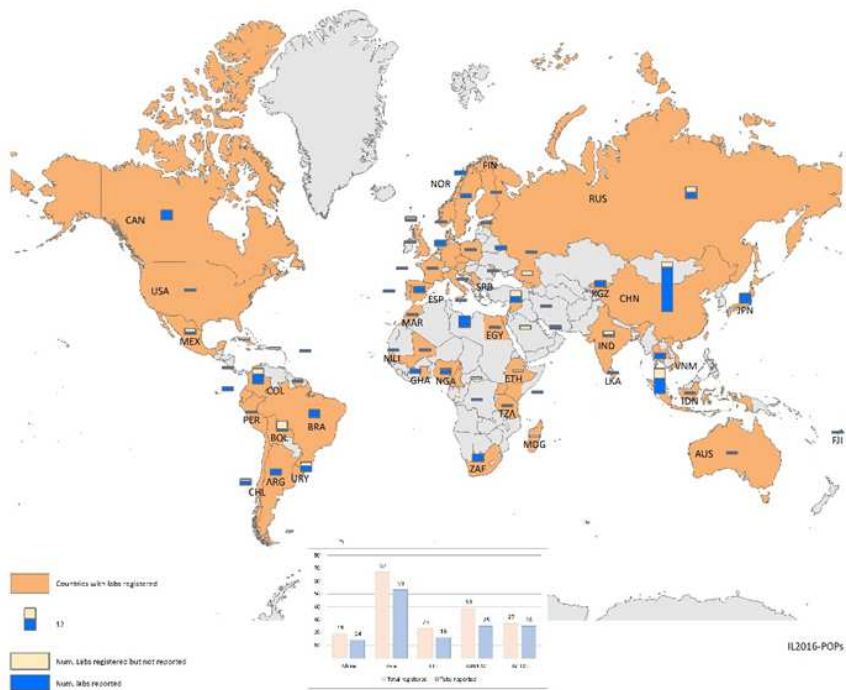




Bi-ennial Global Interlaboratory Assessment on Persistent Organic Pollutants – Third Round 2016/2017



Coordinated by:

Chemicals and Health Branch

United Nations Environment Programme

June 2017



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Assessment on Persistent Organic Pollutants
– Third Round 2016/2017**

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Sketch on title page: World map displaying countries and number of laboratories participating in the “Bi-ennial Global Interlaboratory Assessment on Persistent Organic Pollutants, 3rd Round”; prepared by Haosong Jiao, Chemicals and Health Branch; Economy Division.

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ACRONYMS AND ABBREVIATIONS

AV	Assigned value
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 Include: 29 congeners that were assigned a TEF by WHO/UNEP expert group, namely polychlorinated dibenzo- <i>para</i> -dioxins (7), polychlorinated dibenzofurans (10), and polychlorinated biphenyls (12)
EPA	Environmental Protection Agency (USA)
EtFOSA	N-Ethyl perfluorooctane sulphonamide
EtFOSE	N-Ethyl perfluorooctane sulfonamidoethanol
EU	European Union
FOSA	Perfluorooctane sulphonamides
FOSE	Perfluorooctane sulfonamidoethanols
GC	Gas chromatograph(y)
GC/ECD	Gas chromatography with electron capture detection
GC/MS	Gas chromatography 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
ILAC	International Laboratory Accreditation Cooperation
ISO	International Standardization Organisation
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 sulphonamides
MeFOSE	N-Methyl perfluorooctane sulfonamidoethanol
MS	Mass spectrometer or: mass spectrometry
MS/MS	Tandem mass spectrometry
MTM	Man-Technology-Environment
NA	Not applicable
NAV	Not 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	Polyfluoroalkyl substances
PFCA	Perfluoroalkyl carboxylic acids
PFOS	Perfluoro octylsulphonate
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 third Bi-ennial Global Interlaboratory Assessment on Persistent Organic Pollutants (POPs) was organized in 2016. After invitation to participate in this third round of the proficiency test, 175 laboratories from 66 countries had registered. This is a sharp increase in comparison to the previous interlaboratory assessment where 105 laboratories had registered. The test materials included test solutions of analytical standards, the abiotic matrices sediment, air (extract) and water and the biotic matrices 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 were assessed. These resulted in a report with a wealth of information on POP analysis and huge datasets from which the laboratories can evaluate their own methods and performance. For UN Environment and the organisers of this third round, some global conclusions can be drawn.

The Global Monitoring Programme (GMP) requires that POP laboratories must be capable – at any time – to analyse samples for POPs within a variation of $\pm 25\%$. 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. In the first place the large group of ‘newcomers’ has influenced the overall performance. Clearly, for a number of analytes, in particular for organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs), these laboratories did not perform very well. The new laboratories did not analyse the newer POPs such as the polybrominated diphenyl ethers (PBDEs), polyfluoroalkyl substances (PFASs) and hexabromocyclododecane (HBCD). Therefore, this negative effect on the results was not present for those POPs.

However, also disregarding the newcomers, still many laboratories did not perform up to expectations. Many did not achieve the requested z-scores of 2 for the various matrices. A large number of laboratories only analysed a few matrices and especially the standard solutions. On the other hand, a selected number of laboratories demonstrated a very good performance.

More specifically, sediment was in general experienced as a very difficult matrix. The fish test material, crab in this case, was not much easier. Better results were obtained for air, although it should be added that this extract was fortified. More experienced laboratories showed a good to very good performance for dioxins and dioxin-like (dl)-PCBs, and for PBDEs, PFASs and HBCD (α -HBCD in crab 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 was one of the largest ever organised. Given the huge interest in this study and the need for a substantial increase in quality for many laboratories is strongly advised to continue for several years with this study on a bi-ennial basis.

1 INTRODUCTION

This interlaboratory assessment accompanies United Nations Environment Programme's (UN Environment) capacity building program for laboratories analysing persistent organic pollutants (POPs) that has started in 2005 with Global Environment Facility (GEF) funding and implements 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 (UNEP, 2013a). 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 (here) 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). The needs and support are documented in Conference of the Parties (COP) decisions SC-3/16, SC-4/31, SC-5/18 and SC-6/23(2013b), and in chapter 3 of the guidance document. 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, 2013a). This means that POPs laboratories must be capable – at any time – to analyse samples for POPs within a margin of $\pm 25\%$ (Abalos *et al.*, 2013).

In an interlaboratory assessment, participating laboratories all analyse the same sample within a limited time frame for previously determined analytes and report the results to the coordinator of the study. All results are evaluated together according to international standards, such as established by the International Standardization Organisation (ISO) or the International Laboratory Accreditation Cooperation (ILAC), thus allowing a performance classification.

Whereas proficiency tests or 'round robins' on polychlorinated biphenyls (PCB), organochlorine pesticides (OCPs), and dioxin-like (dl-)POPs are well established for laboratories in OECD countries, challenges can be expected for developing country laboratories since they do not yet have the necessary experience to analyse a large number of POPs in biotic and abiotic matrices at the requested accuracy and within time limits.

To assist laboratories to improve the quality of their analysis, UN Environment has organized regional capacity building and training programmes, which started in 2009. As part of this activity, the first round of the Global Interlaboratory Assessment on Persistent Organic Pollutants was organized in 2010-2011 (Abalos *et al.*, 2013, van Leeuwen *et al.*, 2013) and the second in 2012-2013 (UNEP, 2015). This third round was implemented in 2016/2017.

The "Report on International Intercalibration Studies" (UNEP, 2005) emphasizes the importance of accurate results in POPs analysis, with an analytical variance to be as small as possible in order to make data acceptable and comparable between laboratories, countries, and regions, so as to allow sound decision making. Participation at international intercalibration assessments is considered a prerequisite for existing and well established as well as for newly set-up laboratories because there is a need to permanently check the laboratory's performance and 'prove' their capabilities. From an international quality assurance point of view, world-wide international studies are preferred, but national initiatives could also improve the analytical quality in just that country or a region.

Within the framework of UN Environment's capacity building project for training of laboratory staff on persistent organic pollutants (POP) analysis in developing countries, the Dept of Environment &

Health of the Vrije Universiteit Amsterdam, the Netherlands (VU E&H) and the Man-Technology-Environment (MTM) Research Center, School of Science and Technology at the University of Örebro, Sweden, have organised this third Bi-ennial Global Interlaboratory Assessment on Persistent Organic Pollutants (POPs). The results of the assessment are presented in this report.

The POPs studied included 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*-heptachlorepoxyde, and mirex. The 'new' POPs polybrominated diphenylethers (PBDE), hexachlorocyclohexanes (HCHs), chlordecone (kepone), pentachlorobenzene, α and β -endosulfan, endosulfan sulphate and perfluorinated alkylsulphonates (PFAS) as well as hexachlorobutadiene (HCBd) were also included. Separate test solutions and assessments were prepared for toxaphene (three Parlar congeners) and hexabromobiphenyl (HxBB) (as polybrominated biphenyl (PBB)153). In total, 16 matrices were offered for analysis: nine 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, fish, human milk, human plasma and water (the latter two for PFAS only). The test solutions were ampouled in amber glass ampoules with the target compounds in undisclosed concentrations. The air extracts were also ampouled, sediment was air-dried, the fish (crab) was sterilized in glass jars, the plasma frozen and the human milk was homogenized, frozen and stored at -20 °C prior to shipment. Water was sent in high-density polyethylene (HDPE) bottles.

Hundred and seventy-five laboratories from 66 countries participated (see Appendix I: List of Participants for their names and addresses). However, about one fourth of the laboratories did not submit any result, so that, finally, 133 laboratories from 57 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 participants.

2 MATERIALS AND METHODS

2.1 Identify and Preparation of the Test Samples

2.1.1 Naturally Contaminated Test Samples

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

1. The **sediment** test material was marine sediment from the Elbe River, Germany, 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'** test material consisted of Chinese mitten crab from the Netherlands. After cutting and homogenizing, individual glass jars were filled with the material. 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 **human milk** test material consisted of pooled homogenized human milk from the Swedish mother milk bank in the Örebro region and individual donors. Fifty mL milk was packed in polypropylene bottles and frozen prior shipment.
4. The **human plasma** sample consisted of pooled human blood plasma of individuals in Sweden including potentially exposed professionals and the general population. This sample was intended for the analysis of PFOS with an option of analysis of other PFASs.
5. The **air extract for organochlorine and organobromine POPs analyses – Air (TOL)** was a toluene extract of polyurethane foams (PUF) and glasfiber filters from active samplers taken in Barcelona, Spain. OCPs, dl-POP, and PBDE were spiked to the extract to fortify the natural levels that were too low for the purpose of this interlaboratory study. The extract was ampouled into 1.2 mL glass vials before shipment.
6. The **air extract for PFOS and precursor analyses – Air (MeOH)** was a methanol extract of PUFs from active samplers, taken in Barcelona, Spain. PFOS and PFOS precursors were spiked to the extract. The extract was ampouled into 1.2 mL glass vials before shipment.
7. 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** consisted of a mixture of OCPs in iso-octane in a concentration range of 1 ng/g to 500 ng/g. This test solution was prepared by VU E&H from crystals obtained from Da Vinci Laboratory Solutions B.V. (Rotterdam, The Netherlands). 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, mirex, and pentachlorobenzene.

2. The **test solution for PCB** consisted of a mixture of the indicator PCB (6 congeners) in iso-octane in a concentration range of 1 ng/g to 10 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** consisted of a mixture of 17 2,3,7,8-substituted PCDD/PCDF congeners in nonane in the concentration range of 10 ng/g to 400 ng/g. This test solution was prepared and labelled by Wellington Laboratories (Guelph, Ontario, Canada).
4. The **test solution for dl-PCB** consisted of a mixture of 12 dl-PCB in nonane in the concentration range of 20 ng/g to 500 ng/g. This test solution was prepared, ampouled and labelled by Wellington Laboratories.
5. The **test solution for PBDE/PBB** consisted of a mixture of eight PBDE and PBB153 in nonane in the concentration range of 50 ng/g to 500 ng/g. This test solution was prepared, labelled and packaged by Wellington Laboratories.
6. The **test solution for HxBB** consisted of a solution of PBB153 in iso-octane in the concentration range of 1 ng/g to 10 ng/g. This test solution was prepared by VU E&H out of a stock solutions obtained from Cambridge Isotope Laboratories, Inc.
7. The **test solution for toxaphenes** consisted of a mixture of Parlar 26,50, 62 in nonane in the concentration range of 1 ng/g to 100 ng/g. This test solution was prepared by VU E&H out of individual stock solutions obtained from Cambridge Isotope Laboratories, Inc.
8. The **test solution for HBCD** consisted of a mixture of the α , β , and γ -isomers in toluene in the concentration range of 100 ng/g to 1,000 ng/g. This test solution was prepared, ampouled and labelled by Cambridge Isotope Laboratories, Inc.
9. The **test solution for PFAS** consisted of a mixture of **perfluoroalkyl substances** (perfluoro octylsulphonate (PFOS), Perfluoroalkyl carboxylic acids (PFCAs), perfluoroalkane sulfonic acids (PFSAs), perfluorooctane sulfonamides (FOSAs) and perfluorooctane sulfonamidoethanols (FOSEs)) in methanol in the concentration range of 10 ng/g to 500 ng/g. This test solution was prepared, ampouled and labelled by Wellington Laboratories

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, PBDE, HBCD, and PFAS were distributed by MTM Research Centre. The sediment, fish, and water and the test solutions for OCP, PCB, HxBB, and toxaphene were distributed by VU E&H. All shipments containing human milk or plasma samples were packed in a polystyrene container 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 (MsExcel) 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 (MsExcel) according to laboratory (laboratory code), analyte and test sample. 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, 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. In addition, the definition of “high resolution mass spectrometer” was not interpreted by all laboratories in the same way; here, we understand “HRMS” as sector-field instruments.

The methods used included modified or adapted standard methods including for example EPA 1613 and EU 1948 for the dl-POP analysis. For PCDD/PCDF and dl-PCB, most laboratories reported that high resolution GC/MS (HRGC/HRMS) systems were used – with the limitations mentioned above. Three laboratories, used to analyse PCB, applied GC/ECD instrumentation for the analysis of dioxin-like PCB and reported on toxic equivalents; they did not analyse PCDD/PCDF.

For the separation of dl-POPs, the most common length for GC columns still is 60 m; only for a few instances, shorter columns – 30 m – were used. Only one laboratory reported to use a 50 m column. All participants used an LC-MS/MS method for the analyses of PFASs, and only one reported to have used a GC method for the analyses of the PFOS precursors.

In the other compound classes this is more diversified and GC/ECD, low resolution GC/MS (including GCxGC/MS), but also HRGC/HRMS was used.

Sample extraction was performed using variety of techniques and methods. For the extraction, Soxhlet extraction was still the most popular extraction method, although more and more laboratories used accelerated or pressurized liquid extraction that has become more popular.

Several organic solvents such as toluene, hexane, acetone or dichloromethane were used in different combinations for extraction of especially the fish and sediment sample. Of those, 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 the most preferred solvent for the sediment sample was toluene. For the extraction of PFAS almost all participants used methanol.

Furthermore, a wide variety of sample clean up open column chromatography was used where acid or base loaded silica was most commonly used followed by Florisil and alumina (especially for the OCPs). For the analysis of dioxins, the majority of the laboratories included a carbon column as the final separation step in agreement with the standard methods. Gel permeation chromatography (GPC) was used by only a few laboratories. Activated copper was often used as an extra clean up for the sediment sample.

The participants were encouraged to use appropriate GC columns for the analysis, preferably dual-column sets. Although several co-elution issues are known, especially when using ECD as the final

detection technique, only few laboratories reported that two columns or a confirmation column was used. This was also true for PCDD/PCDF analysis, where the use of a confirmation column is described in most official methods; however, this was hardly used by the participating laboratories. The major reason may be that only 2,3,7,8-substituted congeners or dl-PCB were to be reported. In addition, the human milk sample is known to have only the 2,3,7,8-substituted PCDD and PCDF present and thus, there is no need to separate these congeners from more unipolar non-TEF congeners. The other important reason is that custom-made HRGC columns are available for dl-POPs. Only one laboratory used a more sophisticated GCxGC arrangement.

The methodology for the PBDE analysis is similar to that of the OCPs and PCB. The clean-up and extraction is similar and also the final analysis is performed on similar instrumentation, including HR and LR GC/MS systems.

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 29 laboratories that submitted results for PFAS, only one laboratory used a time-of-flight instrument; all others reported to use LC/MS/MS. For the separation of the analytes, the majority used HPLC columns; however, also UPLC columns are in use. Normally, a C₁₈ based column was used; however, some also used C₈-based columns. One laboratory reported to have applied GC/LRMS (using a DB-WAX column, 30 m x 0.25 mm x 0.25 µm) for the separation of PFOS precursors, *e.g.* Me/EtFOSA and Me/EtFOSE.

2.4 Data Assessment

The data assessment was carried out, likewise the assessment of the previous rounds of the Bi-ennial Global Interlaboratory Assessment on Persistent Organic Pollutants (UNEP, 2014), 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), as is described in the report of the second round (UNEP, 2015).

The z-scores (Thompson and Wood, 1993) 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 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 ≥ 7 :

- An AV is based on the mean when $\geq 25\%$ of values have a z-score of $|z| < 2$.
- Where $< 25\%$ of the data have $|z| < 2$, the value is indicative, *i.e.*, at least 25% must be in good agreement.

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

- An AV is based on the mean when $\geq 70\%$ 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 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 two 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 differing 50% from each other. Consequently, this exercise is 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 $\pm 25\%$. 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.

3 RESULTS

All results of the individual laboratories and the assessments using z-scores include the evaluation of the 133 laboratories that submitted results. The results as mass concentrations submitted by the individual laboratories are given in Appendix II. The z-scores are given in Appendix III. The assessment of the z-scores, according to the keys given in Section 2.4 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 2.4, 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 clarified.

3.1 Participation *per* United Nations Region

In total, 175 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 66 countries. Of these, 133 laboratories from 57 countries 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=53), CEE (n=16), GRULAC (n=25), and WEOG (n=25). In Table 86 to Table 94 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, HBCD and PFASs is still relatively new for many participants, although this round's numbers for toxaphene are encouraging and higher than before. For PFASs the number of participants increased slightly for the test solution, human plasma and air extract in comparison with the previous round, but for the other matrices the participation degree decreased even though the total number of participants to the study increased.

For all other groups, ca. 40-50 (PCDD/PCDF, PBDE) to 75-80 laboratories (PCB/OCP) reported results, although 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	75	60	44	29	-	29	-
PCB	79	66	51	38	-	45	-
PCDD/PCDF	49	40	31	22	-	38	-
dl-PCB	46	37	34	26		38	
PBDE	39	28	23	17	-	25	-
HxBB	13	16	10	9	-	13	-
Toxaphene	14	13	9	6	-	7	-
HBCD	16	7	10	9	-	7	-
PFAS	27	17	15	6	12	11	20

3.2 Compound Group-Specific Results

3.2.1 Organochlorine Pesticides (OCPs)

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

Test Solution P	n			Theoretical							Between	Inclusion
	Total	Numerical	LCV	conc.	AV	Median	Mean	Min	Max	lab CV (%)	rate (%)	
Aldrin	67	63	4	34.9	29.0	31.4	29.0	0.085	491	44	66	
Dieldrin	66	62	4	35.8	32.0	34.2	32.0	0.085	237	41	64	
Endrin	63	61	2	41.1	35.3	40.0	35.3	0.11	389	57	69	
Sum Drins LB (ND = 0)	59	57	2	111.8	91.5	101.5	91.5	0.000	747	50	67	
Sum Drins UB (ND = LOD)	59	59	0	111.8	93.7	105.0	93.7	0.003	747	53	70	
α -Chlordane	43	43	0	46.6	45.4	46.6	45.4	5.04	215	40	74	
γ -Chlordane	43	43	0	48.6	42.9	45.0	42.9	5.16	187	34	71	
Oxychlordane	22	22	0	23.1	22.3	22.3	22.3	4.69	100	22	62	
cis-Nonachlor	19	19	0	89.5	86.2	83.0	86.2	18.05	432	19	61	
trans-Nonachlor	20	20	0	29.4	28.2	29.0	28.2	5.70	131	25	56	
Sum Chlordanes LB (ND = 0)	18	18	0	237.1	232	232	232	48.1	1065	21	64	
Sum Chlordanes UB (ND = LOD)	18	18	0	237.1	232	232	232	48.1	1065	21	64	
Heptachlor	65	62	3	37.9	33.6	37.2	33.6	0.099	312	48	65	
cis-Heptachlorepoxide	45	40	5	17.9	16.4	17.5	16.4	0.003	110	48	66	
trans-Heptachlorepoxide	31	29	2	20.5	15.4	18.0	15.4	0.059	196	66	67	
Sum Heptachlors LB (ND = 0)	23	22	1	76.2	70.2	75.6	70.2	0.000	485	48	69	
Sum Heptachlors UB (ND = LOD)	23	23	0	76.2	72.2	77.8	72.2	0.16	485	45	67	
o,p'-DDT	45	42	3	36.8	32.9	35.8	32.9	0.044	175	50	64	
p,p'-DDT	66	65	1	71.0	60.9	63.6	60.9	0.11	333	52	69	
o,p'-DDD	45	45	0	39.2	35.7	35.0	35.7	0.089	181	28	68	
p,p'-DDD	69	67	2	38.6	34.4	36.5	34.4	0.066	248	50	68	
o,p'-DDE	43	42	1	35.5	32.9	34.0	32.9	0.066	171	27	65	
p,p'-DDE	72	70	2	40.7	35.8	37.0	35.8	0.082	275	34	66	
Sum DDTs LB (ND = 0)	41	41	0	261.7	253	251	253	0.45	1227	39	74	
Sum DDTs UB (ND = LOD)	41	41	0	261.7	257	252	257	0.45	1227	39	73	
α -HCH	65	64	1	23.1	26.8	29.1	26.8	0.069	137	39	69	
β -HCH	62	59	3	13.1	10.5	11.3	10.5	0.036	257	53	68	
γ -HCH	66	63	3	25.4	19.2	20.0	19.2	0.039	305	39	65	
Sum HCHs LB (ND = 0)	59	58	1	61.6	57.5	59.5	57.5	0.000	507	38	67	
Sum HCHs UB (ND = LOD)	59	59	0	61.6	57.3	60.0	57.3	0.14	507	40	68	
α -Endosulfan	52	49	3	61.3	50.5	57.1	50.5	0.022	394	51	62	
β -Endosulfan	51	51	0	67.9	51.5	58.8	51.5	4.64	274	56	70	
Endosulfan sulfate	40	39	1	73.1	60.0	62.6	60.0	1.29	1446	75	76	
Sum Endosulfans LB (ND = 0)	39	39	0	202.3	154	162	154	11.4	1688	64	75	
Sum Endosulfans UB (ND = LOD)	39	39	0	202.3	154	162	154	11.4	1688	64	75	
Chlordecone	1	1	0	312.3	NAV	NAV	NAV	139	139	NAV	NAV	
Hexachlorobenzene	47	44	3	28.5	25.6	26.5	25.6	5.55	415	28	63	
Hexachlorobutadiene	4	2	2	0.0	NAV	NAV	NAV	8.18	33	NAV	NAV	
Mirex	30	30	0	133.8	136	133	136	8.17	617	15	65	
Pentachlorobenzene	20	20	0	60.0	77.6	80.4	77.6	0.30	368	17	67	

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

Test Solution P	% 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
Analyte					
Aldrin	38	43	7	24	19
Dieldrin	38	38	17	18	21
Endrin	36	32	13	29	24
Sum Drins LB (ND = 0)	34	36	12	27	22
Sum Drins UB (ND = LOD)	34	39	14	22	25
α -Chlordane	25	47	23	12	19
γ -Chlordane	25	58	12	14	16
Oxychlordane	13	64	0	18	18
cis-Nonachlor	11	58	11	11	21
trans-Nonachlor	11	55	0	20	25
Sum Chlordanes LB (ND = 0)	10	61	11	11	17
Sum Chlordanes UB (ND = LOD)	10	61	11	11	17
Heptachlor	37	40	9	22	25
cis-Heptachlorepoide	26	42	9	11	27
trans-Heptachlorepoide	18	29	6	26	32
Sum Heptachlors LB (ND = 0)	13	48	9	9	30
Sum Heptachlors UB (ND = LOD)	13	52	9	9	30
o,p'-DDT	26	40	7	22	24
p,p'-DDT	38	39	11	23	26
o,p'-DDD	26	64	9	11	16
p,p'-DDD	39	39	13	23	22
o,p'-DDE	25	53	14	14	16
p,p'-DDE	41	54	8	19	15
Sum DDTs LB (ND = 0)	23	46	22	15	17
Sum DDTs UB (ND = LOD)	23	49	20	17	15
α -HCH	37	46	17	17	18
β -HCH	35	42	3	24	26
γ -HCH	38	53	5	23	15
Sum HCHs LB (ND = 0)	34	53	7	22	17
Sum HCHs UB (ND = LOD)	34	54	3	24	19
α -Endosulfan	30	38	4	27	25
β -Endosulfan	29	25	18	37	20
Endosulfan sulfate	23	25	13	38	23
Sum Endosulfans LB (ND = 0)	22	26	13	44	18
Sum Endosulfans UB (ND = LOD)	22	26	13	44	18
Chlordecone	1	0	0	0	0
Hexachlorobenzene	27	53	11	11	19
Hexachlorobutadiene	2	0	0	0	0
Mirex	17	70	3	10	17
Pentachlorobenzene	11	70	5	10	15

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

Sediment	n									Between	Inclusion
	Total	Numerical	LCV	AV	Median	Mean	Min	Max	lab CV (%)	rate (%)	
Aldrin	47	15	32	NAV	0.90	0.11	0.004	12779	467	32	
Dieldrin	46	28	18	NAV	0.90	0.55	0.008	7081	195	46	
Endrin	43	16	27	NAV	1.24	0.50	0.008	15870	259	34	
Sum Drins LB (ND = 0)	39	26	13	NAV	1.74	1.10	0.000	35730	179	66	
Sum Drins UB (ND = LOD)	39	38	1	NAV	2.21	1.25	0.003	35730	201	60	
α-Chlordane	30	10	20	0.04	0.21	0.04	0.010	750	520	37	
γ-Chlordane	30	10	20	NAV	0.35	0.06	0.009	2256032	596	35	
Oxychlordane	13	2	11	NAV	NAV	NAV	0.43	16	NAV	NAV	
cis-Nonachlor	13	5	8	NAV	0.004	0.004	0.004	13	6	58	
trans-Nonachlor	14	6	8	NAV	0.01	0.01	0.010	17	52	59	
Sum Chlordanes LB (ND = 0)	11	6	5	NAV	0.05	0.04	0.000	76	44	63	
Sum Chlordanes UB (ND = LOD)	10	10	0	0.07	0.10	0.07	0.037	76	103	58	
Heptachlor	47	18	29	NAV	2.69	0.99	0.007	3325	250	29	
cis-Heptachlorepoide	32	10	22	NAV	7.33	1.69	0.004	1065	334	30	
trans-Heptachlorepoide	21	5	16	NAV	0.61	0.05	0.081	20	419	35	
Sum Heptachlors LB (ND = 0)	17	6	11	NAV	0.62	0.25	0.000	156	317	60	
Sum Heptachlors UB (ND = LOD)	17	17	0	NAV	0.60	0.25	0.008	225	301	59	
o,p'-DDT	34	29	5	2.8	3.4	2.8	0.006	180	126	60	
p,p'-DDT	51	41	10	NAV	21.7	17.8	1.90	1923	88	59	
o,p'-DDD	31	28	3	7.1	8.5	7.1	1.12	650	58	60	
p,p'-DDD	55	46	9	15.6	19.2	15.6	0.32	9537	83	62	
o,p'-DDE	30	23	7	0.68	0.81	0.68	0.10	35	39	46	
p,p'-DDE	56	49	7	10.9	12.9	10.9	0.54	17101	75	67	
Sum DDTs LB (ND = 0)	27	27	0	62.2	74.9	62.2	0.54	699	88	76	
Sum DDTs UB (ND = LOD)	27	27	0	62.7	74.9	62.7	0.59	704	87	76	
α-HCH	47	31	16	1.3	1.7	1.3	0.006	13426	96	51	
β-HCH	44	31	13	NAV	4.4	3.2	0.10	299	72	49	
γ-HCH	47	31	16	0.61	0.77	0.61	0.02	1087	69	47	
Sum HCHs LB (ND = 0)	41	31	10	5.5	7.2	5.5	0.000	14812	94	64	
Sum HCHs UB (ND = LOD)	41	41	0	NAV	7.2	5.3	0.006	14812	123	66	
α-Endosulfan	37	12	25	NAV	28.1	3.0	0.35	479323	511	30	
β-Endosulfan	38	19	19	NAV	4.8	2.9	0.030	230283	208	40	
Endosulfan sulfate	30	14	16	NAV	3.6	1.7	0.10	46	246	37	
Sum Endosulfans LB (ND = 0)	29	19	10	NAV	3.5	2.6	0.000	350	107	65	
Sum Endosulfans UB (ND = LOD)	28	27	1	NAV	3.5	2.4	0.085	350	161	62	
Chlordecone	3	1	2	NAV	NAV	NAV	23.0	23	NAV	NAV	
Hexachlorobenzene	35	30	5	15.9	17.3	15.9	0.003	36	43	62	
Hexachlorobutadiene	3	2	1	NAV	NAV	NAV	2.150	30	NAV	NAV	
Mirex	19	5	14	NAV	0.007	0.006	0.006	22	38	45	
Pentachlorobenzene	15	13	2	2.5	2.7	2.5	1.07	14	51	76	

Table 5: Summary of laboratory performance OCPs, sediment

Sediment	% 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	27	0	0	0	0
Dieldrin	26	0	0	0	0
Endrin	25	0	0	0	0
Sum Drins LB (ND = 0)	22	0	0	0	0
Sum Drins UB (ND = LOD)	22	0	0	0	0
α -Chlordane	17	17	0	0	17
γ -Chlordane	17	0	0	0	0
Oxychlordane	7	0	0	0	0
cis-Nonachlor	7	0	0	0	0
trans-Nonachlor	8	0	0	0	0
Sum Chlordanes LB (ND = 0)	6	0	0	0	0
Sum Chlordanes UB (ND = LOD)	6	50	10	0	40
Heptachlor	27	0	0	0	0
cis-Heptachlorepoxide	18	0	0	0	0
trans-Heptachlorepoxide	12	0	0	0	0
Sum Heptachlors LB (ND = 0)	10	0	0	0	0
Sum Heptachlors UB (ND = LOD)	10	0	0	0	0
o,p'-DDT	19	24	0	18	44
p,p'-DDT	29	0	0	0	0
o,p'-DDD	18	39	13	6	32
p,p'-DDD	31	22	7	22	33
o,p'-DDE	17	37	3	7	30
p,p'-DDE	32	27	14	18	29
Sum DDTs LB (ND = 0)	15	26	15	22	37
Sum DDTs UB (ND = LOD)	15	26	15	26	33
α -HCH	27	19	2	13	32
β -HCH	25	0	0	0	0
γ -HCH	27	30	0	6	30
Sum HCHs LB (ND = 0)	23	20	5	22	29
Sum HCHs UB (ND = LOD)	23	0	0	0	0
α -Endosulfan	21	0	0	0	0
β -Endosulfan	22	0	0	0	0
Endosulfan sulfate	17	0	0	0	0
Sum Endosulfans LB (ND = 0)	17	0	0	0	0
Sum Endosulfans UB (ND = LOD)	16	0	0	0	0
Chlordecone	2	0	0	0	0
Hexachlorobenzene	20	37	17	20	11
Hexachlorobutadiene	2	0	0	0	0
Mirex	11	0	0	0	0
Pentachlorobenzene	9	40	0	33	13

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

Fish	n									Between	Inclusion
	Total	Numerical	LCV	AV	Median	Mean	Min	Max	lab CV (%)	rate (%)	
Aldrin	35	15	20	NAV	2.3	1.1	0.005	10649	250	36	
Dieldrin	33	24	9	2.5	2.9	2.5	0.17	5901	92	61	
Endrin	30	15	15	NAV	0.8	0.3	0.034	13225	287	42	
Sum Drins LB (ND = 0)	28	21	7	2.9	3.2	2.9	0.00	29775	91	66	
Sum Drins UB (ND = LOD)	28	28	0	3.0	3.3	3.0	0.12	29775	112	63	
α-Chlordane	24	8	16	0.02	0.07	0.02	0.016	625	211	40	
γ-Chlordane	24	10	14	0.03	0.09	0.03	0.009	1880026	290	41	
Oxychlordane	15	12	3	0.74	0.78	0.74	0.55	19	21	58	
cis-Nonachlor	12	9	3	0.21	0.22	0.21	0.18	18	13	68	
trans-Nonachlor	14	11	3	0.85	0.85	0.85	0.25	20	26	57	
Sum Chlordanes LB (ND = 0)	11	9	2	1.9	1.9	1.9	0.000	1880651	14	68	
Sum Chlordanes UB (ND = LOD)	11	11	0	2.0	2.1	2.0	0.004	82	18	66	
Heptachlor	38	16	22	NAV	4.5	2.4	0.003	2771	220	30	
cis-Heptachlorepoide	25	14	11	0.77	0.88	0.77	0.05	888	40	42	
trans-Heptachlorepoide	17	3	14	NAV	1.4	0.02	0.29	15	583	39	
Sum Heptachlors LB (ND = 0)	15	9	6	0.94	0.90	0.94	0.000	42	25	78	
Sum Heptachlors UB (ND = LOD)	15	15	0	0.91	1.2	0.91	0.015	225	67	62	
o,p'-DDT	23	9	14	NAV	1.3	0.55	0.010	24	244	32	
p,p'-DDT	37	24	13	NAV	0.76	0.46	0.14	160	164	49	
o,p'-DDD	21	15	6	0.10	0.11	0.10	0.060	18	39	60	
p,p'-DDD	41	30	11	4.7	5.8	4.7	0.20	7948	82	58	
o,p'-DDE	20	13	7	0.16	0.19	0.16	0.080	54	94	53	
p,p'-DDE	42	36	6	NAV	48.6	42.5	0.90	14251	64	67	
Sum DDTs LB (ND = 0)	21	20	1	61.0	64.6	61.0	0.000	118	48	72	
Sum DDTs UB (ND = LOD)	21	21	0	60.2	63.2	60.2	0.91	118	47	72	
α-HCH	36	21	15	NAV	0.64	0.24	0.045	11188	276	48	
β-HCH	35	25	10	3.0	3.4	3.0	0.30	1365	47	50	
γ-HCH	36	16	20	NAV	0.51	0.12	0.011	906	489	39	
Sum HCHs LB (ND = 0)	33	27	6	3.4	3.8	3.4	0.000	12343	100	66	
Sum HCHs UB (ND = LOD)	33	33	0	3.1	3.7	3.1	0.015	12343	109	64	
α-Endosulfan	25	8	17	NAV	9.1	1.0	0.040	479323	497	32	
β-Endosulfan	22	8	14	NAV	27.1	10.3	0.019	191903	192	29	
Endosulfan sulfate	20	7	13	NAV	8.4	4.5	0.033	58	211	30	
Sum Endosulfans LB (ND = 0)	17	11	6	NAV	27.2	23.0	0.000	93	151	83	
Sum Endosulfans UB (ND = LOD)	18	18	0	NAV	5.9	2.6	0.003	243	310	59	
Chlordecone	2	1	1	NAV	NAV	NAV	14.0	14	NAV	NAV	
Hexachlorobenzene	28	25	3	19.2	20.8	19.2	0.67	85	38	62	
Hexachlorobutadiene	4	2	2	NAV	NAV	NAV	27.0	55	NAV	NAV	
Mirex	16	13	3	0.16	0.18	0.16	0.050	18	34	62	
Pentachlorobenzene	14	14	0	NAV	2.3	2.0	0.59	75	56	71	

Table 7: Summary of laboratory performance OCPs, fish

Fish 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	20	0	0	0	0
Dieldrin	19	21	9	15	27
Endrin	17	0	0	0	0
Sum Drins LB (ND = 0)	16	29	4	7	36
Sum Drins UB (ND = LOD)	16	32	4	14	50
α -Chlordane	14	17	0	4	13
γ -Chlordane	14	17	4	4	17
Oxychlordane	9	60	0	13	7
cis-Nonachlor	7	67	0	0	8
trans-Nonachlor	8	57	0	14	7
Sum Chlordanes LB (ND = 0)	6	64	9	0	9
Sum Chlordanes UB (ND = LOD)	6	73	0	0	27
Heptachlor	22	0	0	0	0
cis-Heptachlorepoxide	14	28	0	8	20
trans-Heptachlorepoxide	10	0	0	0	0
Sum Heptachlors LB (ND = 0)	9	53	0	0	7
Sum Heptachlors UB (ND = LOD)	9	47	0	7	47
o,p'-DDT	13	0	0	0	0
p,p'-DDT	21	0	0	0	0
o,p'-DDD	12	52	0	5	14
p,p'-DDD	23	20	7	20	27
o,p'-DDE	11	30	5	5	25
p,p'-DDE	24	0	0	0	0
Sum DDTs LB (ND = 0)	12	43	10	19	24
Sum DDTs UB (ND = LOD)	12	48	14	14	24
α -HCH	21	0	0	0	0
β -HCH	20	34	6	11	20
γ -HCH	21	0	0	0	0
Sum HCHs LB (ND = 0)	19	27	3	15	36
Sum HCHs UB (ND = LOD)	19	36	3	12	48
α -Endosulfan	14	0	0	0	0
β -Endosulfan	13	0	0	0	0
Endosulfan sulfate	11	0	0	0	0
Sum Endosulfans LB (ND = 0)	10	0	0	0	0
Sum Endosulfans UB (ND = LOD)	10	0	0	0	0
Chlordecone	1	0	0	0	0
Hexachlorobenzene	16	46	14	11	18
Hexachlorobutadiene	2	0	0	0	0
Mirex	9	56	6	6	13
Pentachlorobenzene	8	0	0	0	0

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

Human milk	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
Aldrin	22	6	16	NAV	1.1	0.10	0.005	1439	433	34
Dieldrin	21	9	12	1.0	1.1	1.0	0.45	5678	36	38
Endrin	20	5	15	NAV	0.61	0.05	0.0001	3963	496	37
Sum Drins LB (ND = 0)	19	9	10	1.4	1.5	1.4	0.000	9794	63	63
Sum Drins UB (ND = LOD)	19	19	0	1.3	1.5	1.3	0.003	9794	153	59
α-Chlordane	18	5	13	NAV	1.2	0.04	0.070	592	613	43
γ-Chlordane	18	6	12	NAV	1.3	0.11	0.031	944433	591	39
Oxychlordane	10	6	4	NAV	1.2	1.2	0.57	2.8	17	46
cis-Nonachlor	9	6	3	0.30	0.33	0.30	0.26	1.2	24	57
trans-Nonachlor	8	5	3	1.5	1.6	1.5	1.38	2.7	17	54
Sum Chlordanes LB (ND = 0)	8	5	3	3.0	3.1	3.0	0.000	10	13	71
Sum Chlordanes UB (ND = LOD)	8	8	0	NAV	3.1	2.7	0.050	39	52	62
Heptachlor	20	5	15	NAV	0.49	0.06	0.003	1272	363	38
cis-Heptachlorepoide	15	8	7	0.55	0.63	0.55	0.0002	1331	42	44
trans-Heptachlorepoide	12	2	10	NAV			0.0003	1.5	NAV	NAV
Sum Heptachlors LB (ND = 0)	10	5	5	0.65	0.63	0.65	0.000	0.7	6	72
Sum Heptachlors UB (ND = LOD)	10	10	0	0.69	0.83	0.69	0.003	90	85	68
o,p'-DDT	16	9	7	NAV	1.2	1.1	0.004	3.2	50	44
p,p'-DDT	23	15	8	8.8	9.5	8.8	0.009	568	22	35
o,p'-DDD	15	5	10	NAV	0.03	0.03	0.001	3.7	167	54
p,p'-DDD	26	12	14	NAV	0.81	0.50	0.059	3342	149	46
o,p'-DDE	15	7	8	0.09	0.10	0.09	0.000	44	22	49
p,p'-DDE	26	19	7	NAV	48.5	32.7	0.005	2630	130	59
Sum DDTs LB (ND = 0)	13	12	1	60.8	63.9	60.8	0.000	145	63	72
Sum DDTs UB (ND = LOD)	13	13	0	57.3	62.7	57.3	0.22	158	75	74
α-HCH	26	10	16	NAV	0.60	0.34	0.007	2210	177	45
β-HCH	24	13	11	6.5	6.5	6.5	0.0003	575	21	36
γ-HCH	26	14	12	NAV	1.1	0.6	0.0004	1505	221	43
Sum HCHs LB (ND = 0)	24	16	8	7.6	8.1	7.6	0.000	4290	69	69
Sum HCHs UB (ND = LOD)	24	24	0	NAV	8.2	7.2	0.003	4290	138	68
α-Endosulfan	20	5	15	NAV	1.6	0.6	0.001	672	238	36
β-Endosulfan	19	6	13	NAV	25.7	2.3	1.03	96542	527	30
Endosulfan sulfate	16	2	14	NAV	NAV	NAV	1.38	379	NAV	NAV
Sum Endosulfans LB (ND = 0)	16	4	12	NAV	7.5	5.9	0.000	549	73	67
Sum Endosulfans UB (ND = LOD)	16	16	0	NAV	7.2	4.4	0.003	551	192	70
Chlordecone	2	0	2	NAV	NAV	NAV	NAV	NAV	NAV	NAV
Hexachlorobenzene	18	12	6	9.2	8.5	9.2	0.18	19	54	61
Hexachlorobutadiene	1	0	1	NAV	NAV	NAV	NAV	NAV	NAV	NAV
Mirex	11	3	8	NAV	0.10	0.10	0.097	0.13	4	38
Pentachlorobenzene	7	4	3	NAV	0.53	0.38	0.35	4.2	60	59

Table 9: Summary of laboratory performance OCPs, human milk

Human milk	% 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	24	0	5	14
Endrin	11	0	0	0	0
Sum Drins LB (ND = 0)	11	21	5	0	21
Sum Drins UB (ND = LOD)	11	32	5	5	58
α -Chlordane	10	0	0	0	0
γ -Chlordane	10	0	0	0	0
Oxychlordane	6	0	0	0	0
cis-Nonachlor	5	56	0	0	11
trans-Nonachlor	5	50	0	13	0
Sum Chlordanes LB (ND = 0)	5	50	0	0	13
Sum Chlordanes UB (ND = LOD)	5	0	0	0	0
Heptachlor	11	0	0	0	0
cis-Heptachlorepoxide	9	33	0	0	20
trans-Heptachlorepoxide	7	0	0	0	0
Sum Heptachlors LB (ND = 0)	6	40	0	0	10
Sum Heptachlors UB (ND = LOD)	6	40	10	0	50
o,p'-DDT	9	0	0	0	0
p,p'-DDT	13	35	0	4	26
o,p'-DDD	9	0	0	0	0
p,p'-DDD	15	0	0	0	0
o,p'-DDE	9	27	0	7	13
p,p'-DDE	15	0	0	0	0
Sum DDTs LB (ND = 0)	7	46	0	15	31
Sum DDTs UB (ND = LOD)	7	46	0	15	38
α -HCH	15	0	0	0	0
β -HCH	14	33	0	0	21
γ -HCH	15	0	0	0	0
Sum HCHs LB (ND = 0)	14	21	8	17	21
Sum HCHs UB (ND = LOD)	14	0	0	0	0
α -Endosulfan	11	0	0	0	0
β -Endosulfan	11	0	0	0	0
Endosulfan sulfate	9	0	0	0	0
Sum Endosulfans LB (ND = 0)	9	0	0	0	0
Sum Endosulfans UB (ND = LOD)	9	0	0	0	0
Chlordecone	1	0	0	0	0
Hexachlorobenzene	10	28	6	22	11
Hexachlorobutadiene	1	0	0	0	0
Mirex	6	0	0	0	0
Pentachlorobenzene	4	0	0	0	0

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

Air extract (TOL)	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
Aldrin	23	18	5	5.4	5.8	5.4	0.18	262	51	64
Dieldrin	24	21	3	9.2	9.5	9.2	0.016	119	42	59
Endrin	20	13	7	5.2	5.9	5.2	0.018	16	32	44
Sum Drins LB (ND = 0)	20	17	3	20.3	21.0	20.3	0.000	119	51	72
Sum Drins UB (ND = LOD)	20	20	0	17.4	20.3	17.4	0.030	149	80	78
α-Chlordane	21	15	6	6.3	6.1	6.3	1.66	12	19	51
γ-Chlordane	21	18	3	6.1	6.4	6.1	1.48	13805	22	51
Oxychlordane	11	7	4	5.5	5.5	5.5	5.01	7.4	4	47
cis-Nonachlor	9	5	4	0.73	0.73	0.73	0.71	12	4	52
trans-Nonachlor	12	7	5	0.49	0.52	0.49	0.39	25	17	55
Sum Chlordanes LB (ND = 0)	7	6	1	19.1	19.0	19.1	0.000	20	6	69
Sum Chlordanes UB (ND = LOD)	7	7	0	19.0	19.0	19.0	0.050	43	10	53
Heptachlor	21	18	3	5.2	5.3	5.2	0.013	21	18	58
cis-Heptachlorepoide	19	14	5	6.0	6.2	6.0	0.001	41	56	55
trans-Heptachlorepoide	13	9	4	5.2	5.3	5.2	0.013	41	17	37
Sum Heptachlors LB (ND = 0)	12	10	2	16.3	16.8	16.3	0.000	82	30	58
Sum Heptachlors UB (ND = LOD)	12	12	0	NAV	16.1	12.1	0.027	97	103	74
o,p'-DDT	19	16	3	5.8	6.2	5.8	0.016	13	36	61
p,p'-DDT	25	19	6	7.3	7.7	7.3	0.041	35	26	45
o,p'-DDD	19	18	1	5.6	5.9	5.6	0.027	18	43	63
p,p'-DDD	26	21	5	5.6	5.9	5.6	0.016	28	30	51
o,p'-DDE	17	15	2	6.1	6.0	6.1	0.009	13	13	58
p,p'-DDE	25	22	3	8.1	8.3	8.1	0.015	103	35	54
Sum DDTs LB (ND = 0)	17	17	0	37.6	36.0	37.6	0.12	78	25	61
Sum DDTs UB (ND = LOD)	17	17	0	37.1	36.0	37.1	0.12	84	25	62
α-HCH	25	22	3	5.7	6.0	5.7	0.12	128	23	57
β-HCH	24	17	7	5.9	6.6	5.9	0.075	29	56	54
γ-HCH	26	19	7	9.2	9.4	9.2	0.010	14	21	47
Sum HCHs LB (ND = 0)	24	21	3	19.8	21.5	19.8	0.000	67	37	67
Sum HCHs UB (ND = LOD)	23	23	0	19.2	19.1	19.2	0.15	67	42	61
α-Endosulfan	20	16	4	NAV	7.2	6.3	0.019	40	64	63
β-Endosulfan	17	10	7	NAV	6.6	4.1	0.23	297	82	40
Endosulfan sulfate	13	5	8	NAV	0.76	0.65	0.41	2.7	33	58
Sum Endosulfans LB (ND = 0)	13	11	2	NAV	12.1	9.4	0.000	40	66	61
Sum Endosulfans UB (ND = LOD)	13	13	0	NAV	11.9	9.4	0.15	68	86	70
Chlordecone	1	0	1	NAV	NAV	NAV	NAV	NAV	NAV	NAV
Hexachlorobenzene	19	16	3	6.6	6.6	6.6	5.64	550	22	53
Hexachlorobutadiene	2	2	0	NAV	NAV	NAV	8.33	13	NAV	NAV
Mirex	16	15	1	6.6	6.4	6.6	3.11	15	27	74
Pentachlorobenzene	10	5	5	NAV	0.54	0.50	0.43	51	30	50

Table 11: Summary of laboratory performance OCPs, 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
Aldrin	13	35	13	22	9
Dieldrin	14	46	0	25	17
Endrin	11	40	5	0	20
Sum Drins LB (ND = 0)	11	35	10	20	20
Sum Drins UB (ND = LOD)	11	40	0	20	40
α -Chlordane	12	52	5	10	5
γ -Chlordane	12	52	5	14	14
Oxychlordane	6	55	9	0	0
cis-Nonachlor	5	44	0	0	11
trans-Nonachlor	7	42	0	0	17
Sum Chlordanes LB (ND = 0)	4	71	0	14	0
Sum Chlordanes UB (ND = LOD)	4	57	0	14	29
Heptachlor	12	62	0	10	14
cis-Heptachlorepoide	11	32	11	11	21
trans-Heptachlorepoide	7	38	0	8	23
Sum Heptachlors LB (ND = 0)	7	42	8	8	25
Sum Heptachlors UB (ND = LOD)	7	0	0	0	0
o,p'-DDT	11	42	16	16	11
p,p'-DDT	14	48	0	4	24
o,p'-DDD	11	47	0	21	26
p,p'-DDD	15	46	4	8	23
o,p'-DDE	10	71	6	0	12
p,p'-DDE	14	44	8	12	24
Sum DDTs LB (ND = 0)	10	59	6	12	24
Sum DDTs UB (ND = LOD)	10	59	6	12	24
α -HCH	14	56	8	8	16
β -HCH	14	33	4	17	17
γ -HCH	15	46	0	19	8
Sum HCHs LB (ND = 0)	14	50	8	13	17
Sum HCHs UB (ND = LOD)	13	48	9	13	30
α -Endosulfan	11	0	0	0	0
β -Endosulfan	10	0	0	0	0
Endosulfan sulfate	7	0	0	0	0
Sum Endosulfans LB (ND = 0)	7	0	0	0	0
Sum Endosulfans UB (ND = LOD)	7	0	0	0	0
Chlordecone	1	0	0	0	0
Hexachlorobenzene	11	53	5	21	5
Hexachlorobutadiene	1	0	0	0	0
Mirex	9	75	6	6	6
Pentachlorobenzene	6	0	0	0	0

3.2.2 Polychlorinated Biphenyls (PCB)

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

Test Solution Q	n			Theoretical						Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV	conc.	AV	Median	Mean	Min	Max		
PCB 28	77	74	3	11.0	9.1	9.6	9.1	0.04	56	35	70
PCB 52	76	74	2	5.9	5.4	5.4	5.4	0.04	41	24	64
PCB 101	78	72	6	2.5	2.5	2.6	2.5	0.01	38	27	61
PCB 138	77	75	2	9.4	8.3	8.2	8.3	0.01	43	30	64
PCB 153	78	76	2	4.9	4.8	4.9	4.8	0.01	50	28	63
PCB 180	77	75	2	12.2	10.7	10.8	10.7	0.02	52	22	66
Sum Indicator PCB LB (ND = 0)	74	72	2	45.9	41.5	41.8	41.5	0.00	281	25	66
Sum Indicator PCB UB (ND = LOD)	73	73	0	45.9	41.5	42.0	41.5	0.14	348	25	65

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

Test Solution Q	% of the data received	% of z-scores		% of z-scores	
		z <2 Satisfactory	3> z >2 Questionable	6> z >3 Unsatisfactory	% of z-scores z >6 Extreme
PCB 28	44	51	16	21	9
PCB 52	43	59	12	14	12
PCB 101	45	56	9	12	15
PCB 138	44	56	9	22	10
PCB 153	45	56	9	15	17
PCB 180	44	65	10	12	10
Sum Indicator PCB LB (ND = 0)	42	59	11	19	8
Sum Indicator PCB UB (ND = LOD)	42	60	11	18	11

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

Sediment	n			Theoretical						Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV	AV	Median	Mean	Min	Max			
PCB 28	58	50	8	2.2	2.5	2.2	0.06	103527	66	67	
PCB 52	60	54	6	3.4	3.9	3.4	0.18	2145342	75	64	
PCB 101	61	56	5	4.5	5.5	4.5	0.09	5023	63	63	
PCB 138	61	58	3	8.0	8.5	8.0	0.37	362286	53	64	
PCB 153	63	61	2	8.1	9.1	8.1	0.33	125118	66	66	
PCB 180	64	60	4	5.5	6.2	5.5	0.23	24000	55	69	
Sum Indicator PCB LB (ND = 0)	57	56	1	32.8	36.0	32.8	0.00	2760347	69	69	
Sum Indicator PCB UB (ND = LOD)	57	57	0	32.8	36.2	32.8	0.53	2760347	69	70	

Table 15: Summary of laboratory performance indicator PCB, sediment

Sediment	% of the data received	% of z-scores		% of z-scores	
		z <2 Satisfactory	3> z >2 Questionable	6> z >3 Unsatisfactory	% of z-scores z >6 Extreme
PCB 28	33	29	9	24	24
PCB 52	34	23	10	32	25
PCB 101	35	28	11	26	26
PCB 138	35	36	13	13	33
PCB 153	36	35	8	21	33
PCB 180	37	33	16	22	23
Sum Indicator PCB LB (ND = 0)	33	37	11	18	33
Sum Indicator PCB UB (ND = LOD)	33	37	12	18	33

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

Fish	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
PCB 28	48	45	3	19.8	22.0	19.8	2.00	208352	59	66
PCB 52	49	46	3	54.0	57.3	54.0	0.43	545115	55	69
PCB 101	50	47	3	125	133	125	0.79	3383258	55	69
PCB 138	48	48	0	119	139	119	1.37	4646901	63	70
PCB 153	48	47	1	NAV	255	224	0.79	1476921	61	69
PCB 180	50	46	4	73.3	73.9	73.3	0.61	184691	28	57
Sum Indicator PCB LB (ND = 0)	46	46	0	627	674	627	0.63	10445238	51	70
Sum Indicator PCB UB (ND = LOD)	46	46	0	626	674	626	1.07	10445238	51	70

Table 17: Summary of laboratory performance indicator PCB, fish

Fish	% 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	27	29	15	23	27
PCB 52	28	37	14	20	22
PCB 101	29	28	18	30	18
PCB 138	27	31	21	15	33
PCB 153	27	0	0	0	0
PCB 180	29	56	0	18	18
Sum Indicator PCB LB (ND = 0)	26	41	11	20	28
Sum Indicator PCB UB (ND = LOD)	26	41	9	22	28

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

Human milk	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
PCB 28	36	29	7	2.3	3.0	2.3	1.14	5723	65	55
PCB 52	35	30	5	NAV	0.67	0.42	0.09	18598	127	56
PCB 101	35	27	8	0.35	0.46	0.35	0.18	22106	79	56
PCB 138	34	28	6	7.7	7.7	7.7	0.02	588	27	49
PCB 153	36	31	5	11.9	13.1	11.9	0.0004	4347	53	58
PCB 180	36	29	7	7.2	7.2	7.2	0.0002	1887	25	52
Sum Indicator PCB LB (ND = 0)	33	31	2	29.5	31.7	29.5	0.0000	52395	44	66
Sum Indicator PCB UB (ND = LOD)	33	33	0	29.8	31.7	29.8	0.61	52395	41	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	21	28	17	8	28
PCB 52	20	0	0	0	0
PCB 101	20	29	17	6	26
PCB 138	19	44	3	12	24
PCB 153	21	33	8	19	25
PCB 180	21	44	6	11	19
Sum Indicator PCB LB (ND = 0)	19	45	3	18	27
Sum Indicator PCB UB (ND = LOD)	19	52	3	12	33

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

Air extract (TOL)	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
PCB 28	45	44	1	7.0	7.0	7.0	0.01	7513	45	69
PCB 52	45	42	3	9.7	9.7	9.7	0.08	8114	21	59
PCB 101	46	44	2	12.7	13.0	12.7	0.01	12165	31	64
PCB 138	44	41	3	10.6	11.0	10.6	0.02	9527	20	60
PCB 153	45	44	1	10.7	10.8	10.7	0.02	9002	21	62
PCB 180	45	43	2	6.3	6.7	6.3	0.01	5918	34	67
Sum Indicator PCB LB (ND = 0)	43	42	1	56.1	55.3	56.1	0.000	52240	30	71
Sum Indicator PCB UB (ND = LOD)	42	42	0	56.2	55.3	56.2	1.13	52240	29	72

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

Air extract (TOL)	% 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
PCB 28	26	40	18	22	18
PCB 52	26	58	11	9	16
PCB 101	26	52	15	9	20
PCB 138	25	61	7	11	14
PCB 153	26	60	13	4	20
PCB 180	26	51	9	18	18
Sum Indicator PCB LB (ND = 0)	25	53	19	12	14
Sum Indicator PCB UB (ND = LOD)	24	55	21	10	14

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

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

Test Solution K and L	n			Theoretical						Inclusion	
	Total	Numerical	LCV	conc.	AV	Median	Mean	Min	Max	Between lab CV (%)	rate (%)
2,3,7,8-TeCDD	49	49	0	41.8	39.9	39.7	39.9	19.0	84	12	69
1,2,3,7,8-PnCDD	49	49	0	230	220	217	220	84.0	271	12	66
1,2,3,4,7,8-HxCDD	49	49	0	188	177	180	177	21.3	225	20	76
1,2,3,6,7,8-HxCDD	49	49	0	188	185	184	185	21.4	243	12	69
1,2,3,7,8,9-HxCDD	49	49	0	230	220	220	220	26.2	266	17	72
1,2,3,4,6,7,8-HpCDD	49	49	0	272	264	260	264	3.03	343	13	69
OCDD	49	49	0	418	399	397	399	0.14	484	15	71
2,3,7,8-TeCDF	49	49	0	41.8	40.0	40.0	40.0	4.34	49	11	66
1,2,3,7,8-PnCDF	49	49	0	105	100	100	100	3.43	120	15	73
2,3,4,7,8-PnCDF	49	49	0	105	99.6	97.6	99.6	34.8	120	13	67
1,2,3,4,7,8-HxCDF	49	49	0	230	218	217	218	25.9	280	16	72
1,2,3,6,7,8-HxCDF	49	49	0	188	181	180	181	21.1	237	15	70
1,2,3,7,8,9-HxCDF	49	49	0	230	216	223	216	26.1	270	20	75
2,3,4,6,7,8-HxCDF	49	49	0	188	184	182	184	21.1	232	14	72
1,2,3,4,6,7,8-HpCDF	49	49	0	313	307	304	307	3.49	401	12	69
1,2,3,4,7,8,9-HpCDF	49	49	0	313	301	308	301	3.85	439	17	72
OCDF	49	49	0	376	355	358	355	0.16	554	17	72
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	45	45	0	452	450	452	452	164.0	529	10	65
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	45	45	0	452	450	452	452	164.0	529	10	65
PCB 77	43	43	0	153	150	150	150	0.02	265	21	66
PCB 81	43	43	0	69.6	71.7	71.6	71.7	0.02	179	13	60
PCB 126	43	42	1	69.6	69.4	67.9	69.4	6.55	90	18	68
PCB 169	43	43	0	153	154	150	154	4.54	341	17	66
PCB 105	46	46	0	69.6	70.2	69.9	70.2	0.002	41801	14	59
PCB 114	45	44	1	69.6	69.2	68.8	69.2	0.002	60963	17	65
PCB 118	46	46	0	237	224	228	224	0.007	131943	25	64
PCB 123	45	45	0	69.6	69.3	68.2	69.3	0.002	42934	14	66
PCB 156	46	46	0	69.6	70.5	69.4	70.5	0.002	32711	14	65
PCB 157	45	45	0	69.6	69.3	68.8	69.3	0.002	43542	11	62
PCB 167	45	45	0	69.6	69.1	69.3	69.1	0.002	42592	15	62
PCB 189	45	45	0	69.6	69.4	69.0	69.4	0.002	54838	9	60
WHO2005-TEQ (dl-PCB) LB (ND = 0)	42	42	0	11.7	11.9	11.7	11.7	3.52	1620	10	64
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	42	42	0	11.7	11.9	11.7	11.7	3.52	1630	10	64
WHO2005-TEQ (total) LB (ND = 0)	37	37	0	464	463	464	464	171	529	11	67
WHO2005-TEQ (total) UB (ND = LOD)	37	37	0	464	463	464	464	171	529	11	67

Table 23: Summary of laboratory performance dl-POPs, test solutions K and L

Test Solution K and L	% 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
Analyte					
2,3,7,8-TeCDD	28	80	12	6	2
1,2,3,7,8-PnCDD	28	80	14	6	0
1,2,3,4,7,8-HxCDD	28	78	16	4	2
1,2,3,6,7,8-HxCDD	28	84	8	6	2
1,2,3,7,8,9-HxCDD	28	86	2	10	2
1,2,3,4,6,7,8-HpCDD	28	80	12	6	2
OCDD	28	86	8	4	2
2,3,7,8-TeCDF	28	86	6	6	2
1,2,3,7,8-PnCDF	28	86	6	6	2
2,3,4,7,8-PnCDF	28	84	6	10	0
1,2,3,4,7,8-HxCDF	28	82	10	6	2
1,2,3,6,7,8-HxCDF	28	80	12	6	2
1,2,3,7,8,9-HxCDF	28	82	10	6	2
2,3,4,6,7,8-HxCDF	28	84	6	8	2
1,2,3,4,6,7,8-HpCDF	28	80	12	6	2
1,2,3,4,7,8,9-HpCDF	28	78	10	10	2
OCDF	28	80	8	8	4
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	26	82	11	7	0
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	26	82	11	7	0
PCB 77	25	63	14	14	9
PCB 81	25	65	19	9	7
PCB 126	25	70	12	12	5
PCB 169	25	70	5	19	7
PCB 105	26	65	13	11	11
PCB 114	26	71	4	11	11
PCB 118	26	61	7	24	9
PCB 123	26	76	4	11	9
PCB 156	26	72	7	13	9
PCB 157	26	73	7	13	7
PCB 167	26	67	7	11	16
PCB 189	26	69	4	13	13
WHO2005-TEQ (dl-PCB) LB (ND = 0)	24	74	10	7	10
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	24	74	10	7	10
WHO2005-TEQ (total) LB (ND = 0)	21	84	11	5	0
WHO2005-TEQ (total) UB (ND = LOD)	21	84	11	5	0

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

Sediment	n			Between Inclusion						
	Total	Numerical	LCV	AV	Median	Mean	Min	Max	lab CV (%)	rate (%)
2,3,7,8-TeCDD	40	36	4	1.0	1.1	1.0	0.57	2.29	33	71
1,2,3,7,8-PnCDD	40	36	4	1.5	1.6	1.5	0.59	5.13	28	69
1,2,3,4,7,8-HxCDD	40	37	3	1.8	1.9	1.8	0.16	4.56	23	74
1,2,3,6,7,8-HxCDD	40	37	3	3.8	3.9	3.8	0.36	7.79	19	71
1,2,3,7,8,9-HxCDD	40	36	4	3.3	3.4	3.3	0.28	19.1	22	68
1,2,3,4,6,7,8-HpCDD	40	38	2	65.7	65.9	65.7	0.61	111	14	68
OCDD	40	39	1	545	535	545	0.14	1096	16	74
2,3,7,8-TeCDF	40	39	1	17.0	17.1	17.0	1.98	50.3	20	69
1,2,3,7,8-PnCDF	40	38	2	18.3	18.8	18.3	0.52	26.5	17	69
2,3,4,7,8-PnCDF	40	38	2	11.0	10.9	11.0	1.98	21.3	20	61
1,2,3,4,7,8-HxCDF	40	38	2	36.4	36.4	36.4	3.37	62.8	15	66
1,2,3,6,7,8-HxCDF	40	38	2	25.3	25.4	25.3	2.39	38.4	17	73
1,2,3,7,8,9-HxCDF	40	38	2	NAV	6.5	7.6	0.33	20.8	61	78
2,3,4,6,7,8-HxCDF	40	38	2	11.2	11.7	11.2	1.28	21.1	42	75
1,2,3,4,6,7,8-HpCDF	40	39	1	120	120	120	1.07	194.0	16	74
1,2,3,4,7,8,9-HpCDF	40	39	1	43.6	41.7	43.6	0.42	70.6	17	73
OCDF	40	39	1	438	431	438	0.16	831.2	14	70
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	38	38	0	20.2	20.3	20.2	0.02	33.2	16	72
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	38	38	0	20.5	20.5	20.5	0.03	36.0	17	73
PCB 77	35	33	2	174	170	174	0.01	77461	46	71
PCB 81	35	32	3	6.6	7.3	6.6	0.0005	186273	67	66
PCB 126	34	30	4	16.4	17.2	16.4	1.25	1389	24	69
PCB 169	35	29	6	2.8	3.0	2.8	0.07	2161058	44	71
PCB 105	36	33	3	712	831	712	0.02	2639	42	55
PCB 114	36	35	1	44.3	50.6	44.3	0.001	6670	65	59
PCB 118	37	36	1	2506	3014	2506	0.07	10435	59	59
PCB 123	36	33	3	NAV	95.0	75.3	0.002	33920	114	67
PCB 156	37	36	1	804	815	804	0.02	1634	35	69
PCB 157	36	33	3	114	120	114	0.003	1634	31	61
PCB 167	36	34	2	388	401	388	0.03	4900	37	62
PCB 189	36	32	4	117	121	117	0.003	5065	30	59
WHO2005-TEQ (dl-PCB) LB (ND = 0)	34	34	0	1.9	2.0	1.9	0.04	2485891	27	66
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	34	34	0	1.9	2.1	1.9	1.43	2511000	31	66
WHO2005-TEQ (total) LB (ND = 0)	29	29	0	22.9	22.5	22.9	1.05	35.6	18	76
WHO2005-TEQ (total) UB (ND = LOD)	29	29	0	23.1	22.5	23.1	16.0	39.0	20	77

Table 25: Summary of laboratory performance dl-POPs, 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
2,3,7,8-TeCDD	23	58	13	10	10
1,2,3,7,8-PnCDD	23	58	18	13	3
1,2,3,4,7,8-HxCDD	23	78	5	3	8
1,2,3,6,7,8-HxCDD	23	75	8	0	10
1,2,3,7,8,9-HxCDD	23	65	8	8	10
1,2,3,4,6,7,8-HpCDD	23	83	5	5	3
OCDD	23	83	8	0	8
2,3,7,8-TeCDF	23	73	10	5	10
1,2,3,7,8-PnCDF	23	75	10	8	3
2,3,4,7,8-PnCDF	23	60	10	18	8
1,2,3,4,7,8-HxCDF	23	78	5	10	3
1,2,3,6,7,8-HxCDF	23	85	5	3	3
1,2,3,7,8,9-HxCDF	23	0	0	0	0
2,3,4,6,7,8-HxCDF	23	30	23	38	5
1,2,3,4,6,7,8-HpCDF	23	88	3	5	3
1,2,3,4,7,8,9-HpCDF	23	80	10	5	3
OCDF	23	80	5	8	5
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	22	79	11	3	8
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	22	76	13	3	8
PCB 77	20	46	9	23	17
PCB 81	20	34	11	9	37
PCB 126	20	63	6	3	14
PCB 169	20	40	11	17	14
PCB 105	21	38	5	16	30
PCB 114	21	36	11	11	39
PCB 118	21	32	11	16	38
PCB 123	21	0	0	0	0
PCB 156	21	54	16	14	14
PCB 157	21	53	6	14	19
PCB 167	21	50	8	8	28
PCB 189	21	53	6	11	19
WHO2005-TEQ (dl-PCB) LB (ND = 0)	19	68	3	6	24
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	19	68	0	6	26
WHO2005-TEQ (total) LB (ND = 0)	17	76	17	3	3
WHO2005-TEQ (total) UB (ND = LOD)	17	79	14	7	0

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

Fish	n			Between Inclusion						
	Total	Numerical	LCV	AV	Median	Mean	Min	Max	lab CV (%)	rate (%)
2,3,7,8-TeCDD	31	31	0	10.6	10.6	10.6	2.88	25.0	8	68
1,2,3,7,8-PnCDD	31	30	1	1.3	1.3	1.3	0.47	2.77	22	69
1,2,3,4,7,8-HxCDD	31	29	2	0.71	0.71	0.71	0.32	1.94	18	69
1,2,3,6,7,8-HxCDD	31	31	0	2.2	2.3	2.2	0.49	6.34	15	71
1,2,3,7,8,9-HxCDD	30	28	2	0.79	0.83	0.79	0.33	2.11	24	60
1,2,3,4,6,7,8-HpCDD	31	31	0	4.8	4.8	4.8	1.92	11.2	12	68
OCDD	31	31	0	5.3	5.5	5.3	4.30	12.8	27	71
2,3,7,8-TeCDF	31	31	0	35.5	35.6	35.5	0.63	83.2	15	71
1,2,3,7,8-PnCDF	31	31	0	9.8	9.7	9.8	0.44	25.1	14	72
2,3,4,7,8-PnCDF	31	31	0	12.8	13.0	12.8	0.28	35.3	11	69
1,2,3,4,7,8-HxCDF	31	31	0	18.9	19.1	18.9	0.48	45.4	13	72
1,2,3,6,7,8-HxCDF	31	31	0	6.8	6.8	6.8	0.72	16.7	12	66
1,2,3,7,8,9-HxCDF	31	26	5	NAV	0.42	0.46	0.11	2.36	94	65
2,3,4,6,7,8-HxCDF	31	31	0	3.3	3.4	3.3	0.46	8.26	11	62
1,2,3,4,6,7,8-HpCDF	31	31	0	16.1	16.0	16.1	2.56	42.9	9	75
1,2,3,4,7,8,9-HpCDF	31	30	1	0.65	0.66	0.65	0.46	4.42	18	66
OCDF	31	30	1	5.6	5.8	5.6	3.64	13.8	19	69
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	28	28	0	23.0	23.1	23.0	17.51	56.3	9	70
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	28	28	0	23.0	23.1	23.0	17.51	56.3	9	72
PCB 77	32	32	0	2296	2284	2296	4.25	58359	23	74
PCB 81	31	30	1	46.2	52.3	46.2	12.2	70398	52	68
PCB 126	32	31	1	198	204	198	71.7	67480496	16	63
PCB 169	32	30	2	25.2	26.0	25.2	9.12	363890	13	56
PCB 105	34	34	0	13758	13823	13758	1811	32206	19	67
PCB 114	33	32	1	774	825	774	296	86463	28	64
PCB 118	33	33	0	77533	78708	77533	4533	263352	39	72
PCB 123	32	30	2	883	977	883	19.0	17434	84	65
PCB 156	34	34	0	10847	10924	10847	3523.0	24273	18	63
PCB 157	33	33	0	2063	2104	2063	492.4	10605	22	65
PCB 167	33	33	0	8034	8091	8034	771.8	22035	12	63
PCB 189	33	33	0	1411	1397	1411	22.6	3060	15	69
WHO2005-TEQ (dl-PCB) LB (ND = 0)	30	30	0	24.6	25.2	24.6	8.02	68025203	15	65
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	30	30	0	24.6	25.5	24.6	8.02	68025203	17	66
WHO2005-TEQ (total) LB (ND = 0)	27	27	0	48.2	48.4	48.2	25.5	159	10	70
WHO2005-TEQ (total) UB (ND = LOD)	27	27	0	48.3	48.4	48.3	25.5	159	11	70

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

Fish 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	18	84	6	6	3
1,2,3,7,8-PnCDD	18	77	3	13	3
1,2,3,4,7,8-HxCDD	18	71	10	10	3
1,2,3,6,7,8-HxCDD	18	84	0	13	3
1,2,3,7,8,9-HxCDD	17	60	3	13	17
1,2,3,4,6,7,8-HpCDD	18	84	0	13	3
OCDD	18	68	10	6	16
2,3,7,8-TeCDF	18	84	6	3	6
1,2,3,7,8-PnCDF	18	84	0	6	10
2,3,4,7,8-PnCDF	18	84	3	6	6
1,2,3,4,7,8-HxCDF	18	84	3	6	6
1,2,3,6,7,8-HxCDF	18	81	10	3	6
1,2,3,7,8,9-HxCDF	18	0	0	0	0
2,3,4,6,7,8-HxCDF	18	77	6	6	10
1,2,3,4,6,7,8-HpCDF	18	90	3	0	6
1,2,3,4,7,8,9-HpCDF	18	81	0	3	13
OCDF	18	74	6	6	10
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	16	93	0	4	4
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	16	93	0	4	4
PCB 77	18	78	3	6	13
PCB 81	18	39	10	26	23
PCB 126	18	69	3	9	16
PCB 169	18	63	6	6	19
PCB 105	19	68	9	15	9
PCB 114	19	58	6	15	18
PCB 118	19	48	12	21	18
PCB 123	18	28	6	22	38
PCB 156	19	65	15	12	9
PCB 157	19	61	9	18	12
PCB 167	19	76	3	12	9
PCB 189	19	82	3	6	9
WHO2005-TEQ (dl-PCB) LB (ND = 0)	17	73	3	3	20
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	17	73	3	3	20
WHO2005-TEQ (total) LB (ND = 0)	15	85	0	7	7
WHO2005-TEQ (total) UB (ND = LOD)	15	85	0	7	7

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

Human milk Analyte	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
2,3,7,8-TeCDD	22	16	6	0.34	0.35	0.34	0.10	0.69	55	76
1,2,3,7,8-PnCDD	22	17	5	0.66	0.72	0.66	0.02	1.96	55	64
1,2,3,4,7,8-HxCDD	22	14	8	0.31	0.34	0.31	0.07	1.10	65	72
1,2,3,6,7,8-HxCDD	22	18	4	2.1	2.2	2.1	0.28	3.60	28	65
1,2,3,7,8,9-HxCDD	22	17	5	0.75	0.73	0.75	0.14	1.30	40	71
1,2,3,4,6,7,8-HpCDD	22	20	2	4.6	4.8	4.6	2.59	8.90	23	64
OCDD	22	21	1	30.4	30.4	30.4	8.95	166	23	63
2,3,7,8-TeCDF	22	19	3	1.8	1.8	1.8	0.28	2.28	19	76
1,2,3,7,8-PnCDF	22	16	6	0.38	0.40	0.38	0.16	0.98	49	70
2,3,4,7,8-PnCDF	22	19	3	2.1	2.1	2.1	0.30	4.18	30	67
1,2,3,4,7,8-HxCDF	22	18	4	0.93	0.94	0.93	0.44	3.00	35	64
1,2,3,6,7,8-HxCDF	22	18	4	0.86	0.89	0.86	0.24	3.00	15	61
1,2,3,7,8,9-HxCDF	22	7	15	NAV	0.27	0.12	0.06	1.40	108	41
2,3,4,6,7,8-HxCDF	22	16	6	0.59	0.60	0.59	0.25	2.60	38	67
1,2,3,4,6,7,8-HpCDF	22	20	2	1.8	1.7	1.8	0.36	24.2	41	74
1,2,3,4,7,8,9-HpCDF	22	15	7	NAV	0.40	0.33	0.05	3.10	108	75
OCDF	22	12	10	NAV	0.90	0.56	0.16	23.8	155	47
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	21	20	1	2.3	2.4	2.3	0.00	4.59	44	70
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	21	21	0	2.7	2.7	2.7	0.64	16.00	48	76
PCB 77	23	22	1	10.4	12.0	10.4	2.68	6040	90	66
PCB 81	23	16	7	NAV	1.8	1.6	0.09	3995	112	54
PCB 126	23	23	0	17.9	17.3	17.9	3.56	11619806	33	71
PCB 169	23	20	3	7.9	7.9	7.9	0.56	5690578	22	61
PCB 105	25	25	0	529	552	529	242	5906	41	76
PCB 114	24	23	1	110	112	110	16.1	1009	26	67
PCB 118	25	25	0	2325	2277	2325	810	27181	24	65
PCB 123	24	24	0	28.7	31.2	28.7	8.24	5523	68	62
PCB 156	25	22	3	1410	1383	1410	167	131218	18	57
PCB 157	24	22	2	227	228	227	39.7	131218	17	54
PCB 167	23	21	2	345	353	345	144	3372	15	56
PCB 189	24	23	1	124	127	124	22.4	2212	19	60
WHO2005-TEQ (dl-PCB) LB (ND = 0)	22	22	0	2.2	2.2	2.2	1.34	17590000	31	75
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	22	22	0	2.2	2.2	2.2	1.34	17580000	31	74
WHO2005-TEQ (total) LB (ND = 0)	21	21	0	4.3	4.3	4.3	2.77	24.0	37	74
WHO2005-TEQ (total) UB (ND = LOD)	21	21	0	4.9	5.0	4.9	3.11	25.0	36	73

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	13	27	23	18	5
1,2,3,7,8-PnCDD	13	32	18	9	18
1,2,3,4,7,8-HxCDD	13	23	14	14	14
1,2,3,6,7,8-HxCDD	13	50	18	9	5
1,2,3,7,8,9-HxCDD	13	50	5	23	0
1,2,3,4,6,7,8-HpCDD	13	55	14	18	5
OCDD	13	59	9	18	9
2,3,7,8-TeCDF	13	68	9	5	5
1,2,3,7,8-PnCDF	13	41	9	9	14
2,3,4,7,8-PnCDF	13	45	23	5	14
1,2,3,4,7,8-HxCDF	13	45	9	14	14
1,2,3,6,7,8-HxCDF	13	59	0	9	14
1,2,3,7,8,9-HxCDF	13	0	0	0	0
2,3,4,6,7,8-HxCDF	13	41	9	18	5
1,2,3,4,6,7,8-HpCDF	13	45	14	18	14
1,2,3,4,7,8,9-HpCDF	13	0	0	0	0
OCDF	13	0	0	0	0
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	12	48	10	19	19
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	12	48	19	19	14
PCB 77	13	26	4	35	30
PCB 81	13	0	0	0	0
PCB 126	13	48	22	17	13
PCB 169	13	61	4	9	13
PCB 105	14	52	8	28	12
PCB 114	14	54	21	4	17
PCB 118	14	64	4	24	8
PCB 123	14	42	4	17	38
PCB 156	14	64	4	8	12
PCB 157	14	58	4	8	21
PCB 167	14	58	4	13	13
PCB 189	14	54	13	8	21
WHO2005-TEQ (dl-PCB) LB (ND = 0)	13	50	32	9	9
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	13	50	32	9	9
WHO2005-TEQ (total) LB (ND = 0)	12	48	29	19	5
WHO2005-TEQ (total) UB (ND = LOD)	12	52	19	19	10

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

Air extract (TOL) Analyte	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
2,3,7,8-TeCDD	38	38	0	27.0	26.9	27.0	0.02	47.9	16	68
1,2,3,7,8-PnCDD	38	38	0	51.6	51.3	51.6	0.05	67.8	20	77
1,2,3,4,7,8-HxCDD	38	38	0	51.2	52.1	51.2	0.04	78.0	18	71
1,2,3,6,7,8-HxCDD	38	38	0	54.9	54.8	54.9	0.05	73.0	15	69
1,2,3,7,8,9-HxCDD	38	38	0	53.4	51.6	53.4	0.04	72.4	22	74
1,2,3,4,6,7,8-HpCDD	38	38	0	117	113	117	0.10	150	17	71
OCDD	38	38	0	130	129	130	0.03	229	20	70
2,3,7,8-TeCDF	38	38	0	31.6	30.5	31.6	1.09	52.7	19	70
1,2,3,7,8-PnCDF	38	38	0	57.9	57.0	57.9	1.39	80.0	14	69
2,3,4,7,8-PnCDF	38	38	0	54.5	53.0	54.5	0.67	82.1	22	75
1,2,3,4,7,8-HxCDF	38	38	0	58.8	59.5	58.8	0.36	85.3	22	73
1,2,3,6,7,8-HxCDF	38	38	0	57.7	57.0	57.7	0.19	86.3	21	74
1,2,3,7,8,9-HxCDF	38	38	0	55.5	55.2	55.5	0.91	72.0	24	78
2,3,4,6,7,8-HxCDF	38	38	0	57.6	56.0	57.6	0.32	77.0	18	71
1,2,3,4,6,7,8-HpCDF	38	38	0	118	120	118	0.17	175	19	71
1,2,3,4,7,8,9-HpCDF	38	38	0	111	110	111	0.42	146	17	72
OCDF	38	38	0	115	117	115	0.03	847	24	68
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	37	37	0	145	142	145	0.10	175	18	76
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	37	37	0	145	142	145	0.10	175	18	76
PCB 77	36	32	4	177	175	177	0.01	573	24	67
PCB 81	36	32	4	44.7	45.0	44.7	0.003	268	31	63
PCB 126	36	31	5	58.6	58.7	58.6	2.91	38156746	11	50
PCB 169	36	31	5	39.8	41.0	39.8	0.86	29342	18	59
PCB 105	38	36	2	2356	2322	2356	0.06	27000	20	65
PCB 114	37	36	1	166	177	166	0.004	8776	38	65
PCB 118	38	36	2	6088	6079	6088	0.14	13836	22	64
PCB 123	36	34	2	133	146	133	0.002	3143	56	69
PCB 156	37	35	2	506	503	506	0.01	1400	16	58
PCB 157	35	31	4	133	130	133	0.003	274	24	66
PCB 167	36	32	4	237	234	237	0.02	1156	13	60
PCB 189	36	32	4	52.5	50.7	52.5	0.001	69	19	67
WHO2005-TEQ (dl-PCB) LB (ND = 0)	35	35	0	7.0	7.4	7.0	0.21	38186695	35	69
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	35	35	0	7.4	7.5	7.4	0.38	38190000	10	52
WHO2005-TEQ (total) LB (ND = 0)	32	32	0	157	156	157	7.39	186	13	73
WHO2005-TEQ (total) UB (ND = LOD)	32	32	0	157	157	157	7.39	186	14	75

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

Air extract (TOL)	% 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	71	13	8	8
1,2,3,7,8-PnCDD	22	84	8	3	5
1,2,3,4,7,8-HxCDD	22	76	8	8	8
1,2,3,6,7,8-HxCDD	22	79	11	3	8
1,2,3,7,8,9-HxCDD	22	76	13	3	8
1,2,3,4,6,7,8-HpCDD	22	76	11	5	8
OCDD	22	74	11	5	11
2,3,7,8-TeCDF	22	76	3	13	8
1,2,3,7,8-PnCDF	22	79	8	5	8
2,3,4,7,8-PnCDF	22	74	13	8	5
1,2,3,4,7,8-HxCDF	22	68	16	8	8
1,2,3,6,7,8-HxCDF	22	74	13	5	8
1,2,3,7,8,9-HxCDF	22	82	8	3	8
2,3,4,6,7,8-HxCDF	22	74	11	8	8
1,2,3,4,6,7,8-HpCDF	22	74	13	5	8
1,2,3,4,7,8,9-HpCDF	22	76	11	5	8
OCDF	22	61	13	16	11
WHO2005-TEQ (PCDD PCDF) LB (ND = 0)	21	84	8	3	5
WHO2005-TEQ (PCDD PCDF) UB (ND = LOD)	21	84	8	3	5
PCB 77	21	56	17	6	11
PCB 81	21	47	11	11	19
PCB 126	21	56	8	8	14
PCB 169	21	53	11	8	14
PCB 105	22	68	5	11	11
PCB 114	21	51	8	14	24
PCB 118	22	66	5	11	13
PCB 123	21	38	8	19	27
PCB 156	22	61	13	5	13
PCB 157	21	56	11	8	11
PCB 167	21	62	5	5	14
PCB 189	21	62	14	5	5
WHO2005-TEQ (dl-PCB) LB (ND = 0)	20	54	17	3	26
WHO2005-TEQ (dl-PCB) UB (ND = LOD)	20	57	11	9	23
WHO2005-TEQ (total) LB (ND = 0)	18	88	6	3	3
WHO2005-TEQ (total) UB (ND = LOD)	18	88	6	3	3

3.2.4 Polybrominated diphenyl ethers (PBDE)

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

Test Solution M	n			Theoretical	AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV								
Analyte				conc.							
BDE 17	31	31	0	348	320	321	320	90.0	425	18	74
BDE 28	37	37	0	174	178	179	178	64.0	267	16	71
BDE 47	38	38	0	696	665	659	665	219	910	16	76
BDE 99	39	39	0	348	342	340	342	0.21	747	8	59
BDE 100	38	38	0	696	673	669	673	191	930	19	70
BDE 153	39	39	0	348	327	316	327	97.8	477	18	66
BDE 154	37	37	0	174	172	173	172	63.7	355	19	69
BDE 183	39	39	0	348	303	304	303	91.7	601	21	69
Sum PBDE LB (ND = 0)	30	30	0		3089	3059	3089	1057	3881	10	63
Sum PBDE UB (ND = LOD)	30	30	0		3089	3059	3089	1057	3881	10	63
PBB 153	16	16	0	696	572	559	572	140	1107	27	68

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

Test Solution M	% of the data received	% of z-scores	% of z-scores	% of z-scores	% of z-scores
		z <2	3> z >2	6> z >3	z >6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
BDE 17	18	81	10	10	0
BDE 28	21	76	11	14	0
BDE 47	22	84	8	8	0
BDE 99	22	82	5	8	5
BDE 100	22	74	8	18	0
BDE 153	22	67	13	21	0
BDE 154	21	73	5	14	8
BDE 183	22	69	13	15	3
Sum PBDE LB (ND = 0)	17	77	7	17	0
Sum PBDE UB (ND = LOD)	17	77	7	17	0
PBB 153	9	69	6	6	19

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

Sediment	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
Analyte										
BDE 47	27	25	2	NAV	1.0	0.96	0.28	24.0	75	67
BDE 99	27	25	2	NAV	1.5	1.5	0.19	51.9	96	69
BDE 100	27	25	2	NAV	0.35	0.34	0.11	22.0	92	65
BDE 153	27	22	5	0.20	0.20	0.20	0.06	15.0	83	65
BDE 154	27	24	3	0.13	0.14	0.13	0.01	41.8	91	72
BDE 183	27	20	7	0.10	0.10	0.10	0.03	13.0	23	57
Sum PBDE LB (ND = 0)	26	25	1	NAV	3.3	3.2	0.00	122	84	70
Sum PBDE UB (ND = LOD)	26	26	0	NAV	3.2	3.1	0.91	122	80	68

Table 35: Summary of laboratory performance PBDE, 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
BDE 47	15	0	0	0	0
BDE 99	15	0	0	0	0
BDE 100	15	0	0	0	0
BDE 153	15	26	7	33	15
BDE 154	15	37	7	26	19
BDE 183	15	48	7	7	11
Sum PBDE LB (ND = 0)	15	0	0	0	0
Sum PBDE UB (ND = LOD)	15	0	0	0	0

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

Fish	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
BDE 47	23	23	0	20.3	19.9	20.3	0.24	26.9	20	74
BDE 99	23	23	0	10.1	10.0	10.1	1.75	16.1	8	60
BDE 100	23	23	0	4.8	4.8	4.8	2.80	18.0	15	72
BDE 153	23	22	1	2.4	2.4	2.4	0.93	16.0	9	62
BDE 154	23	23	0	1.2	1.2	1.2	0.63	34.3	19	72
BDE 183	23	21	2	0.20	0.20	0.20	0.11	21.0	14	66
Sum PBDE LB (ND = 0)	23	23	0	39.3	39.0	39.3	23.7	85.0	14	69
Sum PBDE UB (ND = LOD)	23	23	0	39.3	39.0	39.3	23.7	85.0	14	70

Table 37: Summary of laboratory performance PBDE, fish

Fish	% 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 47	13	78	9	9	4
BDE 99	13	74	4	17	4
BDE 100	13	87	0	9	4
BDE 153	13	78	4	9	4
BDE 154	13	83	0	9	9
BDE 183	13	83	4	0	4
Sum PBDE LB (ND = 0)	13	78	13	4	4
Sum PBDE UB (ND = LOD)	13	78	13	4	4

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

Human milk	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
BDE 47	17	16	1	0.44	0.46	0.44	0.01	3175	31	62
BDE 99	17	15	2	0.11	0.13	0.11	0.06	1021	49	62
BDE 100	17	15	2	0.12	0.13	0.12	0.08	1112	35	62
BDE 153	17	15	2	0.50	0.50	0.50	0.01	6193	21	64
BDE 154	17	9	8	NAV	0.02	0.01	0.007	205	98	54
BDE 183	17	10	7	0.03	0.03	0.03	0.02	553	32	57
Sum PBDE LB (ND = 0)	17	16	1	1.21	1.26	1.21	0.000	12259	29	64
Sum PBDE UB (ND = LOD)	17	17	0	1.24	1.34	1.24	0.07	12259	38	62

Table 39: Summary of laboratory performance PBDE, human milk

Human milk	% 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
Analyte					
BDE 47	10	59	6	0	29
BDE 99	10	53	12	0	24
BDE 100	10	65	0	0	24
BDE 153	10	71	0	0	18
BDE 154	10	0	0	0	0
BDE 183	10	47	0	0	12
Sum PBDE LB (ND = 0)	10	59	6	0	29
Sum PBDE UB (ND = LOD)	10	59	6	0	35

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

Air extract (TOL)	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
Analyte										
BDE 17	20	19	1	1.6	1.6	1.6	0.69	48.2	22	69
BDE 28	23	22	1	3.5	3.4	3.5	0.60	50.7	17	75
BDE 47	25	25	0	9.8	9.7	9.8	1.77	46.0	9	59
BDE 99	25	24	1	14.6	14.8	14.6	2.66	51.8	12	60
BDE 100	24	22	2	5.2	5.2	5.2	1.24	7.81	17	66
BDE 153	25	23	2	3.4	3.4	3.4	0.64	6.50	12	63
BDE 154	24	22	2	3.4	3.4	3.4	0.84	4.30	19	71
BDE 183	24	21	3	1.8	1.8	1.8	0.25	3.03	18	66
Sum PBDE LB (ND = 0)	20	20	0	43.0	43.3	43.0	28.0	166	13	67
Sum PBDE UB (ND = LOD)	19	19	0	43.4	43.9	43.4	28.0	356	13	66

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

Air extract (TOL)	% 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
Analyte					
BDE 17	11	60	15	10	10
BDE 28	13	74	13	0	9
BDE 47	14	76	8	4	12
BDE 99	14	72	8	8	8
BDE 100	14	67	13	13	0
BDE 153	14	72	12	0	8
BDE 154	14	71	13	8	0
BDE 183	14	58	8	17	4
Sum PBDE LB (ND = 0)	11	80	10	5	5
Sum PBDE UB (ND = LOD)	11	79	5	5	11

3.2.5 Hexabromobiphenyl (HxBB)

Table 42: Summary results HxBB, test solution S (ng/g)

Test Solution K and L	n			Theoretical						Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV	conc.	AV	Median	Mean	Min	Max		
PBB 153	13	11	2	11.3	10.4	12.47	10.4	3.24	699	37	62

Table 43: Summary of laboratory performance HxBB, test solution S

Test Solution S	% 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
PBB 153	7	54	8	8	15

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

Sediment	n			Theoretical						Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV	AV	Median	Mean	Min	Max			
PBB 153	16	8	8	NAV	0.99	0.29	0.0008	82717	429	34	

Table 45: Summary of laboratory performance HxBB, 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
PBB 153	9	0	0	0	0

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

Fish	n			Theoretical						Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV	AV	Median	Mean	Min	Max			
PBB 153	10	9	1	0.38	0.40	0.38	0.29	2174964	20	61	

Table 47: Summary of laboratory performance HxBB, fish

Fish	% 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
PBB 153	6	70	0	0	20

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

Human milk	n			Theoretical						Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV	AV	Median	Mean	Min	Max			
PBB 153	9	5	4	NAV	0.04	0.04	0.04	9145	9	58	

Table 49: Summary of laboratory performance HxBB, 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
PBB 153	5	0	0	0	0

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

Air extract (TOL)	n			Theoretical						Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV	AV	Median	Mean	Min	Max			
PBB 153	13	12	1	4.37	5.31	4.37	1.28	357	51	68	

Table 51: Summary of laboratory performance HxBB, 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
PBB 153	7	31	38	8	15

3.2.6 Toxaphenes

Table 52: Summary results toxaphenes, test solution R (ng/g)

Test Solution R	n			Theoretical						Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV	conc.	AV	Median	Mean	Min	Max		
Parlar 26	12	12	0	97.7	89.3	88.2	89.3	6.61	109	11	65
Parlar 50	12	12	0	139	127	126	127	77.0	170	26	84
Parlar 62	10	10	0	100	96.8	97.3	96.8	67.6	115	19	81
Sum toxaphenes LB (ND = 0)	11	11	0	336	317	307	317	2.13	394	14	67
Sum toxaphenes UB (ND = LOD)	11	11	0	336	317	307	317	2.13	394	14	67

Table 53: Summary of laboratory performance toxaphenes, test solution Q

Test Solution R	% 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
Analyte					
Parlar 26	7	83	8	0	8
Parlar 50	7	75	17	8	0
Parlar 62	6	90	10	0	0
Sum toxaphenes LB (ND = 0)	6	82	9	0	9
Sum toxaphenes UB (ND = LOD)	6	82	9	0	9

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

Sediment	n			Theoretical						Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV	AV	Median	Mean	Min	Max			
Parlar 26	12	3	9	NAV	27.0	0.24	0.17	55.8	1548	34	
Parlar 50	13	5	8	NAV	0.73	0.06	0.003	34.2	671	37	
Parlar 62	11	2	9	NAV	NAV	NAV	0.05	24.0	NAV	NAV	
Sum toxaphenes LB (ND = 0)	11	4	7	NAV	0.49	0.28	0.000	64.0	231	65	
Sum toxaphenes UB (ND = LOD)	11	10	1	NAV	0.47	0.36	0.006	64.0	168	67	

Table 55: Summary of laboratory performance toxaphenes, sediment

Sediment	% 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
Analyte					
Parlar 26	7	0	0	0	0
Parlar 50	7	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	0	0	0	0

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

Fish	n			Theoretical						Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV	AV	Median	Mean	Min	Max			
Parlar 26	9	6	3	NAV	0.19	0.10	0.03	2416	155	51	
Parlar 50	9	6	3	NAV	0.13	0.06	0.007	2923	271	52	
Parlar 62	8	2	6	NAV	NAV	NAV	0.01	17.0	NAV	NAV	
Sum toxaphenes LB (ND = 0)	8	5	3	NAV	0.12	0.09	0.000	48.0	127	59	
Sum toxaphenes UB (ND = LOD)	8	8	0	NAV	0.30	0.23	0.04	48.0	131	69	

Table 57: Summary of laboratory performance toxaphenes, fish

Fish	% 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

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

Human milk	n			Between lab CV						Inclusion rate (%)
	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	
Parlar 26	5	3	2	NAV	0.41	0.37	0.11	0.68	97	69
Parlar 50	6	4	2	NAV	0.58	0.57	0.32	0.92	41	61
Parlar 62	5	1	4	NAV	NAV	NAV	0.91	0.91	NAV	NAV
Sum toxaphenes LB (ND = 0)	5	4	1	NAV	0.78	0.64	0.00	2.51	63	66
Sum toxaphenes UB (ND = LOD)	5	5	0	NA0	1.93	1.76	0.30	5.03	112	81

Table 59: Summary of laboratory performance toxaphenes, human milk

Human milk	% 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 60: Summary results toxaphenes, air extract (TOL) (ng/g)

Air extract (TOL)	n			Between lab CV						Inclusion rate (%)
	Total	Numerical	LCV	AV	Median	Mean	Min	Max	(%)	
Parlar 26	6	1	5	NAV	NAV	NAV	0.19	0.19	NAV	NAV
Parlar 50	6	0	6	NAV	NAV	NAV	NAV	NAV	NAV	NAV
Parlar 62	6	0	6	NAV	NAV	NAV	NAV	NAV	NAV	NAV
Sum toxaphenes LB (ND = 0)	6	1	5	NAV	NAV	NAV	0.00	0.19	NAV	NAV
Sum toxaphenes UB (ND = LOD)	6	5	1	NAV	3.00	3.75	1.07	6.00	71	85

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

Air extract (TOL)	% 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

3.2.7 Hexabromocyclododecane (HBCD)

Table 62: Summary results HBCD, test solution O (ng/g)

Test Solution O	n			Theoretical	AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV								
Analyte				conc.							
α -HBCD	16	16	0	1153	1085	1068	1085	284	1330	14	66
β -HBCD	16	16	0	577	549	551	549	284	6054	13	72
γ -HBCD	16	16	0	288	277	289	277	216	530	12	64
Sum HBCD LB (ND = 0)	16	16	0		1861	1857	1861	853	7527	11	62
Sum HBCD UB (ND = LOD)	16	16	0		1861	1857	1861	853	7527	11	62

Table 63: Summary of laboratory performance HBCD, test solution O

Test Solution O	% of the data received	% of z-scores	% of z-scores	% of z-scores	% of z-scores
		z <2	3> z >2	6> z >3	z >6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
α -HBCD	9	75	13	13	0
β -HBCD	9	88	0	6	6
γ -HBCD	9	81	0	6	13
Sum HBCD LB (ND = 0)	9	81	6	6	6
Sum HBCD UB (ND = LOD)	9	81	6	6	6

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

Sediment	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
Analyte										
α -HBCD	7	7	0	NAV	13.80	13.81	2.90	25.2	48	72
β -HBCD	7	7	0	NAV	3.23	2.86	1.00	25.2	91	66
γ -HBCD	7	7	0	19.23	18.20	19.23	5.60	25.2	36	78
Sum HBCD LB (ND = 0)	7	7	0	NAV	33.70	38.64	9.50	75.7	58	75
Sum HBCD UB (ND = LOD)	7	7	0	NAV	33.70	38.64	9.50	75.7	58	75

Table 65: Summary of laboratory performance HBCD, sediment

Sediment	% of the data received	% of z-scores	% of z-scores	% of z-scores	% of z-scores
		z <2	3> z >2	6> z >3	z >6
Analyte		Satisfactory	Questionable	Unsatisfactory	Extreme
α -HBCD	4	0	0	0	0
β -HBCD	4	0	0	0	0
γ -HBCD	4	57	29	14	0
Sum HBCD LB (ND = 0)	4	0	0	0	0
Sum HBCD UB (ND = LOD)	4	0	0	0	0

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

Fish	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
Analyte										
α -HBCD	10	10	0	9.99	10.54	9.99	8.65	21.0	21	74
β -HBCD	10	8	2	0.07	0.10	0.07	0.03	12.0	120	64
γ -HBCD	10	9	1	NAV	0.26	0.24	0.09	17.0	97	63
Sum HBCD LB (ND = 0)	10	10	0	10.04	10.80	10.04	8.78	50.0	20	64
Sum HBCD UB (ND = LOD)	10	10	0	10.08	10.82	10.08	8.98	50.0	20	65

Table 67: Summary of laboratory performance HBCD, fish

Fish	% 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	6	80	0	10	10
β -HBCD	6	50	0	10	20
γ -HBCD	6	0	0	0	0
Sum HBCD LB (ND = 0)	6	70	0	10	20
Sum HBCD UB (ND = LOD)	6	70	0	10	20

Table 68: Summary results HBCD, human milk (lipid weight basis) (ng/g)

Human milk	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
α -HBCD	9	4	5	NAV	0.41	0.18	0.02	165	167	40
β -HBCD	9	2	7	NAV	NAV	NAV	0.0006	165	NAV	NAV
γ -HBCD	9	2	7	NAV	NAV	NAV	0.002	165	NAV	NAV
Sum HBCD LB (ND = 0)	9	4	5	NAV	0.41	0.27	0.000	496	136	67
Sum HBCD UB (ND = LOD)	9	9	0	NAV	0.60	0.35	0.009	496	202	63

Table 69: Summary of laboratory performance HBCD, 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
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 70: Summary results HBCD, air extract (TOL) (ng/g)

Air extract (TOL)	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
α -HBCD	7	6	1	NAV	3.36	3.45	0.06	3.90	12	52
β -HBCD	7	4	3	NAV	0.37	0.28	0.06	0.76	91	56
γ -HBCD	7	4	3	NAV	0.21	0.15	0.05	0.59	81	56
Sum HBCD LB (ND = 0)	7	6	1	NAV	3.68	3.71	0.00	4.66	36	73
Sum HBCD UB (ND = LOD)	7	7	0	NAV	3.86	3.67	0.17	4.66	37	64

Table 71: Summary of laboratory performance HBCD, 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
α -HBCD	4	0	0	0	0
β -HBCD	4	0	0	0	0
γ -HBCD	4	0	0	0	0
Sum HBCD LB (ND = 0)	4	0	0	0	0
Sum HBCD UB (ND = LOD)	4	0	0	0	0

3.2.8 Perfluoroalkyl substances (PFAS)

Table 72: Summary results PFAS, test solution N (ng/g)

Test Solution N	n			Theoretical	AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV								
L-PFOS anion	25	25	0	241	242	248	242	166	454	19	78
br-PFOS anion	15	15	0	48.2	47.5	50.0	47.5	30.9	176	22	66
tot-PFOS LB (ND = 0)	17	17	0		300	294	300	198	381	12	67
tot-PFOS UB (ND = LOD)	17	17	0		299	295	299	198	381	11	67
FOSA	14	14	0	316	279	299	279	8.28	632	30	69
MeFOSA	8	8	0	631	509	527	509	314	677	31	82
EtFOSA	9	9	0	316	NAV	264	233	64.4	311	51	79
MeFOSE	7	7	0	631	532	528	532	377	845	27	75
EtFOSE	6	6	0	316	275	275	275	164	335	28	80
PFOS precursors LB (ND = 0)	6	6	0		1812	1864	1812	1139	2227	25	78
PFOS precursors UB (ND = LOD)	6	6	0		1812	1864	1812	1139	2227	25	78
PFBA	17	17	0	126	115	118	115	92.4	12914	17	70
PFPeA	19	19	0	126	120	118	120	64.0	205	20	74
PFHxA	24	24	0	253	223	222	223	109	342	19	75
PFHpA	24	24	0	126	114	116	114	74.0	241	21	73
PFOA	24	24	0	253	239	240	239	171	370	20	81
PFNA	25	25	0	126	117	119	117	98.0	203	11	68
PFDA	25	25	0	126	118	118	118	75.0	217	14	72
L-PFBS	21	21	0	156	144	147	144	104	328	12	69
L-PFHxS	24	24	0	119	115	116	115	60.0	178	13	70
PFCA_s + PFSA_s LB (ND = 0)	16	16	0		1314	1306	1314	1073	14358	18	70
PFCA_s + PFSA_s UB (ND = LOD)	16	16	0		1314	1306	1314	1073	14358	18	70
PFUnDA	21	5	16	NC	NAV	0.36	0.08	0.16	9.00	167	48
PFDoDA	21	1	20	NC	NAV	NAV	NAV	8.00	8.00	NAV	NAV
PFTTrDA	19	1	18	NC	NAV	NAV	NAV	3.57	3.57	NAV	NAV
PFTeDA	18	2	16	NC	NAV	NAV	NAV	0.29	0.40	NAV	NAV
L-PFHpS	9	1	8	NC	NAV	NAV	NAV	0.12	0.12	NAV	NAV
L-PFDS	18	5	13	NC	NAV	0.17	0.08	0.10	191	85	51

NC = Not contained

Table 73: Summary of laboratory performance PFAS, test solution N

Test Solution N	% 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	80	16	0	4
br-PFOS anion	9	53	33	0	13
tot-PFOS LB (ND = 0)	10	82	18	0	0
tot-PFOS UB (ND = LOD)	10	88	12	0	0
FOSA	8	57	14	14	14
MeFOSA	5	50	38	13	0
EtFOSA	5	0	0	0	0
MeFOSE	4	71	14	14	0
EtFOSE	3	83	0	17	0
PFOS precursors LB (ND = 0)	3	67	33	0	0
PFOS precursors UB (ND = LOD)	3	67	33	0	0
PFBA	10	82	0	12	6
PFPeA	11	79	5	16	0
PFHxA	14	79	13	8	0
PFHpA	14	71	17	8	4
PFOA	14	83	13	4	0
PFNA	14	88	4	8	0
PFDA	14	88	4	4	4
L-PFBS	12	81	10	5	5
L-PFHxS	14	88	4	8	0
PFCA_s + PFSA_s LB (ND = 0)	9	75	13	6	6
PFCA_s + PFSA_s UB (ND = LOD)	9	75	13	6	6
PFUnDA	12	0	0	0	0
PFDoDA	12	0	0	0	0
PFTTrDA	11	0	0	0	0
PFTeDA	10	0	0	0	0
L-PFHpS	5	0	0	0	0
L-PFDS	10	0	0	0	0

Table 74: Summary results PFAS, sediment (ng/g)

Sediment	n								Between	Inclusion
	Total	Numerical	LCV	AV	Median	Mean	Min	Max	lab CV (%)	rate (%)
L-PFOS anion	16	14	2	0.65	0.69	0.65	0.46	5.71	20	63
br-PFOS anion	10	7	3	0.12	0.12	0.12	0.11	1.80	17	52
tot-PFOS LB (ND = 0)	11	10	1	0.76	0.79	0.76	0.00	3.23	13	67
tot-PFOS UB (ND = LOD)	11	11	0	0.79	0.86	0.79	0.65	3.23	23	62

Table 75: 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	9	56	6	13	13
br-PFOS anion	6	50	0	0	20
tot-PFOS LB (ND = 0)	6	73	0	9	9
tot-PFOS UB (ND = LOD)	6	64	0	9	27

Table 76: Summary results PFAS, fish (product basis) (ng/g)

Fish	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
L-PFOS anion	14	13	1	7.85	7.90	7.85	0.89	18.4	4	59
br-PFOS anion	10	8	2	0.56	0.57	0.56	0.24	3.55	56	59
tot-PFOS LB (ND = 0)	11	11	0	8.31	8.35	8.31	4.44	16.1	4	70
tot-PFOS UB (ND = LOD)	11	11	0	8.43	8.38	8.43	4.44	16.1	3	74

Table 77: Summary of laboratory performance PFAS, fish

Fish	% 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	71	0	0	21
br-PFOS anion	6	40	0	30	10
tot-PFOS LB (ND = 0)	6	82	0	9	9
tot-PFOS UB (ND = LOD)	6	82	0	9	9

Table 78: Summary results PFAS, human milk (product basis) (ng/g)

Human milk	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
L-PFOS anion	6	5	1	0.03	0.03	0.03	0.01	0.07	20	59
br-PFOS anion	5	4	1	NAV	0.01	0.01	0.01	0.02	6	65
tot-PFOS LB (ND = 0)	5	5	0	0.04	0.04	0.04	0.04	0.07	12	74
tot-PFOS UB (ND = LOD)	5	5	0	0.04	0.04	0.04	0.04	0.10	12	74

Table 79: 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	3	67	17	0	0
br-PFOS anion	3	0	0	0	0
tot-PFOS LB (ND = 0)	3	100	0	0	0
tot-PFOS UB (ND = LOD)	3	80	0	20	0

Table 80: Summary results PFAS, human plasma (product basis) (ng/g)

Human plasma	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
L-PFOS anion	12	12	0	3.47	3.56	3.47	3.08	4.44	7	67
br-PFOS anion	10	10	0	2.00	2.03	2.00	0.63	5.26	35	73
tot-PFOS LB (ND = 0)	10	10	0	5.52	5.55	5.52	4.24	9.70	16	72
tot-PFOS UB (ND = LOD)	10	10	0	5.59	5.55	5.59	4.24	9.70	19	76
FOSA	6	1	5	NAV	NAV	NAV	0.003	0.003	NAV	NAV
PFBA	6	1	5	NAV	NAV	NAV	0.45	0.45	NAV	NAV
PFPeA	7	0	7	NAV	NAV	NAV	NAV	NAV	NAV	NAV
PFHxA	11	0	11	NAV	NAV	NAV	NAV	NAV	NAV	NAV
PFHpA	11	0	11	NAV	NAV	NAV	NAV	NAV	NAV	NAV
PFOA	11	11	0	1.18	1.13	1.18	0.42	1.86	24	71
PFNA	11	11	0	0.48	0.50	0.48	0.29	0.95	18	72
PFDA	11	8	3	0.17	0.18	0.17	0.14	0.25	12	72
L-PFBS	8	1	7	NAV	NAV	NAV	0.11	0.11	NAV	NAV
L-PFHxS	11	11	0	1.84	1.82	1.84	1.44	2.33	10	70
PFCAs + PFSAs LB (ND = 0)	6	6	0	3.85	3.79	3.85	2.40	4.88	25	80
PFCAs + PFSAs UB (ND = LOD)	6	6	0	NAV	4.79	4.72	2.80	10.9	46	69
PFUnDA	11	8	3	0.16	0.17	0.16	0.12	0.29	12	54
PFDoDA	11	2	9	NAV	NAV	NAV	0.02	0.02	NAV	NAV
PFTTrDA	9	3	6	NAV	0.02	0.02	0.02	0.03	25	80
PFTeDA	9	0	9	NAV	NAV	NAV	NAV	NAV	NAV	NAV
L-PFHpS	4	3	1	NAV	0.15	0.14	0.12	0.17	21	64
L-PFDS	9	1	8	NAV	NAV	NAV	0.21	0.21	NAV	NAV

Table 81: Summary of laboratory performance PFAS, human plasma

Human plasma	% 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	7	83	17	0	0
br-PFOS anion	6	50	30	10	10
tot-PFOS LB (ND = 0)	6	90	0	10	0
tot-PFOS UB (ND = LOD)	6	90	0	10	0
FOSA	3	0	0	0	0
PFBA	3	0	0	0	0
PFPeA	4	0	0	0	0
PFHxA	6	0	0	0	0
PFHpA	6	0	0	0	0
PFOA	6	73	0	27	0
PFNA	6	82	9	0	9
PFDA	6	64	9	0	0
L-PFBS	5	0	0	0	0
L-PFHxS	6	91	9	0	0
PFCAs + PFSAs LB (ND = 0)	3	67	33	0	0
PFCAs + PFSAs UB (ND = LOD)	3	0	0	0	0
PFUnDA	6	55	9	9	0
PFDoDA	6	0	0	0	0
PFTTrDA	5	0	0	0	0
PFTeDA	5	0	0	0	0
L-PFHpS	2	0	0	0	0
L-PFDS	5	0	0	0	0

Table 82: Summary results PFAS, air extract (MeOH) (ng/g)

Air extract (MeOH) Analyte	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
L-PFOS anion	11	11	0	11.8	11.9	11.8	9.36	34.9	31	67
br-PFOS anion	5	2	3	NAV	NAV	NAV	0.37	8.02	NAV	NAV
tot-PFOS LB (ND = 0)	10	10	0	11.2	11.5	11.2	9.36	42.9	26	68
tot-PFOS UB (ND = LOD)	6	6	0	NAV	12.9	11.7	10.8	42.9	23	60
FOSA	8	8	0	NAV	23.0	23.0	7.60	63.2	58	72
MeFOSA	5	5	0	NAV	44.0	37.7	15.1	114	98	80
EtFOSA	6	6	0	NAV	82.0	81.6	20.9	282	99	74
MeFOSE	6	6	0	NAV	105	104	37.0	184	57	81
EtFOSE	5	5	0	NAV	50.0	46.6	44.9	100	13	58
PFOS precursors LB (ND = 0)	5	5	0	NAV	310	311	178	688	4	55
PFOS precursors UB (ND = LOD)	5	5	0	NAV	310	311	178	688	4	55

Table 83: Summary of laboratory performance PFAS, air extract (MeOH)

Air extract (MeOH) Analyte	% 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
		Satisfactory	Questionable	Unsatisfactory	Extreme
L-PFOS anion	6	64	9	9	18
br-PFOS anion	3	0	0	0	0
tot-PFOS LB (ND = 0)	6	60	20	0	20
tot-PFOS UB (ND = LOD)	3	0	0	0	0
FOSA	5	0	0	0	0
MeFOSA	3	0	0	0	0
EtFOSA	3	0	0	0	0
MeFOSE	3	0	0	0	0
EtFOSE	3	0	0	0	0
PFOS precursors LB (ND = 0)	3	0	0	0	0
PFOS precursors UB (ND = LOD)	3	0	0	0	0

Table 84: Summary results PFAS, water (pg/g)

Water Analyte	n			AV	Median	Mean	Min	Max	Between lab CV (%)	Inclusion rate (%)
	Total	Numerical	LCV							
L-PFOS anion	19	18	1	7.4	7.7	7.4	4.07	44.5	33	60
br-PFOS anion	11	11	0	NAV	3.7	3.9	1.40	15.6	73	69
tot-PFOS LB (ND = 0)	11	11	0	10	11	10	5.35	60.1	41	61
tot-PFOS UB (ND = LOD)	11	11	0	10	11	10	7.29	60.1	39	63

Table 85: Summary of laboratory performance PFAS, water

Water Analyte	% 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
		Satisfactory	Questionable	Unsatisfactory	Extreme
L-PFOS anion	11	45	5	15	25
br-PFOS anion	6	0	0	0	0
tot-PFOS LB (ND = 0)	6	45	9	9	36
tot-PFOS UB (ND = LOD)	6	45	18	0	36

3.3 Regional Participation

The following Tables (Table 86 to Table 94) show the number of laboratories reporting results per region. The largest number of laboratories reported results on indicator PCB (89 laboratories) followed by OCP reporting (80 laboratories). The lowest number of laboratories reported results for toxaphene (16 laboratories), HBCD (17 laboratories) and HxBB (19 laboratories). A quite impressive number of laboratories reported results for the more advanced POPs such as for PCDD/PCDF (59 laboratories), dl-PCB (56 laboratories) or PFAS (44 laboratories). 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 – except for HxBB – the test solution for POPs standards had the highest reporting rate. Interestingly for abiotic and biotic matrices, the matrices that are not included in UN Environment’s Global Monitoring Plan – sediment and fish – were more frequently analyzed than the core matrices human milk and air. However, the interest (and capacity) in analyzing core matrices has increased.

Table 86: Number of reporting laboratories for OCPs *per region*

Region	Total	Test solution	Sediment	Fish	Human milk	Air extract
Asia	31	29	22	16	9	11
WEOG	11	11	4	6	3	7
GRULAC	17	17	14	8	9	4
Africa	9	8	9	6	5	4
CEE	12	10	11	8	3	3
Total	80	75	60	44	29	29

Table 87: Number of reporting laboratories for PCB *per region*

Region	Total	Test solution	Sediment	Fish	Human milk	Air extract
Asia	33	31	24	18	13	17
WEOG	16	16	8	9	6	13
GRULAC	15	12	13	7	7	5
Africa	10	7	9	8	7	5
CEE	15	13	12	9	5	5
Total	89	79	66	51	38	45

Table 88: Number of reporting laboratories for PCDD/PCDF *per region*

Region	Total	Test solution	Sediment	Fish	Human milk	Air extract
Asia	30	28	23	15	13	21
WEOG	16	13	9	9	6	12
GRULAC	6	3	3	4	0	2
Africa	2	2	2	1	1	1
CEE	5	3	3	2	2	2
Total	59	49	40	31	22	38

Table 89: Number of reporting laboratories for dl-PCB *per region*

Region	Total	Test solution	Sediment	Fish	Human milk	Air extract
Asia	26	24	18	15	13	17
WEOG	15	12	8	9	7	12
GRULAC	4	2	2	3	0	2
Africa	3	3	3	3	3	3
CEE	8	5	6	4	3	4
Total	56	46	37	34	26	38

Table 90: Number of reporting laboratories for PBDE *per region*

Region	Total	Test solution	Sediment	Fish	Human milk	Air extract
Asia	20	17	13	10	9	10
WEOG	13	13	6	9	5	11
GRULAC	3	3	3	1	1	1
Africa	3	3	3	1	1	1
CEE	5	3	3	2	1	2
Total	44	39	28	23	17	25

Table 91: Number of reporting laboratories for HxBBB *per region*

Region	Total	Test solution	Sediment	Fish	Human milk	Air extract
Asia	10	6	8	3	3	7
WEOG	5	3	4	5	4	4
GRULAC	0	0	0	0	0	0
Africa	4	4	4	2	2	2
CEE	0	0	0	0	0	0
Total	19	13	16	10	9	13

Table 92: Number of reporting laboratories for toxaphenes *per region*

Region	Total	Test solution	Sediment	Fish	Human milk	Air extract
Asia	8	6	7	5	3	4
WEOG	4	4	2	3	2	1
GRULAC	3	3	3	0	1	2
Africa	0	0	0	0	0	0
CEE	1	1	1	1	0	0
Total	16	14	13	9	6	7

Table 93: Number of reporting laboratories for HBCDs *per region*

Region	Total	Test solution	Sediment	Fish	Human milk	Air extract
Asia	8	7	3	5	5	3
WEOG	7	7	2	4	3	3
GRULAC	0	0	0	0	0	0
Africa	2	2	2	1	1	1
CEE	0	0	0	0	0	0
Total	17	16	7	10	9	7

Table 94: Number of reporting laboratories for PFAS *per region*

Region	Total	Test solution	Sediment	Fish	Human milk	Human plasma	Air extract	Water
Asia	10	9	5	5	3	6	3	6
WEOG	14	14	9	8	3	6	6	9
GRULAC	1	1	0	0	0	0	1	1
Africa	2	2	2	1	0	0	1	2
CEE	2	1	1	1	0	0	0	2
Total	29	27	17	15	6	12	11	20

4 DISCUSSION

4.1 Methodological Considerations

It is always a challenge to identify trends in an interlaboratory assessment dataset and to explain the underlying methodological causes. 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 be elucidated.

The POPs concentrations in all matrices except human milk are presented on a wet weight basis. The interlaboratory comparison of lipid weight concentrations is rather vulnerable to interlaboratory variation in determination of lipid content (Karl *et al.*, 2012). Furthermore, the combination of high lipid content and low concentrations tend to cause higher RSD values (Wells and De Boer, 2006). Participants were asked, however, to report the lipid content of human milk, so it could be used when needed for interpretation of the data.

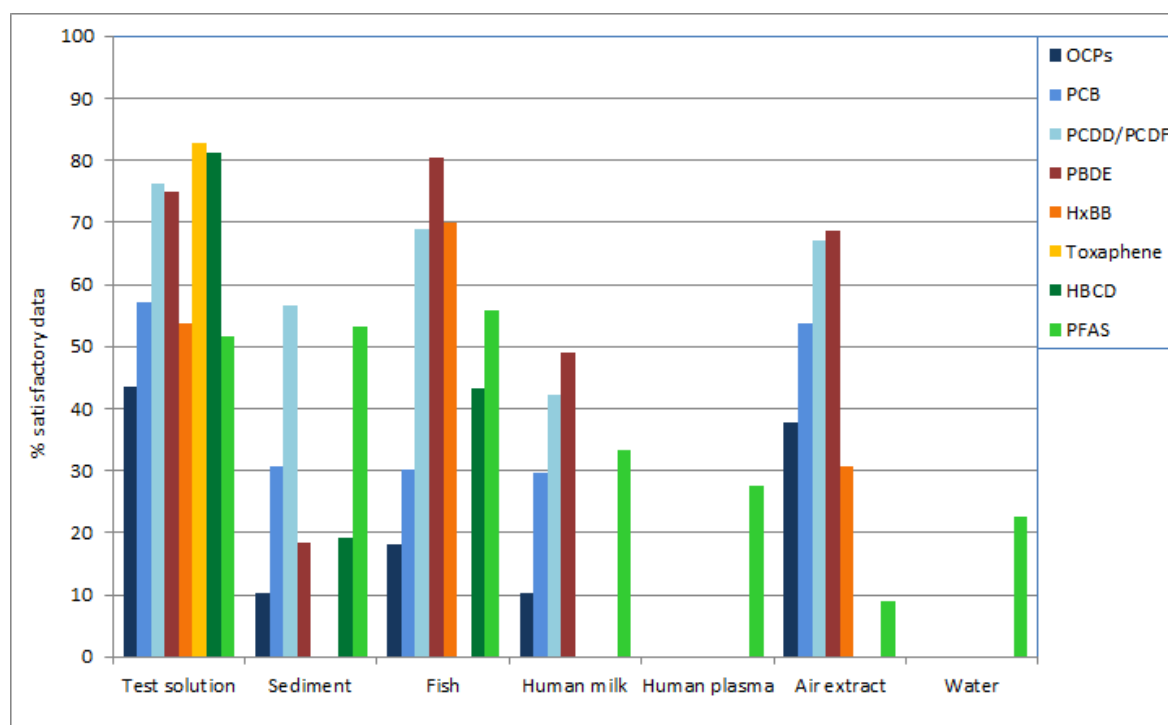


Figure 1 Percentage of laboratories with satisfactory z-scores in the analysis of OCPs, PCB, PCDD/PCDF, PBDE, HxBB, toxaphene, HBCD and PFAS, with the compounds included, which did not receive an assigned value.

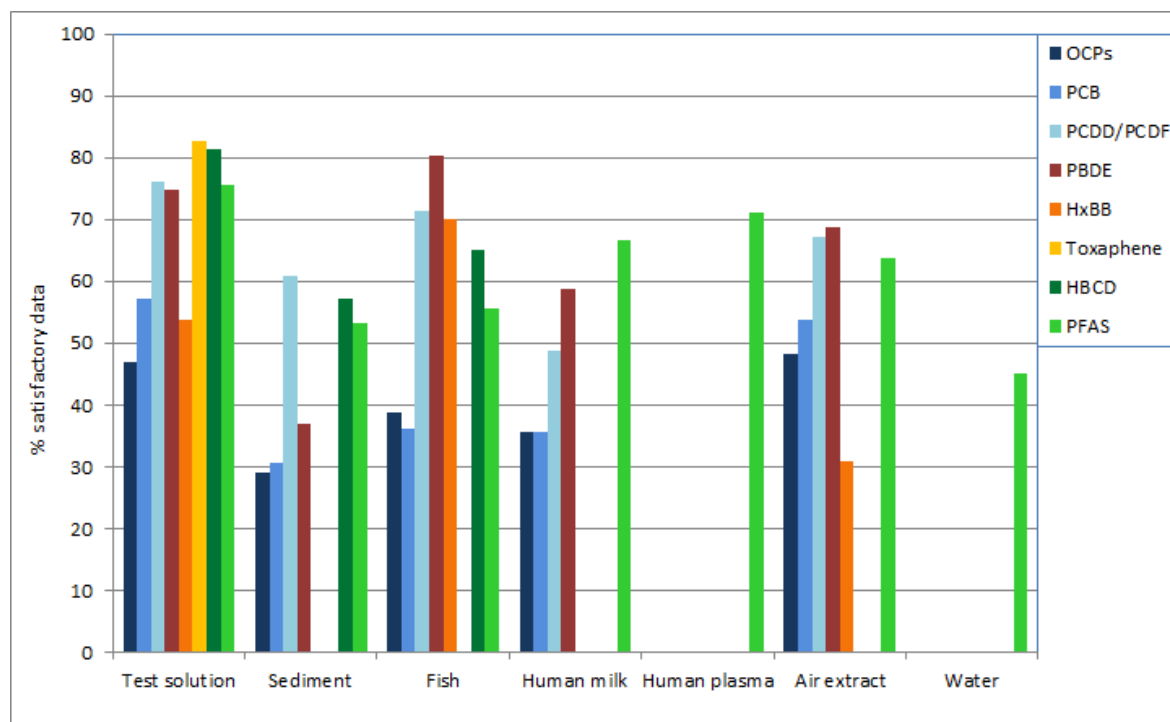


Figure 2 Percentage of laboratories with satisfactory z-scores in the analysis of OCPs, PCB, PCDD/PCDF, PBDE, HxBB, toxaphene, HBCD and PFAS, for all the compounds, which received an assigned value.

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 PCBs 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/Fs, PBDEs, PFAS and surprisingly also toxaphene, showed a better performance, although in fact with the target of 25% CV the performance should be closer to 100%.

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 dramatic. The substantial percentage of newcomers in this exercise may have contributed to this poor result, but more experienced laboratories should also do better. The air extract results show somewhat better results, which is probably due to the absence of matrix and the fortification of POP concentrations. However, 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 with improvement for the plasma compared to the previous round when the percentage of satisfactory z-scores was still below 50%.

Overall, there are still too few laboratories submitting satisfactory results.

4.2 Analyte Group - Specific Performance

4.2.1 Organochlorine Pesticides

The individual results for the OCPs for the test solution show between-lab model CV values of 41%-57% for the drins, 19%-40% for the chlordanes and 27%-52% for the DDTs (Table 2). This is illustrated in Figure 3 for dieldrin (41%), in which the individual results from each laboratory are given in addition to the consensus value as calculated by the Cofino statistics and the UN Environment criteria of 12.5% ($z = 1$) and 25% ($z = 2$). Although the concentrations of OCPs in the test solution were all in the same range as in the second round, the percentage of satisfactory z-scores decreased for each individual compound, and the overall satisfactory z-scores decreased dramatically from 61% in the second round to 44% in this round (Figure 1). Those results are very disappointing. Laboratories should collectively be able to determine OCPs in a test solution, so without any matrix, much better than $\pm 25\%$. Now we have 75 laboratories that analysed 28 OCPs of which for only five (oxychlordane, cis and trans-nonachlor, mirex and pentachlorobenzene) a CV of $<25\%$ was obtained. Given the increase in the total number of participating laboratories, it is fair to suppose that a substantial number of newcomers may not have their OCP method under control. However, the performance of some of the meanwhile more experienced is also disappointing.

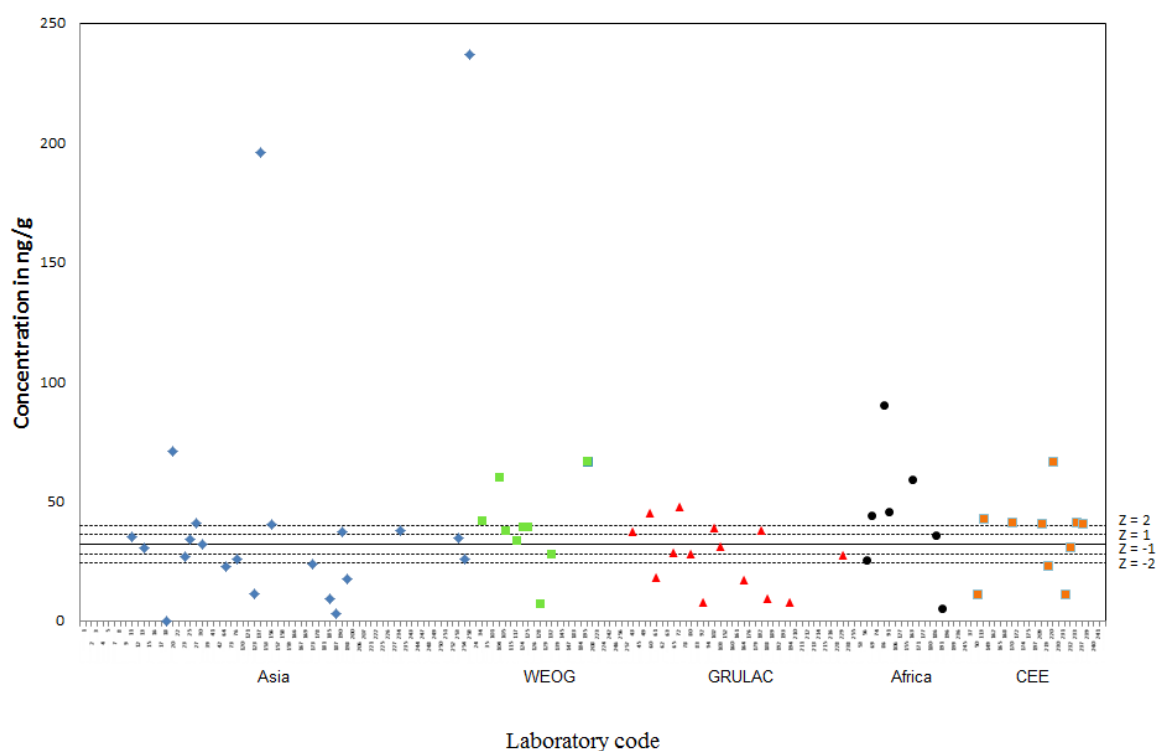


Figure 3 Results for dieldrin in the test solution. Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

The results for OCPs in the air extract showed between-lab model CV values of 32%-51% for the drins, 4%-22% for the chlordanes and 13%-43% for the DDTs (Table 10). In the second round endosulfan sulphate was analysed for the first time, which resulted in a model CV value of 91%. Although in this round again only eight laboratories analysed this compound in air, the CV value

improved to 33%. Unfortunately, the β -endosulfan CV value is still at an unsatisfactory level of 82%. The results of the other two compounds with poor results in the second round, hexachlorobenzene (CV = 68%), and endrin (CV = 58%) were much better in this round with CV values of respectively 22% and 32% even though the concentrations in this round were lower. The average between-lab model CV value over all OCPs in the air extract was equal to the second round with 32%, although the average concentration decreased with a factor of 10. This is a hopeful sign. Improvements are possible and necessary, but apparently, the absence of large matrix effects such as in sediment and fish helps the laboratories in their analysis. So, from the analytical point of view, including air in the GMP was a very good choice.

As expected, the model CV values for the other materials are larger than for the test solution and the air extract. For the sediment an extremely high average model CV value of 196% has been calculated for OCPs, with the highest variation calculated for α -, and γ -chlordane and α -endosulfan, 520%, 596%, and 511% respectively. For 18 of the 28 compounds it was not even possible to calculate an assigned value for the sediment.

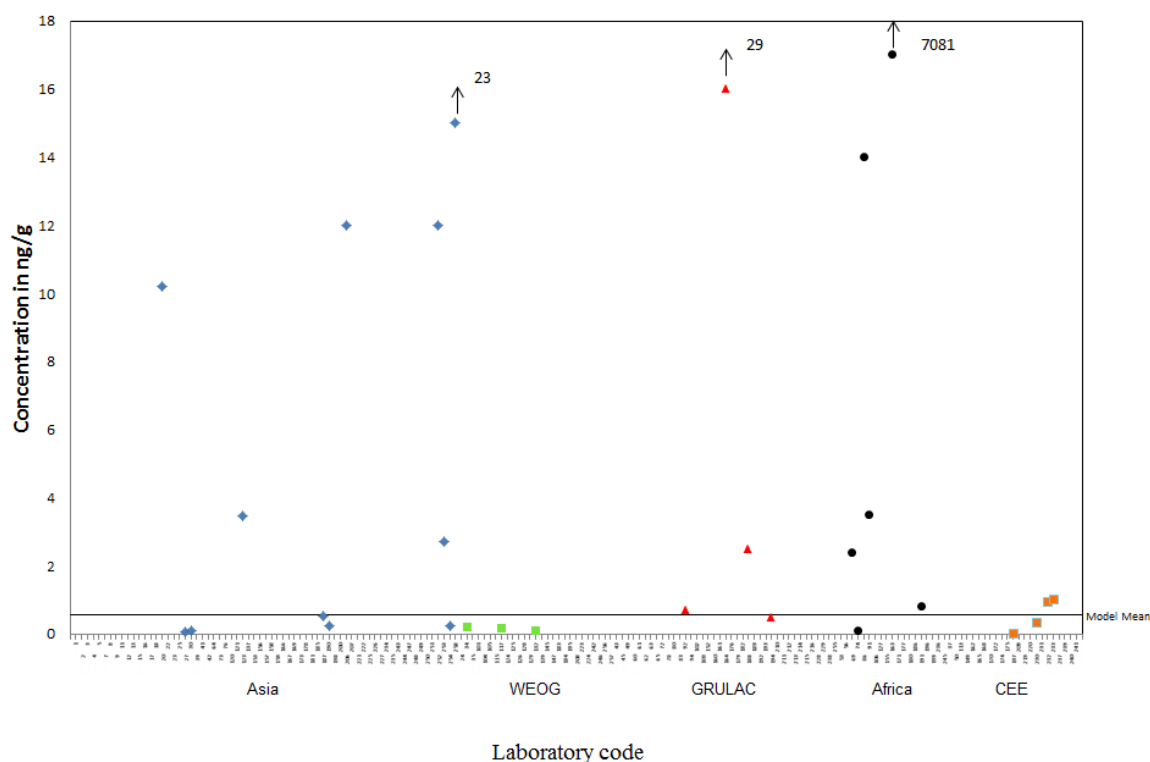


Figure 4 Results for dieldrin in the sediment sample.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The model mean value given by straight line. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

As an example of the large inter-laboratory variation, the results of dieldrin in the sediment sample (CV 195%) are given in Figure 4. The outliers on the high side are most likely caused by interferences in the chromatogram. To determine dieldrin, sulphuric acid treatment is not allowed, as that causes degradation of dieldrin (as well as endrin and some other OCPs). Consequently, the dieldrin peak in GC-ECD chromatograms is often hindered by interferences. The use of a mass spectrometric detector would overcome this problem, which is clearly shown by the results in Figure 5, which shows the results of dieldrin in the sediment sample arranged by detection system. Except one result, all results obtained with MS, are lower than the model mean value. Although sensitivity is sometimes an issue with MS for OCPs, the much better isolation from interferences with MS is a

substantial improvement. Although more expensive, MS/MS is even a better alternative as that also offers a better sensitivity.

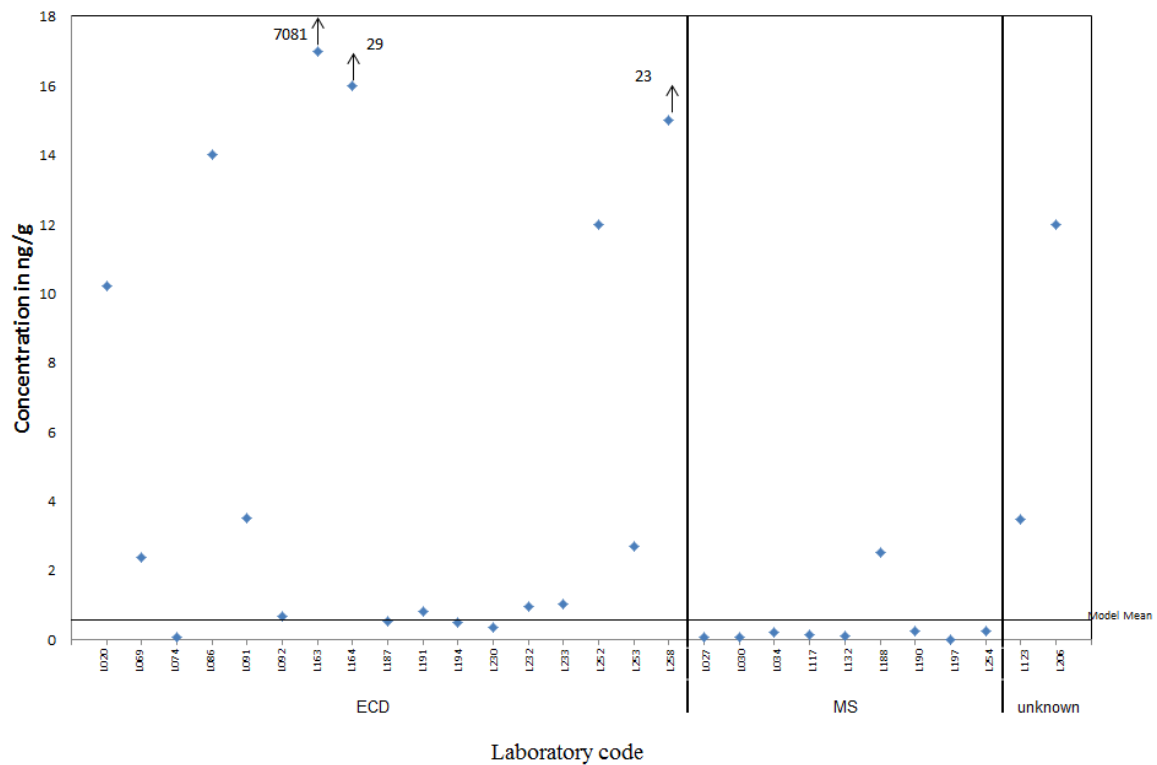


Figure 5 Results for dieldrin in the sediment sample arranged by detection method. Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines.

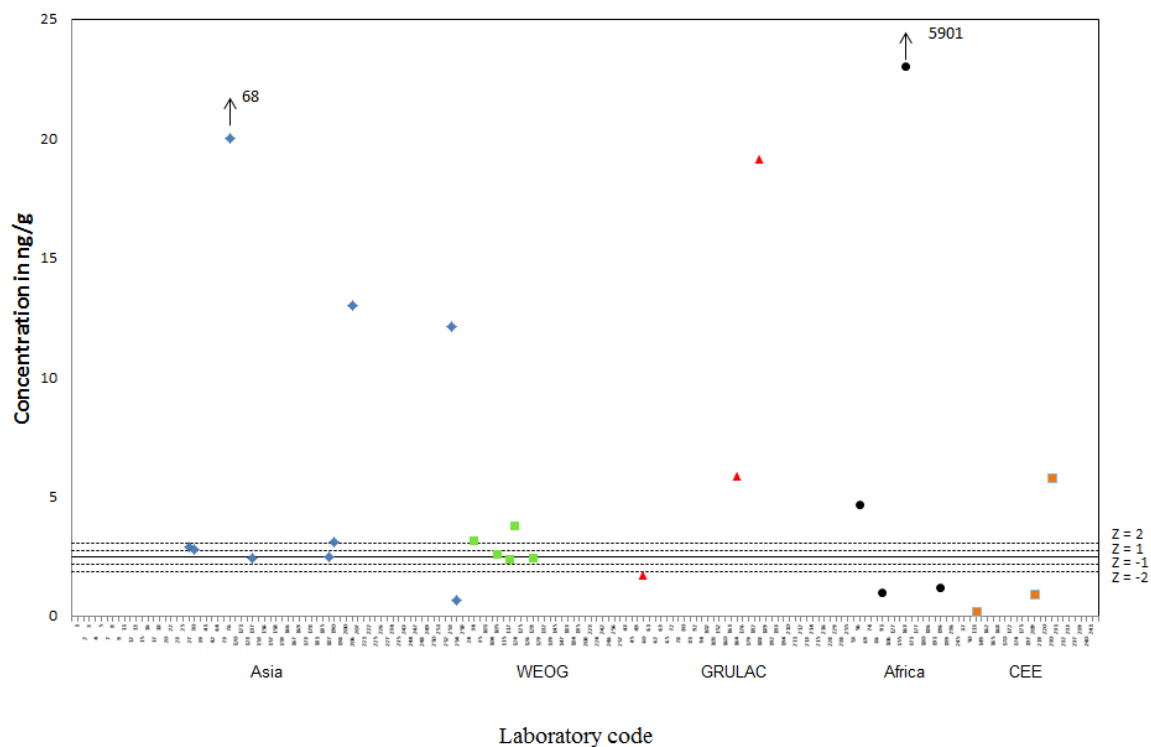


Figure 6 Results for dieldrin in the crab sample. Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

The largest variation was seen for the OCPs in sediment, crab and human milk. Often, less than 50% of the data showed satisfactory z-scores (see Table 5, Table 7 and Table 9). For sediment for none of the compounds more than 50% received a satisfactory z-score. For human milk only for cis- and nonachlor 56% and 50% received a satisfactory z-score. For six other compounds in the milk sample between 20% and 50% received a satisfactory z-score. For all other compounds ($n=20$) it was not possible to calculate an assigned value. For only five compounds in the fish material, more than 50% received a satisfactory z-score, and the model between lab CVs ranged from 13% for cis-nonachlor to 583% for trans-heptachlorepoxide, with an average CV value of 175%. The variation for dieldrin in the fish sample was better than in sediment, but the CV was with 92% still much too high (Figure 6). Figure 7 shows the results of dieldrin in the fish sample arranged by detection system. Although the results are not as clear as for the sediment sample, again it can be observed that detection with MS results in a lower CV than detection with ECD.

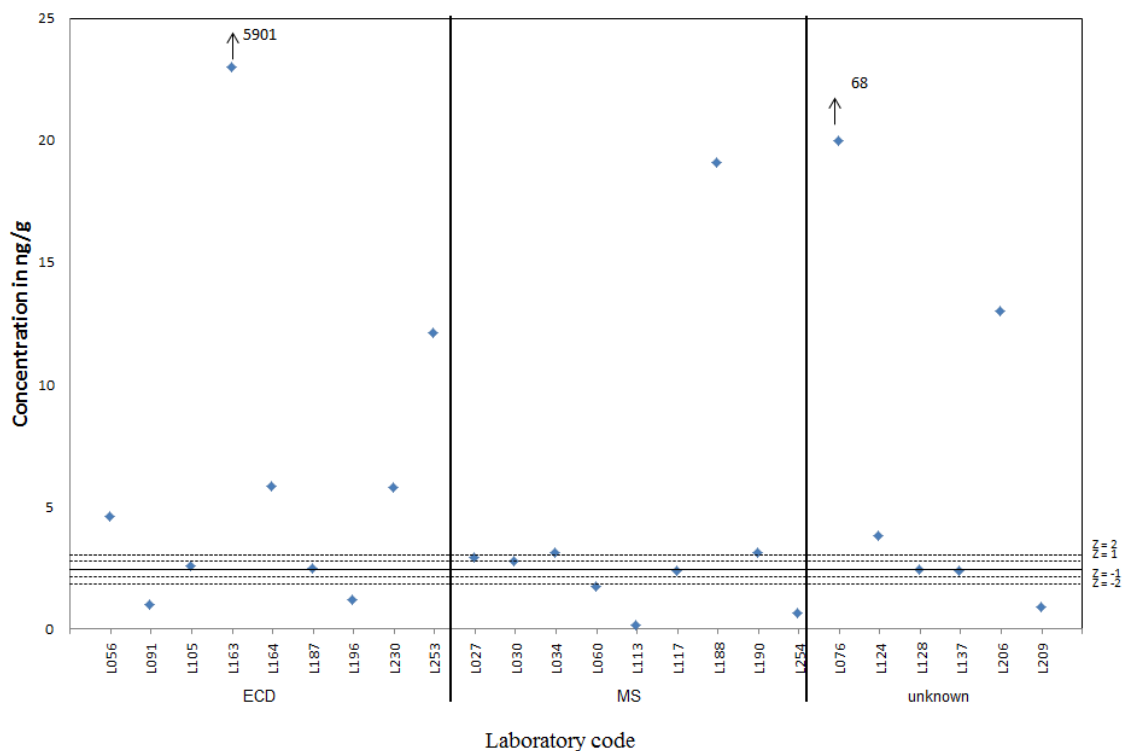


Figure 7 Results for dieldrin in the fish sample arranged by detection method. Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines.

4.2.2 Polychlorinated Biphenyls

The overall performance for the analyses of the indicator PCB was clearly better than for the OCPs. The test solution was somewhat better analysed than the OCP solution, but also here much improvement is needed (see 4.2.1). 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 present study (Figure 1 and Figure 8, Table 13). The best results were obtained for the air extract for which between-lab CV values of 20-45% were found (Table 20). The performance for the air extract with 54% satisfactory z-scores in the present study (Table 21) in comparison with 22% in the previous round. Possibly, the higher PCB concentrations in this round, 35-65-fold higher than in previous round, may have helped. The results for the other test materials show a larger variation. The between-lab CV values for human milk ranged from 25% to 127% (Table 18), for fish from 58% to 63% (Table 16) and for sediment from 53% to 75% (Table 14). Although the concentrations in sediment were in the same range as in the previous study the overall percentage of satisfactory z-scores received decreased dramatically for the sediment material from 62% in the second round to 31% in the present study (Table 15). For the crab sample the concentration in the present study was ten times higher than in the previous round, but still the percentage of satisfactory z-scores decreased from 44% to 30% (Table 17). Matrix effects caused by a difference between fish and crab could have played a role, but also for the human milk sample the percentage of satisfactory z-scores decreased (second round, 55%; present study 30%) (Table 19). However, for this matrix the concentration in the present study was 40 times lower than in the second round of the study.

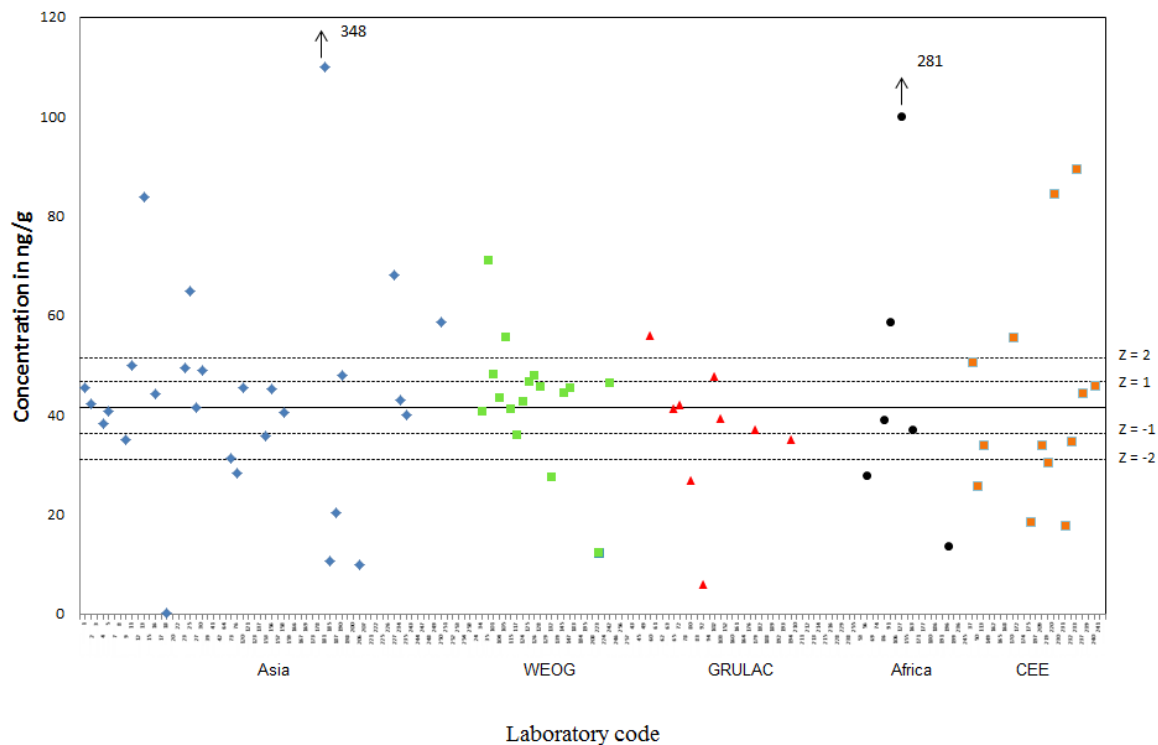


Figure 8 Results for sum of indicator PCB in the test solution.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

Plotting the CB153 results in sediment *per* detection method (Figure 9) shows that all results obtained by ECD received a not-satisfactory z-score either positive or negative, while with MS the majority received a satisfactory z-score. In general PCBs do not suffer from much interference when analysed by ECD. The first fractions after clean up containing the PCBs normally consist of non-polar solvents only, which do not contain the interferences that are present in the much more polar second fraction that contains the OCPs. GC/ECD should therefore be a good method for PCB analysis as it combines a very high sensitivity with a very good selectivity. However, meanwhile more and more laboratories in the WEOG group, which are more experienced than those in the other groups do their PCB analysis by MS. That probably explains the picture that emerges from Figure 9. Some of the participants reported that PCB 153 coeluted with PCB 168 on their column. Quantifying PCB 153 and PCB 168 together as PCB 153 would result in a higher z-score. This coelution was, however, not reported by the participants who received a positive unsatisfactory z-score.

The data in Figure 9 contains some obvious outliers. By using the Cofino statistics the outliers do not have a great influence on the model. If this model was not used, the interlaboratory variation would have been much higher.

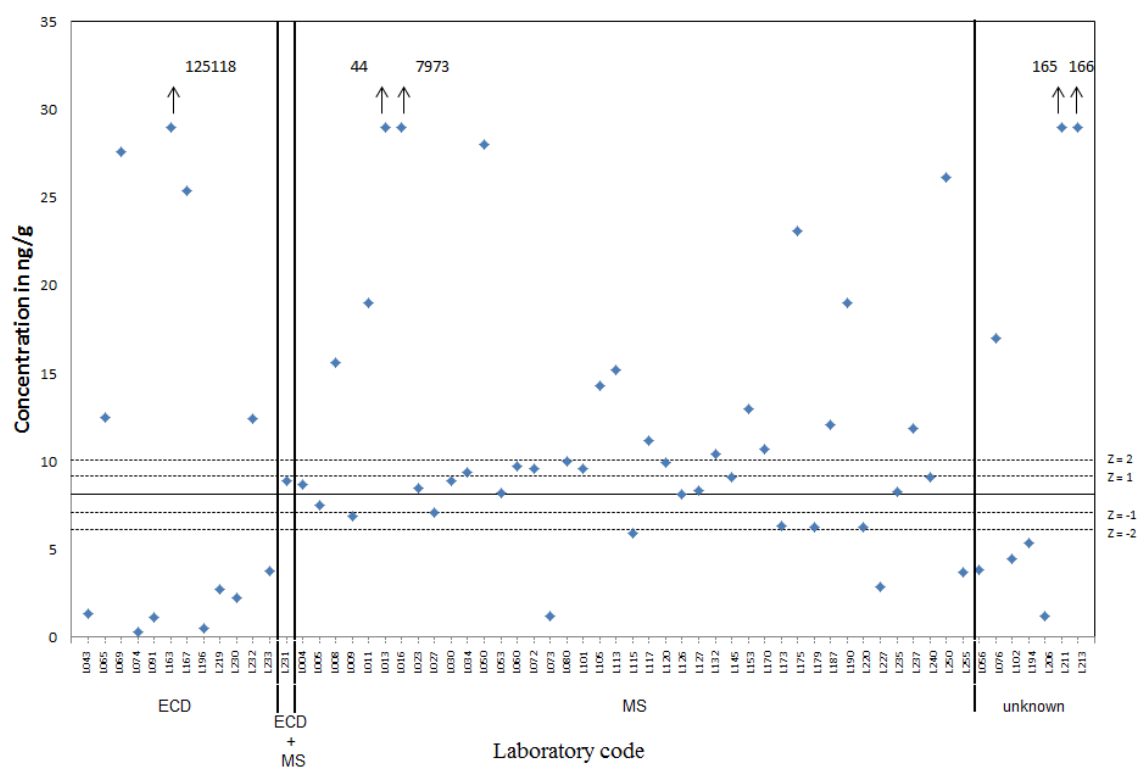


Figure 9 Results for CB 153 in the sediment sample arranged by detection method. Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines.

4.2.3 Dioxin-like POPs

A total of 66 laboratories reported at least one results 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, more than 4,000 satisfactory performance results have been generated in this interlaboratory assessment. However, the regional distribution varies highly as can be seen in Table 95. 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. However, it should be mentioned that also in the Africa, GRULAC and the CEE regions capacity exists.

Table 95: 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	4	80
Asia	32	2,132
CEE	8	245
GRULAC	6	295
WEOG	16	1,288
Grand Total	66	4,040

The most common extraction procedure was Soxhlet extraction but also liquid-liquid extraction was used. An increasing number of laboratories uses accelerated or pressurized extraction methods. The vast majority of samples was analysed using one 60 m column; the use of two columns was not very evident from the information provided. A number of laboratories used shorter – 30 m length – HRGC columns; one laboratory reported to use a 50 m column.

Information on instrumentation was available for 66 laboratories and 5,705 z-scores ranked either S, Q, or U (C and I z-scores were not included). Of these, 48 laboratories used HRMS (sector-field instruments), ten LRMS (whereby one had MS/MS), for five laboratories, no information was provided and three analysed dl-POPs with ECD. The vast majority of z-score results was generated with HRMS detection (84%); they also had by far the highest percentage of satisfactory results (79%) within their instrumentation level. For LRMS instruments, the picture is less favourable: less than half of their z-scores ranked satisfactory. Inacceptable seems to be use of ECD for the detection of dl-POPs: of 105 z-scores, only 12 (or 2% were satisfactory). It shall be noted that these three laboratories did not analyse PCDD/PCDF but indicator PCB. In addition, the number of analytes reported by these laboratories were quite low

The global picture across all test samples for PCDD/PCDF is shown in Figure 10 and for dl-PCB in Figure 11.

With respect to the PCDD/PCDF, the CV values were very good for the test solution (CV = 10 for lower bound (LB) and upper bound (UB)) and the fish test sample (CV=9, for LB and UB), both on WHO2005-TEQ basis. For individual congeners, the CV values ranged from 12 to 20 for the test solution and from 8 to 27 for the fish. For the fish, an unexpectedly high CV value (CV=94) was found for 1,2,3,7,8,9-HxCDF (Figure 10). Throughout the study, this congener had the highest CV values and consensus values could not be assigned for the sediment, fish and human milk sample.

Also for the sediment sample and the air extract, the performance of the laboratories were satisfactory with CV values around 20 for all congeners and the toxicity equivalent (TEQ) at LB and UB. Surprisingly, the CV values for all congeners and the TEQ were high for the human milk sample: the CV values for congeners ranged from 15 to 155 and the CVs for WHO₂₀₀₅-TEQ were 44 for LB and 48 for UB, resp. The higher CV values for the human milk sample may be explained by the low concentrations of congeners in the sample, which were close to the LOQ (at or below 1 µg/g lipid).

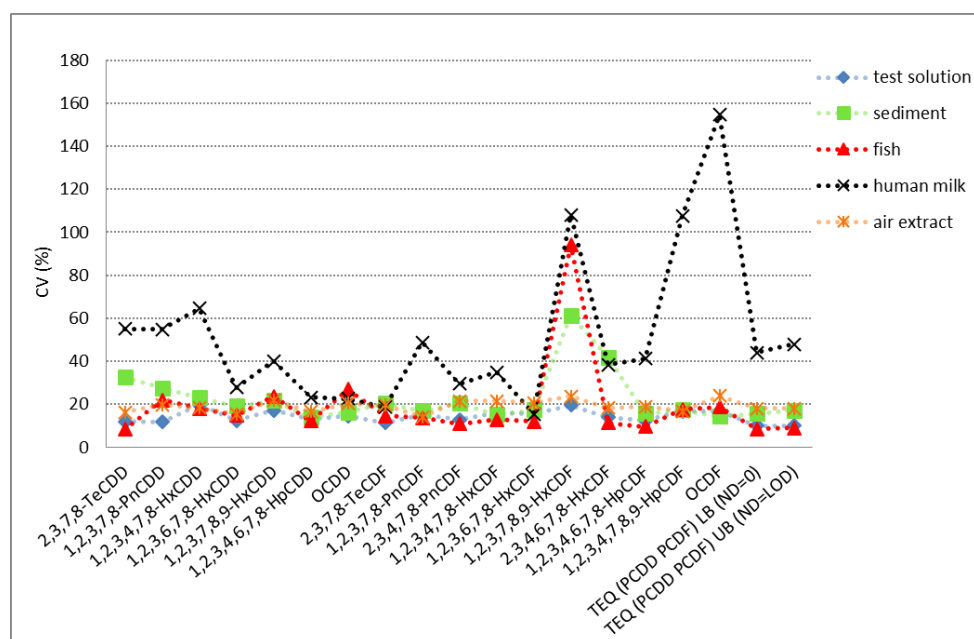


Figure 10: Performance of laboratories for analysis of PCDD/PCDF per congener and TEQ (as %CV)

With respect to the dl-PCB, the CV values on WHO₂₀₀₅-TEQ basis also were very good for the test solution (CV = 10 for LB and UB) and satisfactory for the fish test sample (CV=15 for LB and CV=17 for

UB) (Figure 11). For the individual twelve congeners, the CV values ranged from 9 to 25 for the test solution and from 12 to 28 for the fish. However, for the fish, some higher CV values were encountered for PCB 81 (CV=52), PCB 118 (CV=39) and PCB 123 (CV=84).

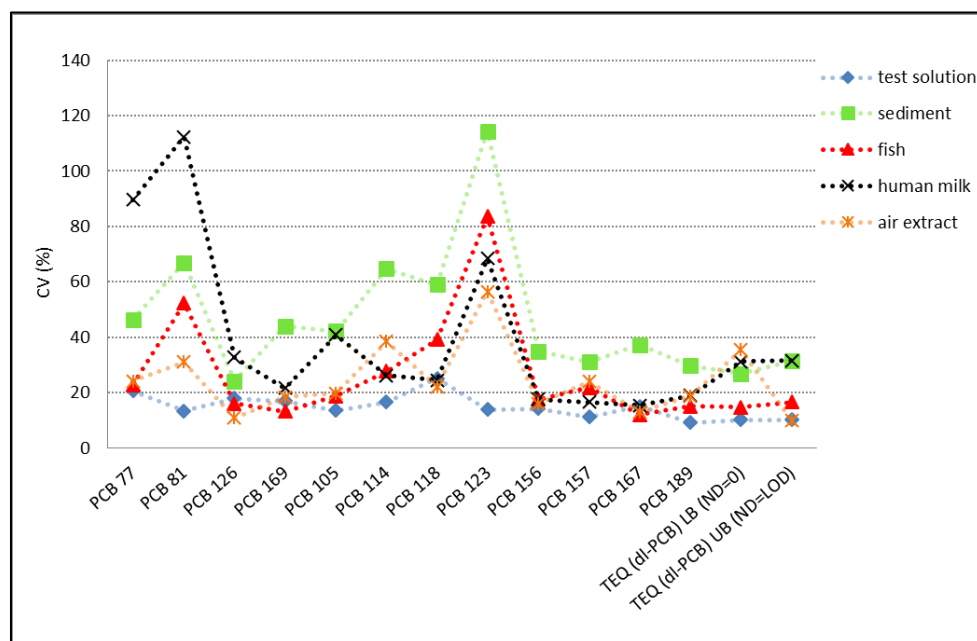


Figure 11: Performance of laboratories for analysis of dl-POPs per congener and TEQ (as %CV)

For all other test samples, the CV values on WHO₂₀₀₅-TEQ basis were very similar: 27 and 31 for LB and UB, *resp.* for the sediment; 31 for LB and UB for the human milk, and 35 and 10 for LB and UB, *resp.*, for the air extract the sediment sample and the air extract, the CV values were around 20 for all congeners and the TEQ at LB and UB. For the human milk sample, the CV values were not as high as for the PCDD/PCDF, maybe due to the higher concentrations of the individual congeners (ranged from 7.9 pg/g to 2,325 pg/g lipid).

4.2.4 Polybrominated Diphenyl Ethers

The individual results for the PBDE in the test solution and the air extract were relatively good with between-lab CV values of 8%-21% (Table 32) and 9%-22% (Table 40) respectively. Also the results of PBDE in the crab were relatively good (CVs 8%-14%, Table 36), and comparable with the results of the test solution, although the matrix was more complex and the concentrations in the fish were 30-1500 times lower. The results for PBDE in the sediment and the human milk were less satisfying, with CV values of 23%-96% (Table 34) and 21%-98% (Table 38) respectively.

Except for the sediment the performance for the PBDE analysis was significantly better than in the second round of the study. For the test solution an average of 75% received satisfactory z-scores in the present study in comparison with 59% in the previous round. The performance for the air extract improved from 48% in the second round to 69% satisfactory z-scores in the present study. For fish the improvement is even more significant with an increase from 29% in the second round to 80% in the present study. This improvement for the fish sample could be partially explained by the ten-fold higher concentration of PBDEs in the present study. For human milk the concentration is 25-35 times lower in the present study, but also there the performance increased from 36% to 49% satisfactory z-scores.

For the sediment sample the concentration of PBDEs was only two to three times lower than in the second round of the study, but only an average of 19% of the participants received a satisfactory z-score in the present study, compared to 66% in the previous round. For three of the compounds (BDE 47, BDE 99, BDE 100) the variation was so high (Table 34) that no assigned value could be calculated by the model.

The better performance for PBDEs compared to that of OCPs and indicator PCBs may be related to a selective effect. Most laboratories doing the PBDE analysis have an MS available and may have more experience compared to many laboratories doing OCP and PCB analysis. The latter group includes more newcomers. The sediment matrix was apparently so complicated that it caused difficulties for all laboratories, experienced and inexperienced.

Individual results from each laboratory for BDE 47 in sediment (CV 75%), for BDE 47 in fish (CV 20%), and for BDE 47 in air (CV 31%) are shown in Figure 12- Figure 14.

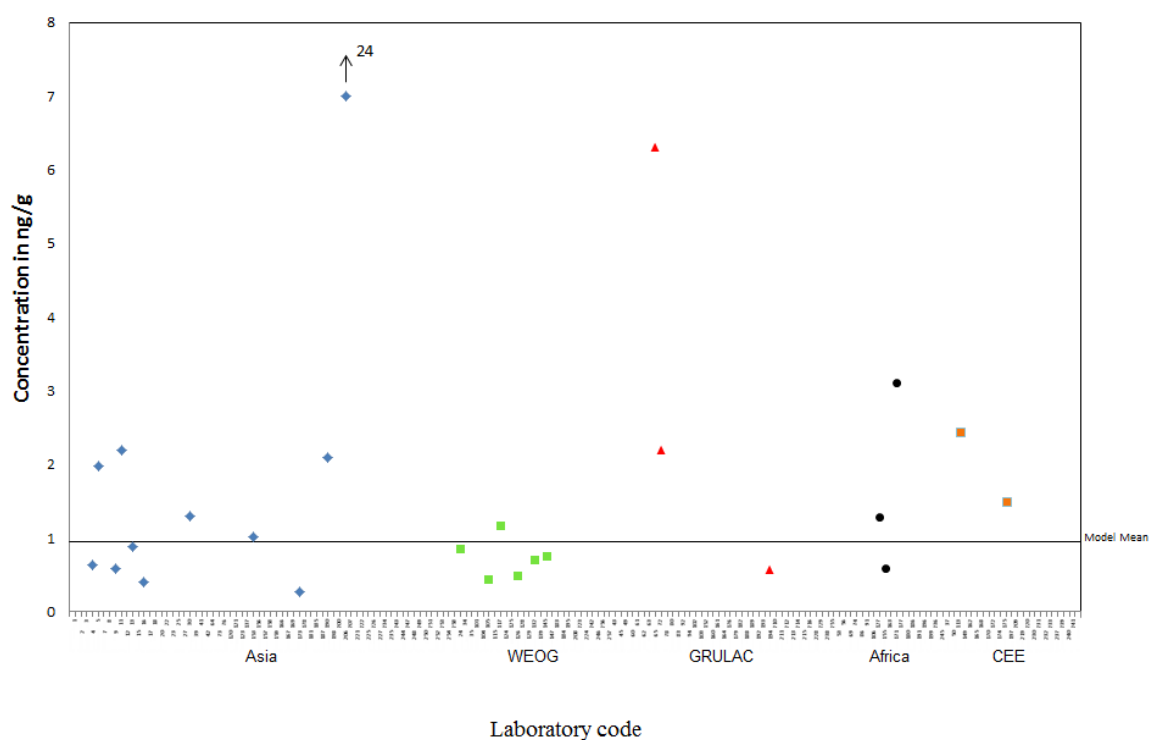


Figure 12 Results for BDE 47 in the sediment sample.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The model mean value given by straight line. The blue \diamond symbols represent Asia, The green \square symbols represent WEOG, the red \triangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \square symbols represent CEE.

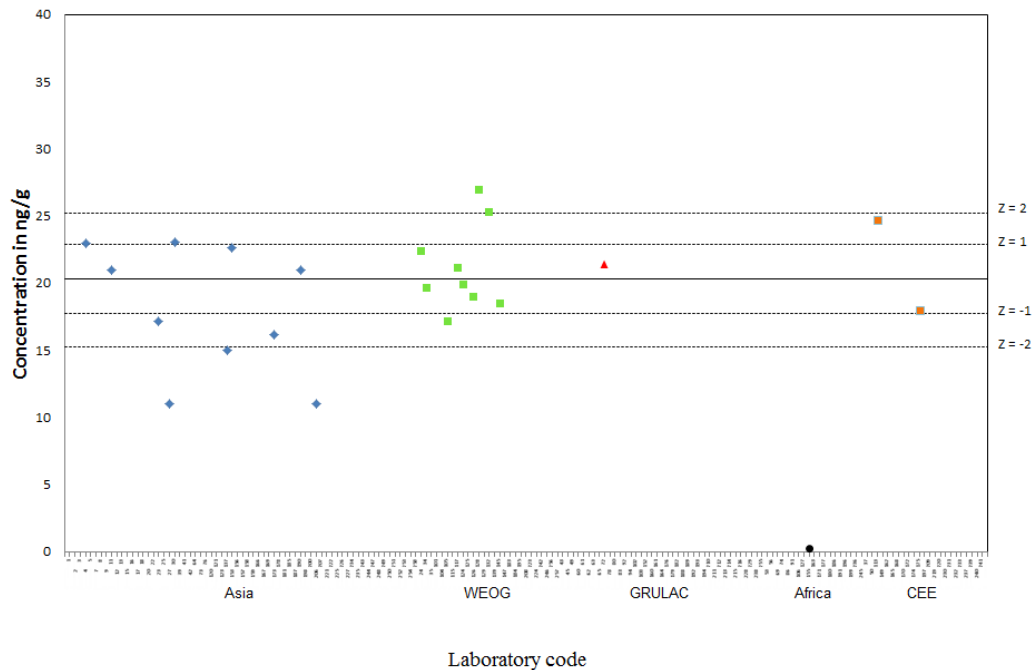


Figure 13 Results for BDE 47 in the fish sample. Laboratory code on the x-axis, concentration in ng/kg on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

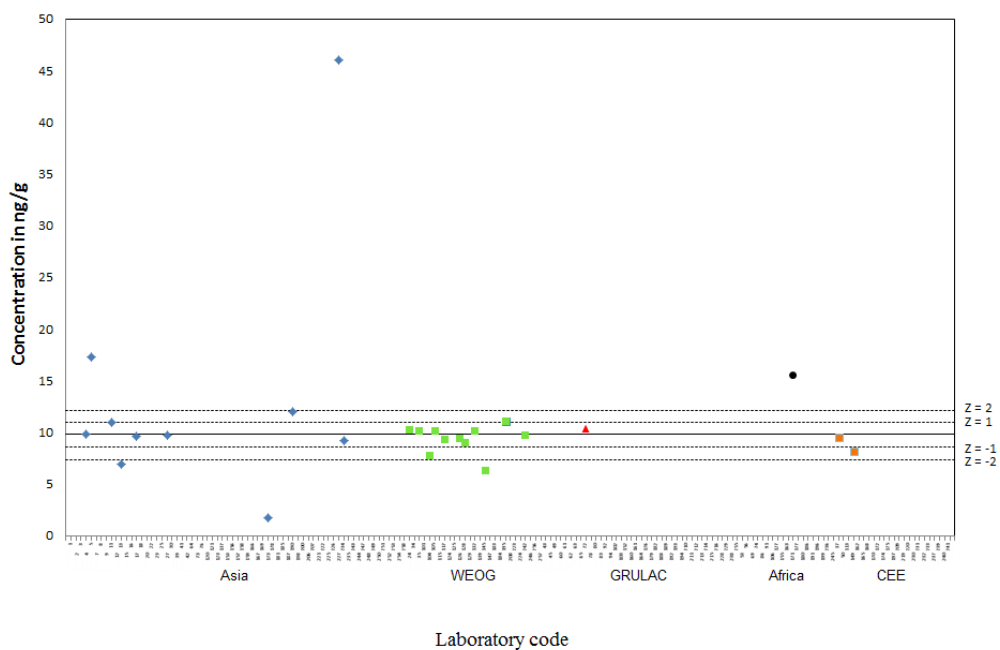


Figure 14 Results for BDE 47 in the air extract. Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

4.2.5 Hexabromobiphenyl

PBB 153 could be analysed in the test solution M, together with the PBDEs, and in the test solution S where PBB 153 was provided as the sole compound. Analyzing only one HxBB was a service to laboratories that are not experienced in PBDE analysis; they should be given the chance to address this compound in individual basis – and perhaps in the context of PCB analysis. Sixteen participants analysed the compound in the test solution M, and 13 in the test solution S. Of those participants, ten analysed both solutions. Six analysed only solution M, and three analysed only solution S. The z-scores received by participants for this compound are shown in Figure 15 for both solutions. As can be observed, the z-scores received by participants analysing both solutions are in good agreement except for two participants who received extremely high z-scores for either solution S, or for both solutions. Those two participants used the same analyses method for both solutions, so it may be expected that they performed equally for both, except for the fact that the concentration in test solution M was about 50 times higher than the concentration in test solution S. The theoretical concentration in solution S (11.3 ng/g (Table 42)) was still much higher than the detection limits reported by those two participants for the other matrices. Two other participants reported LCV values for test solution S of 5 ng/g and 10 ng/g respectively.

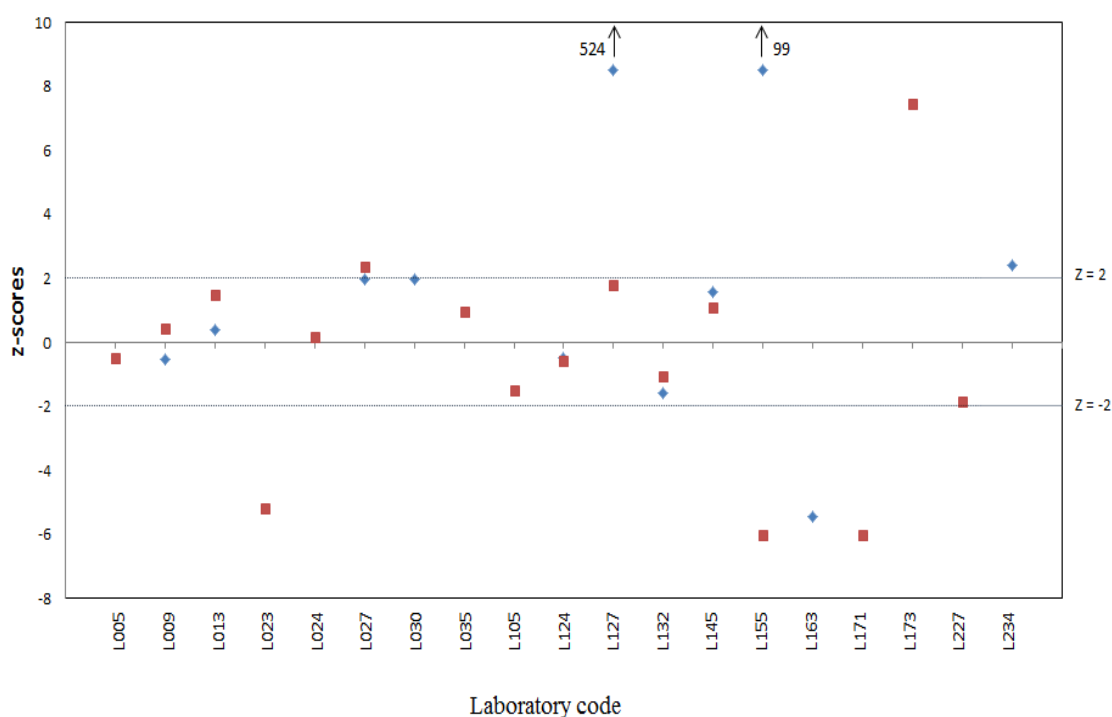


Figure 15 Z-scores for PBB 153 in test solution M and test solution S. Laboratory code on the x-axis, z-scores on the y-axis. $z = \pm 2$ (25%) are given by the dotted lines. The red (■) symbols represent test solution M, The blue (◆) symbols represent test solution S.

The individual results for PBB 153 in the test solution M were a little better than for solution S with between-lab CV values of 27% (Table 32), and 37% (Table 42) respectively. In addition, the overall performance was a little better for solution M with 69% of the participants receiving a satisfactory z-score for PBB 153 in solution M (Table 33) and 54% of the participants for solution S (Table 43). Most likely, this difference in performance is caused by the 50-fold difference in concentration.

Although it is expected that results are worse for a matrix than for a test solution, the performance for PBB 153 in fish was much better. 70% of the participants received a satisfactory z-score (Table 47) and the model between-lab CV was 20% (Table 46, Figure 16).

The performance for PBB 153 in the air extract was less satisfying with 31% (Table 51) of participants receiving a satisfactory z-score and a model between-lab CV of 51% (Table 50). For the sediment the variation was much too high (429%, Table 44, Figure 17) to calculate an assigned value. Although eight participants did report a numerical value, eight others reported below LOD. Most likely the concentration of PBB 153 was too low (model mean 0.29 ng/g) for most of the participants to perform a proper quantitative analyses on such a difficult matrix as this sediment.

For the human milk it was not possible to calculate an assigned value either. The concentration of PBB 153 in this matrix was so low (model mean 0.04 ng/g) that only five participants were able to report a numerical value (Table 48).

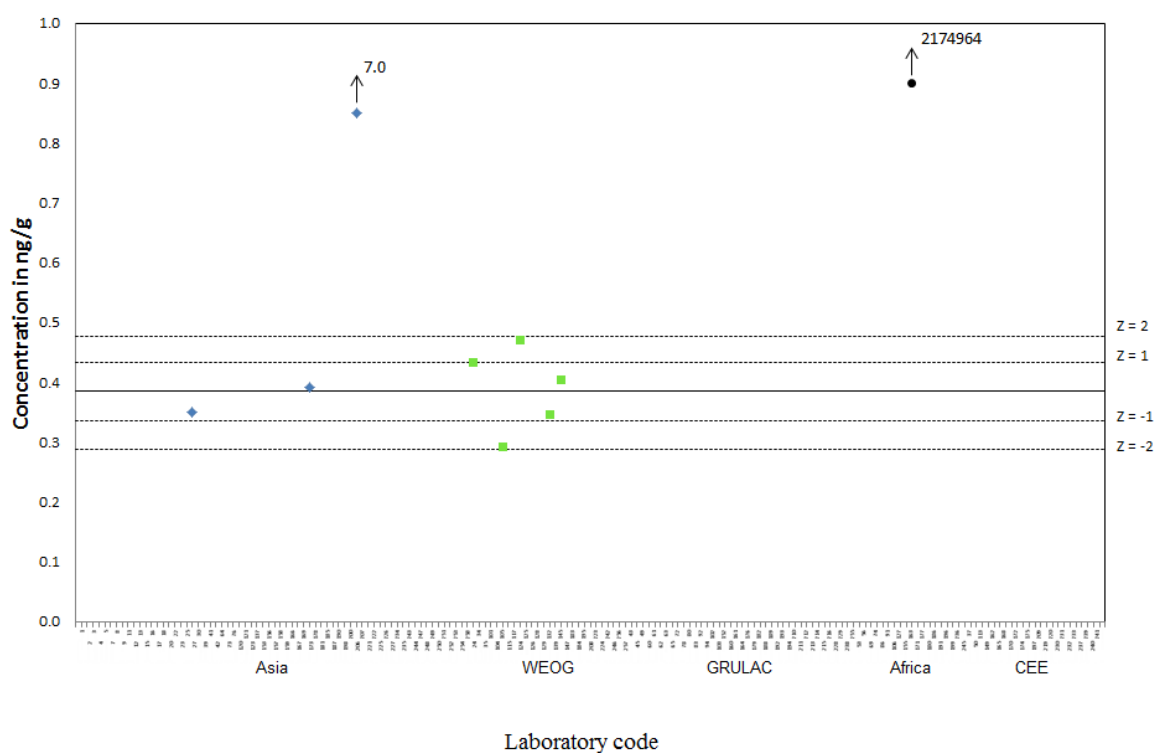


Figure 16 Results for PBB 153 in the fish sample.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \diamond symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

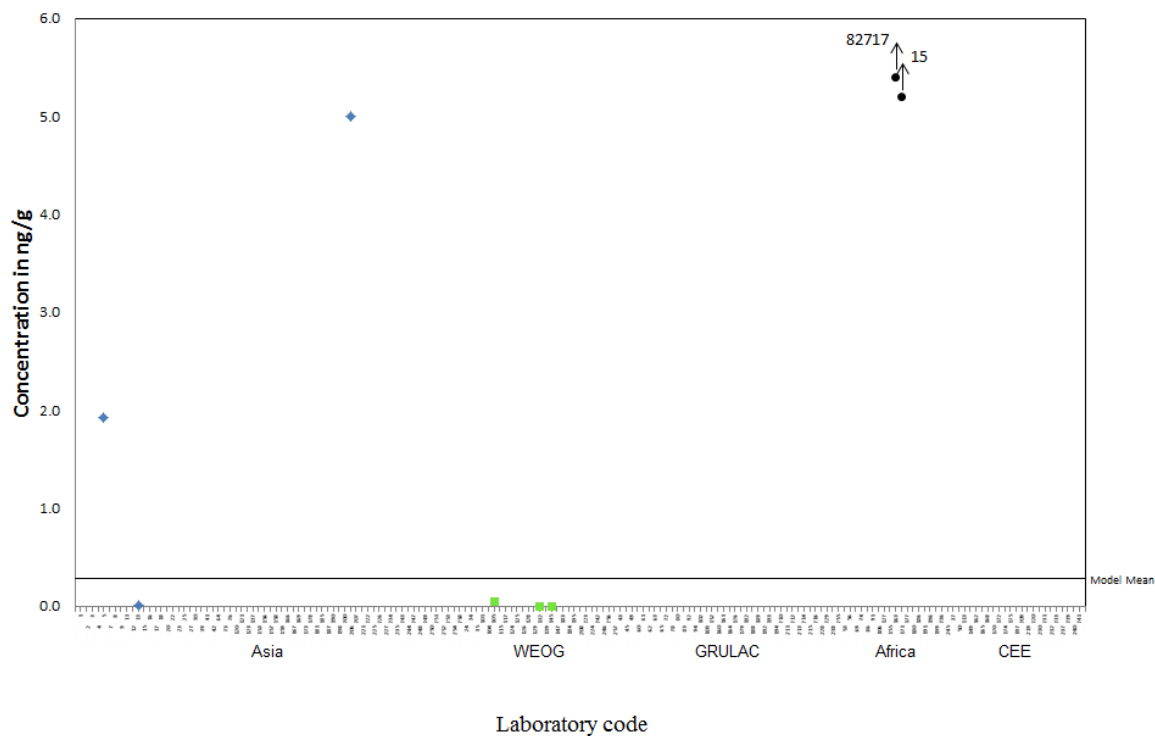


Figure 17 Results for PBB 153 the sediment sample. Laboratory code on the x-axis, concentration in ng/g on the y-axis. The model mean value given by straight line. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

4.2.6 Toxaphenes

In the first two rounds of the bi-ennial interlaboratory assessment on POPs (UNEP, 2015), toxaphene was not included since no or very limited capacity was available among the participating laboratories. In the present study 16 of the laboratories participated in the analyses of toxaphenes. 14 of those participants analysed the test solution, although not every participant analysed all three requested toxaphene congeners. 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 (Table 52)), and all results received were numerical values above the LOD. The performance for toxaphenes in the test solution was good with an average of 83% of the participants receiving satisfactory z-scores (Table 53, Figure 1). The individual results for the toxaphenes in the test solution showed between-lab CV values of 11%-26% (Table 52). This is illustrated for the sum of toxaphenes (UB) (14%) in Figure 18, in which the individual results from each laboratory are given in addition to the consensus value as calculated by the Cofino statistics and the UN Environment criteria of 12.5% ($z = 1$) and 25% ($z = 2$).

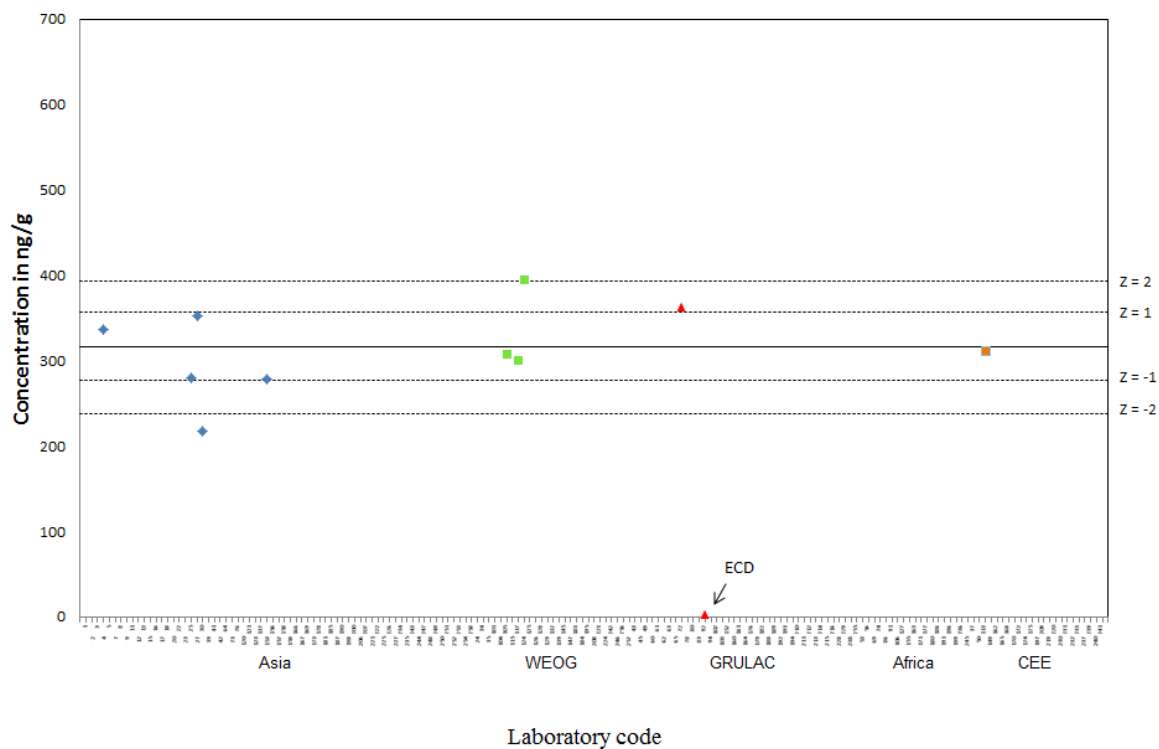


Figure 18 Results for the sum of toxaphenes (UB) in the test solution.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

Of the 16 participants, 13 reported which detection system was used for the analyses of toxaphenes. All of them used an MS method except one participant, who used an ECD. Remarkable is that the result obtained with the ECD for the sum of toxaphenes (UB) in the test solution is much lower than the results obtained with MS (Figure 18). Since this is only one result, it is not possible to draw any conclusions out of it. It may be related to the low response of toxaphene on ECD, due to the aliphatic character of toxaphene.

The participation degree for the other matrices was lower than for the test solution (Table 1). Besides that, toxaphene concentrations in the test matrices were environmentally relevant concentrations and much lower than the concentrations in the test solution. As a result, only a few participants reported a numerical value for toxaphenes in the other test materials, and as a result of that, no assigned values could be calculated at all. Extremely high model CVs were calculated for sediment (168%-1548%, Table 54), fish (127%-271%, Table 56) and human milk (41%-112%, Table 58). For the air extract no CVs could be calculated (Table 60) since only one participant reported one numerical value for one of the toxaphenes. For a next round, environmental samples containing more relevant toxaphene concentrations should be used.

As an example of the extremely high model CVs received, results from the 5 laboratory reporting a numerical value for the Parlar 50 congener in sediment (CV 671%) are shown in Figure 19.

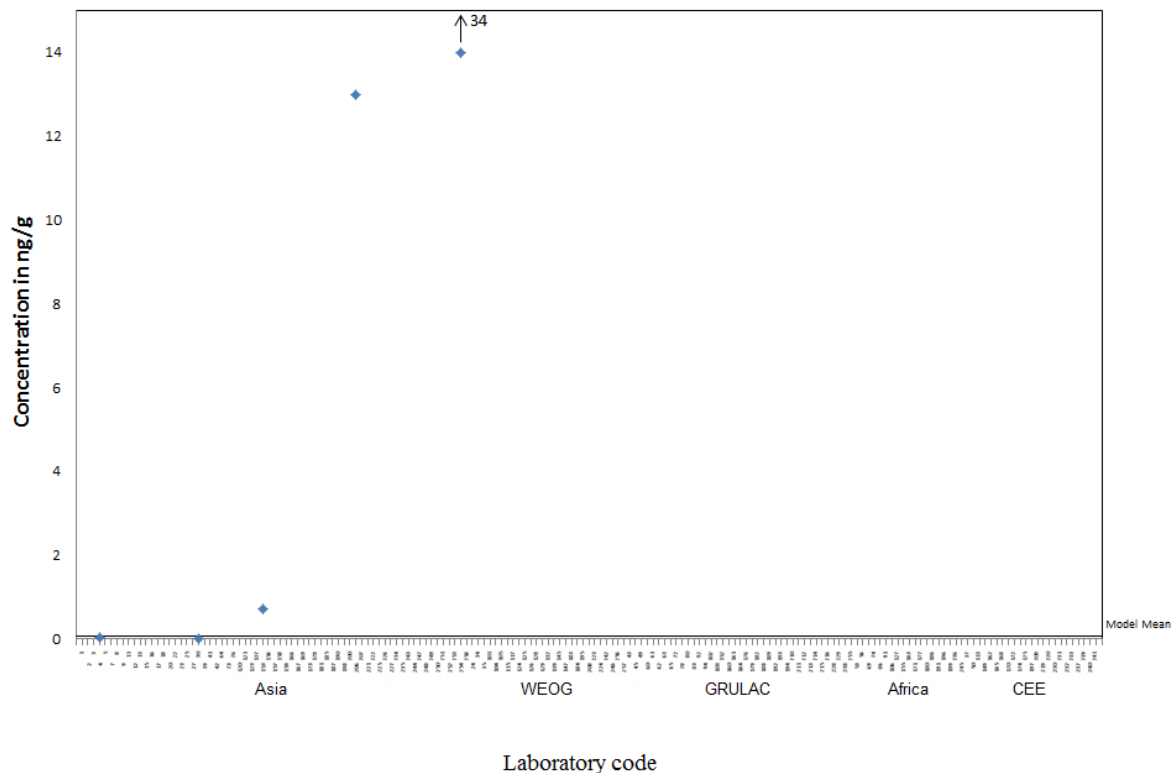


Figure 19 Results for the Parlars 50 in the sediment sample.

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

4.2.7 Hexabromocyclododecane

Considering the fact that HBCDs were included in the study for the first time, the performance for HBCDs in the test solution is very good with an average of 81 % of the participants receiving a satisfactory z-score. The individual results for the HBCDs in the test solution show between-lab model CV values of 12%-14%. Again, as for the PBDEs, the participating laboratories may have been more experienced and obviously had an LC/MS and labelled internal standards available. As an example of the low CV values, the individual results reported for γ -HBCD in the test solution are shown in Figure 20 *per* laboratory.

For the individual HBCDs in the fish material model CV values were higher (21%-120%), but for the most relevant isomer in fish, α -HBCD, the model CV value was only 21%, and 80% of the participants received a satisfactory z-score for this compound. In sediment the most relevant compound is γ -HBCD. The model CV for this compound in sediment (36%) was an acceptable outcome given that this group was included for the first time. For the other two isomers the variation was higher, and no assigned values could be calculated. Unfortunately, for the human milk sample and for the air extract it was not possible to calculate assigned values, either due to a low participation degree or low concentrations.

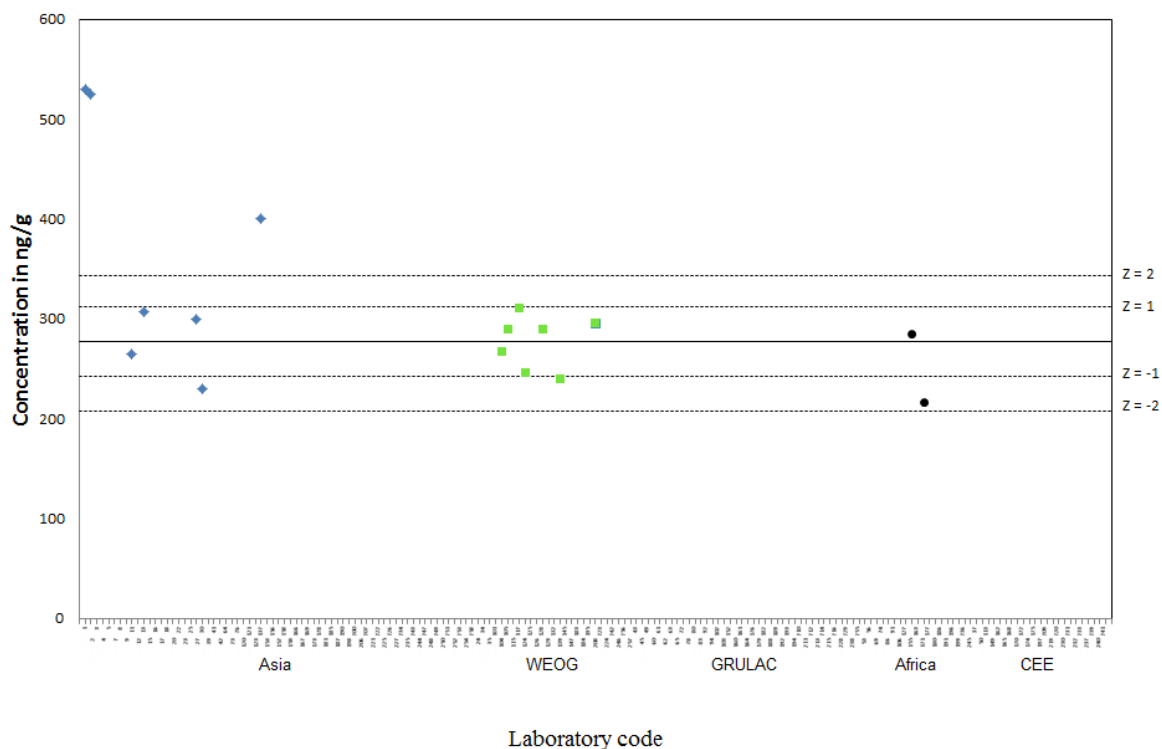


Figure 20 Results for γ -HBCD in the test solution.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

4.2.8 Perfluoroalkyl Substances

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 29 laboratories that submitted results for PFAS, only one laboratory used a time-of-flight instrument; all others reported to use LC/MS/MS. For the separation of the analytes, the majority used HPLC columns; however, also UPLC columns are in use. Normally, a C_{18} based column was used; however, some also used C_8 -based columns. One laboratory reported to have applied GC/LRMS (using a DB-WAX column, 30 m x 0.25 mm x 0.25 μ m) for the separation of PFOS precursors, *e.g.* Me/EtFOSA and Me/EtFOSE.

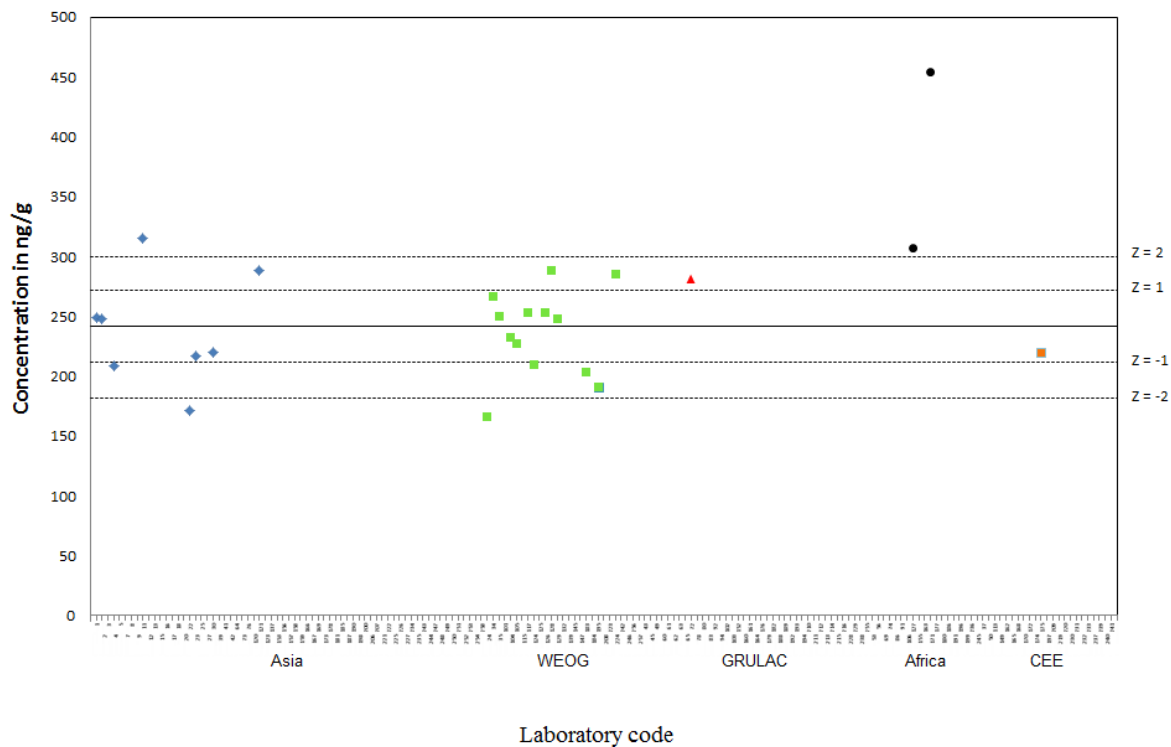


Figure 21 Results for L-PFOS anion in the test solution. Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

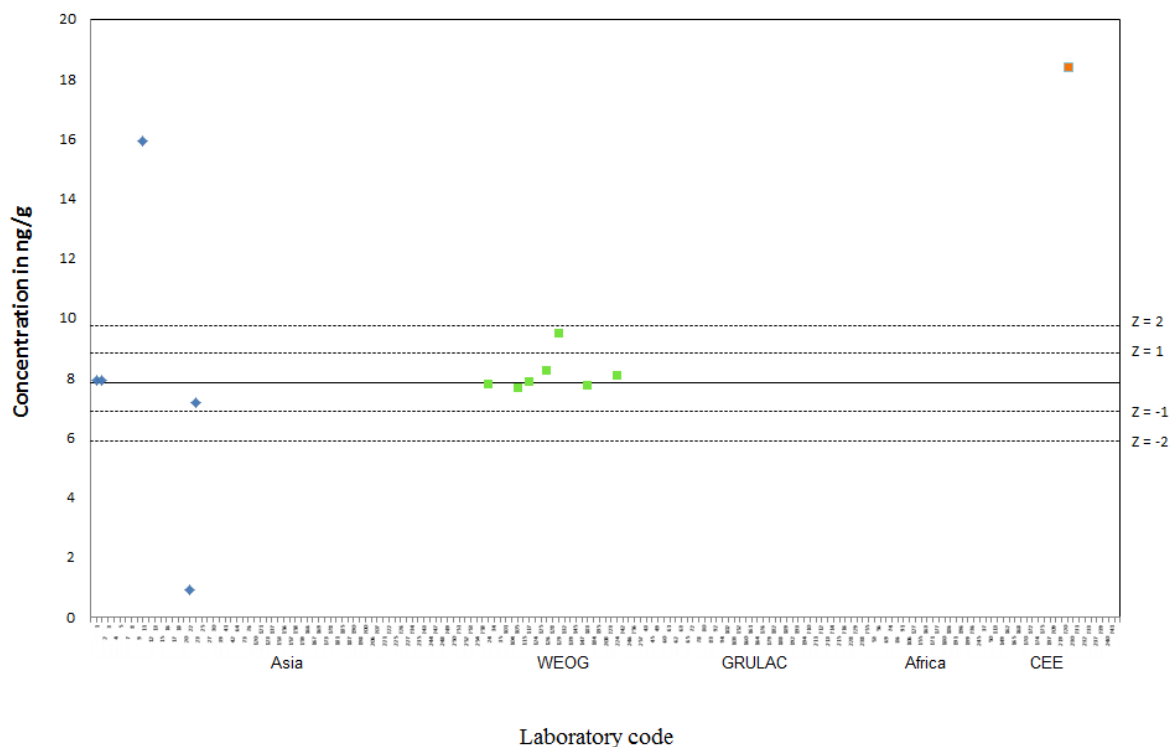


Figure 22 Results for L-PFOS anion in the fish sample.

Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

The global picture of laboratory's performance in PFAS analysis – as percentage variation of the CV – is shown in Figure 23. 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.

The standard test solution did not contain any PFUnDA, PFDoDA, PFTTrDA, PFTeDA, L-PFHpS or L-PFDS (see Table 72).

As can be seen from Figure 23 the performance of the laboratories was satisfactory for the PFOS anion in the test solution, the sediment and the human milk and human plasma sample. For the fish and the water sample, difficulties were observed for the branched PFOS. The results for the air extract were less impressive.

In general, the small number of laboratories reporting results for PFAS hampers the assessment of this group of POPs.

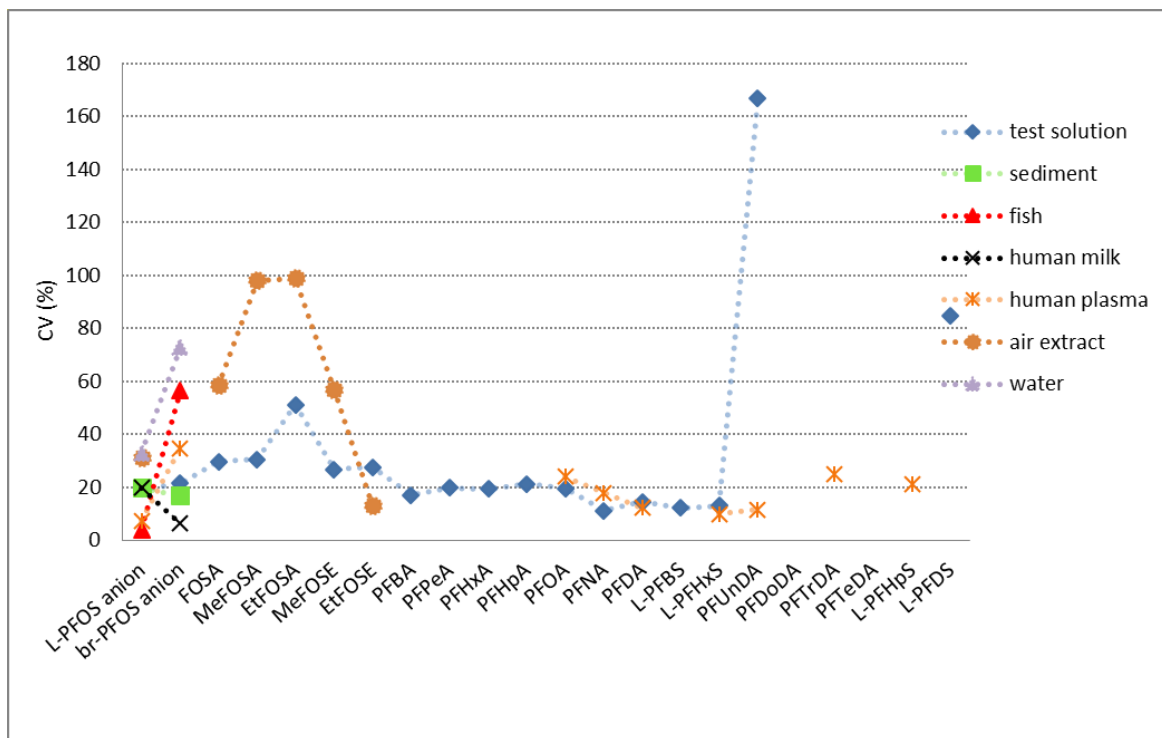


Figure 23: Performance of laboratories for PFAS according to sample type (as CV).

4.3 Performance on Sum Parameters

4.3.1 Sum OCPs

In this section, the performance of participants on the sums of drins, chlordanes, heptachlors, DDTs, HCHs and endosulfan (Figure 24) is discussed. Although such an evaluation provides valuable data, it should be noted that the results on the statistical evaluation of the sum parameters is only indicative, as some participants only reported on one or two compounds of a compound group, while others reported results for all OCPs.

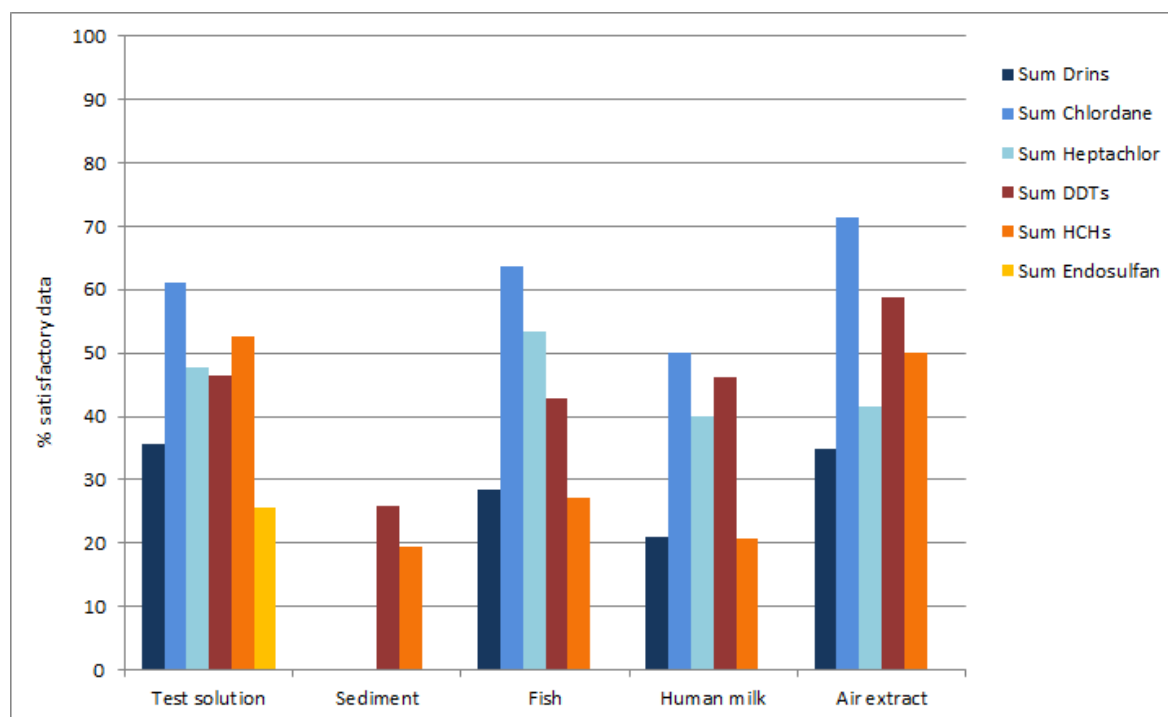


Figure 24 Percentage of laboratories with satisfactory z-scores for sum OCPs (LB).

The group of OCPs is one of the most difficult-to-analyse groups. This also appears from Figure 24, where the percentages of satisfactory z-scores are plotted. Even for the standard test solution, this percentage does not reach more than 60%, which is much too low for a reliable analysis. Obviously, this will have influenced all other results. On top of that matrix effects have made the analysis more complicated, in particular for sediment, but also for fish and human milk. The air results are somewhat better, probably due to a low matrix influence and the fortification of the test extract.

It is recommended that the labs should focus on the precise preparation and storage of their test solutions. In addition, MS should preferably be used for this group of compounds, as ECD is not reliable due to negative peaks and other interferences in the chromatograms. Figure 25 shows that major difficulties are met for endosulfan and heptachlor.

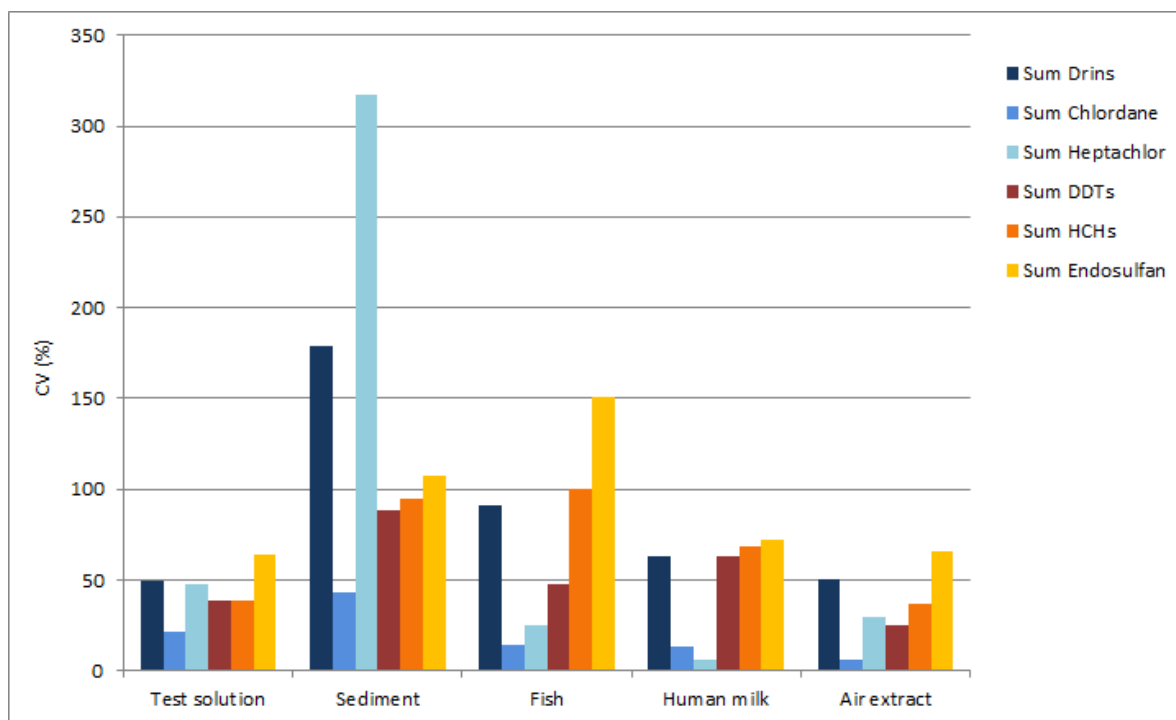


Figure 25 Variation in CV values for sum OCPs (LB).

4.3.2 Sum PCB

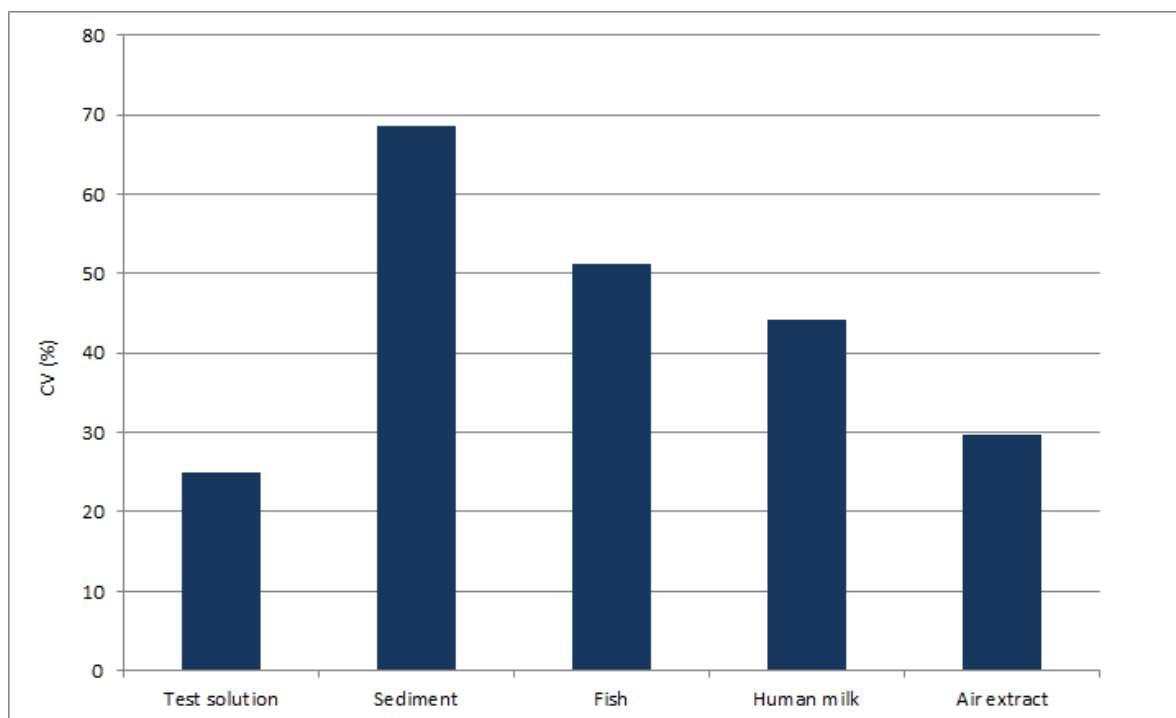


Figure 26 Variation in CV values for sum PCBs (LB).

Figure 26 and 27 show that also for indicator-PCB sediment was the most difficult test materials. The standard test solution shows a better performance than that of the OCPs, although improvement is still possible. Satisfactory z-scores are not higher than 60%, which is not enough, given the targets of the GMP.

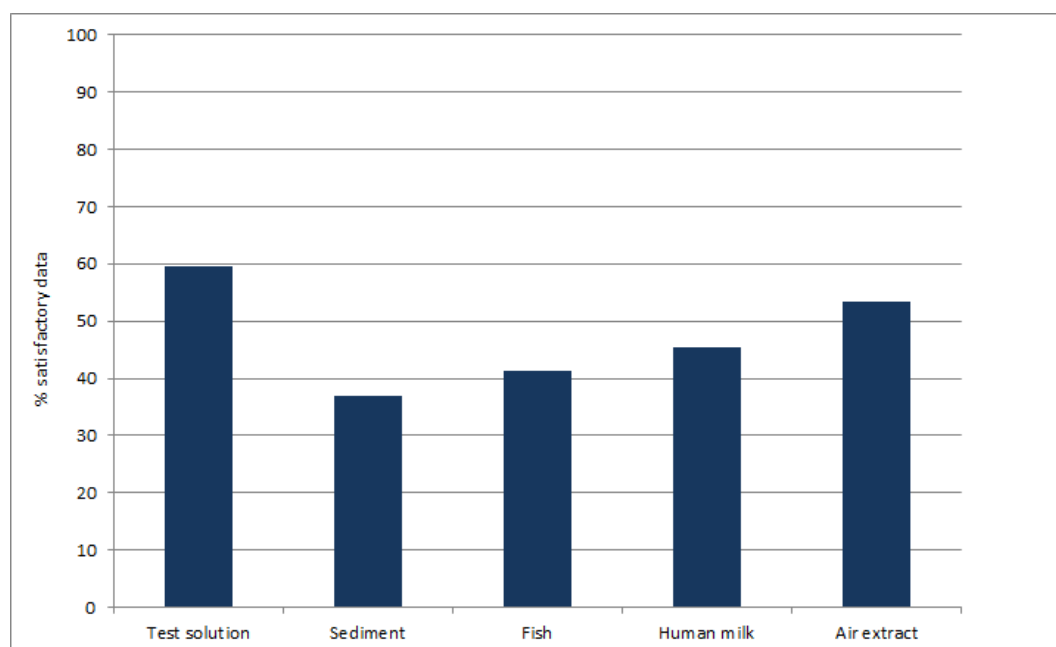


Figure 27 Percentage of laboratories with satisfactory z-scores for sum PCBs (LB).

4.3.3 Sum PBDE

In Figure 28, the performance of the participants on the sum PBDE is shown. It should again be noted that the results on the statistical evaluation of the sum parameters is only indicative, as several participants only reported on one or two PBDE congeners. CVs. The laboratories that performed this type of analysis were more experienced. It is recommended that also the new laboratories start to develop the methods for the determination of PBDEs.

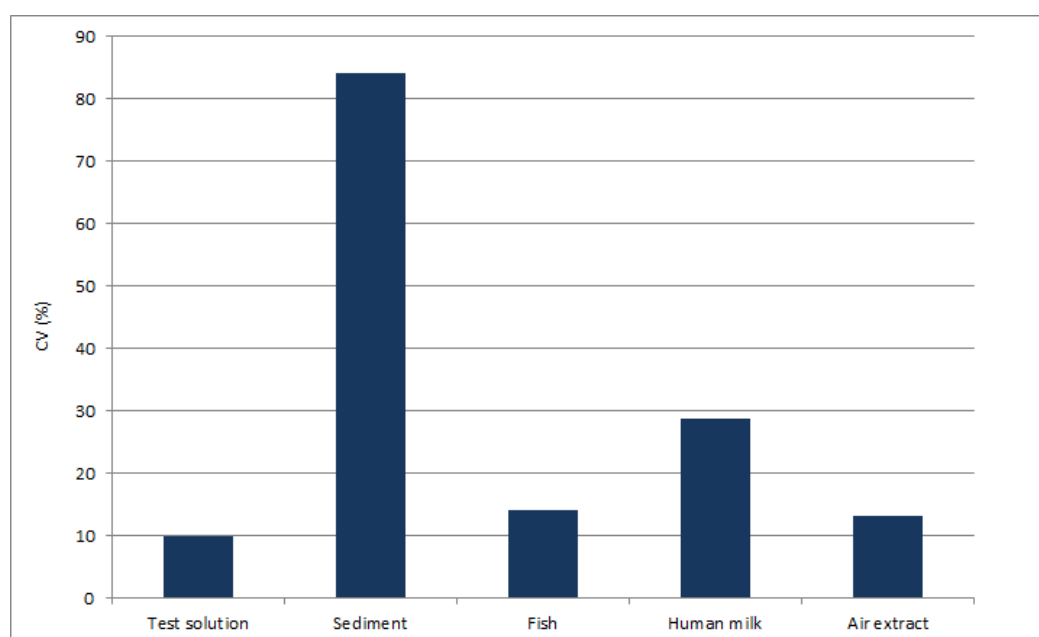


Figure 28 Variation in CV values for sum PBDEs (LB).

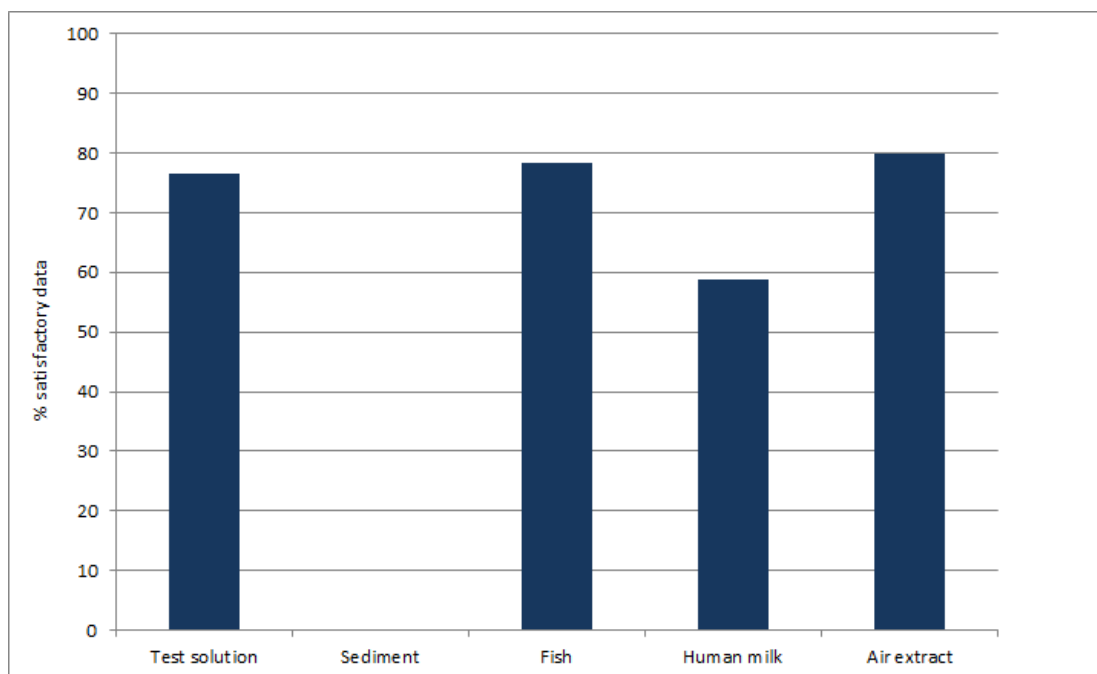


Figure 29 Percentage of laboratories with satisfactory z-scores for sum PBDEs (LB).

4.3.4 Sum toxaphenes

Unfortunately the matrix-based test materials contained very low levels of toxaphene. Therefore, the laboratories who analysed for toxaphene could only obtain reasonable results for the test solution. No z-scores could be calculated for either test materials. It is good to see that indeed they were able to analyse the test solution with satisfactory results. It is recommended for a next round to either fortify the matrix-based test materials with toxaphene or use or naturally contaminated test materials. Laboratories are encouraged to develop methods for toxaphene analysis.

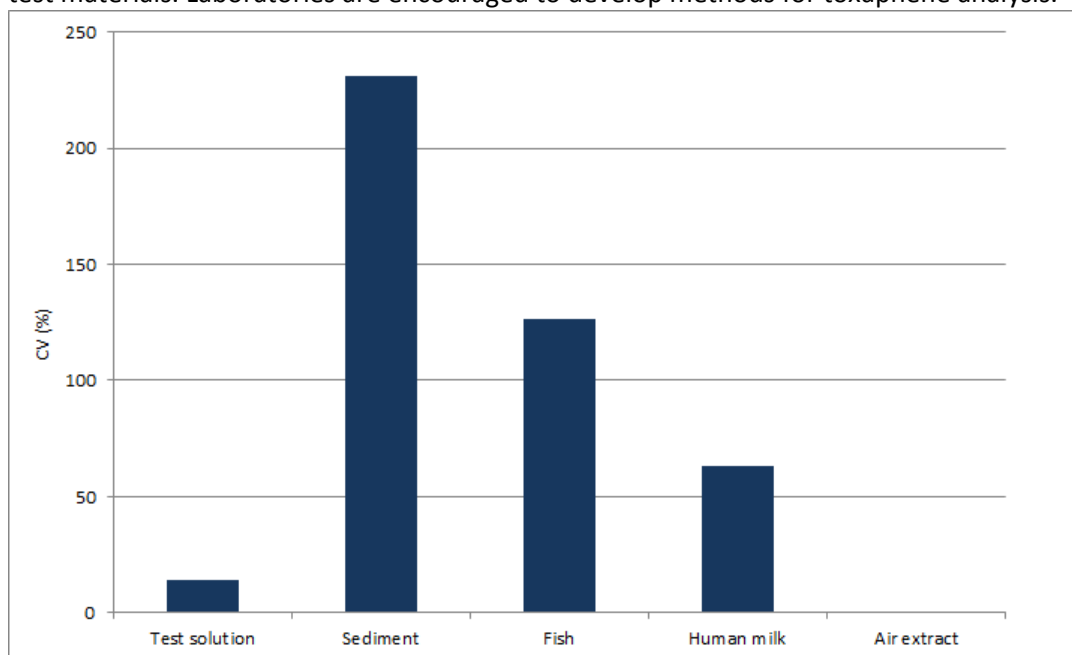


Figure 30 Variation in CV values for sum toxaphenes (LB).

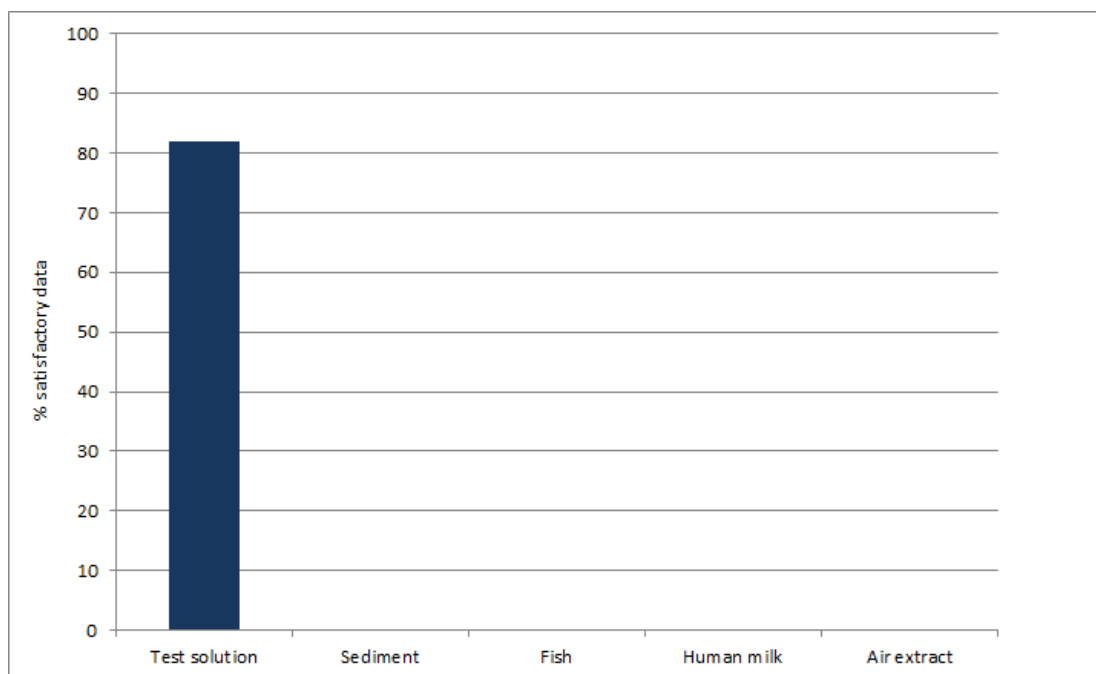


Figure 31 Percentage of laboratories with satisfactory z-scores for sum toxaphenes (LB).

4.3.5 Sum HBCD

The individual results for HBCD were better than the sum-HBCD results. This is because in fish a-HBCD is a major compound while the other two congeners are relatively low in concentration and in sediment the g-HBCD is clearly present while the a- and b- congeners are both low. The test solution showed a good performance for all three congeners. In conclusion the laboratories showed a good ability to analyse HBCDs, apart from the congeners present at low levels only. More laboratories should develop methods for HBCD congeners, which of course implies that they have to invest in a proper LC/MS.

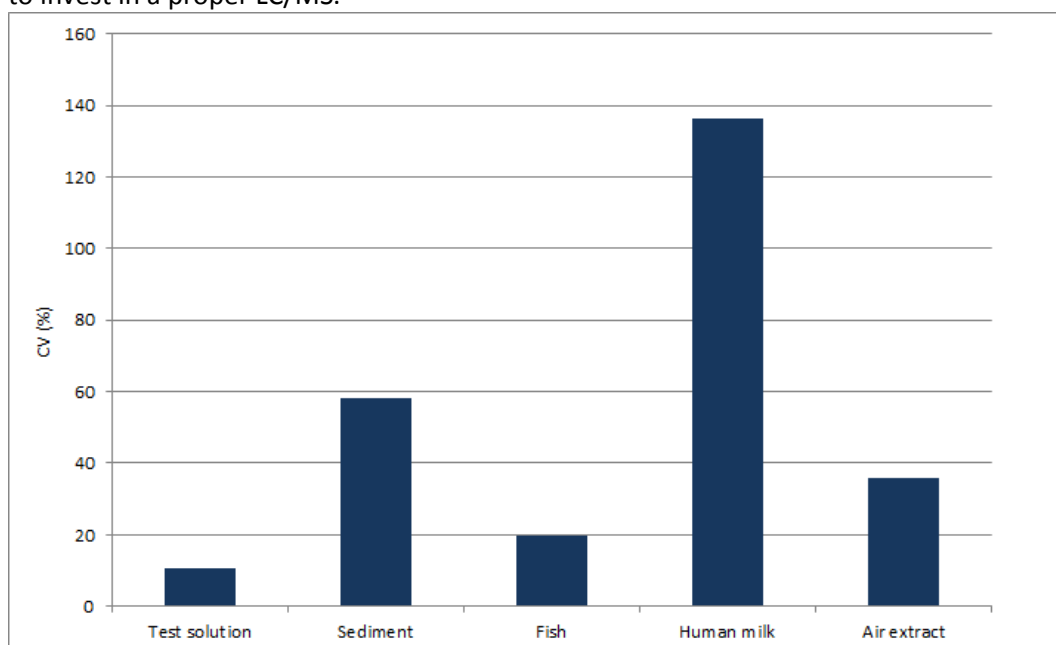


Figure 32 Variation in CV values for sum HBCDs (LB).

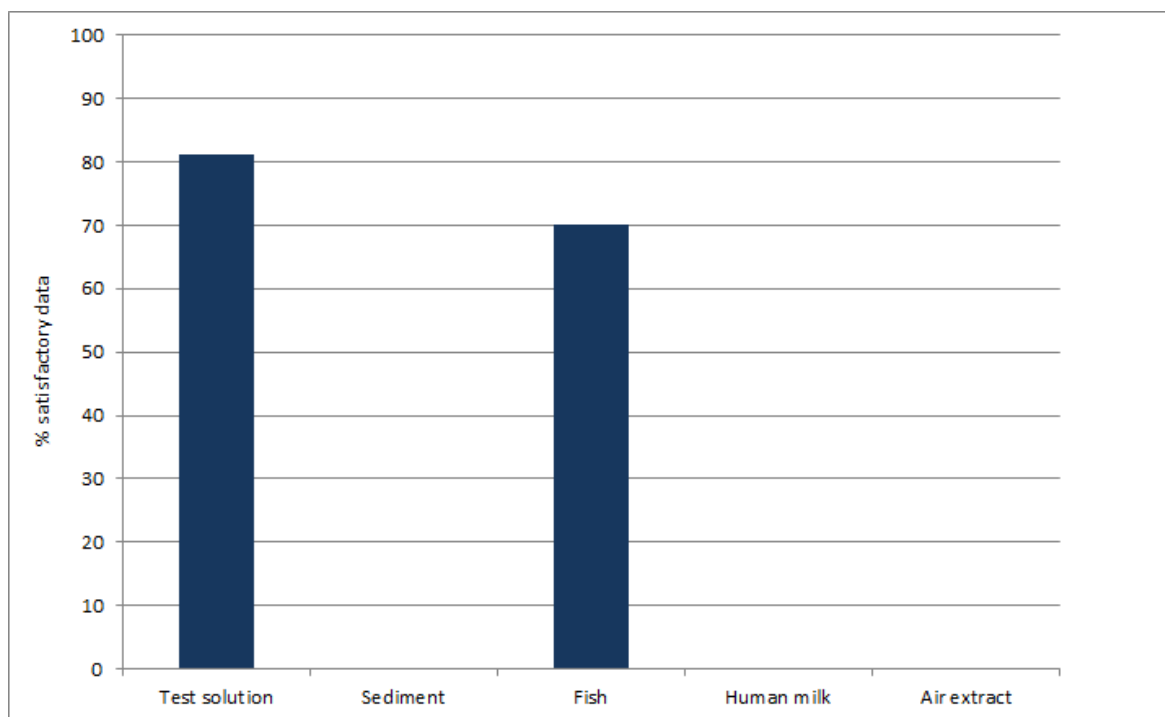


Figure 33 Percentage of laboratories with satisfactory z-scores for sum HBCDs (LB).

4.3.6 Sum PCDD/PCDF and dl-PCB as TEQ

The toxicity equivalency factors (TEFs) and the use of toxic equivalents (TEQs) have been established to summarize 17 or 12 congeners, respectively as one number. It can be seen from Figure 10 and Figure 11, that typically the CV values for the TEQ are smaller than for individual congeners. Therefore, more laboratories perform satisfactory for the TEQ despite there may be some results characterized by Q or U for individual congeners.

Some examples on performance with an emphasis on the TEQ-based results are provided in the following Figures (from Figure 34 - Figure 37). For illustration and further assessments, they are discussed on a regional basis. It can be seen that outliers are reported from all regions including the “experienced” WEOG region; especially with respect to dl-PCB in the air extract (Figure 37). Extreme outlier could be assigned to individual laboratories that reported concentrations three and more orders of magnitude higher than other laboratories or the assigned value (Figure 36 and Figure 37).

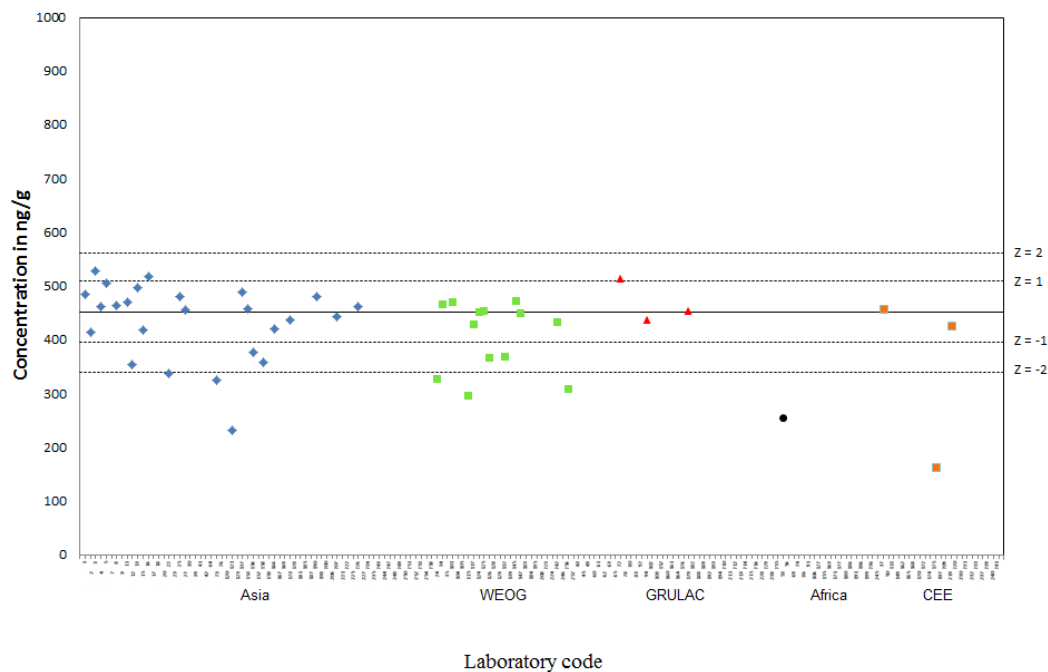


Figure 34 Results for the PCDD/PCDF TEQ in the test solution. Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

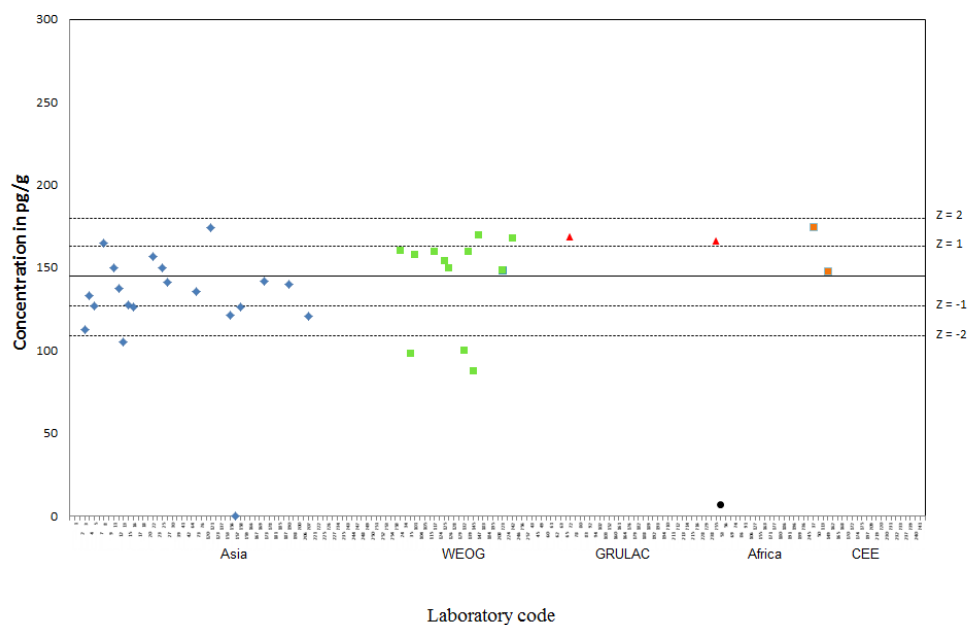


Figure 35 Results for the PCDD/PCDF TEQ in the air extract. Laboratory code on the x-axis, concentration in pg/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

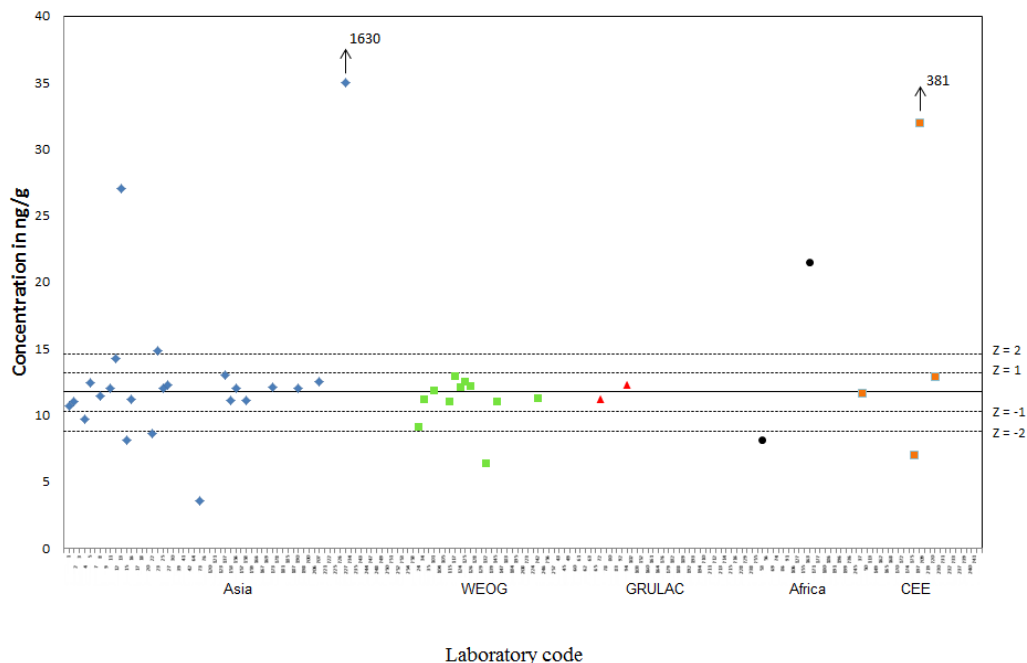


Figure 36 Results for the di-PCB TEQ in the test solution. Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

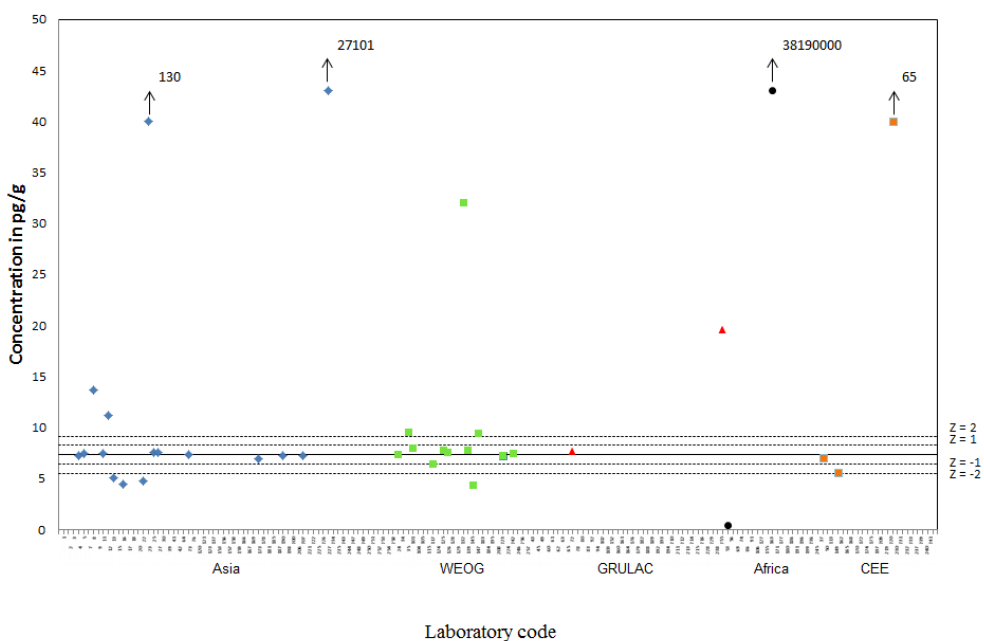


Figure 37 Results for the di-PCB TEQ in the air extract. Laboratory code on the x-axis, concentration in ng/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

Basically, no difference could be seen between laboratories participating for the first time (9) and laboratories that have participated previously (28). The following two figures (Figure 38 and Figure 39) show the results for the WHO₂₀₀₅-TEQ for PCDD/PCDF and dl-PCB in the air extract.

For the PCDD/PCDF, six laboratories reported results below the concentration of 109 pg WHO₂₀₀₅-TEQ/g which represents -2 z-scores (Figure 38), namely: Five that previously participated with one laboratory from Asia (L013, 105 pg WHO₂₀₀₅-TEQ/g), three from WEOG (L035, 98.2 pg WHO₂₀₀₅-TEQ/g; L132, 99.9 pg WHO₂₀₀₅-TEQ/g and L145, 87.5 pg WHO₂₀₀₅-TEQ/g) and one from Africa (L053, 7.0 pg WHO₂₀₀₅-TEQ/g); one of the new laboratories from Asia highly underreported the concentration (L157, 0.1 pg WHO₂₀₀₅-TEQ/g).

The situation is similar but less positive for the dl-PCB (Figure 39) where nine laboratories participated for the first time; of these, only four provided results within the ± 2 z-score range and five were outside (<5 pg WHO₂₀₀₅-TEQ/g or >9 pg WHO₂₀₀₅-TEQ/g). From the 26 laboratories that previously participated, ten reported results outside the ± 2 z-score range and 16 were inside.

Among the laboratories that reported results outside of the ± 2 -z-score range, three laboratories used ECD detectors for the determination of all dl-PCB and in all matrices. The two very extreme values in **Error! Reference source not found.** (27,000 pg WHO₂₀₀₅-TEQ/g and >38,000,000 pg HO₂₀₀₅-TEQ/g) were provided by two new laboratories in Africa; the “experienced” laboratory from Africa reported only very few congeners and not for the TEQ.

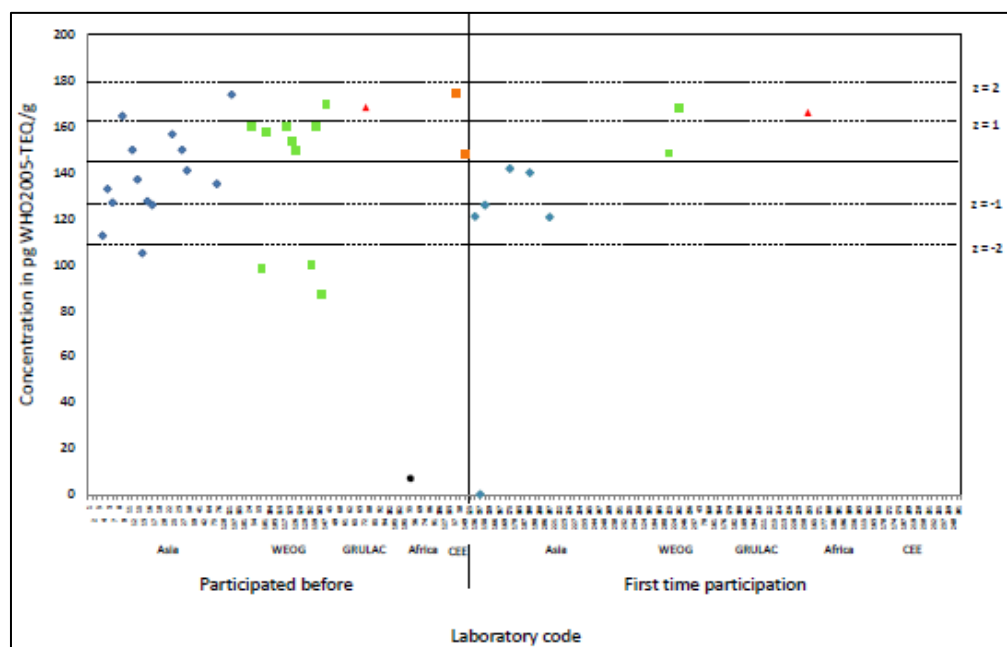


Figure 38: Comparison of results for PCDD/PCDF (as WHO₂₀₀₅-TEQ) in the air extract for first time and previously participating laboratories
 Laboratory code on the x-axis, concentration in pg/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa, and the orange \blacksquare symbols represent CEE.

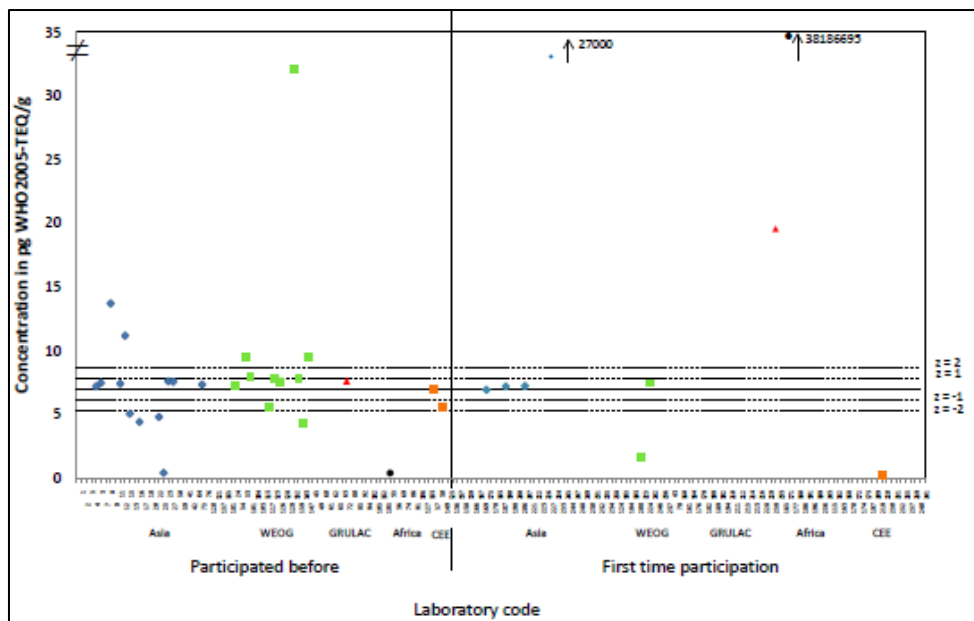


Figure 39: Comparison of results for PCDD/PCDF (as WHO₂₀₀₅-TEQ) in the air extract for first time and previously participating laboratories
 Laboratory code on the x-axis, concentration in pg/g on the y-axis. The assigned value given by straight line, $z = \pm 1$ (12.5%) and $z = \pm 2$ (25%) are given by the dotted lines. The blue \blacklozenge symbols represent Asia, The green \blacksquare symbols represent WEOG, the red \blacktriangle symbols represent GRULAC, the black \bullet symbols represent Africa and the orange \blacksquare symbols represent CEE.

5 COMPARISON WITH THE PREVIOUS TWO ROUNDS OF UN ENVIRONMENT'S INTERLABORATORY ASSESSMENT

In 2010/2011, UN Environment organized the first global interlaboratory assessment on POPs (Abalos *et al.*, 2013, Van Leeuwen *et al.*, 2013). In the first assessment, test solutions, a sediment, a fish, a human milk sample and fly ash were tested. Only OCP, PCB and dl-POPs were tested. Overall, the performance obtained on the test solution was reasonable to good. However, a substantial number of laboratories struggled with the analysis of the other matrices.

In the second assessment participants performed much better for the analysis of PCB in sediment, fish and human milk (Figure 40). CVs for PCB in the test solution in the second assessment (18%-28%) were, however, larger than in the assessment of 2010/2011 (8%-19%). In this third round

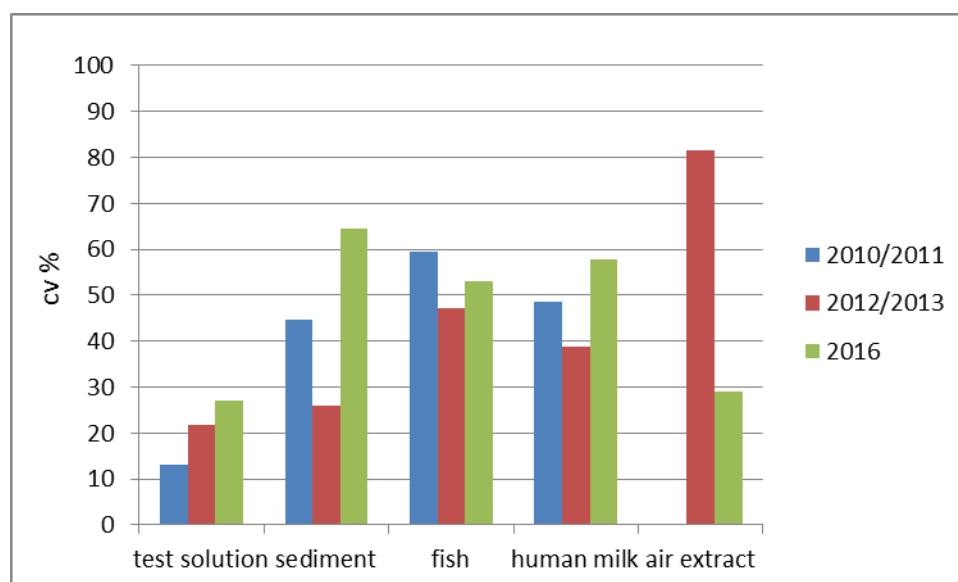


Figure 40 Comparison of performances between interlaboratory assessments for the indicator PCB analyses.

The performance for the test solution went again backwards (Fig. 40). Also the human milk and sediment showed poorer results compared to the two previous rounds. For the sediment it may be explained by a difficult matrix with high background, due to a polluted river from which the sediment was taken (river Elbe, Germany). The human milk sample showed lower concentrations of most analytes compared to the human milk samples used in the previous rounds. The poorer performance for the test solution is most likely due to a sudden increased participation degree in this exercise. A number of unexperienced laboratories, participating for the first time may have negatively influenced the overall between-lab CV. This effect is stronger for the test solution and for PCB and OCPs as many of these new labs did not analyse other matrices or other compounds. Figure 41 shows a similar pattern for OCPs as for PCB in the test solution and sediment. Figure 42 gives more details for the OCP test solution.

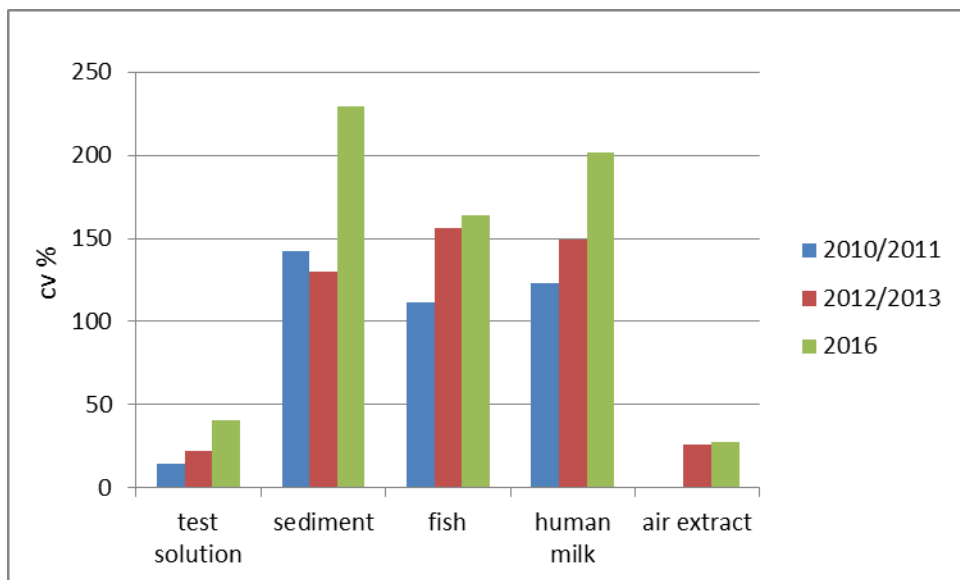


Figure 41 Comparison of performances between interlaboratory assessments for the OCP analyses.

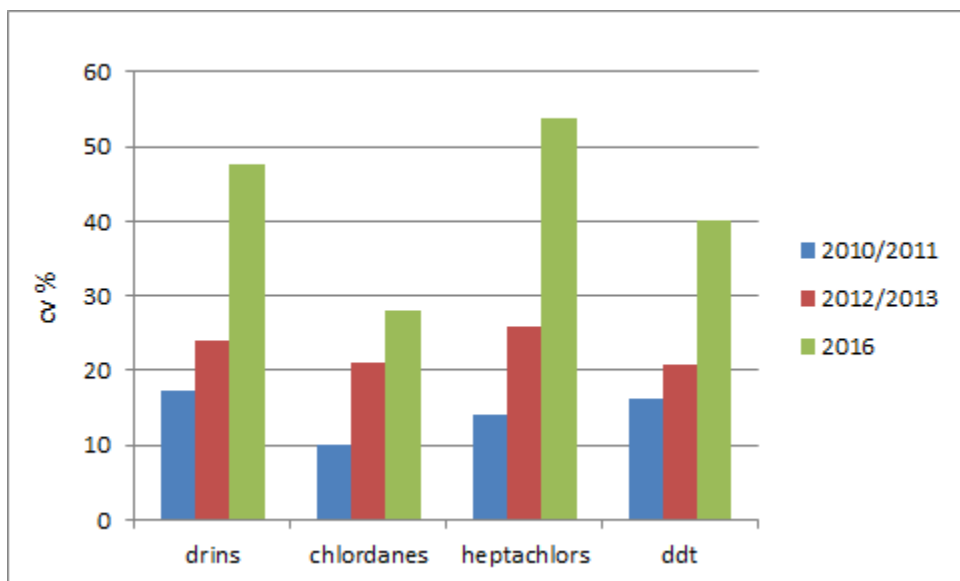


Figure 42 Comparison of performances between interlaboratory assessments for the analyses of drins, chlordane, heptachlors and DDTs in test solutions.

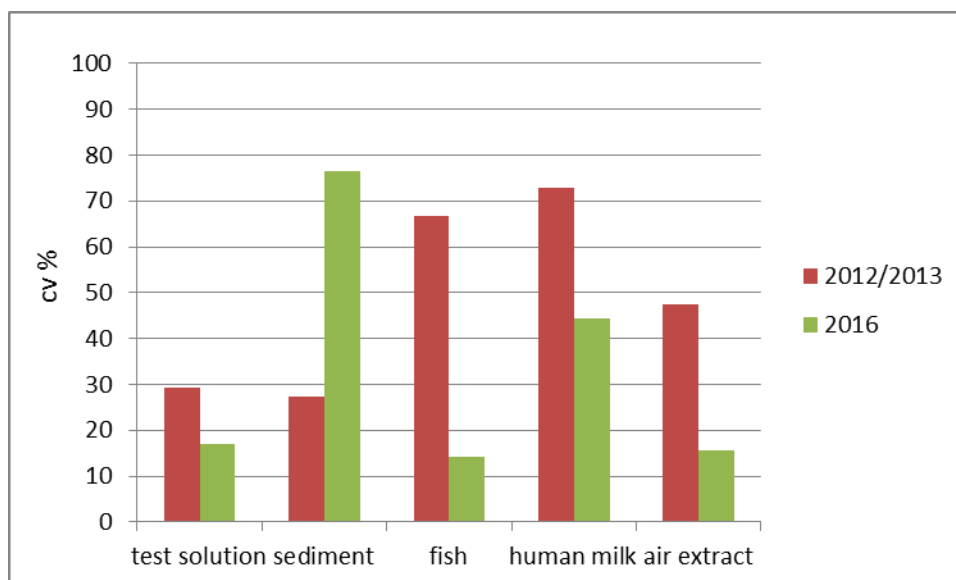


Figure 43 Comparison of performances between interlaboratory assessments for the PBDE analyses.

The PBDE performance was generally better in this round, apart, again, for the sediment (Figure 43). The first round did not include PBDE analysis. The improvement for fish, human milk and air is encouraging. CVs for fish, test solution and air are all below the desired 25%.

6 CONCLUSIONS AND RECOMMENDATIONS

6.1 Technical Conclusions

This exercise was characterized by a strong increase in participation of laboratories. As such, this is encouraging as 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 and for a number of matrices such as the test solution and for some compound groups such as the OCPs and indicator PCB the poorer performance of the newcomers has negatively influenced the overall performance. For the more advanced POPs, such as dl-POPs and PFAS, no difference was found between laboratories that participated in earlier rounds and those that participated for the first time.

Laboratories that carry out POP analyses on a daily basis, and with proper instrumentation, such as the WEOG group do show better results. The challenge of this program is of course to bring the laboratories in continents such as Africa at the same level. This is possible but only when governments support their laboratories and let them carry out monitoring programs and analyses on a regular basis.

Two components of this study were encouraging. The overall performance of the participants for the air extracts was clearly better than for the fish and sediment. However, it shall be noted that the air extract was fortified. Further improvements are still possible, but air is apparently a matrix that many laboratories can handle. This is a great support for the GMP as air is an important matrix in the GMP. The other encouraging achievement is that for the first time some data on toxaphene were generated. The results for the test solution were very reasonable. Unfortunately, the other test materials contained very little toxaphene. For a next round test materials with substantial levels of toxaphene should be used.

As in the previous two assessments, the results for dioxin-like POPs are good. However, it should be mentioned that there is larger variation for the dl-PCB. The majority of the laboratories analyses both groups of dl-POPs, *i.e.*, PCDD/PCDF and dl-PCB. The laboratories carrying out these analyses are apparently well aware of the required quality issues and have high quality 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). Three laboratories seem to originate from “basic POPs” laboratories and expanded their spectrum of PCB to dioxin-like PCB. These laboratories failed with their intention: It is not possible to identify and quantify dl-PCB in naturally contaminated samples (at baseline contamination) with GC/ECD instrumentation. The capacity and good performance of dl-POPs analysis remains to be located in Asia (China and Japan) and the WEOG regions; however, in each of the other regions there is at least one laboratory performing dl-POPs analysis with HRGC/HRMS instrumentation.

The analysis of brominated flame retardants, PBDEs and HBCD was in general encouraging. 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 results are good for PFAS, including PFOS, but only a relatively small number of laboratories submitted results and for quite a number of compounds and matrices, no consensus value could be assigned. Capacity now exists in all five UN

regions; however, the vast majority is located in the Asia-Pacific and WEOG region. All but one laboratory used LC/MS/MS instrumentation; the exemption was one laboratory in WEOG using a TOF instrument.

None of the 133 participating laboratories were able to carry out all analyses that were offered in this assessment. This shows that none of the laboratories have methods at its disposal for all Stockholm Convention POPs for all samples types and laboratories are often specialized on a certain compound class or sample type. Several regions and countries were under-represented concerning the analysis of several of the compound classes or sample types.

With respect to logistics, unexpected difficulties occurred due to stricter 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.

Because of the growing number of analytes and test materials, more time for analysis should be given to the laboratories in the next round of this exercise.

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 UN Environment 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.

6.2 Recommendations

Based on the results in the second assessment, 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. Training, instruction and capacity building is necessary in the developing regions (CEE, Africa, GRULAC and parts of the Asian and Pacific region) for all POPs with particular attention to clean up of difficult matrices such as sediment and fish.
3. 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.
4. Laboratories analysing OCPs are encouraged to use GC-MS and ^{13}C labelled standards to improve their analysis.
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 chlordane. At the moment there is very little capacity in the various UN regions for these POPs.
7. As it is extremely difficult to obtain test materials with a relevant contamination degree for all POPs, the materials may need to be fortified for some of the POPs, in order to provide materials with realistic levels that can be detected with the current methods, so that z-scores can be calculated for all compounds.
8. Due to the high number of POPs to be analysed in this study, participants should be given more time in the next round to complete their analyses on time.
9. Participants should consider to more often use a second GC column to check possible co-elutions.

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8 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 UN Environment Chemicals and Health Branch's Website.

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