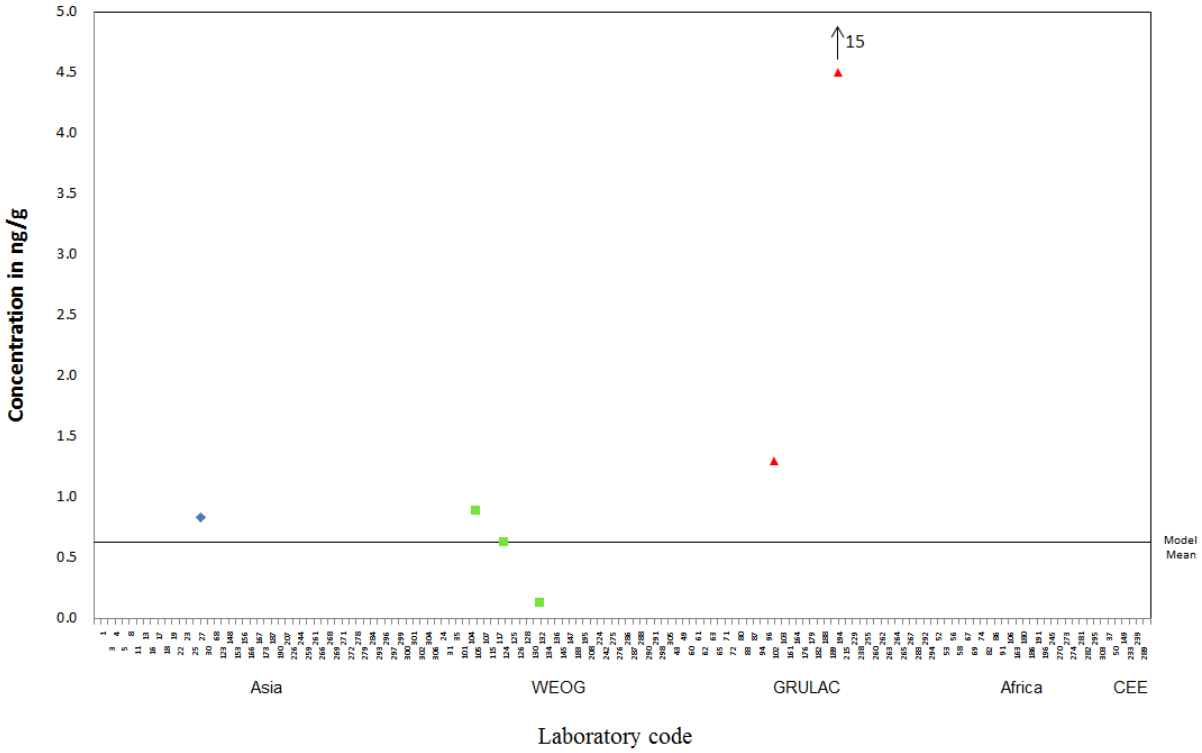

Figure 23 Results for the toxaphene congener Parlar 50 in the fish sample.
Laboratory code on the x-axis, concentration in ng/g on the y-axis. The model mean value is given by the straight line, The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

For the unspiked sediment sample (Table 44), and for the spiked air extract (Table 50), only one or two participants reported a numerical value. For the human milk sample only four participants were able to report a numerical value for Parlar 26 and Parlar 50. No result > LCV was submitted for Parlar 62 in the human milk (Table 48). As a result, no assigned value could be calculated.

3.5.7 Hexabromocyclododecane

HBCDs were included in the study for the second time this round. The performance for HBCDs in the test solution was already good the first time the isomers were included, with an average of 81% of the participants receiving a satisfactory z-score. In the present round the performance on the test solution was even better with an average of 87% satisfactory results, and an average between-lab model CV value of 13% (Figure 24, Table 52, Table 53).

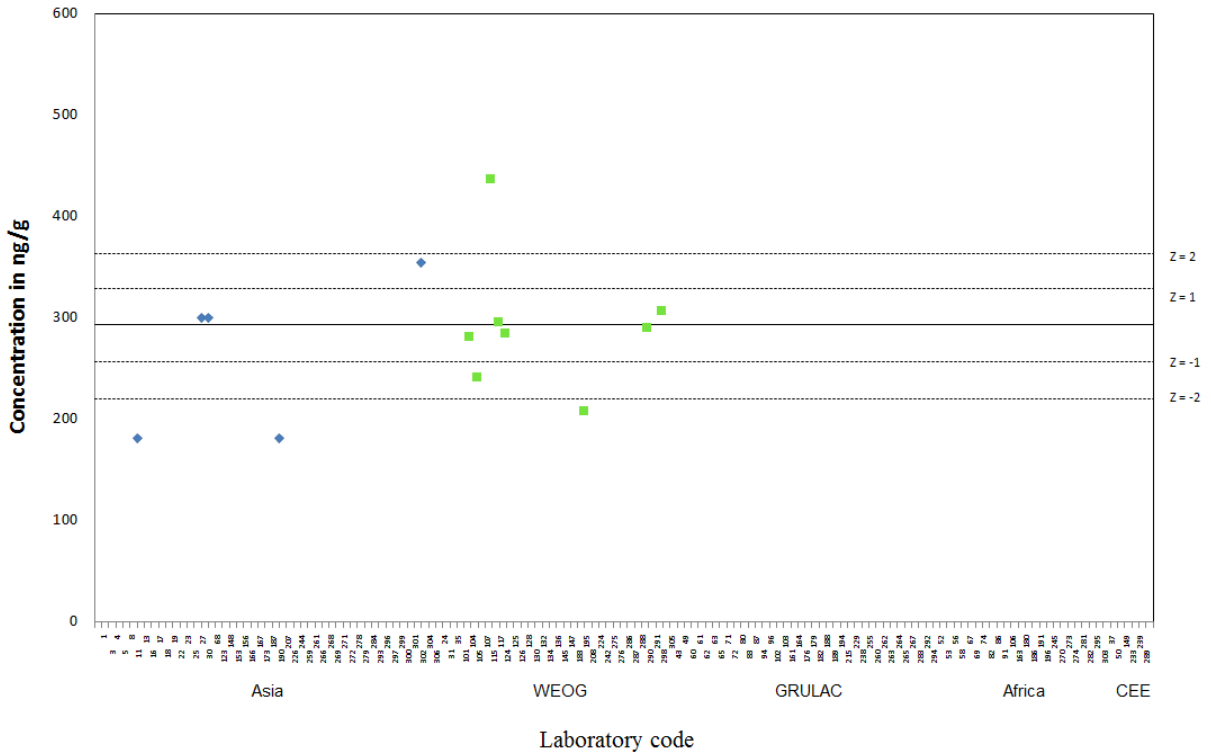

Figure 24 Results for γ-HBCD in the test solution.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

Only six participants handed in results for HBCDs in the air extract, of which four reported numerical values. Those results were in very good agreement with a CV of 11-14% for the individual isomers.

The most relevant isomer for fish is α-HBCD. Although in the fish sample this compound was present in a very low concentration (model mean: 0.03 ng/g), still seven participants out of nine were able to report a numerical value of which only one result was not satisfactory.

Model between lab CV values for the isomers of HBCD in the sediment sample (11-36%) were lower than in the third round (36-91%), but too few participants reported a numerical value (n=6) with too little agreement to be able to calculate a assigned value (category 2, see section 2.2). Detailed information for γ-HBCD in the sediment sample is shown in the 'kilt' plots in Figure 25.

Unfortunately, for the human milk sample it was not possible to calculate assigned value due to low participation degree.

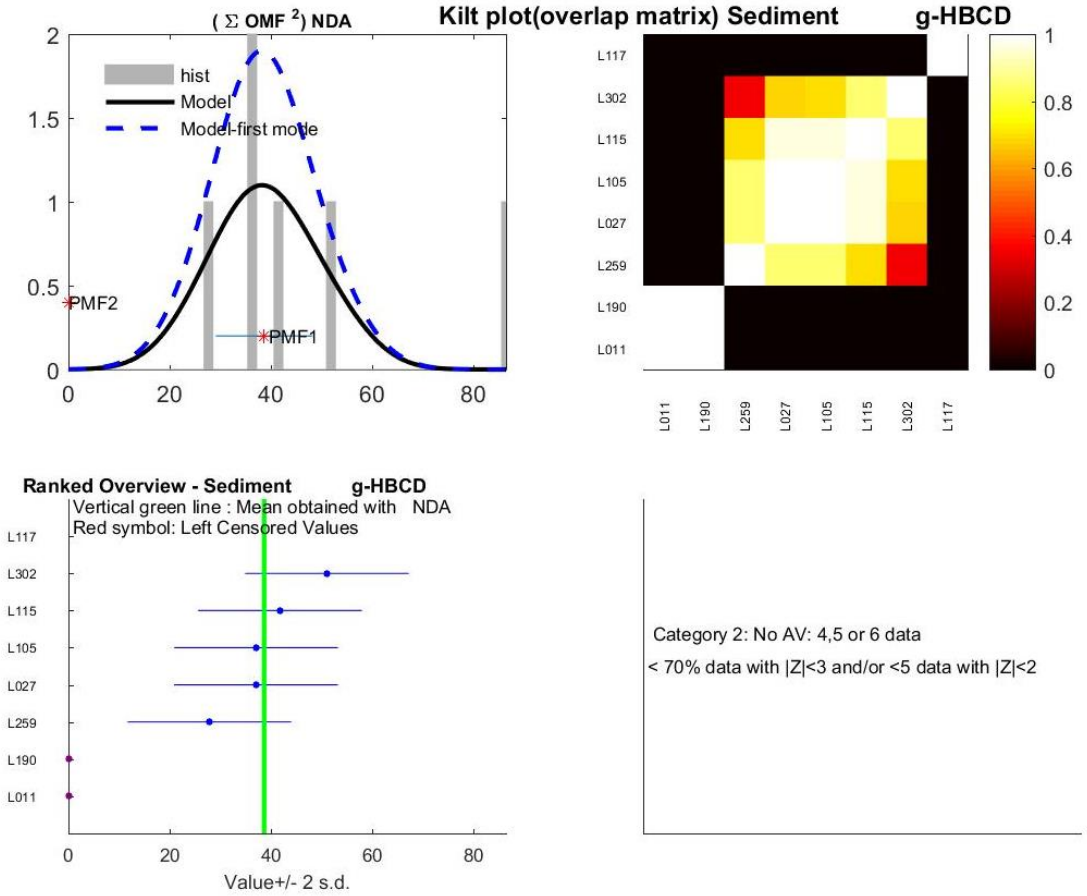

Figure 25 Plots detailed information: γ-HBCD in sediment

Overall, the performance on the diastereomers of HBCD was good, except for the test solution. The number of participants was low, especially considering the fact that the isomers of HBCD are listed at the monitoring list of the Stockholm Convention (UNEP, 2013, 2019c).

3.5.8 Perfluoroalkyl Substances

In total, there were 28 PFAS determinands for test solution, air and human plasma and 21 for human milk, water, and sediments (without the PFOS precursors and their sums). For sum parameters, upper-bound (UB) and lower-bound (LB) values were to be reported. For LB values, all values below the limit of detection (LOD) were set zero (<LOD=0) to calculate the sum of the analytes (referred to as 'tot-PFAS class); likewise, for UB values, the values below the limit of detection (LOD) were set at the LOD to calculate the sum of the analytes (<LOD=LOD).

A total of 39 laboratories submitted results and for 1869 datasets (PFAS compound and matrix) z-scores could be assigned (Table 85). Of the z-scores, 1228 were satisfactory corresponding to 66% of all z-scores for PFAS. 328 or 18% were unsatisfactory and 174 or 9% were questionable. 3% (corresponding to 63 z-scores) and 4% (corresponding to 76 z-scores) had insufficient statistical power and results corresponded to C (consistent) and I (inconsistent), respectively. The highest number of z-scores (>100) were for laboratories that analysed abiotic and biotic matrices; *i.e.* L126 (115), L107 (113), L027 (109), and L105 (103). Three laboratories had only unsatisfactory results.

Table 85: Summary of performance of the 39 laboratories submitting results for PFAS (all PFAS and all matrices included)

z-sore interpretation	#S	#Q	#U	#C	#I	#Res
Number of z-scores	1228	174	328	63	76	1869
Percentage	66%	9%	18%	3%	4%	100%

The distribution of PFAS laboratories and the number of satisfactory results ("S") in terms of z-score interpretation according to region is shown in Table 86.

Table 86: Number of laboratories and number of satisfactory results per region for PFAS

Region	No of Labs	No of S results (PFAS)
Africa		
Asia	11	331
CEE	2	37
GRULAC	1	57
WEOG	25	803
Grand Total	**39**	**1228**

All participating laboratories used in-house methods for sample preparation, clean-up, extraction and instrumental analysis. It shall be noted that not all laboratories provided information on their methods according to the reporting format. Unfortunately, no systematic information could be obtained as to digestion steps applied before extraction. One laboratory reported acid digestion and one laboratory sonication for sediment and fish. Manual extraction was much more used than automated systems (88.4% vs. 11.6%). Methanol was the most frequently used solvent (70%), acetonitrile was used in 8.8% of the samples. 103 of the samples (or 55.1%) were cleaned-up with SPE and 39 (or 20.9%) with LLE; only one laboratory (0.5%) used QuEChERS in one sample. 43.6% of the laboratories reported use of an extra column (isolator column) whereas 56.4% did not use such column.

The **test solution** contained L- and br-PFOS, five precursor compounds, 11 carboxylic acids, three sulfonic acids (without PFOS) and one fluorotelomer sulfonic acid; thus, a total of 22 compounds to be reported together with three sum parameters, each of them for lower-bound (LB) and upper bound (UB) (Table 62). Up to 29 laboratories reported values and none of them was left-censored. Assigned values could be calculated for 27 of the 28 parameters. Only for the LB of the five precursors, no AV could be calculated. The theoretical value for the sum of these five was 631 ng/g; the coefficient of variation (CV) between laboratories was 49%. For the UB value, the CV was very narrow with 4%.

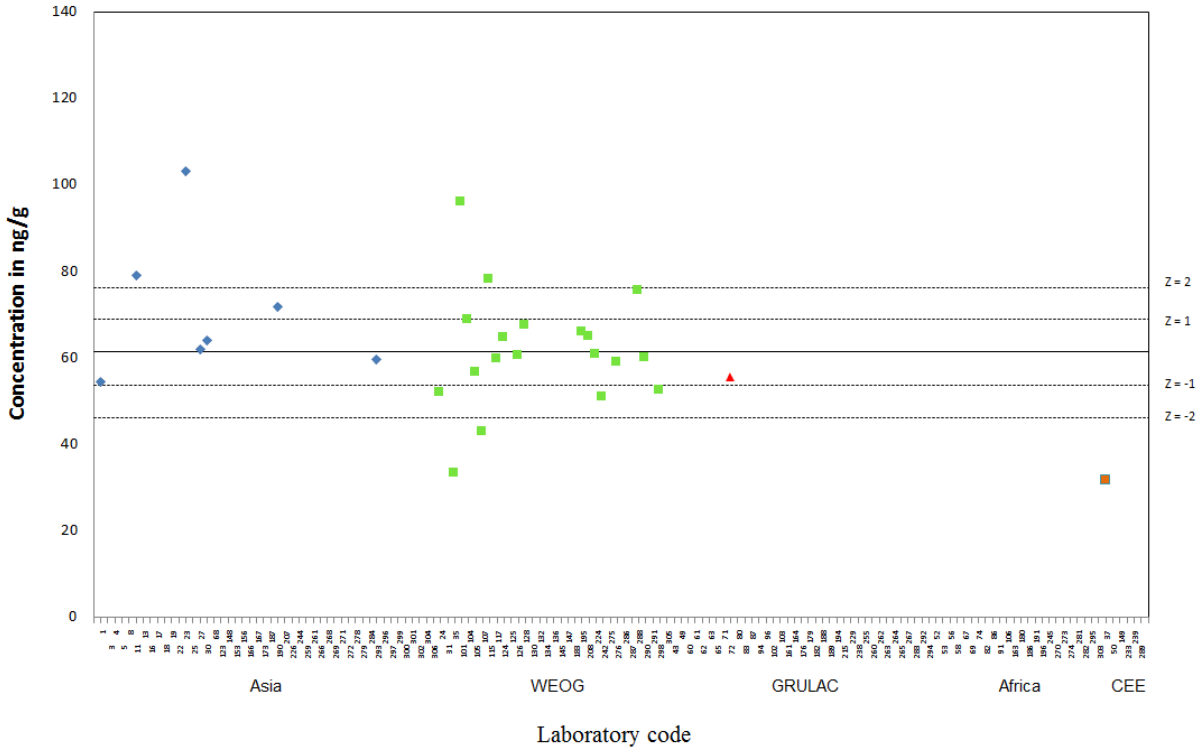

Figure 26 Results for L-PFOS anion in the test solution.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

About 21 laboratories submitted results for the **fish sample** (Table 66). No consensus values could be calculated for five carboxylic acids and two sulfonic acids; the high number of laboratories with LCV should be noted, indicating that the concentrations were very low. Of the 22 laboratories, no consensus value could be assigned for PFOA and the CV was very high (174%); for PFHxS, the CV was also quite high (167%) but the statistical power was sufficient to calculate an AV. The CVs for L-PFOS and the tot-PFOS were excellent (between 11 and 16); only for br-PFOS the CV was higher with 32% (but still acceptable). It is interesting to note that br-PFOS could be quantified at an AV of 0.52 ng/g.

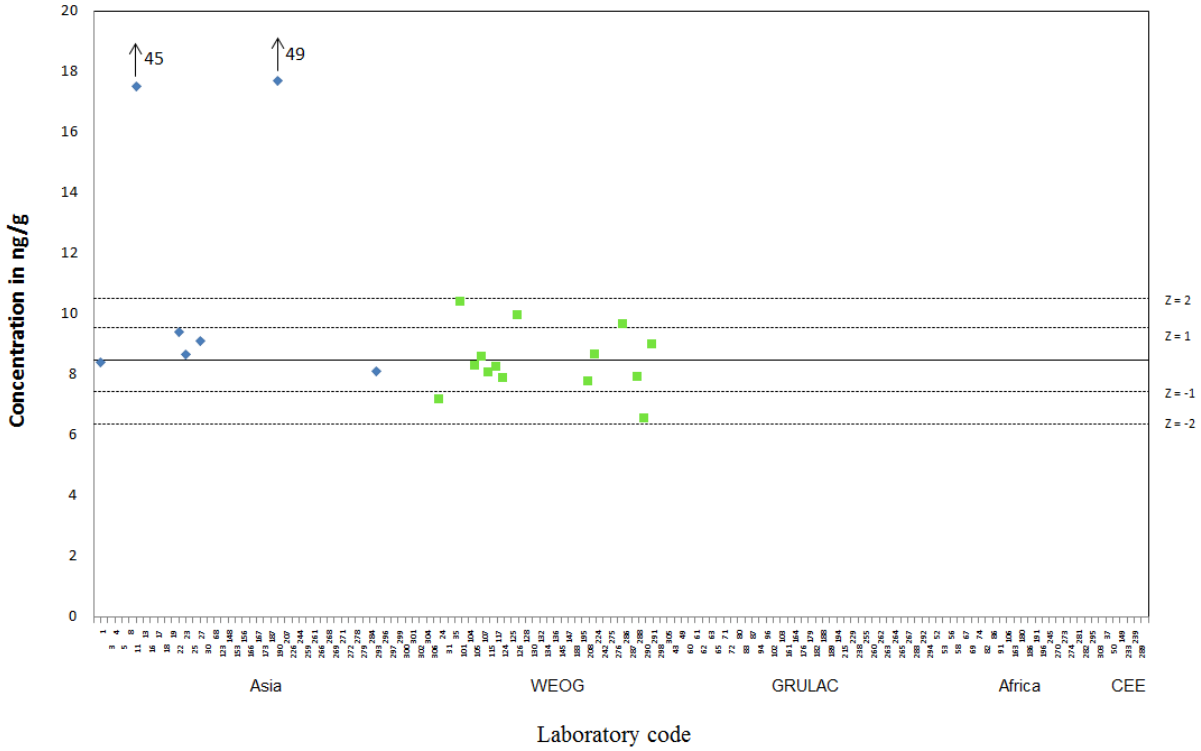

Figure 27 Results for L-PFOS anion in the fish sample.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ♦ symbols represent Asia, the green ■ symbols represent WEOG, the red ▲ symbols represent GRULAC, the black ● symbols represent Africa and the orange ■ symbols represent CEE.

The global picture of laboratory´s performance in PFAS analysis – as percentage variation of the CV – is shown in Figure 28. It shall be noted that the broader spectrum of PFAS has been analysed for the test solution of analytical standards and human plasma only. For water, sediment, fish and human milk only the linear and branched PFOS isomers and their sum were requested. The air test sample included the precursor FOSAs and FOSEs. As can be seen, the human milk sample posed some problems to the laboratories; possibly due to the low concentrations (20 pg/g wet weight for L-PFOS) (Table 68). For most of the analytes, it was not possible to calculate an AV. For the congeners where an AV could be assigned, the CVs were quite high (from 38% for PFOA to more than 100 for br-PFOS, tot-PFOS UB, and PFHxS). Even higher CVs were obtained for PFNA (CV=611) and PFUnDA (CV=231).

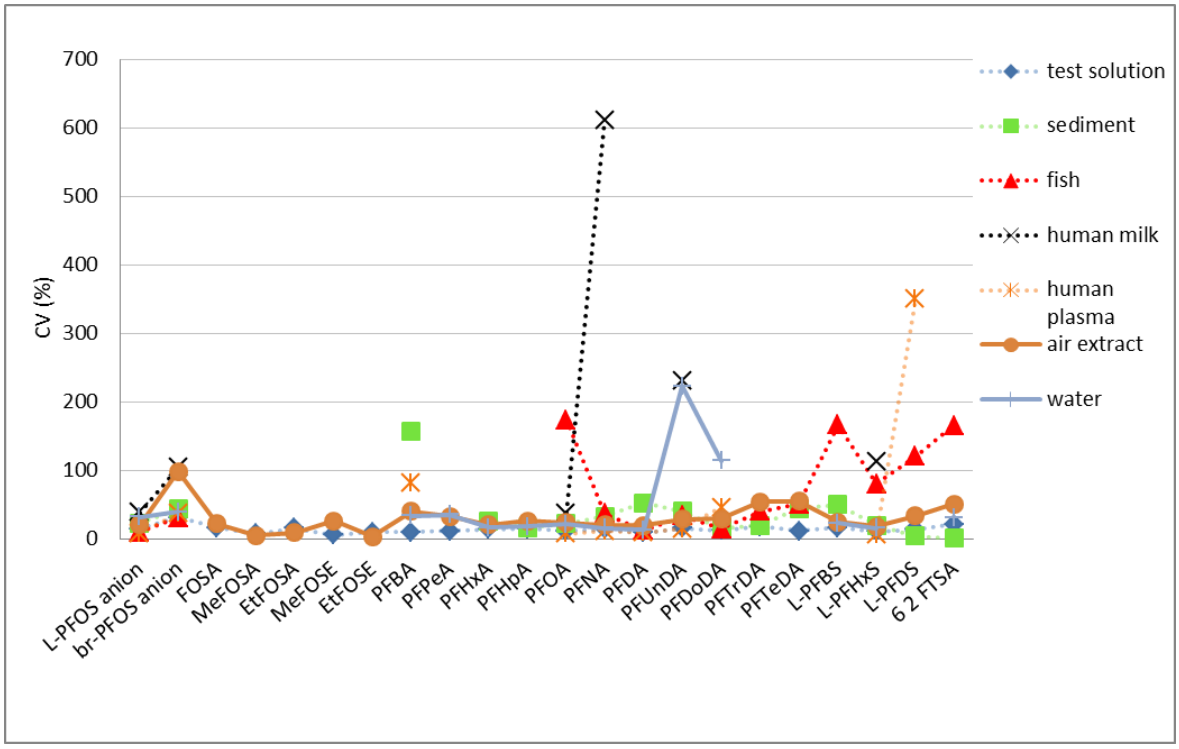

Figure 28: Performance of laboratories for PFAS according to sample type (as CV).

4 COMPARISON WITH THE PREVIOUS ROUNDS OF UNEP'S INTERLABORATORY ASSESSMENT

The present study was the fourth round of the interlaboratory assessment on POPs organized by UNEP. In the first assessment, test solutions, sediment, fish, human milk, and fly ash were tested for OCP, PCB and dl-POPs. In the second round two additional compound classes were added, e.g. PBDE and PFASs. Transformer oil was included in the second round for the analyses of PCB only, and water and human serum were included for PFASs analyses. Fly ash was not included anymore. In the third assessment transformer oil was excluded from the study. Toxaphene, HBCD, and HxBB were added. In the present study the same matrices, and compound classes as in the third round could be analysed again.

Table 87 shows the degree of laboratory participation *per* compound class and matrix in this IL4. Only laboratories are listed for POPs groups where z-scores could be assigned. It is striking, that for example the number of laboratories for PCB in fish is "0", due to the fact that no z-score could be assigned for any or the six congeners nor the sum of PCB.

For all POPs groups, the number of participating laboratories with z-scores was lower than in the previous third Round (IL3), which had a record-high number of participating laboratories. Only for PFASs, a slight increase was observed (25 in IL3 and 29 in IL4). Clearly, the analysis of HxBB, toxaphene and HBCD is still low for many participants. Dioxin laboratories were fewer than in previous rounds (41 in IL4). For PFASs the number of participants is similar to the number of laboratories for PBDE, although the number of laboratories for PFASs increased slightly whereas the number of laboratories for PBDE decreased. The number of basic POPs laboratories (analyzing OCPs or indicator PCB) is 57 and 56, respectively.

As can be seen from Table 87, there is still a large number of laboratories where z-scores could be assigned to test solutions only. For all matrices except water and human plasma, the numbers are comparable with up to 36 to 40 laboratories.

Table 87: Number of laboratories that reported results (and z/score assigned) per compound class (maximum number of labs is given).

POP group/ Test sample	Test Solutions	Sediment	Fish	Air	Human Milk	Human Plasma	Water
OCP	57	37	28	22	21		
PCB	56	40	0	36	37		
dl-POPs	41	35	38	34	24		
PBDE	28	22	25	22	13		
HxBB	10	12	13	9	0		
HBCD	13	0	9	6	0		
PFAS	29	13	25	18	15	16	20
Toxaphene	10	0	9	0	0		
Maximum	**57**	**40**	**38**	**36**	**37**	**16**	**20**

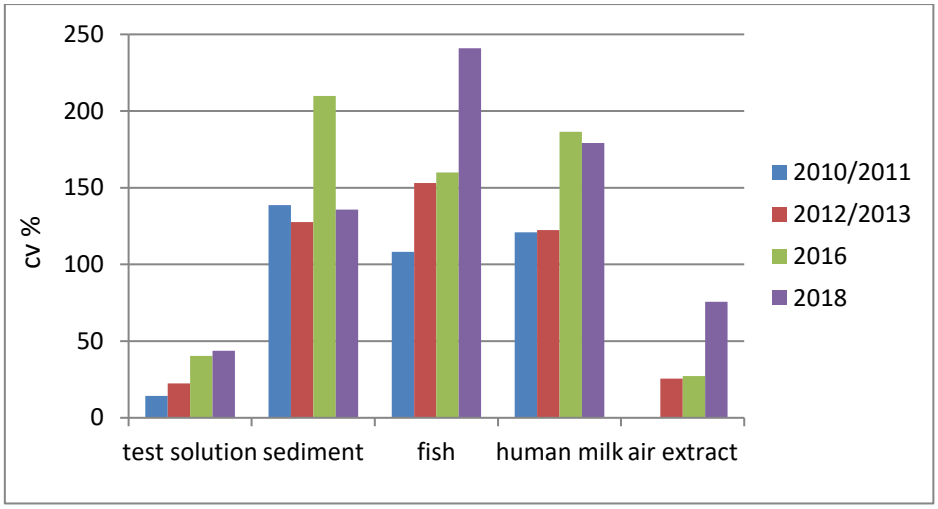

Figure 29 Comparison of performances between interlaboratory assessments for the OCP analyses (for OCPs determined in all rounds).

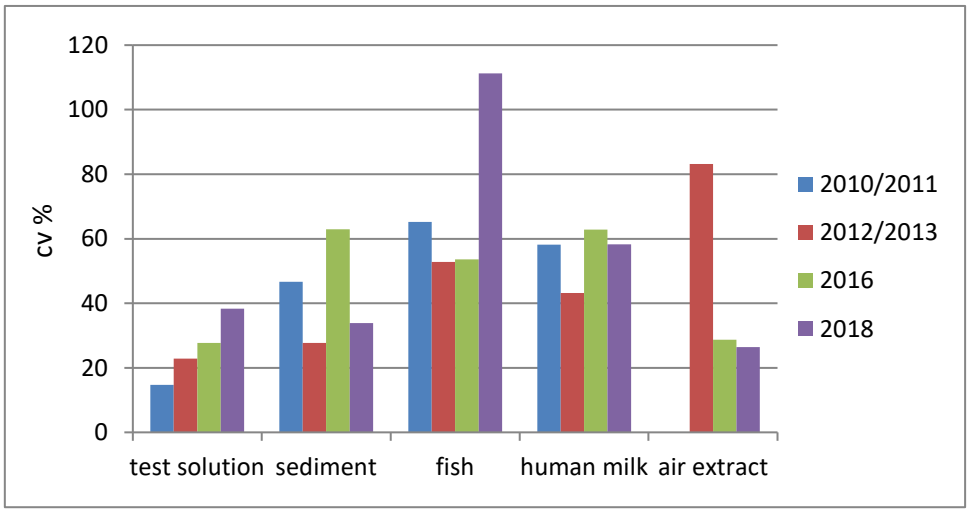

Figure 30 Comparison of performances between interlaboratory assessments for the indicator PCB analyses.

The performance for OCPs in the test solution went significantly backwards in the third round compared to the first two assessments, and in the fourth round the performance was even worse (Figure 29). Also, the performance of PCBs in the test solution went again backwards (Figure 30). It should be expected that all participants would be able to analyse a test solution with a good agreement, since no matrix is present. In the third round a lot of new participants were included in the study, and in the fourth round again 46 laboratories participated for the first time. It could be that some of those new participants are less familiar with the setup of the UNEP interlaboratory study, what might cause a bigger variance in the results. One of the issues not all participants are aware of, although it is clearly requested in all of the documents, is that the results on the test solutions should be reported on mass base, and not on volume base. Since test solutions have to be analyses by laboratories located all over the world, with all different climates, temperatures and pressures, reporting a test solution on volume base, would create an error due to differences in density, while expressing the results on weight base would give a more solid result. Reporting results on volume base instead of reporting on weight base might have resulted in a lower assigned value, a lower z-score for labs who reported their result on volume base, and a higher z-score for laboratories who reported their result on weight base.

The performance on the sediment sample is more or less stable for the OCP analyses, except for the results obtained in the third round, which might have to do with the very polluted location the sediment sample originated from in that assessment. Sediment is a difficult matrix for OCP analyses. A clean-up is required, but not all OCPs are stable for sulphuric acid treatment. It has been observed that it does make a difference which detection method has been used for OCP analyses (section 3.5.1, Figure 6, Figure 7).

For the other matrices, the fish, the human milk, and the air extract, the variance between the laboratories increased for OCP analyses compared to the first two assessments. The fish sample contained low concentrations of POPs, which might be a challenge to laboratories, however why the performance on the human milk, and on the air extract are decreasing for OCPs is not clear.

17 laboratories participated on the analyses of OCPs and 19 laboratories on the analyses of PCB in all four rounds. Trends in individual results of those laboratories over the four laboratories cannot clearly be detected. As an example the z-scores obtained by participant L103 for OCPs in the test solution in the four rounds are shown in Figure 31, and the z-scores of participant L030 obtained for PCB in the human milk sample in the four rounds are shown in Figure 32.

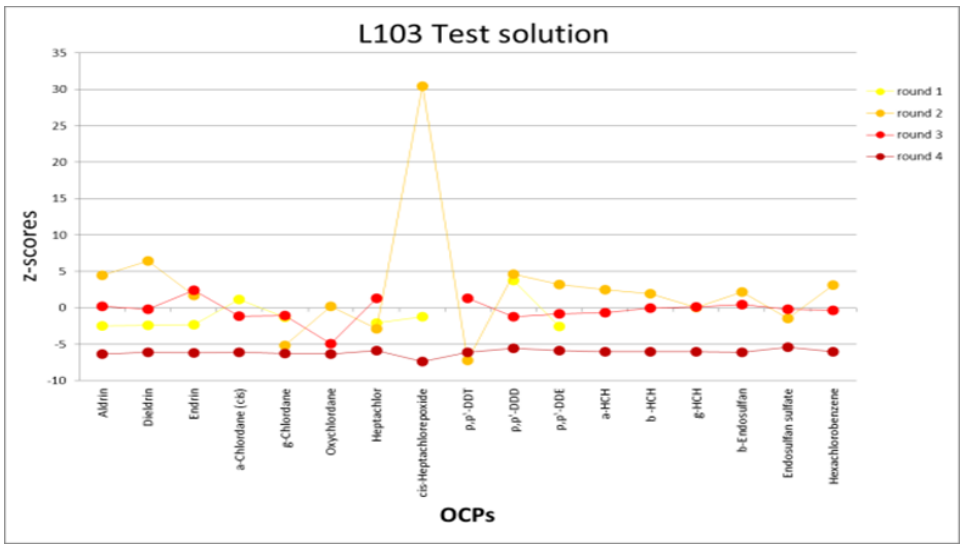

Figure 31 z-scores obtained by L103 for OCP analyses in the test solutions in four rounds of the UNEP ILS.

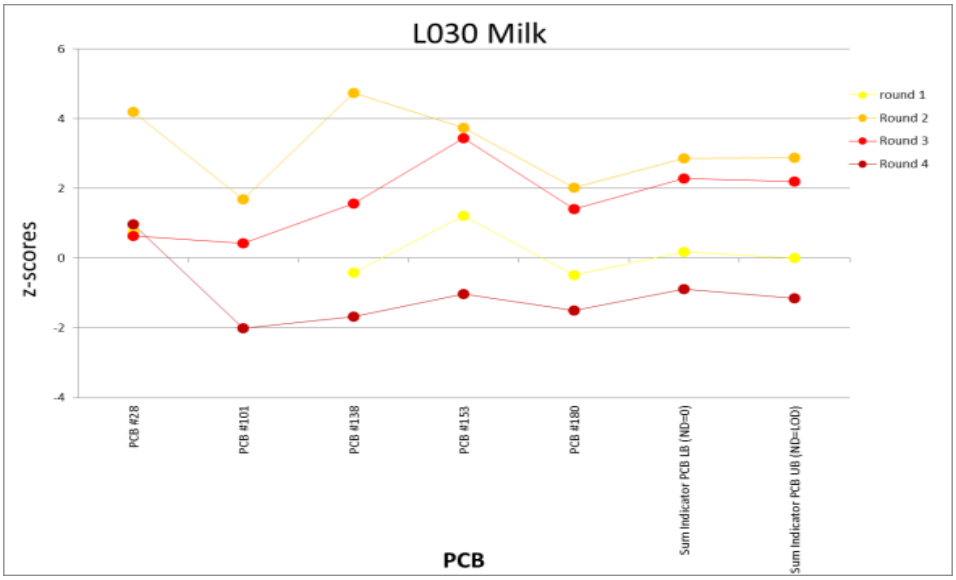

Figure 32 z-scores obtained by L030 for PCB analyses in the human milk sample in four rounds of the UNEP ILS.

Although no trends can be indicated in the results of individual laboratories, it is important that laboratories asses their own results per round. In case of unsatisfactory or questionable z-scores participants should try to identify the cause of the deviation, to be able to optimize their analyses and to be able to keep their performance on OCP analyses good.

The performance for the PBDE analyses in the third round was generally better than in the second round, except for the sediment sample (Figure 33). In this round the performance on the sediment sample was much better, and the CV% was even lower than in the second round of the study. The human milk appeared to be a difficult matrix for PBDE analyses in the second round. In the third round the performance improved, and in this round it improved even more. The performance on the test solution and on the air extract stayed the same compared to the third round, when the performance was already good.

Overall the performance on the PBDE analyses was relatively good, except for the fish sample, like also for the OCP and PCB analyses, which might be due to the low concentrations present in the fish.

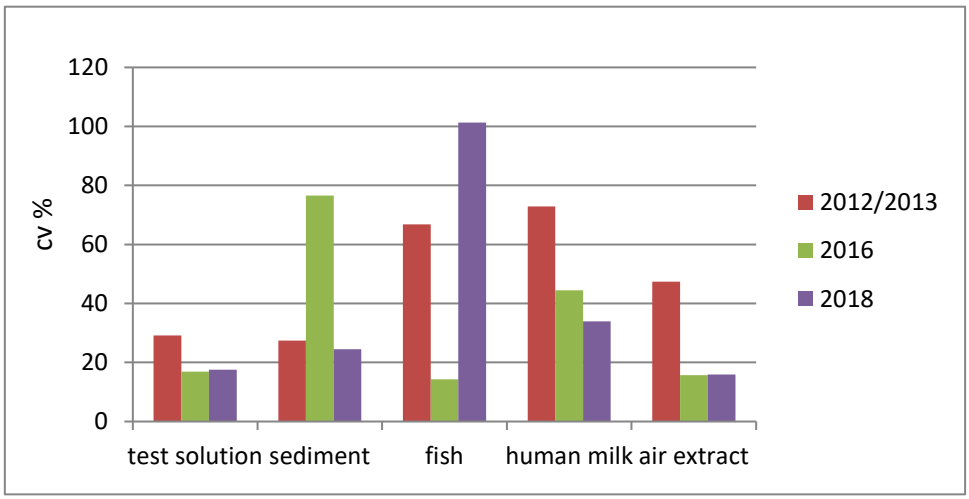

Figure 33 Comparison of performances between interlaboratory assessments for the PBDE analyses.

5 CONCLUSIONS AND RECOMMENDATIONS

5.1 Technical Conclusions

Again, a number of new laboratories entered this study. It is encouraging to see that many laboratories show an interest in this type of studies and apparently are also working on setting up methods and improving their performance. However, for the comparison with previous exercises, this is a handicap. The new participants are less experienced. For a number of matrices a poorer performance of the newcomers could be identified. Obviously, this has a negative impact of the overall results.

One of the main reasons of the flawed results is the lack of routine in many laboratories. A substantial number of laboratories even only carry out POP analyses for this interlaboratory study without further analyses in the rest of the year. Such a situation will never lead to satisfactory results. Daily or at least weekly or monthly experience in the analysis of POPs is essential to produce reliable results. We therefore recommend restricting this exercise to those laboratories that truly report data to the Global Monitoring Plan or can demonstrate that regular analyses in their laboratory are carried out.

Many participants report results for only one or maybe two analyte groups or only one or two matrices. The statistical evaluation is hampered by lower participation numbers. In some cases the assigned values could not be established or were established but based on a rather low number of laboratory results. To prevent errors in the assigned values, we plan to raise the minimum number of numerical observations to 10 or higher (now 7, see 2.4) for the next round.

Clearly, OCP results produced by GC/ECD are of lower quality that those based on GC/MS. Negative peaks present in the second fraction of the extracts after clean up cause serious errors in the determination and labelled standards cannot be used to correct for that.

OCP, PCB and PBDE results in the fish test material were disappointing. Many laboratories apparently struggle when the fat content of a fish sample is very low such as in this case for the pike perch. Yet, many fishes have low fat contents of around 1 %. More in general it can be observed that many laboratories struggle when concentrations are relatively low in test materials. The air extract analysis resulted as in the previous round in better results, which was at least partly due to fortifying of the extract.

Although overall the performance of the dioxin laboratories remained to be stable and quite impressive, in comparison to the three previous assessments, the results for dioxin-like POPs were not as good as before. Especially the fish matrix but also the human milk test sample generated higher CV values than before. Typically, laboratories analyse both groups of chemicals, PCDD/PCDF and dl-PCB, whereby there is larger variation for the dl-PCB congeners and the WHO_{2005}-TEQ values. The laboratories carrying out these analyses are apparently well aware of the required quality issues and have expensive and sophisticated instrumentation for this task such as high-resolution mass spectrometers (sector-field instruments) coupled to HRGC. A few laboratories used low resolution mass spectrometers (quadrupole instruments). We also assume that these laboratories in general are quite stable as to personnel and analyze large number of samples per year. There is still one laboratory that applies “basic POPs” analysis using ECD detector and no internal standards (whereby it is not clear if not internal standard at all or no labelled standard). New dioxin laboratories are emerging with good performance but few sample types (one in Asia-Pacific, one in GRULAC).

The analysis of brominated flame retardants, PBDE and HBCD was in general encouraging, the fish test material excluded. However, only a small number of the more experienced laboratories participated in this exercise and extension to a wider suite of laboratories is highly desired. This is in particular

desirable, as several of the laboratories involved in this study will sooner or later face the challenge of e-waste screening for flame retardants before prior to possible recycling. Although this is not a GMP-related task it is an important activity for the Stockholm Convention.

The number of laboratories analysing toxaphene slowly increases. The results of the test solution and fortified fish test material were encouraging. Results were less good for the non-spiked air and sediment test materials.

In comparison with previous interlaboratory assessments (Fiedler et al., 2020), more PFAS laboratories participated in this IL4. As in IL3, besides PFOS, which is listed in the Stockholm Convention (UNEP, 2009, 2019a) and recommended for analysis in the GMP guidance document (UNEP, 2019b), a wider spectrum of perfluoroalkane carboxylic acids and sulfonic acids were included in the assessment. PFOA was already included although PFOA, its salts and PFOA-related compounds were listed in 2019 only (UNEP, 2019). Subsequently, the number of z-scores that were achieved continuously increased since the second round (IL 2) (see Table 88). Although in general, PFAS laboratories are at the higher end of performance in the UNEP-coordinated laboratories and the number of satisfactory results increased; it must be noted that the performance decreased from 85% satisfactory results in IL2 to 66% in IL4. Whereas the number of PFAS laboratories and the number of good results increase, care should be taken to choose proficient laboratories and carefully assess the successful participation as to the PFAS analyte and the matrix.

Table 88: Summary of z-score results for PFAS in IL2, IL3, and IL4

PFAS	# S	# Q	# U	# C	# I	**Total**	% S	% Q	% U	% C	% I
IL2	377	39	19	3	4	**435**	85	4	9	1	1
IL3	461	64	89	8	8	**630**	73	14	10	1	1
IL4	1228	174	328	63	76	**1869**	66	9	18	3	4

With respect to logistics, difficulties occurred again with strict regulations at customs and domestic transport. Some of the biological test materials, fish or human milk – had to be sent twice or could not be shipped with express mail.

In contrast to other interlaboratory assessments, laboratories were allowed to have a second look at their data after the compilation of all results. About 100 laboratories submitted new results files whereby only editorial corrections were allowed to be undertaken. Commonly occurring errors included the following:

- Errors with units for reporting (dimensions) or volume basis instead of mass basis;
- Sequence of congeners in this assessment does not correspond to chromatographic elution sequence or sequence in the laboratory´s normally used template;
- Errors with the summation of congeners to report sums of parameters;
- Lack of understanding to calculate the toxic equivalent (for dioxin-like POPs);
- Errors with the choice of the TEF scheme;
- Incorrect handling of LODs to report lower-bound or upper-bound values.

The results of this assessment emphasise the need for all laboratories to pay more attention to quality assurance (QA) and more extensive method validation. It is imperative that authorities, management and others provide the resources necessary for an adequate QA-scheme in each laboratory. Regular, routine analyses instead of one-off projects would help to build up the required level of experience for this type of analysis.

Based on the results achieved in this assessment, it is concluded that a long-term commitment to organise similar assessments on a regular basis (1-2 years) will be needed to obtain a reasonable-to-

good comparability of POP laboratories world-wide. Results need to be discussed at workshops or in mutual exchange programmes (*e.g. per* continent). To achieve the UNEP criteria for all regions, provision of training and information on methods and QA/QC will still be needed, especially for the new POPs added to the convention.

5.2 Recommendations

Based on the results in this third exercise, the following recommendations are proposed:

1. Continuation of the bi-ennial scheme of interlaboratory assessment studies is needed to monitor and improve the overall level of performance of POPs analysis of the analytical laboratories worldwide, including in developing countries.

2. Laboratories need to carry out POP analyses **on a regular basis** in order not to lose the built up knowledge. Governments should support their laboratories herein, as only participation in this interlaboratory study and occasional training will not be enough to guarantee reliable analytical results for POPs. Admission criteria for the next round should take this aspect into account.

4. Laboratories analysing OCPs strongly are encouraged to use GC-MS and ^{13}C labelled standards to improve their analysis. This and previous rounds have shown that GC/ECD results are not reliable for most of the OCPs.

5. Participating laboratories are encouraged to train their own technicians by repeatedly analysing certified reference materials and internal laboratory reference materials.

6. Laboratories are encouraged to develop methods for toxaphene, brominated flame retardants, PFASs, hexachlorobutadiene and chlordecone. At the moment there is very little capacity in the various UN regions for these POPs.

7. Participants should consider to more often use a second GC column to check possible co-elutions.

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7 APPENDICES

Appendix I - List of Participants

Appendix II – Original Data

Appendix III – z-Scores

Appendix IV – z-Score assessment

Appendix V – Statistical Evaluation

Please note: Appendices II to VII are electronically available from the WebSite at Örebro University https://www.oru.se/english/research/research-environments/ent/mtm/research-projects/global-monitoring-plan/Downloads18-19

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