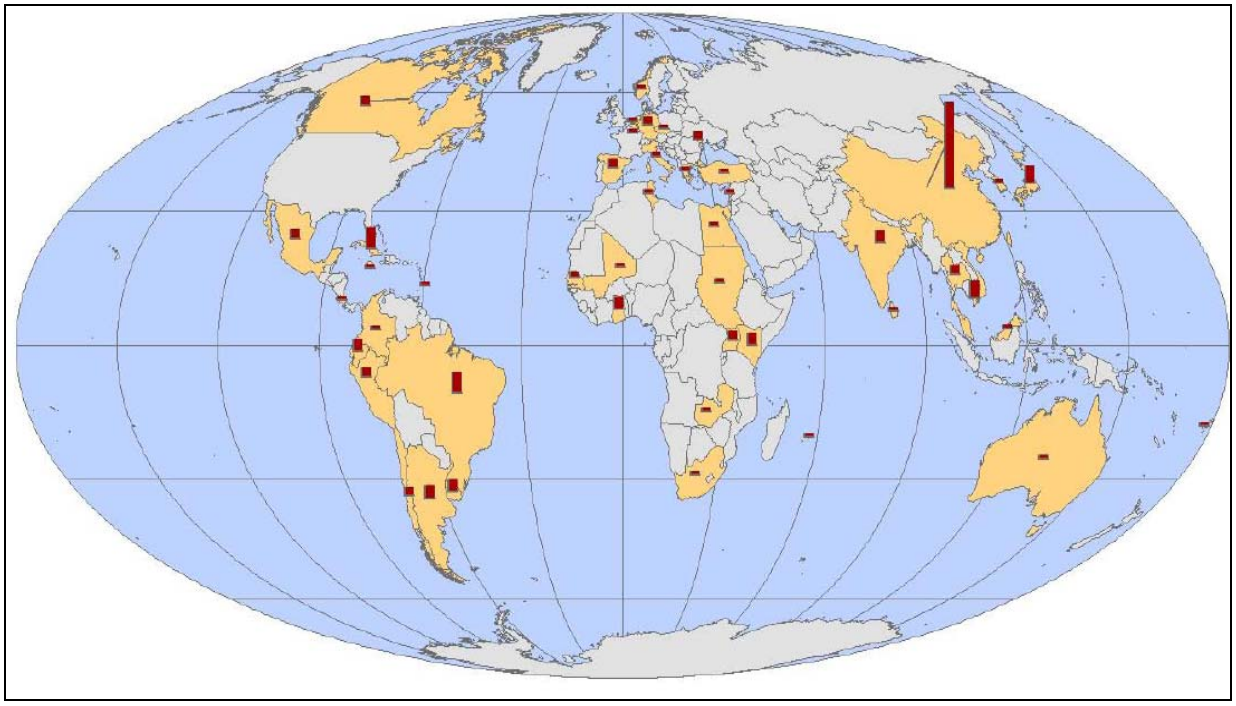
Bi-ennial Global Interlaboratory Assessment on Persistent Organic Pollutants – First Round 2010/2011



Coordinated by:

Chemicals Branch

United Nations Environment Programme/DTIE

March 2012

IOMC

INTER-ORGANIZATION PROGRAMME FOR THE SOUND MANAGEMENT OF CHEMICALS

A cooperative agreement among FAO, ILO, UNDP, UNEP, UNIDO, UNITAR, WHO, World Bank and OECD

Sketch on title page: World map displaying countries and number of laboratories participating in this interlaboratory proficiency assessment;
prepared by Dr. Heidelore Fiedler, UNEP/DTIE Chemicals Branch

This publication was developed in the IOMC context. The contents do not necessarily reflect the views or stated policies of individual IOMC Participating Organizations.

The Inter-Organisation Programme for the Sound Management of Chemicals (IOMC) was established in 1995 following recommendations made by the 1992 UN Conference on Environment and Development to strengthen co-operation and increase international co-ordination in the field of chemical safety. The Participating Organisations are FAO, ILO, UNDP, UNEP, UNIDO, UNITAR, WHO, World Bank and OECD. The purpose of the IOMC is to promote co-ordination of the policies and activities pursued by the Participating Organisations, jointly or separately, to achieve the sound management of chemicals in relation to human health and the environment.

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Bi-ennial Global Interlaboratory Assessment on Persistent Organic Pollutants

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Acronyms and Abbreviations

AV	Assigned value
CE	Constant error
CEE	Central and Eastern Europe
CSIC	Consejo Superior de Investigaciones Científicas
DDT	Dichlorodiphenyltrichloroethane
dl-PCB	Dioxin-like polychlorinated biphenyls
dl-POPs	Dioxin-like POPs
GC	Gas chromatograph(y)
GC/ECD	Gas chromatography with electron capture detection
GC/MS	Gas chromatography with mass spectrometric detection
GEF	Global Environment Facility
GPC	Gel permeation chromatography
GRULAC	Group of Latin America and Caribbean
HCB	Hexachlorobenzene
HCH	Hexachlorocyclohexane
HRGC	High resolution gas chromatography
HRMS	High resolution mass spectrometry
ILAC	International Laboratory Accreditation Cooperation
ISO	International Organization for Standardization and
IVM	Institute for Environmental Studies
LB	Lowerbound
LCV	Left-censored values (values below detection limit)
LP	Laboratory performance
LRMS	Low resolution mass spectrometry
MS	Mass spectrometer or: mass spectrometry
MSWI	Municipal waste incinerator
MTM	Man-Technology-Environment
NA	Not applicable
ND	Not detected
NDA	Normal distribution assumption
OCP	Organochlorine pesticide
OECD	Organisation for Economic Co-operation and Development

PCB	Polychlorinated biphenyl
PCDD/PCDF	Polychlorinated dibenzo-*para*-dioxins/polychlorinated dibenzofurans
PDF	Probability density function
PE	Proportional error
PMF	Main mode
POPs	Persistent organic pollutants
QUASIMEME	Quality Assurance of Information for Marine Environmental Monitoring in Europe
QA/QC	Quality assurance/quality control
RSD	Relative standard deviation
SAICM	Strategic Approach for International Chemicals Management
SD	Standard deviation
TCDD	2,3,7,8-Tetrachloro-*p*-dibenzodioxin
TEF	Toxicity equivalency factor
TEQ	Toxic equivalent
TEQ_{PCB}	Toxic equivalent based on dl-PCB
$TEQ_{PCDD/PCDF}$	Toxic equivalent based on PCDD and PCDF (dl-PCB not included)
TEQ_{total}	Toxic equivalent based on PCDD, PCDF, and dl-PCB
UB	Upperbound
UNEP	United Nations Environment Programme
WEOG	Western European and Other Groups

Definitions

Basic POPs	Include: Organochlorine pesticides (aldrin, chlordane, DDT, dieldrin, endrin, heptachlor, hexachlorobenzene, mirex, toxaphene) and polychlorinated biphenyls
Dioxin-like POPs	Include: 29 congeners that were assigned a TEF by WHO/IPCS expert group, namely polychlorinated dibenzo-*para*-dioxins, polychlorinated dibenzofurans, and polychlorinated biphenyls

Summary

Objective

The Chemicals Branch of the United Nations Environment Programme (UNEP) with the assistance of funds from the Global Environment Facility (GEF), SAICM Quick Start Programme, and the government of Norway organized the interlaboratory assessment on persistent organic pollutants, named the "Bi-ennial Global Interlaboratory Assessment on Persistent Organic Pollutants (POPs)". The assessment has been implemented during 2010-2011. Its goal was to test the capabilities of laboratories in the analysis of the twelve initial POPs listed in the Stockholm Convention. The UNEP-coordinated Global Interlaboratory Assessment was performed according to internationally agreed standards (following ISO-International Organization for Standardization and ILAC-International Laboratory Accreditation Cooperation). Such proficiency tests are valuable management tools to allow external quality controls of the performance of a laboratory that undertake chemical analysis.

The basis for the interlaboratory assessment is laid down in the Databank of Operational POPs Laboratories, which was developed by the UNEP/GEF Global project on POPs laboratory capacity building[1] from 2005 to 2007. Since that time, the Chemicals Branch maintains this databank and makes it publicly available on its web-site (http://212.203.125.2/databank/Home/Welcome.aspx). Presently there are more than 230 POPs laboratories registered.

The mandate for the international laboratory comparison studies comes from recommendations by the Conference of the Parties to the Stockholm Convention as expressed in the guidance document for the Global Monitoring Plan in article 16 of the Convention (UNEP 2007a, UNEP 2011). In chapter 4 of the guidance document for the GMP, it states "Interlaboratory exercises are often used to assess the effectiveness of QA/QC practices among several participating labs and to provide a measure of interlab comparability. This usually involves the circulation and analysis of a common standard or reference sample, often at two or more concentration levels".

With respect to POPs concentrations in humans and the environment, the quantitative objective for the Global Monitoring Plan is set in chapter 3 "To detect a 50 % decrease within a time period of 10 years with a statistical power of 80 % at a significance level of 5 %." (UNEP 2007a, UNEP 2011). In order to achieve this objective in POPs laboratory analysis, UNEP has set a criterion of 25 % for relative standard deviations (RSDs) (corresponding to -12.5 % to +12.5 % below or above the consensus value).

Methodology

The assessment focused on the analysis of the twelve initial POPs listed in Annexes A, B, and C of the Stockholm Convention on Persistent Organic Pollutants (UNEP 2009).

[1] Assessment of Existing Capacity and Capacity Building Needs to Analyze POPs in Developing Countries, WebSite http://www.chem.unep.ch/Pops/laboratory/default.htm

Whereas proficiency tests are well established for laboratories in OECD countries, challenges were expected for developing country participating laboratories since they do not yet have the necessary experience to analyze a large number of POPs in biotic and abiotic matrices at the requested accuracy.

The first "Global Interlaboratory Assessment on Persistent Organic Pollutants" (POPs) under the Stockholm Convention was organised in the Asian/Pacific region in 2009/2010 and for the African and Latin American region in 2010/2011. In addition to these laboratories, several laboratories from OECD countries participated in both rounds. The participating laboratories had a choice to analyse different matrices: three test solutions, a fish material, sediment, fly ash and human milk. In total, 103 laboratories worldwide participated in the present assessment. Of these, 83 laboratories submitted data on at least one of the sample types. All results were statistically evaluated according to the procedures used in the QUASIMEME proficiency-testing scheme.

The 83 POPs laboratories participating in this UNEP interlaboratory assessment were from 47 countries representing all UN regions. The distribution of the laboratories per group of POPs and region was as follows:

1. Simple POPs (PCB and organochlorine pesticides), 12 laboratories came from the Western and other Groups (WEOG) region and 61 laboratories came from the other four UN regions (10 from Africa, 35 from Asia, 3 from Central and Eastern Europe (CEE), and 23 from the Group of Latin America and the Caribbean (GRULAC));

2. Complex POPs (polychlorinated dibenzo-*p*-dioxins, polychlorinated dibenzofurans; PCDD/PCDF, dioxin-like polychlorinated biphenyls; dl-PCB), 10 laboratories came from WEOG region and 40 came from the other four UN regions (3 from Africa, 32 from Asia, 1 from CEE, and 4 from GRULAC).

A large number of laboratories, especially in the Asian region, reported data for the PCDD/PCDF and dioxin-like PCB. For the indicator PCB and organochlorine pesticides (OCPs) more results were included from the African and Latin American region. The analysis of pesticides is generally considered less complicated and less complex instrumentation is needed.

Results

The best results were obtained for the test solutions, and, as regards to chemical compounds, for PCDD/PCDF. The fish sample was the most difficult matrix for the participants as were the OCP as a compound class.

The results for the PCDD/PCDF and dioxin-like PCB, although reported on congener basis, were assesses in terms of the toxic equivalent (TEQ) of the sample. Toxic equivalents were reported for the PCDD/PCDF ($TEQ_{PCDD/PCDF}$) and for the dl-PCB (TEQ_{PCB}). In general, the results on TEQ basis were good whereby typically, the $TEQ_{PCDD/PCDF}$ had lower RSDs than the TEQ_{PCB}. . In some cases the results from this assessment were even better than reported earlier for this complex analysis. The $TEQ_{PCDD/PCDF}$ results showed an RSD (relative standard deviation) or between-lab CV (coefficient of variation) of 11%-17% for the human milk, whereas between-lab CV values for OCP in milk were regularly higher than 100%. This was also true for fish, in which the $TEQ_{PCDD/PCDF}$ between-lab CV was 26% while the between-lab CV for OCPs in fish varied between 33% and 207%. Most re-

sults were roughly comparable to an earlier, smaller interlab assessment organised by a UNEP/GEF project for the Stockholm Convention during a pilot project on capacity building in 2006/2007, but the more sophisticated statistical model used now gives a better insight in the results and shows more clearly the contrast between the mean results and the outliers.

Conclusions and Recommendations

This interlaboratory intercomparison assessment had the largest number of participating POPs laboratories in comparison to earlier studies. It also had a large number of sample matrices offered to the participating laboratories. This interlaboratory assessment has been successful in using the momentum for generating awareness on the need of accurate and precise data. POPs laboratories around the world had committed themselves to take part in this comparison between laboratories using real samples. Overall, the assessment has laid down a sound basis for laboratories to know where they stand with their analytical capacities and how their performance compares with others.

According to this assessment, the UNEP criterion of 25% RSD has been met only for dioxin-like POPs, *i.e.,* for $TEQ_{PCDD/PCDF}$ in standard solution, sediment, and human milk and for TEQ_{PCB} in human milk. Further analytical improvement is required for OCP and PCB in all sample matrices.

The poorer PCB and OCP results were mainly caused by the use of the electron capture detector (ECD), whereas the more sophisticated mass-selective (MS) detection was normally applied for PCDD/PCDF analysis. However, for many laboratories investment in MS detection will be a major cost issue. Whereas there are obvious advantages in using MS detection, additional difficulties will arise with, *e.g.,* training demands and maintenance of these apparatus. Further, the presence of MS instrumentation alone does not guarantee better results in this stage.

The results emphasise the need for all laboratories to pay more attention to quality assurance (QA) and method development. With the addition of new compounds to the list of the POPs under the Stockholm Convention, POPs laboratories are encouraged to expand their spectrum of analytes and matrices to match the national and global needs. Accordingly, analytical schemes will be developed and tested in further rounds of interlaboratory comparison assessments.

1. INTRODUCTION

This interlaboratory assessment accompanies UNEP's capacity building program for POPs laboratories that has started in 2005 with GEF funding and implements the recommendations by the Conference of the Parties to the Stockholm Convention as expressed in the guidance document for the Global Monitoring Plan in article 16 of the Convention (UNEP 2007a, UNEP 2011). In chapter 4, the guidance document states "Interlaboratory exercises are often used to assess the effectiveness of 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 subsequence guidance developed for the Global Monitoring Plan (GMP). The needs and support are documented in COP decisions SC-3/16, SC-4/31 and 5/18 and in chapter 3 of the GMP Guidance Document. To provide reliable monitoring information for the Parties to the Stockholm Convention, the guidance in the GMP document aims to "confirm a 50% decline in the levels of POPs within a 10 year period" (UNEP 2007a, UNEP 2011). This means that POPs laboratories must be capable – at any time – to analyze samples for POPs within a margin of ±12.5 %.

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

Whereas proficiency tests or "round robins" on polychlorinated biphenyls (PCB), organochlorine pesticides (OCPs), and dioxin-like 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 analyze 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, the United Nations Environment Programme (UNEP) 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 has been organized, which included various types of biotic and abiotic matrices.

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 to allow sound decision making. A well established approach that has been working very well to improve the analytical quality and to quantify the uncertainty in analytical data is the organization of interlaboratory comparison studies. Today, interlabora-

tory studies are an important QA/QC tool for POPs analysis and are being organized on a regular basis for a variety of matrices and groups of POPs. These studies have growing numbers of participating laboratories and show that the number of laboratories able to perform analysis with acceptable variation is increasing. Participation at international intercalibration studies is considered a prerequisite for existing, well established and 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.

In the scoring system to rank performance of POPs laboratories, successful participation in international interlaboratory studies is rank highest, namely with 50%. Detailed information on scoring criteria is available in the Handbook for POPs Laboratory Databank (UNEP 2007b)

Within the framework of the United Nations Environment Programme's (UNEP) Capacity Building project for training of laboratory staff on persistent organic pollutants (POP) analysis in developing countries, the Institute for Environmental Studies of the VU University Amsterdam, The Netherlands (IVM) and the MTM Research Center, School of Science and Technology at the University of Örebro, Sweden, have organised the Bi-ennial Global Interlaboratory Assessment on Persistent Organic Pollutants (POPs). The first phase of this assessment was financed by the Norwegian Government and was conducted in Asia in 2009/2010. The second phase of the project was embedded into four regional projects financed by the Global Environment Facility (GEF) and two projects by the Quick Start Programme (QSP) of the Strategic Approach for International Chemicals Management (SAICM); they covered the African and Latin American regions in 2010/2011. In addition to the developing countries from Asia-Pacific, POPs laboratories from developed countries were invited to participate as well at their own costs.

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, mirex, dieldrin, endrin, aldrin, chlordanes, hexachlorobenzene, heptachlor and *cis*-heptachlorepoxide. Toxaphene was not included since no or only limited capacity was available among the participating laboratories.

In total, five matrices were offered for analysis: standard solutions for organochlorine pesticides, for indicator PCB, and for dioxin-like POPs, sediment, fish, fly ash (for dioxin-like POPs only), and human milk. The test solutions in amber glass ampoules with the target compounds in undisclosed concentrations were sent to the participating laboratories. The sediment was air-dried, the fish consisted of a freeze-dried sample, and the human milk was homogenised and frozen before shipment.

Hundred and three laboratories from 47 countries participated (see Appendix I for their names and addresses as well as the abbreviations (lab codes) that have been used throughout this report). All codes are confidential and kept with the organizers; they will only be revealed to third parties after permission of the participant.

2. MATERIALS AND METHODS

Chemicals Branch of UNEP/DTIE (Division of Technology, Industry and Economics) invited POPs laboratories around the world to participate in this Global Interlaboratory Assessment. Interested laboratories were given a choice of analytes and test samples according to the Table 1 below.

Table 1: Registration form for POPs laboratories to participate in the Interlaboratory Assessment – choice of analytes and test matrices

My laboratory is interested in analyzing the following matrices and POPs and provide the analytical results according to the reporting scheme and timetable (analysis within ca. 8 weeks after receipt):					
Matrix of Inter-calibration Sample	Persistent Organic Pollutant				Instrumentation (Indicative: ECD, LRMS, HRMS)
Standard Solution	Pesticides ☐	PCB_7 ☐	PCDD/PCDF ☐	dl-PCB ☐	LRMS,
Soil/Sediment	Pesticides ☐	PCB_7 ☐	PCDD/PCDF ☐	dl-PCB ☐	,
Fly Ash	Pesticides ☐	PCB_7 ☐	PCDD/PCDF ☐	dl-PCB ☐	,
Fish	Pesticides ☐	PCB_7 ☐	PCDD/PCDF ☐	dl-PCB ☐	,
Mother's Milk	Pesticides ☐	PCB_7 ☐	PCDD/PCDF ☐	dl-PCB ☐	LRMS,

The coordinating laboratories of this POPs Global Interlaboratory Assessment, Institute for Environmental Studies IVM), VU University Amsterdam, Netherlands and MTM Centre of the University of Örebro, Sweden, received and maintained the list of participating laboratories. A form for reporting results in MsExcel® was prepared and sent to the registered laboratories together with the sample that they had chosen for the interlaboratory assessment. Laboratories were given a period of eight weeks to report back their results using the defined format.

2.1 Preparation of the Test Samples

The ash test material was a fly ash from a MSWI incinerator from Sweden taken after the bag house filter and wet scrubber. The ash was used as received (dry) and homogenised at the MTM Research Center at the Örebro University. The ash contained medium levels of the target compounds (dl-POPs).

The sediment originated from Norway and was air-dried at 40 °C and sieved (0.5 mm pore size). After homogenisation, individual plastic containers were filled with the test matrix and stored at room temperature until shipment. The samples were obtained from WEPAL.

The fish material consisted of a freeze-dried trout from the Great Lakes, made available by Dr. Eric Reiner from the Ontario Ministry of Environment, Laboratory Services Branch, Ontario, Canada.

The human milk consisted of pooled, homogenised milk from the Swedish mother milk bank in the Stockholm area. The milk samples were frozen and stored at -20 °C before shipment.

All above samples were naturally contaminated with the target analytes.

Standard 1A consisted of a mixture of PCDD/PCDFs and dl-PCBs in the concentration range of 10- 500 pg/µl (ng/ml). This standard was prepared, ampouled and labelled by Wellington Laboratories (Guelph, Ontario, Canada).

Standard 1B consisted of a mixture of the indicator PCB (PCB 28, 52, 101, 138, 153 and 180) in the concentration range of 0.1-5 ng/µl (µg/ml). This standard was prepared, ampouled and labelled by Wellington Laboratories (Guelph, Ontario, Canada).

Standard 1C consisted of a mixture of organochlorine pesticides (OCP) in the concentration range of 10-50 pg/µl (ng/ml). This standard was prepared by IVM from a standard solution obtained from Cambridge Isotope Laboratories (Andover, USA). After preparation, the aliquots were ampouled, labelled and stored at room temperature. The OCP present in the solution were HCB, aldrin, dieldrin, endrin, *p,p'*-DDT, *p,p'*-DDE, *p,p'*-DDD, *o,p'*-DDT, *o,p'*-DDE, *o,p'*-DDD, *trans*-chlordane (*gamma*), *cis*-chlordane (*alpha*), *trans*-nonachlor, *cis*-nonachlor, oxychlordane, heptachlor, *trans*- heptachloroepoxide (HEPO), *cis*-HEPO, mirex, α-HCH, β-HCH, γ-HCH and δ-HCH. Although present, the HCHs were not part of the assessment since they are not included in the list of the initial twelve POPs under the Stockholm Convention.

The fish, human milk and ash samples and standards 1A and 1B were distributed by MTM, whereas the sediment sample and standard 1C were distributed by IVM.

2.2 Methods Used by Participating Laboratories

The participants were not restricted in the methodology used for the analysis of the target compounds. Although it is for example advisable to analyse dl-POPs (*i.e.,* PCDD, PCDF, dl-PCB) with gas chromatography (GC) - high resolution mass spectrometry (HRMS) systems, also results from low resolution mass spectrometry (LRMS) instrumentation were accepted. The use of 'high resolution' capillary GC is considered mandatory to achieve the separation needed for an accurate determination of the analytes. The laboratories used their own sample extraction and clean-up protocols, spiking schemes and internal QA/QC. The reporting forms for the different classes of chemicals and the different matrices prepared in Microsoft EXCEL were used by all participating laboratories to submit their results.

For the analysis of the PCDD/PCDF and dl-PCB most laboratories used GC-HRMS systems but some used GC-LRMS systems. The majority of the labs used three columns to clean-up the samples after extraction: a multi-layer silica, an alumina or Florisil column and a carbon-based column. Several labs used an automated clean-up system in which these three columns were incorporated. A few GC-HRMS labs did not use all clean-up columns and omitted the alumina or the carbon column from the clean-up procedure. The fly ash samples were often treated with acid before Soxhlet extraction or pressurized liquid extraction systems using toluene or toluene-based mixtures. Only one laboratory used dichloromethane as extraction solvent. Also for the sediment sample, Soxhlet and pressurized liquid extraction systems were used with toluene or dichloromethane/hexane mixtures.

The freeze-dried fish sample was extracted by pressurized liquid extraction systems, Soxhlet or liquid/liquid extraction (sometimes after KOH/ethanol decomposition of the sample). A large variety of extraction methods were used for the milk samples ranging from liquid/liquid to supercritical fluid extraction after mixing with an absorbent or pressurized extraction systems or Soxhlet extraction.

Only a limited number of labs analysed the seven indicator PCB in the fly ash samples using similar clean-up steps as for PCDD/PCDF and dl-PCB (Soxhlet, multilayer silica and or alumina) and detection using GC-HRMS. A variety of GC columns with different polarities and dimensions were used for optimal separation. GC/ECD was sometimes used for the indicator PCB in fly ash.

Methods to analyse the sediment for non-dl-POPs did not show much variation: most laboratories used GC/LRMS and GC/ECD. The marker PCB in the fish and the milk sample were extracted by liquid/liquid, Soxhlet and pressurized liquid extraction systems and fat removal was achieved by alumina, multi-layer silica, gel permeation or concentrated sulphuric acid (H_2SO_4). From the submitted data it was often not clear if the marker PCB were analysed together with the dl-PCB, as a separate fraction apart from the dioxin analysis or by applying a complete separate extraction and clean-up procedure.

The analytical procedures to analyse the pesticides varied widely from using HRGC/HRMS, HRGC/LRMS to GC/ECD to detect the target compounds. Again, in several cases it was not clear from the data if a combined or separate pesticide analyses was performed.

Surprisingly and especially in the Asian region several labs used GC/HRMS to analyse the pesticides. This is an interesting development and seems to be characteristic for the Asian

region where GC-HRMS capacity seems to be more widely available. For all samples, a wide variety of sample extraction and clean-up methods were used including Soxhlet, pressurized liquid extraction systems, liquid/liquid, ultrasonic extraction, GPC, multilayer silica, alumina and Florisil.

2.3 Data Assessment

The data assessment was carried out according to the principles employed in the data assessment of the QUASIMEME proficiency testing organisation (www.quasimeme.org). All data received from the participants were entered into a database and assessed using a standard procedure to allow direct comparison between participants. The approach of the assessment is based on the standard, ISO 13528 (2005), the IUPAC International Harmonised Protocol for Proficiency Testing (Advanced Draft) by Thompson *et al.* (2006). Additions or differences in the assessment from these standards are given or referred to in this report. However, the assigned value, the RSD (CV) values and the laboratory assessment using z-scores are based on the Cofino Model (Cofino *et al.*, 2000). For information also the RSD values that were not corrected for outliers are given. These values have also been used in the Figures 11-17, as those better reflect the realistic performance of all laboratories in the regions. The last column of the Tables 1, 3, 5, 7, 9, 11, 13, 15, 17, 19, 21, 23, 25 and 27 shows the so-called 'Inclusion rate'. This values is a percentage that reflects how many of the data are included in the 'Between-lab CV', shown in the column left from the Inclusion rate column. The higher the inclusion rate, the lower the number of outliers. A higher inclusion rate also tells that the Between lab RSD is more representative for the entire group of participants that produced that specific matrix-determinand combination.

Comparison between the robust statistics method for calculation of a mean and the Cofino model continues to be made, and where there are any significant discrepancies between the two methods, further investigative analysis was undertaken. The Cofino model is generally able to separate the effects of the method on the results and provide a more reliable estimate of the measurement relating to the method. The standard, ISO 13528, includes statistics for proficiency testing schemes, and uses robust statistics as a basis for the assessment. However, it is generally acknowledged that robust statistics cannot cope with more than 10% extreme values, particularly with a skewed distribution. The Cofino model is able to routinely cope with these types of distribution and provide the best estimate of the consensus value, which may be used as the assigned value.

The Cofino model has been developed for the routine QUASIMEME assessments. The Cofino model uses a Normal Distribution Assumption (NDA). The assigned value is based on the Cofino NDA model without any trimming of the data. This approach includes all data in the evaluation and no subjective truncation or trimming is made. This model has been further developed to include Left Censored Values (LCV)[2]. The development of these models has been fully documented and published (Cofino *et al.*, 2000; Cofino *et al.*, 2005; Wells *et al.*, 2004). An overview of the assessment with explanation and examples is given in the Assessment Rules for the Evaluation of the QUASIMEME LP Studies Data (Wells and Scurfield, 2004).

[2] *Left Censored Values* is the correct nomenclature for "less than" values

The details of the Cofino Model were provided elsewhere (Wells *et al.*, 2004; Wells and Scurfield, 2004) but in summary, the approach is as follows:

- All data included in the assessment
- No data trimmed or down weighted
- Assigned values (AV) based on Cofino NDA model
- All LCV are also included, provided certain criteria are met

2.3.1 Plots

The performance of the laboratories in this assessment is illustrated in the z-score histograms. Where the assigned value for an analyte is indicative, the values are plotted as their original reported concentrations. The rules for confirming whether the consensus value should be an assigned value or an indicative value are given in the Assessment Rules for the Evaluation of the QUASIMEME LP Studies Data (Wells and Scurfield, 2004) with relevant examples.

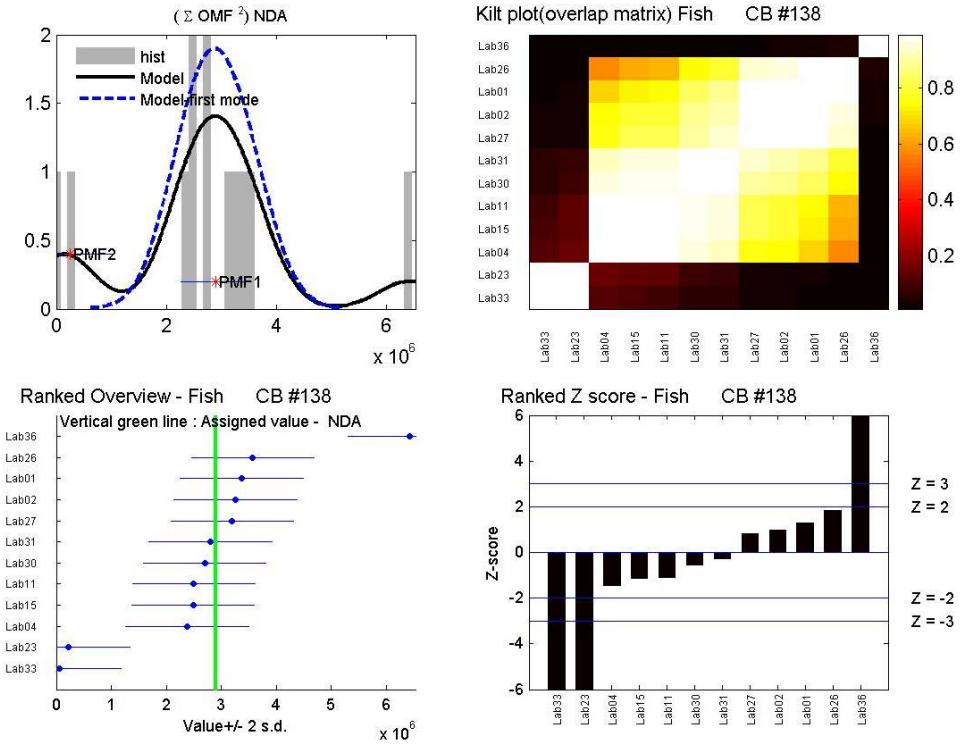


Figure 1 Graphical output of the Cofino Model statistics for PCB 138 in the fish sample.

Figure 1 presents the four plots that are normally are given for each analyte. The upper left plot provides an impression of the probability density function (PDF) for all data (black) and for the first mode (blue dotted) (PMF1) of the data. Superimposed on these PDFs is a histogram of the individual measurements, given in grey. This plot shows the distribution of the data as a whole, and of the data in the main mode (PMF1) on which the assigned value is based.

The "Kilt Plot" (Overlap Matrix) (upper right plot of Figure 1) provides an overview of the degree of overlap of each pair of data. It gives a clear indication of the degree of homogeneity of the data. As a key, the white areas indicate maximum overlap of the PDFs and, therefore, highest agreement (an overlap of one implies that the two laboratories of the pair report exactly the same results), while the black area show the pairs in poor agreement.

The lower left plot in Figure 1 is a ranked overview of all data with an error bar of ± 2 SD. The numerical values are given in blue and the left censored values are given in red.

Finally, the ranked z-score plot (lower right in Figure 1) is based on the mean of the data, which is normally also the assigned value. However, if there is any adjustment required to the assigned value as a result of the assessment, *e.g.,* use of the nominal concentration or a trimmed value, then the final z-score given in the z-score histograms will reflect these changes. In this assessment, no such adjustments are made and therefore, the z-score plot (lower right) is the definite plot for obtaining the individual lab z-scores.

For each matrix-determinand combination a set of these four graphs is available. They can all be found in Appendix IV.

2.3.2 The Assigned Values and Indicative Values

The Assigned Value (AV) is obtained from the main mode of the data using the Cofino Model (bleu dotted line in upper left panel in Figure 1, top-left), and is centered around the highest density of values. Unless otherwise stated, the assigned value is based on this consensus value of *all* data. Although *all* data are included in the assessment, those values that lie some distance from AV contribute less to the mean than values, which occur at or near the mean.

In some instances, it is not possible to set an AV, and an indicative value is given. No assessment of laboratory performance is given where an indicative value is set. An overview of the assessment, with explanation, decision flowcharts and examples, is given in the paper *Assessment Rules for the evaluation of the QUASIMEME Laboratory Performance Studies Data*, available on the QUASIMEME website, www.quasimeme.org. A summary of the categories is given below:

Category 1

For data with the number of numerical observations ≥ 7

An assigned value is based on the mean when ≥ 33% of values have a z-score of $|z| < 2$. Where < 33% of the data have $|z| < 2$ the value is indicative, *i.e.,* at least 33% must be in good agreement.

Category 2

For data with the number of numerical observations > 3 and < 7

An assigned value is based on the mean when ≥ 70% of values have a z-score of $|z| < 3$ and a minimum of 4 observations have $|z| < 2$. Otherwise, the value is indicative. *i.e.,* for small datasets, 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 with the number of numerical observations < 4

No assigned value is given. Normally the median value is given as an indicative value.

Category 4

For data with the high Total Error% >100% in combination with bad performance, no assigned value is given.

2.3.3 The z-score Assessment

A z-score (Thompson and Wood, 1993) is calculated for each participant's data for each matrix / analyte combination which is given an assigned value. The z-score is calculated as follows:

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

It is emphasized that in many interlaboratory studies the between-laboratory standard deviation obtained from the statistical evaluation of the assessment is used as 'total error' in the formula above. In the QUASIMEME data assessment, the total error is estimated independently taking the needs of present-day international monitoring programs as starting point. For each analyte in a particular matrix, a proportional error (PE) and a constant error (CE) have been defined. The total error depends on the magnitudes of these errors and on the assigned value:

$$\text{Total Error} = \frac{\text{Assigned Value} \times \text{Proportional Error (\%)}}{100} + 0.5 \times \text{Constant Error}$$

The values for PE and CE were developed by QUASIMEME. The values are based on the following criteria:

- Consistency of the required standard of performance to enable participating laboratories to monitor their assessment over time.
- Achievable targets in relation to the current state of the art and the level of performance needed for national and international monitoring programmes.

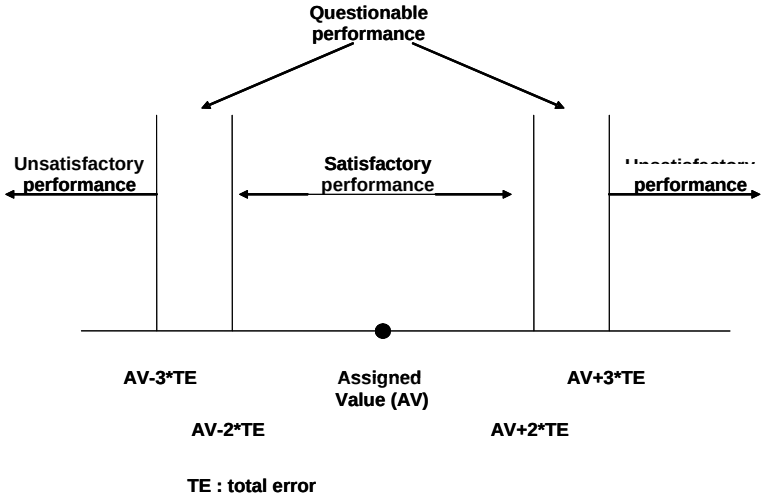
The assessment is based on ISO 43 as z-scores. The QUASIMEME model is designed to provide a consistent interpretation over the whole range of concentration of analytes provided, including an assessment where Left Censored Values (LCVs) are reported.

The PE in this assessment was set at 12.5% for all matrices. This applies to all analytes. The CE has been set for each analyte or analyte group (*e.g.,* polychlorinated biphenyls). This value was initially set to reflect the limit of determination, but is at present more closely related to the overall laboratory performance. The magnitude of the CE is set to provide a constant assessment in terms of z-score regardless of concentration. Therefore, at low concentrations the level of accuracy required to obtain a satisfactory z-score is less stringent than at a high concentrations.

Following usual practices *e.g.*, ISO 43, the z-scores can be interpreted as follows to assure the quality of their data:

$\|z\| < 2$	Satisfactory performance
$2 < \|z\| < 3$	Questionable performance
$\|z\| > 3$	Unsatisfactory performance

The following figure illustrates the interpretation of the z-scores:



$|z| > 6$ frequently points to gross errors (mistakes with units during reporting, calculation or dilution errors, and so on).

It is not possible to calculate a z-score for left censored values (LCVs). QUASIMEME provides a simple quality criterion:

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

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

z score key: S – Satisfactory
Q – Questionable
U – Unsatisfactory

LCV key: C – Consistent
I – Inconsistent

No data: B – Blank

2.4 UNEP Criteria for Data Assessment

After the conclusion of the first phase of the Interlaboratory Assessment, where 37 POPs laboratories from Asia and some from WEOG participated, a regional workshop was organized in Hong Kong, SAR (26-28 February 2010, see Report of the Final Results Workshop of First Worldwide UNEP Intercalibration Study on POPs – Asia Region, Regal Riverside Hotel, Shatin, Hong Kong SAR, People's Republic of China). The workshop report and the presentations given can be downloaded from Chemicals Branch's website http://www.chem.unep.ch/Pops/GMP/Asia/Report%20Final%20WS%20for%20Asia%20Intercalibration%20Study-HKG%202010.pdf.

Asian participating laboratories were invited to share and discuss in detail the preliminary results, especially in relation to the UNEP RSD criteria of 12.5%. This stringent criterion was set by Chemicals Branch of UNEP to assure that the target decrease of POPs concentration in the core matrices can be monitored: The Global Monitoring Plan (GMP) aims to show a 50% decline in levels of the POPs over a ten-year period. To demonstrate this decline is one of the decisive factors in the effectiveness evaluation of the Stockholm Convention (Article 16).

Especially when there is a large variation in the data set and outlier removal does not improve the RSDs or is not possible due to the distribution of the data, it is important to calculate the assigned values as accurate as possible. This importance has been illustrated in section 2.3, where the Cofino statistical approach is explained. A detailed discussion on the different statistical approaches, outlier removal and set of floating RSD values to calculated z-scores will be given in an upcoming issue of Trends in Analytical Chemistry (TrAC) in 2012, using the unique data of this First Round of the Bi-ennial Global Interlaboratory Assessment on POPs as an example.

3. RESULTS

All results of the individual laboratories are given in Appendix II. All z-scores are given in Appendix III. As mentioned above (section 2.3.1.) Appendix IV shows the four plots that characterize the results for each matrix-determinand combination. Finally, Appendix V gives all z-score plots. The submitted results have been evaluated statistically and whenever the data met the requirements (as mentioned in chapter 2), an assigned value was established. z-scores were calculated based on the assigned value. Summaries of the assigned values and the percentage of satisfactory to unsatisfactory z-scores are presented below. Whenever numerical 'less than' values (left censored values, LCV) were reported, it is mentioned whether these LCVs are consistent with the assigned value. Apart from the assigned value, mean and median value, the tables also include the geomean, which is obtained by the multiplication of n results and taking the n-root of that multiplication:

$$\text{geomean} = \sqrt[n]{a_1.a_2.....a_n}$$

3.1 Organochlorine Pesticides (OCPs)

Table 2: Summary results OCPs, standard solution

Standard													
Contaminant	**Unit**	**Target**	**Assigned**	**Mean**	**Median**	**Geomean**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
Aldrin	ng/ml	31	35	36	35	35	22	78	9	26	46	13	68
Dieldrin	ng/ml	31	36	38	36	37	14	92	12	31	50	17	68
Endrin	ng/ml	31	37	37	36	35	7,3	61	10	28	42	22	72
Sum Drins LB (ND = 0)	**ng/ml**	**92**	**104**	**101**	**103**	**97**	**34**	**194**	**27**	**27**	**47**	19	70
Sum Drins UB (ND = LOD)	**ng/ml**	**92**	**107**	**111**	**106**	**108**	**67**	**244**	**30**	**27**	**40**	16	74
trans-Chlordane	ng/ml	31	35	38	35	37	20	100	14	36	37	11	65
cis-Chlordane	ng/ml	31	35	36	35	35	13	68	9	24	37	15	74
trans-Nonachlor	ng/ml	31	35	39	35	38	27	81	12	32	26	5	57
cis-Nonachlor	ng/ml	31	34	39	34	37	29	70	11	29	19	10	68
Oxychlordane	ng/ml	31	34	40	34	37	26	155	26	63	23	9	63
Heptachlor	ng/ml	31	35	37	36	36	20	71	9,3	25	43	16	66
cis-Heptachlorepoxide	ng/ml	31	34	35	34	33	19	90	11	32	34	13	66
trans-Heptachlorepoxide	ng/ml	30	35	37	35	37	30	73	10	26	22	13	78
Sum Chlordane LB (ND = 0)	**ng/ml**	**245**	**NA**	**195**	**196**	**170**	**30**	**308**	**87**	**45**	**42**	59	85
Sum Chlordane UB (ND = LOD)	**ng/ml**	**245**	**283**	**271**	**280**	**257**	**110**	**621**	**96**	**35**	**22**	9	64
p,p'-DDT	ng/ml	31	36	37	36	34	8	67	13	35	46	18	66
o,p'-DDT	ng/ml	31	35	37	35	35	13	71	10	28	31	18	71
p,p'-DDE	ng/ml	31	35	38	35	36	11	137	18	46	51	15	69
o,p'-DDE	ng/ml	31	35	36	35	35	21	68	7,9	22	34	10	67
p,p'-DDD	ng/ml	31	35	41	37	38	9.1	93	18	44	47	25	66
o,p'-DDD	ng/ml	31	36	38	37	37	25	75	10	25	35	11	71
Sum DDTs LB (ND = 0)	**ng/ml**	**184**	**183**	**183**	**191**	**170**	**59**	**389**	**67**	**36**	**45**	30	72
Sum DDTs UB (ND = LOD)	**ng/ml**	**184**	**208**	**204**	**210**	**194**	**82**	**389**	**64.1**	**31**	**31**	17	67
Mirex	ng/µm	31	36	43	37	39	28	202	31.9	73	28	8	70
Hexachlorobenzene	ng/ml	31	36	39	36	37	21	90	12.3	32	41	15	70

Table 3: Summary of laboratory performance OCPs, standard solution

Standard				% of the data re-ceived	% of Zscores |Z|<2	% of Zscores 3>|Z|>2	% of Zscores 6>|Z|>3	% of Zscores |Z|>6
Contaminant	**Unit**	**Target**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
Aldrin	ng/ml	31	35	57	83	4	6	4
Dieldrin	ng/ml	31	36	60	76	14	6	4
Endrin	ng/ml	31	37	51	69	14	14	2
Sum Drins LB (ND = 0)	**ng/ml**	**92**	**104**	**58**	**73**	**17**	**6**	**4**
Sum Drins UB (ND = LOD)	**ng/ml**	**92**	**107**	**49**	**78**	**15**	**2**	**5**
trans-Chlordane	ng/ml	31	35	45	76	8	8	8
cis-Chlordane	ng/ml	31	35	45	81	8	8	3
trans-Nonachlor	ng/ml	31	35	31	77	15	0	8
cis-Nonachlor	ng/ml	31	34	24	80	0	10	5
Oxychlordane	ng/ml	31	34	28	87	4	4	4
Heptachlor	ng/ml	31	35	55	74	9	9	2
cis-Heptachlorepoxide	ng/ml	31	34	42	83	9	3	3
trans-Heptachlorepoxide	ng/ml	30	35	27	91	0	5	5
Sum Chlordane LB (ND = 0)	**ng/ml**	**245**	**NA**	**51**	**NA**	**NA**	**NA**	**NA**
Sum Chlordane UB (ND = LOD)	**ng/ml**	**245**	**283**	**27**	**77**	**9**	**9**	**5**
p,p'-DDT	ng/ml	31	36	55	72	4	17	7
o,p'-DDT	ng/ml	31	35	39	78	6	9	3
p,p'-DDE	ng/ml	31	35	61	76	12	6	6
o,p'-DDE	ng/ml	31	35	42	86	3	6	3
p,p'-DDD	ng/ml	31	35	58	63	6	17	13
o,p'-DDD	ng/ml	31	36	42	89	6	0	6
Sum DDTs LB (ND = 0)	**ng/ml**	**184**	**183**	**54**	**56**	**20**	**20**	**4**
Sum DDTs UB (ND = LOD)	**ng/ml**	**184**	**208**	**37**	**68**	**10**	**19**	**3**
Mirex	ng/ml	31	36	34	93	0	0	7
Hexachlorobenzene	ng/ml	31	36	49	78	10	7	5

Table 4: Summary results OCPs, sediment

Sediment

Contaminant	Unit	Assigned	Mean	Median	Min	Max	SD	%RSD	n	Between Lab CV (%)	Inclusion rate (%)
Aldrin	µg/kg	0.018	1.37	0.041	0.004	16.8	4.28	313	15	250	50
Dieldrin	µg/kg	NA	6.49	2.05	0.009	42.5	11.3	174	16	216	44
Endrin	µg/kg	NA	1.02	0.513	0.007	6.56	1.99	194	10	178	56
Sum Drins LB (ND = 0)	**µg/kg**	**NA**	**6.31**	**0.159**	**0**	**59.3**	**17.7**	**280**	**11**	103	58
Sum Drins UB (ND = LOD)	**µg/kg**	**NA**	**4.40**	**0.300**	**0.023**	**59.3**	**13.2**	**299**	**21**	120	61
trans-Chlordane	µg/kg	NA	0.962	0.070	0.020	12.2	3.02	314	16	109	62
cis-Chlordane	µg/kg	NA	0.148	0.091	0.038	352	0.122	82	10	109	56
trans-Nonachlor	µg/kg	0.033	0.340	0.037	0.001	3.09	0.872	256	12	107	60
cis-Nonachlor	µg/kg	NA	0.053	0.013	0.011	0.157	0.065	122	6	14	63
Oxychlordane	µg/kg	NA	0.816	0.344	0.001	2.10	1.13	138	3	451	36
Heptachlor	µg/kg	NA	2.19	0.206	0.006	17.3	5.40	246	10	258	46
cis-Heptachlorepoxide	µg/kg	0.012	0.281	0.013	0.011	1.60	0.590	210	7	22	50
trans-Heptachlorepoxide	µg/kg	NA	1.82	1.37	0.698	3.40	1.41	77	3	348	39
Sum Chlordane LB (ND = 0)	**µg/kg**	**NA**	**1.53**	**0.366**	**0.054**	**12.2**	**3.19**	**209**	**14**	123	63
Sum Chlordane UB (ND = LOD)	**µg/kg**	**NA**	**2.63**	**0.631**	**0.163**	**19.8**	**5.49**	**209**	**20**	99	70
p,p'-DDT	µg/kg	18.1	18.0	18.2	0.010	71.7	13.6	76	32	48	66
o,p'-DDT	µg/kg	NA	7.06	3.67	0.061	42.2	10.8	153	22	78	63
p,p'-DDE	µg/kg	15.4	26.0	16.4	0.093	464	73.3	282	38	44	74
o,p'-DDE	µg/kg	1.52	2.30	1.62	0.002	9.79	2.46	107	23	32	55
p,p'-DDD	µg/kg	32.0	30.5	33.1	0.008	64.7	17.5	57	36	59	76
o,p'-DDD	µg/kg	21.4	19.2	21.0	0.015	32.6	8.44	44	25	33	67
Sum DDTs LB (ND = 0)	**µg/kg**	**96.6**	**102**	**95.4**	**0.885**	**479**	**82.2**	**80**	**27**	26	68
Sum DDTs UB (ND = LOD)	**µg/kg**	**99.0**	**101**	**96.7**	**0.600**	**479**	**85.6**	**85**	**26**	26	67
Mirex	µg/kg	0.013	0.881	0.015	0.010	5.36	1.70	193	10	53	58
Hexachlorobenzene	µg/kg	33.9	34.4	31.9	0.027	108	22.1	64	30	59	76

Table 5: Summary of laboratory performance OCPs, sediment

Sediment			% of the	% of Zscores	% of Zscores	% of Zscores	% of Zscores
			data received	|Z|<2	3>|Z|>2	6>|Z|>3	|Z|>6
Contaminant	**Unit**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
% Lipids	%	NA	NA	NA	NA	NA	NA
Aldrin	ng/kg	18	31	19	4	0	35
Dieldrin	ng/kg	NA	35	NA	NA	NA	NA
Endrin	ng/kg	NA	25	NA	NA	NA	NA
Sum Drins LB (ND = 0)	**ng/kg**	**NA**	**13**	**NA**	**NA**	**NA**	**NA**
Sum Drins UB (ND = LOD)	**ng/kg**	**NA**	**27**	**NA**	**NA**	**NA**	**NA**
trans-Chlordane	ng/kg	NA	30	NA	NA	NA	NA
cis-Chlordane	ng/kg	NA	25	NA	NA	NA	NA
trans-Nonachlor	ng/kg	33	19	31	6	0	38
cis-Nonachlor	ng/kg	NA	13	NA	NA	NA	NA
Oxychlordane	ng/kg	NA	16	NA	NA	NA	NA
Heptachlor	ng/kg	NA	30	NA	NA	NA	NA
cis-Heptachlorepoxide	ng/kg	12	20	24	0	0	18
trans-Heptachlorepoxide	ng/kg	NA	14	NA	NA	NA	NA
Sum Chlordane LB (ND = 0)	**ng/kg**	**NA**	**17**	**NA**	**NA**	**NA**	**NA**
Sum Chlordane UB (ND = LOD)	**ng/kg**	**NA**	**25**	**NA**	**NA**	**NA**	**NA**
p,p'-DDT	ng/kg	18 093	40	39	12	15	30
o,p'-DDT	ng/kg	NA	30	NA	NA	NA	NA
p,p'-DDE	ng/kg	15 445	47	38	21	23	15
o,p'-DDE	ng/kg	1 521	35	48	0	14	17
p,p'-DDD	ng/kg	32 023	45	32	14	32	19
o,p'-DDD	ng/kg	21 380	33	59	4	22	7
Sum DDTs LB (ND = 0)	**ng/kg**	**96 611**	**33**	**59**	**15**	**15**	**11**
Sum DDTs UB (ND = LOD)	**ng/kg**	**99 034**	**31**	**58**	**15**	**12**	**15**
Mirex	ng/kg	13	18	33	7	0.0	27
Hexachlorobenzene	ng/kg	33 914	37	42	10	26	19

Table 6: Summary results OCPs, fish (lipid weight basis)

Fish												
Contaminant	**Unit**	**Assigned**	**Mean**	**Median**	**Geom ean**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
% Lipids	%	4.9	5.0	5.2	1.7	0.2	20	4.31	85	36	54	66
Aldrin	µg/kg	NA	0.070	0.015	0.082	0.00001	0.717	0.162	231	18	207	51
Dieldrin	µg/kg	NA	0.371	0.338	0.127	0.00001	1.33	0.314	85	28	76	67
Endrin	µg/kg	NA	0.105	0.014	0.019	0.00001	0.677	0.189	179	15	132	57
Sum Drins LB (ND = 0)	**µg/kg**	**0.316**	**0.413**	**0.343**	**0.141**	**0.00001**	**1.48**	**0.367**	**89**	**28**	103	77
Sum Drins UB (ND = LOD)	**µg/kg**	**0.324**	**0.417**	**0.343**	**0.291**	**0.036**	**1.10**	**0.310**	**74**	**20**	35	54
trans-Chlordane	µg/kg	NA	0.154	0.099	0.075	0.00001	0.484	0.126	82	24	99	77
cis-Chlordane	µg/kg	0.247	0.293	0.287	0.121	0.00001	1.12	0.274	93	25	55	68
trans-Nonachlor	µg/kg	0.956	2.65	1.06	0.365	0.00001	35.3	7.34	277	21	94	79
cis-Nonachlor	µg/kg	NA	0.560	0.497	0.194	0.00001	2.28	0.521	93	16	71	75
Oxychlordane	µg/kg	0.095	0.095	0.096	0.061	0,63	0.211	0.053	56	14	40	62
Heptachlor	µg/kg	NA	0.083	0.034	0.010	0.00001	0.332	0.105	127	10	221	43
cis-Heptachlorepoxide	µg/kg	0.029	0.062	0.035	0.032	0.02	0.236	0.057	91	20	66	57
trans-Heptachlorepoxide	µg/kg	NA	0.063	0.07	0.068	0.35	0.301	0.108	171	10	240	62
Sum Chlordane LB (ND = 0)	**µg/kg**	**NA**	**2.86**	**1.56**	**0.670**	**0.07**	**36.7**	**6.90**	**241**	**26**	124	80
Sum Chlordane UB (ND = LOD)	**µg/kg**	**1.77**	**4.12**	**2.10**	**1.63**	**35**	**36.7**	**8.26**	**200**	**17**	81	73
p,p'-DDT	µg/kg	NA	1.21	0.884	0.467	1.25	5.00	1.25	103	28	107	79
o,p'-DDT	µg/kg	0.200	0.345	0.222	0.147	0.00001	1.08	0.289	84	19	69	55
p,p'-DDE	µg/kg	8.88	11.3	9.77	2.84	0.00001	47.0	10.2	91	34	80	73
o,p'-DDE	µg/kg	NA	0.059	0.037	0.025	0.00001	0.243	0.065	110	20	62	56
p,p'-DDD	µg/kg	0.723	0.866	0.750	0.336	0.00001	4.00	0.761	88	31	64	71
o,p'-DDD	µg/kg	0.040	0.091	0.045	0.033	0.00001	0.635	0.141	155	17	58	52
Sum DDTs LB (ND = 0)	**µg/kg**	**NA**	**11.6**	**11.3**	**3.61**	**0.05**	**42.3**	**9.32**	**81**	**32**	87	78
Sum DDTs UB (ND = LOD)	**µg/kg**	**12.3**	**14.2**	**13.0**	**8.55**	**0.077**	**42.3**	**9.13**	**64**	**21**	59	74
Mirex	µg/kg	3.16	2.94	3.20	0.940	0.00001	5.95	1.98	67	20	64	74
Hexachlorobenzene	µg/kg	NA	0.049	0.047	0.023	0.00001	0.130	0.037	76	28	99	83

Table 7: Summary of laboratory performance OCPs, fish

Fish			% of the	% of Zscores	% of Zscores	% of Zscores	% of Zscores
			data received	|Z|<2	3>|Z|>2	6>|Z|>3	|Z|>6
Contaminant	**Unit**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
% Lipids	%	4.9%	40	48	3	9	39
Aldrin	ng/kg	NA	30	NA	NA	NA	NA
Dieldrin	ng/kg	NA	36	NA	NA	NA	NA
Endrin	ng/kg	NA	24	NA	NA	NA	NA
Sum Drins LB (ND = 0)	**ng/kg**	**316**	**34**	**36**	**4**	**11**	**50**
Sum Drins UB (ND = LOD)	**ng/kg**	**324**	**25**	**48**	**5**	**10**	**33**
trans-Chlordane	ng/kg	NA	29	NA	NA	NA	NA
cis-Chlordane	ng/kg	247	30	36	16	16	32
trans-Nonachlor	ng/kg	956	25	33	10	10	48
cis-Nonachlor	ng/kg	NA	19	NA	NA	NA	NA
Oxychlordane	ng/kg	95	18	33	33	7	20
Heptachlor	ng/kg	NA	22	NA	NA	NA	NA
cis-Heptachlorepoxide	ng/kg	29	27	36	5	14	36
trans-Heptachlorepoxide	ng/kg	NA	13	NA	NA	NA	NA
Sum Chlordane LB (ND = 0)	**ng/kg**	**NA**	**34**	**NA**	**NA**	**NA**	**NA**
Sum Chlordane UB (ND = LOD)	**ng/kg**	**1 770**	**22**	**39**	**0**	**17**	**44**
p,p'-DDT	ng/kg	NA	34	NA	NA	NA	NA
o,p'-DDT	ng/kg	200	27	36	5	14	32
p,p'-DDE	ng/kg	8884	41	35	6	15	44
o,p'-DDE	ng/kg	NA	28	NA	NA	NA	NA
p,p'-DDD	ng/kg	723	37	39	6	19	35
o,p'-DDD	ng/kg	40	24	35	5	10	35
Sum DDTs LB (ND = 0)	**ng/kg**	**NA**	**41**	**NA**	**NA**	**NA**	**NA**
Sum DDTs UB (ND = LOD)	**ng/kg**	**12 268**	**28**	**43**	**4**	**22**	**30**
Mirex	ng/kg	3 156	24	40	10	5	45
Hexachlorobenzene	ng/kg	NA	34	NA	NA	NA	NA

Table 8: Summary results OCPs, milk

Milk												
Contaminant	**Unit**	**Assigned**	**Mean**	**Median**	**Geomean**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
% Lipids	%	2.6%	2.4%	2.4%	1.2%	0.02%	14%	2.58	107	29	30	64
Aldrin	ng/kg	NA	12	0.81	0.97	0.06	41	18	153	4	332	35
Dieldrin	ng/kg	1.7	3.0	1.9	1.9	0.99	20	4.9	163	11	34	66
Endrin	ng/kg	NA	5.8	1.3	1.3	0.02	20	8.3	142	4	272	37
Sum Drins LB (ND = 0)	**ng/kg**	**1.9**	**9.4**	**2.0**	**2.8**	**0.06**	**61**	**17**	**178**	**15**	50	66
Sum Drins UB (ND = LOD)	**ng/kg**	**2.1**	**12**	**2.4**	**4.3**	**0.06**	**61**	**18**	**148**	**20**	29	61
trans-Chlordane	ng/kg	NA	4.0	0.78	0.51	0.01	32	9.1	230	10	203	65
cis-Chlordane	ng/kg	NA	2.0	0.95	0.70	0.02	10	2.8	143	10	145	64
trans-Nonachlor	ng/kg	4.2	4.5	4.5	2.9	0.03	12	3.1	70	10	63	74
cis-Nonachlor	ng/kg	0.94	1.7	1.0	1.1	0.58	9.0	2.6	147	8	38	74
Oxychlordane	ng/kg	2.8	4.4	2.8	3.1	1.6	23	6.1	139	8	14	50
Heptachlor	ng/kg	NA	28	2.3	3.1	0.04	127	47	166	7	224	40
cis-Heptachlorepoxide	ng/kg	1.1	3	1.3	1.8	0.33	11	3.7	119	9	50	58
trans-Heptachlorepoxide	ng/kg	NA	13	13	6.8	2.0	23	15	119	1	132	38
Sum Chlordane LB (ND = 0)	**ng/kg**	**NA**	**27**	**10**	**12**	**1.0**	**143**	**40**	**149**	**16**	85	69
Sum Chlordane UB (ND = LOD)	**ng/kg**	**NA**	**28**	**11**	**14**	**1.0**	**143**	**38**	**137**	**18**	93	71
p,p'-DDT	ng/kg	2.7	26	3.1	5,2	0.44	302	71	273	13	40	56
o,p'-DDT	ng/kg	0.5	29	0.37	0.80	0.23	284	90	311	8	34	60
p,p'-DDE	ng/kg	NA	50	49	25	0.34	228	48	95	20	64	72
o,p'-DDE	ng/kg	0.08	0.34	0.09	0.15	0.07	2.2	0,7	197	7	38	62
p,p'-DDD	ng/kg	0.12	4.1	0.17	0,44	0.05	25	8.2	198	10	121	55
o,p'-DDD	ng/kg	NA	8.6	1.4	2,0	0.16	33	13	154	5	253	38
Sum DDTs LB (ND = 0)	**ng/kg**	**56**	**83**	**60**	**38**	**0.48**	**405**	**100**	**120**	**21**	40	68
Sum DDTs UB (ND = LOD)	**ng/kg**	**57**	**90**	**61**	**49**	**0.48**	**405**	**99**	**110**	**21**	45	72
Mirex	ng/kg	0.29	18	0.31	0,5	0.05	227	63	340	9	31	57
Hexachlorobenzene	ng/kg	7.9	8.7	8.0	6.5	0.47	29	6.5	74	11	41	65

Table 9: Summary of laboratory performance OCPs, milk (lipid weight basis)

Milk			% of the	% of Zscores	% of Zscores	% of Zscores	% of Zscores
			data received	|Z|<2	3>|Z|>2	6>|Z|>3	|Z|>6
Contaminant	**Unit**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
% Lipids	%	2.6%	31	65	4	8	23
Aldrin	ng/kg	NA	19	NA	NA	NA	NA
Dieldrin	ng/kg	1.7	24	40	10	15	10
Endrin	ng/kg	NA	19	NA	NA	NA	NA
Sum Drins LB (ND = 0)	**ng/kg**	**1.9**	**20**	**47**	**6**	**18**	**29**
Sum Drins UB (ND = LOD)	**ng/kg**	**2.1**	**23**	**53**	**11**	**0**	**32**
trans-Chlordane	ng/kg	NA	19	NA	NA	NA	NA
cis-Chlordane	ng/kg	NA	19	NA	NA	NA	NA
trans-Nonachlor	ng/kg	4.2	17	43	7	29	21
cis-Nonachlor	ng/kg	0.94	13	64	18	0	18
Oxychlordane	ng/kg	2.8	17	50	14	7	14
Heptachlor	ng/kg	NA	19	NA	NA	NA	NA
cis-Heptachlorepoxide	ng/kg	1.1	19	44	0	13	31
trans-Heptachlorepoxide	ng/kg	NA	8	NA	NA	NA	NA
Sum Chlordane LB (ND = 0)	**ng/kg**	**NA**	**22**	**NA**	**NA**	**NA**	**NA**
Sum Chlordane UB (ND = LOD)	**ng/kg**	**NA**	**23**	**NA**	**NA**	**NA**	**NA**
p,p'-DDT	ng/kg	2.7	25	43	10	5	33
o,p'-DDT	ng/kg	0.35	19	38	6	6	19
p,p'-DDE	ng/kg	NA	31	NA	NA	NA	NA
o,p'-DDE	ng/kg	0.08	18	47	0	0	27
p,p'-DDD	ng/kg	0.12	28	26	17	0	30
o,p'-DDD	ng/kg	NA	17	NA	NA	NA	NA
Sum DDTs LB (ND = 0)	**ng/kg**	**56**	**24**	**45**	**15**	**15**	**25**
Sum DDTs UB (ND = LOD)	**ng/kg**	**57**	**24**	**45**	**15**	**15**	**25**
Mirex	ng/kg	0.29	20	41	12	12	18
Hexachlorobenzene	ng/kg	7.9	24	35	20	15	15

3.2 Polychlorinated Biphenyls (PCB)

Table 10: Summary results marker PCB, standard

Standard												
Contaminant	**Unit**	**Target**	**Assigned**	**Mean**	**Median**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
PCB #28	ng/µl	1.25	1.15	1.17	1.15	0.57	2.11	0.28	24	42	19	75
PCB #52	ng/µl	1.25	1.20	1.20	1.20	0.52	2.23	0.28	23	42	13	69
PCB #101	ng/µl	1.25	1.17	1.18	1.17	0.59	2.15	0.25	21	42	14	67
PCB #138	ng/µl	1.25	1.16	1.22	1.19	0.37	3.82	0.52	43	40	14	67
PCB #153	ng/µl	1.25	1.21	1.23	1.21	0.83	2.19	0.23	19	42	12	67
PCB #180	ng/µl	1.25	1.13	1.21	1.17	0.71	3.49	0.43	36	41	16	73
Sum Marker PCB LB (ND = 0)	**ng/µl**	**8.75**	**7.17**	**6.68**	**7.10**	**0.01**	**9.39**	**1.88**	**28**	**35**	8	64
Sum Marker PCB UB (ND = LOD)	**ng/µl**	**8.75**	**7.22**	**6.74**	**7.11**	**1.00**	**9.39**	**1.78**	**26**	**35**	8	64

Table 11: Summary of laboratory performance marker PCB, standard

Standard				% of the	% of Zscores	% of Zscores	% of Zscores	% of Zscores
				data received	|Z|<2	3>|Z|>2	6>|Z|>3	|Z|>6
Contaminant	**Unit**	**Target**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
% Lipids	%	NA	NA	NA	NA	NA	NA	NA
PCB #28	ng/ul	1.25	1.15	51	83	7	7	2
PCB #52	ng/ul	1.25	1.20	52	86	7	5	2
PCB #101	ng/ul	1.25	1.17	53	86	7	2	2
PCB #118	ng/ul	1.25	NA	27	NA	NA	NA	NA
PCB #138	ng/ul	1.25	1.16	51	81	10	2	5
PCB #153	ng/ul	1.25	1.21	52	88	9	0	2
PCB #180	ng/ul	1.25	1.13	52	86	5	2	5
Sum Marker PCB LB (ND = 0)	**ng/ul**	**8.75**	**7.17**	**43**	**86**	**6**	**3**	**6**
Sum Marker PCB UB (ND = LOD)	**ng/ul**	**8.75**	**7.22**	**43**	**86**	**6**	**3**	**6**

Table 12: Summary results marker PCB, ash

Ash											
Contaminant	**Unit**	**Assigned**	**Mean**	**Median**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
PCB #28	µg/kg	NA	1.33	0.160	0.006	6.68	2.29	172	11	191	59
PCB #52	µg/kg	NA	0.348	0.146	0.024	2.16	0.604	173	12	130	73
PCB #101	µg/kg	0.131	0.238	0.156	0.039	0.700	0.223	94	11	82	71
PCB #118	µg/kg	0.170	**0.331**	**0.177**	**0.083**	**2.06**	**0.549**	**166**	**12**	**49**	**77**
PCB #138	µg/kg	NA	**0.294**	**0.171**	**0.052**	**1.00**	**NA**	**NA**	**10**	**105**	**69**
PCB #153	µg/kg	NA	0.231	0.122	0.010	1.00	0.298	129	10	108	69
PCB #180	µg/kg	0.183	0.905	0.203	0.160	7.47	2.08	229	12	25	56
Sum Marker PCB LB (ND = 0)	**µg/kg**	**NA**	**2.66**	**1.38**	**0.450**	**8.27**	**2.48**	**93**	**11**	**94**	**63**
Sum Marker PCB UB (ND = LOD)	**µg/kg**	**NA**	**9.20**	**1.60**	**0.450**	**86.4**	**22.4**	**244**	**14**	**110**	**60**

Table 13: Summary of laboratory performance marker PCB, ash

Ash			% of the data received	% of Zscores |Z|<2	% of Zscores 3>|Z|>2	% of Zscores 6>|Z|>3	% of Zscores |Z|>6
Contaminant	**Unit**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
% Lipids	%	NA	NA	NA	NA	NA	NA
PCB #28	ng/kg	NA	13	NA	NA	NA	NA
PCB #52	ng/kg	NA	16	NA	NA	NA	NA
PCB #101	ng/kg	131	16	31	8	23	23
PCB #118	ng/kg	170	17	36	14	29	7
PCB #138	ng/kg	NA	14	NA	NA	NA	NA
PCB #153	ng/kg	NA	14	NA	NA	NA	NA
PCB #180	ng/kg	183	17	50	0	0	36
Sum Marker PCB LB (ND = 0)	**ng/kg**	**NA**	**13**	**NA**	**NA**	**NA**	**NA**
Sum Marker PCB UB (ND = LOD)	**ng/kg**	**NA**	**17**	**NA**	**NA**	**NA**	**NA**

Table 14: Summary results marker PCB, sediment

Sediment											
Contaminant	**Unit**	**Assigned**	**Mean**	**Median**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
PCB #28	µg/kg	4.22	4.23	4.20	0.077	9.06	2.20	52	25	53	77
PCB #52	µg/kg	6.22	5.93	6.39	0.335	10.2	2.38	40	27	32	73
PCB #101	µg/kg	NA	6.07	6.56	0.006	19.9	3.78	62	31	52	75
PCB #118	µg/kg	3.28	4.44	3.31	0.178	16.9	3.74	84	26	31	69
PCB #138	µg/kg	NA	10.7	10.7	0.899	21.0	5.58	52	27	59	79
PCB #153	µg/kg	12.9	12.4	12.7	0.006	28.8	7.01	57	30	38	64
PCB #180	µg/kg	8.87	8.34	9.24	0.175	14.0	3.95	47	29	46	77
Sum Marker PCB LB (ND = 0)	**µg/kg**	**52.3**	**50.8**	**55.3**	**2.37**	**93.6**	**21.3**	**42**	**27**	**39**	**74**
Sum Marker PCB UB (ND = LOD)	**µg/kg**	**52.6**	**51.2**	**55.4**	**2.37**	**93.6**	**21.3**	**42**	**26**	**38**	**74**

Table 15: Summary of laboratory performance marker PCB, sediment

Sediment			% of the data received	% of Zscores |Z|<2	% of Zscores 3>|Z|>2	% of Zscores 6>|Z|>3	% of Zscores |Z|>6
Contaminant	**Unit**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
% Lipids	%	NA	NA	NA	NA	NA	NA
PCB #28	ng/kg	4 222	30	44	12	32	12
PCB #52	ng/kg	6 224	33	59	15	19	7
PCB #101	ng/kg	NA	39	NA	NA	NA	NA
PCB #118	ng/kg	3 284	33	59	4	15	19
PCB #138	ng/kg	NA	34	NA	NA	NA	NA
PCB #153	ng/kg	12 851	36	57	3	17	23
PCB #180	ng/kg	8 869	36	43	17	27	10
Sum Marker PCB LB (ND = 0)	**ng/kg**	**52 275**	**33**	**52**	**11**	**30**	**7**
Sum Marker PCB UB (ND = LOD)	**ng/kg**	**52 551**	**31**	**58**	**8**	**27**	**8**

Table 16: Summary results marker PCB, fish (lipid weight basis)

Fish											
Contaminant	**Unit**	**Assigned**	**Mean**	**Median**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
PCB #28	µg/kg	NA	232	114	3.520	3380	601	258	30	68	71
PCB #52	µg/kg	372	362	381	10.1	737	197	54	32	48	71
PCB #101	µg/kg	NA	2000	1580	41.7	20000	3.280	164	34	64	77
PCB #118	µg/kg	NA	3440	1520	55.4	55100	9870	287	30	113	78
PCB #138	µg/kg	3000	9650	3200	61.5	201300	35600	369	31	65	74
PCB #153	µg/kg	NA	6780	3970	7.80	112700	18900	279	34	93	79
PCB #180	µg/kg	2010	2710	2290	57.7	19900	3430	127	32	53	66
Sum Marker PCB LB (ND = 0)	**µg/kg**	**12200**	**12200**	**12600**	**290**	**34100**	**7990**	**66**	**33**	**47**	**66**
Sum Marker PCB UB (ND = LOD)	**µg/kg**	**13200**	**13900**	**13300**	**346**	**34100**	**7850**	**57**	**29**	**38**	**63**

Table 17: Summary of laboratory performance marker PCB, fish

Fish			% of the data received	% of Zscores |Z|<2	% of Zscores 3>|Z|>2	% of Zscores 6>|Z|>3	% of Zscores |Z|>6
Contaminant	**Unit**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
% Lipids	%	NA	NA	NA	NA	NA	NA
PCB #28	ng/kg	NA	39	NA	NA	NA	NA
PCB #52	ng/kg	371 820	40	42	18	12	27
PCB #101	ng/kg	NA	42	NA	NA	NA	NA
PCB #118	ng/kg	NA	37	NA	NA	NA	NA
PCB #138	ng/kg	2 999 317	39	44	0	19	38
PCB #153	ng/kg	NA	42	NA	NA	NA	NA
PCB #180	ng/kg	2 009 904	40	42	12	12	33
Sum Marker PCB LB (ND = 0)	**ng/kg**	**12 184 302**	**40**	**42**	**15**	**6**	**36**
Sum Marker PCB UB (ND = LOD)	**ng/kg**	**13 247 498**	**35**	**52**	**7**	**10**	**31**

Table 18: Summary results marker PCB, milk (lipid weight basis)

Milk											
Contaminant	**Unit**	**Assigned**	**Mean**	**Median**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
PCB #28	µg/kg	1.37	16.9	1.48	0.170	248	58.1	345	18	43	60
PCB #52	µg/kg	NA	9.14	0.546	0.240	92.7	22.0	241	18	95	57
PCB #101	µg/kg	NA	3.84	0.935	0.080	29.2	7.33	191	20	117	62
PCB #118	µg/kg	4.85	12.4	5.05	0.030	124	25.8	207	22	46	62
PCB #138	µg/kg	20.1	19.4	20.2	2.02	28.3	6.05	31	21	26	72
PCB #153	µg/kg	33.8	29.1	32.7	0.170	48.1	14.6	50	23	36	69
PCB #180	µg/kg	20.2	19.1	20.2	0.030	39.8	9.03	47	24	32	68
Sum Marker PCB LB (ND = 0)	**µg/kg**	**82.2**	**97.3**	**78.1**	**0.010**	**432**	**80.3**	**83**	**23**	**23**	**65**
Sum Marker PCB UB (ND = LOD)	**µg/kg**	**83.3**	**96.1**	**83.5**	**1.00**	**432**	**82.1**	**85**	**23**	**16**	**62**

Table 19: Summary of laboratory performance marker PCB, milk

Milk			% of the data received	% of Zscores |Z|<2	% of Zscores 3>|Z|>2	% of Zscores 6>|Z|>3	% of Zscores |Z|>6
Contaminant	**Unit**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
% Lipids	%	NA	NA	NA	NA	NA	NA
PCB #28	ng/kg	1 373	25	38	10	10	29
PCB #52	ng/kg	NA	27	NA	NA	NA	NA
PCB #101	ng/kg	NA	28	NA	NA	NA	NA
PCB #118	ng/kg	4 852	**28**	**39**	**13**	**17**	**26**
PCB #138	ng/kg	20 066	**27**	**55**	**27**	**9**	**5**
PCB #153	ng/kg	33 841	29	50	21	13	13
PCB #180	ng/kg	20 243	30	60	4.0	16	16
Sum Marker PCB LB (ND = 0)	**ng/kg**	**82 174**	**28**	**65**	**4**	**13**	**17**
Sum Marker PCB UB (ND = LOD)	**ng/kg**	**83 331**	**25**	**67**	**10**	**5**	**19**

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

Table 20: Summary results dl-POPs, standard

Standard												
Contaminant	**Unit**	**Target**	**Assigned**	**Mean**	**Median**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
2,3,7,8-TeCDD	pg/µl	30	29	29	30	24	34	2.7	9,4	37	11	81
1,2,3,7,8-PeCDD	pg/µl	60	58	58	58	39	93	8.1	14	37	8	73
1,2,3,4,7,8-HxCDD	pg/µl	60	55	54	55	28	67	6.3	12	37	8	72
1,2,3,6,7,8-HxCDD	pg/µl	60	56	57	57	49	69	5.0	9	37	9	78
1,2,3,7,8,9-HxCDD	pg/µl	60	56	56	56	29	69	7.1	13	37	11	77
1,2,3,4,6,7,8-HpCDD	pg/µl	120	119	119	119	79	145	13	11	37	9	74
OCDD	pg/µl	120	126	125	126	85	153	14	11	37	9	73
2,3,7,8-TeCDF	pg/µl	30	31	31	31	25	38	2.8	9	37	6	63
1,2,3,7,8-PeCDF	pg/µl	60	62	62	61	42	86	7.5	12	37	10	75
2,3,4,7,8-PeCDF	pg/µl	60	59	60	58	47	120	12	20	37	13	80
1,2,3,4,7,8-HxCDF	pg/µl	60	59	59	59	41	70	6.5	11	37	10	74
1,2,3,6,7,8-HxCDF	pg/µl	60	59	60	59	39	77	7.1	12	37	9	73
1,2,3,7,8,9-HxCDF	pg/µl	60	57	58	57	45	67	4.7	8	37	6	68
2,3,4,6,7,8-HxCDF	pg/µl	60	57	56	57	35	67	5.7	10	37	7	71
1,2,3,4,6,7,8-HpCDF	pg/µl	120	121	121	121	91	170	14	12	37	10	78
1,2,3,4,7,8,9-HpCDF	pg/µl	120	118	120	120	83	216	22	19	37	12	75
OCDF	pg/µl	120	119	120	120	85	161	16	13	37	12	78
TEQ (PCDD/PCDF) LB (ND=0)	**pg TEQ/µl**	**172**	**166**	**167**	**167**	**137**	**232**	**17**	**10**	**37**	9	78
TEQ (PCDD/PCDF) UB (ND=LOD)	**pg TEQ/µl**	**172**	**166**	**167**	**167**	**137**	**233**	**17**	**10**	**37**	9	78
PCB #77	pg/µl	40	37	38	37	11	76	11	28	31	21	78
PCB #81	pg/µl	40	39	39	40	13	91	12	31	31	22	79
PCB #126	pg/µl	40	38	40	39	28	96	12	29	31	16	77
PCB #169	pg/µl	40	39	41	40	28	79	9.5	23	31	16	76
PCB #105	pg/µl	40	39	40	40	12	86	11	28	31	14	74
PCB #114	pg/µl	40	40	40	40	21	88	11	26	32	15	74
PCB #118	pg/µl	40	39	40	39	23	87	10	25	33	12	73
PCB #123	pg/µl	40	40	41	40	30	94	11	27	31	13	74
PCB #156	pg/µl	40	39	40	39	21	95	12	29	33	17	79
PCB #157	pg/µl	40	40	41	40	26	82	9.7	24	32	13	70
PCB #167	pg/µl	40	39	37	38	13	50	7.9	21	31	14	72
PCB #189	pg/µl	40	36	38	37	23	98	12	32	32	16	77
TEQ (PCB) LB (ND=0)	**pg TEQ/µl**	**4.5**	**4.3**	**4.4**	**4.4**	**0.02**	**10.6**	**1.5**	**34**	**32**	16	76
TEQ (PCB) UB (ND=LOD)	**pg TEQ/µl**	**4.5**	**4.3**	**4.5**	**4.4**	**3.2**	**10.6**	**1.3**	**28**	**31**	15	77
TEQ Total LB (ND=0)	**pg TEQ/µl**	**176**	**168**	**171**	**168**	**140**	**237**	**18**	**11**	**31**	9	78
TEQ Total UB (ND=LOD)	**pg TEQ/µl**	**176**	**167**	**170**	**168**	**140**	**237**	**18**	**11**	**30**	9	78

Table 21: Summary of laboratory performance dl-POPs, standard

Standard				% of the data received	% of Zscores |Z|<2	% of Zscores 3>|Z|>2	% of Zscores 6>|Z|>3	% of Zscores |Z|>6
Contaminant	**Unit**	**Target**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
2,3,7,8-TeCDD	pg/ul	30	29	74	100	0	0	0
1,2,3,7,8-PeCDD	pg/ul	60	58	74	95	3	3	0
1,2,3,4,7,8-HxCDD	pg/ul	60	55	74	97	0	3	0
1,2,3,6,7,8-HxCDD	pg/ul	60	56	74	100	0	0	0
1,2,3,7,8,9-HxCDD	pg/ul	60	56	74	97	0	3	0
1,2,3,4,6,7,8-HpCDD	pg/ul	120	119	74	97	3	0	0
OCDD	pg/ul	120	126	74	95	5	0	0
2,3,7,8-TeCDF	pg/ul	30	31	74	100	0	0	0
1,2,3,7,8-PeCDF	pg/ul	60	62	74	95	3	3	0
2,3,4,7,8-PeCDF	pg/ul	60	59	74	97	0	0	3
1,2,3,4,7,8-HxCDF	pg/ul	60	59	74	97	3	0	0
1,2,3,6,7,8-HxCDF	pg/ul	60	59	74	92	8	0	0
1,2,3,7,8,9-HxCDF	pg/ul	60	57	74	100	0	0	0
2,3,4,6,7,8-HxCDF	pg/ul	60	57	74	97	0	3	0
1,2,3,4,6,7,8-HpCDF	pg/ul	120	121	74	97	0	3	0
1,2,3,4,7,8,9-HpCDF	pg/ul	120	118	74	89	5	3	3
OCDF	pg/ul	120	119	74	89	11	0	0
TEQ (PCDD/PCDF) LB (ND=0)	**pg TEQ/ul**	**172**	**166**	**74**	**97**	**0**	**3**	**0**
TEQ (PCDD/PCDF) UB (ND=LOD)	**pg TEQ/ul**	**172**	**166**	**74**	**97**	**0**	**3**	**0**
PCB #77	pg/ul	40	37	62	81	10	6	3
PCB #81	pg/ul	40	39	62	81	13	3	3
PCB #126	pg/ul	40	38	62	87	10	0	3
PCB #169	pg/ul	40	39	62	84	10	3	3
PCB #105	pg/ul	40	39	62	87	6	3	3
PCB #114	pg/ul	40	40	64	88	6	3	3
PCB #118	pg/ul	40	39	66	88	6	3	3
PCB #123	pg/ul	40	40	62	90	6	0	3
PCB #156	pg/ul	40	39	66	88	6	3	3
PCB #157	pg/ul	40	40	64	84	13	0	3
PCB #167	pg/ul	40	39	62	84	10	6	0
PCB #189	pg/ul	40	36	64	91	6	0	3
TEQ (PCB) LB (ND=0)	**pg TEQ/ul**	**4.5**	**4.3**	**64**	**84**	**9**	**0**	**6**
TEQ (PCB) UB (ND=LOD)	**pg TEQ/ul**	**4.5**	**4.3**	**62**	**87**	**10**	**0**	**3**
TEQ Total LB (ND=0)	**pg TEQ/ul**	**176**	**168**	**62**	**97**	**0**	**3**	**0**
TEQ Total UB (ND=LOD)	**pg TEQ/ul**	**176**	**167**	**60**	**97**	**0**	**3**	**0**

Table 22: Summary results dl-POPs, ash

Ash											
Contaminant	**Unit**	**Assigned**	**Average**	**Median**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
2,3,7,8-TeCDD	ng/kg	19	18	19	8.0	27	4.7	26	26	28	80
1,2,3,7,8-PeCDD	ng/kg	84	79	83	30	110	21	27	27	23	75
1,2,3,4,7,8-HxCDD	ng/kg	89	87	90	29	130	24	28	27	31	82
1,2,3,6,7,8-HxCDD	ng/kg	243	242	240	82	409	73	30	27	29	76
1,2,3,7,8,9-HxCDD	ng/kg	151	152	144	77	235	40	26	27	29	79
1,2,3,4,6,7,8-HpCDD	ng/kg	1980	1900	1900	93	3030	677	36	27	34	78
OCDD	ng/kg	6110	5720	6010	240	8700	2110	37	27	34	79
2,3,7,8-TeCDF	ng/kg	144	149	150	53	337	59	40	27	31	73
1,2,3,7,8-PeCDF	ng/kg	326	323	307	104	516	100	31	27	28	72
2,3,4,7,8-PeCDF	ng/kg	476	476	481	190	775	131	27	27	22	72
1,2,3,4,7,8-HxCDF	ng/kg	636	622	638	227	922	177	29	27	30	79
1,2,3,6,7,8-HxCDF	ng/kg	722	717	712	254	1100	214	30	27	27	72
1,2,3,7,8,9-HxCDF	ng/kg	NA	193	108	27	979	217	113	27	80	73
2,3,4,6,7,8-HxCDF	ng/kg	1190	1070	1119	206	1630	407	38	27	25	65
1,2,3,4,6,7,8-HpCDF	ng/kg	3950	3.880	3820	1092	5680	1210	31	27	35	81
1,2,3,4,7,8,9-HpCDF	ng/kg	572	568	601	190	957	177	31	27	31	78
OCDF	ng/kg	4940	4640	4750	210	7130	1780	38	27	37	80
TEQ (PCDD/PCDF) LB (ND=0)	**ng TEQ/kg**	**757**	**737**	**757**	**310**	**1032**	**189**	**26**	**27**	23	75
TEQ (PCDD/PCDF) UB (ND=LOD)	**ng TEQ/kg**	**763**	**739**	**758**	**310**	**1032**	**193**	**26**	**26**	23	75
PCB #77	ng/kg	169	614	165	25	9780	2050	334	22	23	67
PCB #81	ng/kg	161	586	156	20	9930	2090	356	22	14	60
PCB #126	ng/kg	335	704	328	90	9380	1940	275	22	13	59
PCB #169	ng/kg	219	4670	230	85	91200	19000	408	23	23	65
PCB #105	ng/kg	176	225	180	93	741	157	70	21	36	76
PCB #114	ng/kg	65	64	67	27	111	23	36	20	28	67
PCB #118	ng/kg	159	736	168	93	11100	2390	325	21	49	79
PCB #123	ng/kg	49	576	49	19	11100	2420	420	21	15	64
PCB #156	ng/kg	221	871	218	83	14800	3110	357	22	17	62
PCB #157	ng/kg	211	327	210	73	3260	658	202	22	20	67
PCB #167	ng/kg	98	990	96	32	10400	2970	300	23	22	62
PCB #189	ng/kg	307	364	306	12	2284	439	121	22	23	68
TEQ (PCB) LB (ND=0)	**ng TEQ/kg**	**36**	**109**	**35**	**0.01**	**1071**	**273**	**250**	**24**	18	60
TEQ (PCB) UB (ND=LOD)	**ng TEQ/kg**	**36**	**133**	**36**	**10.0**	**1210**	**327**	**246**	**22**	13	61
TEQ Total LB (ND=0)	**ng TEQ/kg**	**804**	**763**	**778**	**327**	**1071**	**213**	**28**	**23**	21	68
TEQ Total UB (ND=LOD)	**ng TEQ/kg**	**805**	**780**	**811**	**327**	**1210**	**236**	**30**	**22**	26	72

Table 23: Summary of laboratory performance dl-POPs, ash

Ash			% of the data received	% of Zscores |Z|<2	% of Zscores 3>|Z|>2	% of Zscores 6>|Z|>3	% of Zscores |Z|>6
Contaminant	**Unit**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
2,3,7,8-TeCDD	ng/kg	19	52	69	19	12	0
1,2,3,7,8-PeCDD	ng/kg	84	54	70	15	15	0
1,2,3,4,7,8-HxCDD	ng/kg	89	54	67	19	15	0
1,2,3,6,7,8-HxCDD	ng/kg	243	54	63	19	19	0
1,2,3,7,8,9-HxCDD	ng/kg	151	54	67	22	11	0
1,2,3,4,6,7,8-HpCDD	ng/kg	1 976	54	52	30	15	4
OCDD	ng/kg	6 114	54	59	19	15	7
2,3,7,8-TeCDF	ng/kg	144	54	59	19	15	7
1,2,3,7,8-PeCDF	ng/kg	326	54	63	11	26	0
2,3,4,7,8-PeCDF	ng/kg	476	54	70	11	19	0
1,2,3,4,7,8-HxCDF	ng/kg	636	54	74	7	19	0
1,2,3,6,7,8-HxCDF	ng/kg	722	54	67	4	30	0
1,2,3,7,8,9-HxCDF	ng/kg	NA	54	NA	NA	NA	NA
2,3,4,6,7,8-HxCDF	ng/kg	1 185	54	59	15	22	4
1,2,3,4,6,7,8-HpCDF	ng/kg	3 951	54	59	22	19	0
1,2,3,4,7,8,9-HpCDF	ng/kg	572	54	67	15	19	0
OCDF	ng/kg	4 936	54	52	26	19	4
TEQ (PCDD/PCDF) LB (ND=0)	**ng TEQ/kg**	**757**	**54**	**70**	**19**	**11**	**0**
TEQ (PCDD/PCDF) UB (ND=LOD)	**ng TEQ/kg**	**763**	**52**	**65**	**23**	**12**	**0**
PCB #77	ng/kg	169	46	65	9	9	13
PCB #81	ng/kg	161	46	70	4	13	9
PCB #126	ng/kg	335	46	65	4	22	4
PCB #169	ng/kg	219	46	61	9	22	9
PCB #105	ng/kg	176	46	48	13	22	9
PCB #114	ng/kg	65	44	50	14	27	0
PCB #118	ng/kg	159	48	33	21	25	8
PCB #123	ng/kg	49	46	70	0	13	9
PCB #156	ng/kg	221	46	61	9	22	4
PCB #157	ng/kg	211	46	70	0	22	4
PCB #167	ng/kg	98	46	61	9	17	13
PCB #189	ng/kg	307	46	65	9	13	9
TEQ (PCB) LB (ND=0)	**ng TEQ/kg**	**36**	**48**	**63**	**4**	**17**	**17**
TEQ (PCB) UB (ND=LOD)	**ng TEQ/kg**	**36**	**44**	**68**	**5**	**18**	**9**
TEQ Total LB (ND=0)	**ng TEQ/kg**	**804**	**46**	**70**	**13**	**17**	**0**
TEQ Total UB (ND=LOD)	**ng TEQ/kg**	**805**	**44**	**64**	**14**	**23**	**0**

Table 24: Summary results dl-POPs, sediment

Sediment											
Contaminant	**Unit**	**Assigned**	**Average**	**Median**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
2,3,7,8-TeCDD	ng/kg	3.3	4.6	3.4	0.9	38.0	6.6	144	28	18	67
1,2,3,7,8-PeCDD	ng/kg	4.3	4.7	4.5	2.4	9.6	1.6	34	27	30	73
1,2,3,4,7,8-HxCDD	ng/kg	3.5	3.9	3.6	2.3	6.4	1.2	30	25	24	68
1,2,3,6,7,8-HxCDD	ng/kg	10.2	10.9	10,0	6.6	19.3	3.1	29	27	26	73
1,2,3,7,8,9-HxCDD	ng/kg	8.6	8.5	8.7	5.1	12	1.8	21	27	22	75
1,2,3,4,6,7,8-HpCDD	ng/kg	147	160	150	61	373	60	38	28	23	69
OCDD	ng/kg	1110	1080	1100	486	1510	267	25	28	22	72
2,3,7,8-TeCDF	ng/kg	41	41	43	21	60	10	25	28	22	69
1,2,3,7,8-PeCDF	ng/kg	50	56	51	31	204	32	56	27	25	75
2,3,4,7,8-PeCDF	ng/kg	30	30	31	16	48	8.2	27	27	32	79
1,2,3,4,7,8-HxCDF	ng/kg	112	110	113	59	160	25	22	28	22	77
1,2,3,6,7,8-HxCDF	ng/kg	78	74	76	31	106	18	24	28	17	71
1,2,3,7,8,9-HxCDF	ng/kg	19	27	18	7,.7	68	16	59	28	54	62
2,3,4,6,7,8-HxCDF	ng/kg	NA	29	26	10	48	12	43	28	54	81
1,2,3,4,6,7,8-HpCDF	ng/kg	384	396	385	163	1040	149	38	28	17	70
1,2,3,4,7,8,9-HpCDF	ng/kg	131	131	130	62	200	31	23	28	22	76
OCDF	ng/kg	1290	1250	1280	516	1890	329	26	28	21	71
TEQ (PCDD/PCDF) LB (ND=0)	**ng TEQ/kg**	**64**	**64**	**64**	**35**	**103**	**16**	**25**	**28**	19	69
TEQ (PCDD/PCDF) UB (ND=LOD)	**ng TEQ/kg**	**64**	**62**	**63**	**35**	**89**	**14**	**23**	**27**	17	67
PCB #77	ng/kg	402	1420	401	89	18200	3810	268	24	17	67
PCB #81	ng/kg	15	736	16	3.0	15200	3110	422	24	29	61
PCB #126	ng/kg	37	62	37	30	560	111	180	22	14	74
PCB #169	ng/kg	6.4	6.9	6.4	4.3	11	1.6	24	19	11	67
PCB #105	ng/kg	906	1090	915	492	2970	541	50	24	21	71
PCB #114	ng/kg	44	625	45	25	13200	2730	437	23	15	62
PCB #118	ng/kg	3500	4280	3590	2380	16900	2910	68	24	15	71
PCB #123	ng/kg	NA	881	86	31	13700	2840	323	23	98	60
PCB #156	ng/kg	1050	1020	1010	258	1490	246	24	24	14	68
PCB #157	ng/kg	155	563	160	103	8620	1800	320	22	15	67
PCB #167	ng/kg	538	535	520	270	719	102	19	23	18	76
PCB #189	ng/kg	204	218	202	131	457	64.9	30	22	16	71
TEQ (PCB) LB (ND=0)	**ng TEQ/kg**	**5**	**4**	**5**	**0.80**	**16**	**3**	**67**	**25**	14	56
TEQ (PCB) UB (ND=LOD)	**ng TEQ/kg**	**5**	**11**	**5**	**0.9**	**150**	**30**	**288**	**23**	12	57
TEQ Total LB (ND=0)	**ng TEQ/kg**	**66**	**65**	**66**	**35**	**107**	**18**	**28**	**24**	18	65
TEQ Total UB (ND=LOD)	**ng TEQ/kg**	**67**	**68**	**66**	**35**	**150**	**24**	**35**	**23**	16	65

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

Sediment			% of the	% of Zscores	% of Zscores	% of Zscores	% of Zscores
Contaminant	Unit	Assigned	data received	|Z|<2 Satisfactory	3>|Z|>2 Questionable	6>|Z|>3 Unsatisfactory	|Z|>6 Extreme
2,3,7,8-TeCDD	ng/kg	3	56	71	7	11	11
1,2,3,7,8-PeCDD	ng/kg	4	56	57	21	14	4
1,2,3,4,7,8-HxCDD	ng/kg	4	54	63	15	11	4
1,2,3,6,7,8-HxCDD	ng/kg	10	56	64	21	4	7
1,2,3,7,8,9-HxCDD	ng/kg	9	56	68	25	4	0
1,2,3,4,6,7,8-HpCDD	ng/kg	147	56	64	14	14	7
OCDD	ng/kg	1 106	56	61	29	11	0
2,3,7,8-TeCDF	ng/kg	41	56	68	18	14	0
1,2,3,7,8-PeCDF	ng/kg	50	56	71	14	7	4
2,3,4,7,8-PeCDF	ng/kg	30	56	61	14	21	0
1,2,3,4,7,8-HxCDF	ng/kg	112	56	75	11	14	0
1,2,3,6,7,8-HxCDF	ng/kg	78	56	75	14	11	0
1,2,3,7,8,9-HxCDF	ng/kg	19	56	39	11	11	39
2,3,4,6,7,8-HxCDF	ng/kg	NA	56	NA	NA	NA	NA
1,2,3,4,6,7,8-HpCDF	ng/kg	384	56	71	18	7	4
1,2,3,4,7,8,9-HpCDF	ng/kg	131	56	75	14	11	0
OCDF	ng/kg	1 289	56	71	14	14	0
TEQ (PCDD/PCDF) LB (ND=0)	**ng TEQ/kg**	**64**	**56**	**75**	**7**	**18**	**0**
TEQ (PCDD/PCDF) UB (ND=LOD)	**ng TEQ/kg**	**64**	**54**	**78**	**7**	**15**	**0**
PCB #77	ng/kg	402	48	71	13	0	17
PCB #81	ng/kg	15	48	50	13	13	25
PCB #126	ng/kg	37	46	83	4	4	4
PCB #169	ng/kg	6	42	71	5	14	0
PCB #105	ng/kg	906	48	79	0	8	13
PCB #114	ng/kg	44	46	65	13	4	17
PCB #118	ng/kg	3 497	50	80	4	4	8
PCB #123	ng/kg	NA	46	NA	NA	NA	NA
PCB #156	ng/kg	1 050	50	80	4	8	4
PCB #157	ng/kg	155	46	74	9	0	13
PCB #167	ng/kg	538	48	79	13	4	0
PCB #189	ng/kg	204	46	74	13	4	4
TEQ (PCB) LB (ND=0)	**ng TEQ/kg**	**4.9**	**50**	**64**	**0**	**20**	**16**
TEQ (PCB) UB (ND=LOD)	**ng TEQ/kg**	**4.8**	**46**	**65**	**4**	**17**	**13**
TEQ Total LB (ND=0)	**ng TEQ/kg**	**66**	**48**	**71**	**4**	**25**	**0**
TEQ Total UB (ND=LOD)	**ng TEQ/kg**	**67**	**46**	**74**	**4**	**17**	**4**

Table 26: Summary results dl-POPs, fish (lipid weight basis)

Fish											
Contaminant	**Unit**	**Assigned**	**Average**	**Median**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
% Lipids	**%**	NA	6.0%	5.9%	1.2%	12%	1.9%	32	28	NC	NC
2,3,7,8-TeCDD	ng/kg	196	189	184	16	340	74	39	23	33	75
1,2,3,7,8-PeCDD	ng/kg	41	41	42	3.2	125	25	60	20	32	66
1,2,3,4,7,8-HxCDD	ng/kg	NA	6,0	2,4	0.7	34	8,9	149	15	87	61
1,2,3,6,7,8-HxCDD	ng/kg	14	15	14	1.0	65	13	88	20	33	62
1,2,3,7,8,9-HxCDD	ng/kg	NA	8,9	2,8	0.5	64	18	198	13	112	58
1,2,3,4,6,7,8-HpCDD	ng/kg	NA	37	12	1.8	370	85	228	18	102	62
OCDD	ng/kg	NA	280	64	1.2	4000	862	308	21	135	68
2,3,7,8-TeCDF	ng/kg	212	189	210	8.8	298	81	43	23	34	75
1,2,3,7,8-PeCDF	ng/kg	19	26	21	2.3	120	25	95	22	44	67
2,3,4,7,8-PeCDF	ng/kg	114	117	115	8.6	246	48	41	22	26	70
1,2,3,4,7,8-HxCDF	ng/kg	7.2	22	8.2	1.7	136	36	167	21	47	64
1,2,3,6,7,8-HxCDF	ng/kg	NA	20	10	2.4	91	25	128	18	88	64
1,2,3,7,8,9-HxCDF	ng/kg	NA	9	2,5	0.3	45	13	142	14	119	50
2,3,4,6,7,8-HxCDF	ng/kg	NA	9	5,5	1.3	37	9,3	100	20	78	71
1,2,3,4,6,7,8-HpCDF	ng/kg	NA	47	21	1.8	309	72	155	18	132	56
1,2,3,4,7,8,9-HpCDF	ng/kg	NA	30	3,7	0.3	224	65	215	14	123	52
OCDF	ng/kg	NA	47	13	2.0	415	101	212	16	130	53
TEQ (PCDD/PCDF) LB (ND=0)	**ng TEQ/kg**	**325**	**309**	**322**	**27**	**546**	**121**	**39**	**23**	30	73
TEQ (PCDD/PCDF) UB (ND=LOD)	**ng TEQ/kg**	**328**	**311**	**322**	**27**	**546**	**122**	**39**	**23**	31	74
PCB #77	ng/kg	33500	37000	33700	40	86500	22700	61	21	41	66
PCB #81	ng/kg	2640	7750	3200	40	36500	10600	137	21	68	64
PCB #126	ng/kg	11500	21900	11300	34	148000	37200	169	21	45	70
PCB #169	ng/kg	1160	1170	1200	87	202-	422	36	19	27	71
PCB #105	ng/kg	753000	726000	748000	136	1360000	338000	47	21	38	73
PCB #114	ng/kg	52300	54300	53300	674	126000	27500	51	21	23	64
PCB #118	ng/kg	1650000	1600000	1690000	990	3270000	958000	60	22	58	75
PCB #123	ng/kg	NA	104000	73800	200	363000	95000	92	21	56	71
PCB #156	ng/kg	204000	214000	205000	2380	552000	121000	57	22	21	60
PCB #157	ng/kg	53300	570000	49900	3700	170000	33600	59	20	28	65
PCB #167	ng/kg	157000	165000	149000	9500	412000	88000	53	21	37	70
PCB #189	ng/kg	32000	30500	30200	2100	53000	12700	42	20	31	73
TEQ (PCB) LB (ND=0)	**ng TEQ/kg**	**1590**	**2590**	**1620**	**1**	**15150**	**3750**	**145**	**21**	38	66
TEQ (PCB) UB (ND=LOD)	**ng TEQ/kg**	**1620**	**2230**	**1620**	**116**	**15150**	**3190**	**143**	**19**	32	68
TEQ Total LB (ND=0)	**ng TEQ/kg**	**1940**	**2620**	**1920**	**176**	**15500**	**3180**	**122**	**19**	26	68
TEQ Total UB (ND=LOD)	**ng TEQ/kg**	**1940**	**2620**	**1920**	**176**	**15500**	**3190**	**122**	**19**	26	68

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

Fish			% of the data received	% of Zscores |Z|<2	% of Zscores 3>|Z|>2	% of Zscores 6>|Z|>3	% of Zscores |Z|>6
Contaminant	**Unit**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
% Lipids	**%**	NA	NA	NA	NA	NA	NA
2,3,7,8-TeCDD	ng/kg	196	46	43	39	9	9
1,2,3,7,8-PeCDD	ng/kg	41	44	45	23	9	14
1,2,3,4,7,8-HxCDD	ng/kg	NA	46	NA	NA	NA	NA
1,2,3,6,7,8-HxCDD	ng/kg	14	46	43	17	4	22
1,2,3,7,8,9-HxCDD	ng/kg	NA	46	NA	NA	NA	NA
1,2,3,4,6,7,8-HpCDD	ng/kg	NA	44	NA	NA	NA	NA
OCDD	ng/kg	NA	46	NA	NA	NA	NA
2,3,7,8-TeCDF	ng/kg	212	46	65	9	17	9
1,2,3,7,8-PeCDF	ng/kg	19	46	39	22	17	17
2,3,4,7,8-PeCDF	ng/kg	114	44	59	18	9	14
1,2,3,4,7,8-HxCDF	ng/kg	7	46	43	9	17	22
1,2,3,6,7,8-HxCDF	ng/kg	NA	44	NA	NA	NA	NA
1,2,3,7,8,9-HxCDF	ng/kg	NA	46	NA	NA	NA	NA
2,3,4,6,7,8-HxCDF	ng/kg	NA	44	NA	NA	NA	NA
1,2,3,4,6,7,8-HpCDF	ng/kg	NA	44	NA	NA	NA	NA
1,2,3,4,7,8,9-HpCDF	ng/kg	NA	46	NA	NA	NA	NA
OCDF	ng/kg	NA	44	NA	NA	NA	NA
TEQ (PCDD/PCDF) LB (ND=0)	**ng TEQ/kg**	**325**	**46**	**57**	**22**	**13**	**9**
TEQ (PCDD/PCDF) UB (ND=LOD)	**ng TEQ/kg**	**328**	**46**	**61**	**17**	**13**	**9**
PCB #77	ng/kg	33 467	42	48	10	14	29
PCB #81	ng/kg	2 644	42	33	14	10	43
PCB #126	ng/kg	11 459	42	38	24	14	24
PCB #169	ng/kg	1 164	38	63	16	16	5
PCB #105	ng/kg	753 201	42	57	10	19	14
PCB #114	ng/kg	52 292	42	62	10	10	19
PCB #118	ng/kg	1 650 662	44	36	14	23	27
PCB #123	ng/kg	NA	42	0	0	0	0
PCB #156	ng/kg	203 994	44	64	0	14	23
PCB #157	ng/kg	53 260	40	60	5	20	15
PCB #167	ng/kg	156 898	42	43	29	10	19
PCB #189	ng/kg	31 993	40	55	20	15	10
TEQ (PCB) LB (ND=0)	**ng TEQ/kg**	**1 586**	**42**	**52**	**14.3**	**10**	**24**
TEQ (PCB) UB (ND=LOD)	**ng TEQ/kg**	**1 616**	**38**	**58**	**16**	**11**	**16**
TEQ Total LB (ND=0)	**ng TEQ/kg**	**1 936**	**38**	**53**	**26**	**11**	**11**
TEQ Total UB (ND=LOD)	**ng TEQ/kg**	**1 936**	**38**	**53**	**26**	**11**	**11**

Table 28: Summary results dl-POPs, milk (lipid weight basis)

Milk											
Contaminant	**Unit**	**Assigned**	**Average**	**Median**	**Min**	**Max**	**SD**	**%RSD**	**n**	**Between Lab CV (%)**	**Inclusion rate (%)**
% Lipids	**%**	NA	3.5%	2.7%	1.6%	20%	3.8%	111	**22**	NC	NC
2,3,7,8-TeCDD	ng/kg	0.43	0.71	0.47	0.10	4.1	1.0	140	14	37	67
1,2,3,7,8-PeCDD	ng/kg	1.3	1.3	1.3	0.29	1.8	0.40	32	15	32	75
1,2,3,4,7,8-HxCDD	ng/kg	0.64	0.66	0.65	0.23	1.2	0.27	40	14	41	76
1,2,3,6,7,8-HxCDD	ng/kg	4.3	4.0	4.1	0.48	5.5	1.3	33	16	19	69
1,2,3,7,8,9-HxCDD	ng/kg	0.77	1.07	0.80	0.30	5.2	1.2	111	15	40	74
1,2,3,4,6,7,8-HpCDD	ng/kg	4.4	4.5	4.4	2.0	6.8	1.3	30	16	25	71
OCDD	ng/kg	33	45	32	6.1	270	59	130	17	18	66
2,3,7,8-TeCDF	ng/kg	0.40	0.86	0.44	0.25	4.5	1.2	140	12	54	65
1,2,3,7,8-PeCDF	ng/kg	0.24	0.40	0.31	0.10	1.3	0.32	81	11	55	55
2,3,4,7,8-PeCDF	ng/kg	3.8	3.6	3.7	0.93	4.7	0.90	25	16	15	74
1,2,3,4,7,8-HxCDF	ng/kg	1.3	1.3	1.3	0.92	1.6	0.22	17	15	22	79
1,2,3,6,7,8-HxCDF	ng/kg	1.3	1.9	1.3	1.0	10	2.2	121	16	16	74
1,2,3,7,8,9-HxCDF	ng/kg	NA	0.17	0.15	0.05	0.33	0.13	74	6	115	61
2,3,4,6,7,8-HxCDF	ng/kg	0.89	1.2	0.93	0.08	6.5	1.4	116	16	18	67
1,2,3,4,6,7,8-HpCDF	ng/kg	4.5	7.1	4.5	2.4	52	11	161	17	14	57
1,2,3,4,7,8,9-HpCDF	ng/kg	0.13	1.6	0.15	0.09	13	4.3	259	9	69	52
OCDF	ng/kg	NA	13	0.85	0.37	138	39	315	12	79	66
TEQ (PCDD/PCDF) LB (ND=0)	**ng TEQ/kg**	**4.53**	**4.54**	**4.53**	**1.59**	**7.48**	**1.28**	**28**	**17**	26	79
TEQ (PCDD/PCDF) UB (ND=LOD)	**ng TEQ/kg**	**4.88**	**5.71**	**4.80**	**1.59**	**23**	**4.49**	**79**	**17**	16	73
PCB #77	ng/kg	NA	1870	8.4	2.6	24100	6690	358	13	110	60
PCB #81	ng/kg	1.3	121	1.3	0.18	1440	414	343	12	50	71
PCB #126	ng/kg	22	26	22	1.7	96	20	77	16	26	68
PCB #169	ng/kg	19	18	18	8.5	24	4.0	23	15	14	68
PCB #105	ng/kg	913	1650	950	14	15100	3300	200	19	20	60
PCB #114	ng/kg	222	363	230	7.5	1810	427	117	18	28	68
PCB #118	ng/kg	4340	5130	4480	17	25400	5080	99	21	21	60
PCB #123	ng/kg	53	460	55	2.1	3750	1000	218	16	43	62
PCB #156	ng/kg	3610	3160	3470	11	4370	1.223	39	20	18	71
PCB #157	ng/kg	581	543	570	18	850	181	33	17	20	70
PCB #167	ng/kg	747	663	716	22	948	228	34	18	17	69
PCB #189	ng/kg	347	321	340	11	447	104	32	17	21	75
TEQ (PCB) LB (ND=0)	**ng TEQ/kg**	**5.1**	**5.9**	**5.2**	**0.30**	**19**	**3.9**	**67**	**18**	24	60
TEQ (PCB) UB (ND=LOD)	**ng TEQ/kg**	**5.2**	**11**	**5.6**	**0.30**	**76**	**17**	**164**	**17**	22	62
TEQ Total LB (ND=0)	**ng TEQ/kg**	**9.6**	**10**	**10**	**1.9**	**21**	**3.9**	**38**	**17**	11	58
TEQ Total UB (ND=LOD)	**ng TEQ/kg**	**10**	**15**	**10**	**0.5**	**99**	**21**	**140**	**18**	17	58

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

Milk			% of the data received	% of Zscores |Z|<2	% of Zscores 3>|Z|>2	% of Zscores 6>|Z|>3	% of Zscores |Z|>6
Contaminant	**Unit**	**Assigned**		**Satisfactory**	**Questionable**	**Unsatisfactory**	**Extreme**
% Lipids	**%**	NA					
2,3,7,8-TeCDD	ng/kg	0.43	32	44	25	6	13
1,2,3,7,8-PeCDD	ng/kg	1.29	34	65	6	18	0
1,2,3,4,7,8-HxCDD	ng/kg	0.64	30	47	13	27	7
1,2,3,6,7,8-HxCDD	ng/kg	4.32	34	76	6	6	6
1,2,3,7,8,9-HxCDD	ng/kg	0.77	32	44	19	25	6
1,2,3,4,6,7,8-HpCDD	ng/kg	4.45	34	59	12	24	0
OCDD	ng/kg	32.7	34	71	6	12	12
2,3,7,8-TeCDF	ng/kg	0.40	30	40	7	20	13
1,2,3,7,8-PeCDF	ng/kg	0.24	32	31	6	19	13
2,3,4,7,8-PeCDF	ng/kg	3.82	34	82	6	6	0
1,2,3,4,7,8-HxCDF	ng/kg	1.29	34	82	6	0	0
1,2,3,6,7,8-HxCDF	ng/kg	1.27	32	88	0	6	6
1,2,3,7,8,9-HxCDF	ng/kg	NA	28	NA	NA	NA	NA
2,3,4,6,7,8-HxCDF	ng/kg	0.89	32	75	6	0	19
1,2,3,4,6,7,8-HpCDF	ng/kg	4.48	34	65	12	18	6
1,2,3,4,7,8,9-HpCDF	ng/kg	0.13	32	31	0	0	25
OCDF	ng/kg	NA	30	0	0	0	0
TEQ (PCDD/PCDF) LB (ND=0)	**ng TEQ/kg**	**4.53**	**34**	**82**	**6**	**12**	**0**
TEQ (PCDD/PCDF) UB (ND=LOD)	**ng TEQ/kg**	**4.88**	**34**	**82**	**6**	**6**	**6**
PCB #77	ng/kg	NA	32	NA	NA	NA	NA
PCB #81	ng/kg	1.26	30	27	20	13	20
PCB #126	ng/kg	21.7	34	59	6	12	18
PCB #169	ng/kg	18.6	32	63	25	6	0
PCB #105	ng/kg	913	40	65	5	5	20
PCB #114	ng/kg	222	38	58	11	5	21
PCB #118	ng/kg	4 344	42	62	5	5	29
PCB #123	ng/kg	53.1	38	32	21	0	32
PCB #156	ng/kg	3 612	40	75	10	5	10
PCB #157	ng/kg	581	36	72	6	11	6
PCB #167	ng/kg	747	36	72	11	11	6
PCB #189	ng/kg	347	36	72	11	6	6
TEQ (PCB) LB (ND=0)	**ng TEQ/kg**	**5.13**	**36**	**56**	**6**	**22**	**17**
TEQ (PCB) UB (ND=LOD)	**ng TEQ/kg**	**5.24**	**34**	**59**	**12**	**0**	**29**
TEQ Total LB (ND=0)	**ng TEQ/kg**	**9.63**	**34**	**65**	**18**	**6**	**12**
TEQ Total UB (ND=LOD)	**ng TEQ/kg**	**10.1**	**36**	**56**	**11**	**11**	**22**

4. DISCUSSION

In total 103 laboratories from Asia, Africa, Europe and North-, Central and South-America participated in the present assessment. Of these, 83 laboratories submitted data on the test solution, the sediment, human milk, fish or fly ash samples.

Interlaboratory studies can provide some explanations of the relationship between the methods used and the results obtained. Unfortunately, poorly performing laboratories are often confronted with multiple difficulties. That makes it difficult to determine the exact sources of error. The present results and draft report were presented at a UNEP regional workshop in Hong Kong, China (February 2010), in Amsterdam, the Netherlands (February/March 2011) and Barcelona, Spain (March 2011). During these workshops, the participants evaluated their own performance and improvement of methodology. During a pilot project on capacity building, a few of the laboratories that participated in this assessment received training in analysis of POPs in biota and sediments. These laboratories also participated in the present assessment.

The participating laboratories were divided into five regions Africa, Asia, CEE (Central and Eastern Europe), GRULAC (Latin American and Caribbean), WEOG (Western European and Others (OECD countries)). For PCB and OCPs, there were ten laboratories from Africa, 35 from Asia, three from CEE, 23 from GRULAC and 12 from the WEOG region (83 in total). For the marker PCB the regional distribution was similar. For the dl-POPs, 49 laboratories participated in total, with three laboratories from Africa - of these, one was a high resolution GC/MS laboratory, one used bio assays (total dl-POPs results only), and one was a low resolution GC/MS laboratory reporting only the dl-PCB - one laboratory from the CEE region, four laboratories from GRULAC , 32 laboratories from Asia and ten from the WEOG region.

4.1 Methodological Considerations

It is a challenge to identify trends in interlaboratory assessment datasets and to explain the underlying methodological causes for the differences in results obtained. Deviations from the assigned value can be caused by a number of factors and errors, which often cannot be easily identified or quantified.

Especially for inexperienced laboratories that participate for the first time in an interlaboratory assessment, systematic errors are a source of deviations from the assigned value. Systematic errors may range from imperfect calibration of the measurement instruments to reporting concentrations in wrong units. For this like any other interlaboratory assessment, factors that may influence the interpretation and the outcome include the following (de Boer and Wells, 2006):

- The number of laboratories submitting results for each group of contaminants;
- The concentrations of the target compounds in the test materials;
- Variations in the analytical methods used by the participants,.

Further, errors can be introduced during calculation of results or dilution of the sample.

Nonetheless, based on the results and previous experience with interlaboratory studies, several problems could be elucidated such as:

- The POPs concentrations for the fatty samples – mothers' milk and fish – were to be reported on lipid basis. Since there exist various methods to determine the fat content of a sample, also the results from this interlaboratory comparison are vulnerable to interlaboratory variation in determination of lipid content (Miskiewicz and Gibbs, 1992). Furthermore, the combination of high lipid content and low POPs concentrations tend to cause higher RSD values (de Boer and Wells, 2006).
- The reported fat contents in the fish and the milk samples showed a large variation. The mean lipid percentage for the fish was 6.0% with an RSD of 32% with the lowest lipid content of 1.2% and the highest of 12% reported. For the milk sample an even larger variation was seen from a minimum lipid percentage of 3.8% to a maximum of 20% resulting in a mean value of 3.5% with an uncorrected RSD between all participants of 111%. This has a major effect on the reported concedntrations on a lipid basis. In an interlaboratory study on brominated flame retardants, the authors suggested that a high variability in lipid content occurred because the laboratories did not adapt standard analytical protocols to the new matrices (de Boer and Wells, 2006). When organising an interlaboratory assessment precaution should be made to reduce the variability in determination of lipid content as it may hamper interlaboratory comparison using lipid based concentrations.
- Errors in the reported results by orders of magnitude: A number of laboratories reported values deviating one or more orders of magnitude from the consensus or mean concentration assigned to the different samples. This is probably due to some confusion on the units used in the assessment. The original units in ng/kg (pg/g) resulted for some of the samples in large numbers. In this round of interlaboratory assessment, laboratories were allowed to correct such obvious mistakes (after discussions between the submitting laboratory and the coordinator of the Interlaboratory assessment). However, even after control by the participants of the preliminary data, some individual results still deviated considerably.
- Uneven participation of laboratories for certain test matrices: As can be seen from the summary of the results in chapter 3, there was a high variability between acceptance or preference of matrices, compound class and regions. This is indicated by the number of laboratories with satisfactory z-scores in tables 2-28 and summarised in Figure 2. Within the matrices, the highest number of laboratories analyzed the standard solution. With respect to the POPs analytes, DDT and PCB were widely analyzed whereas only a few laboratories submitted results for mirex. On average, more than 50% of the laboratories submitted data for the test solutions, whereas 54% submitted PCDD/PCDF data on fly ash. Between 30%-40% of the laboratories submitted data on the other three matrices; sediment, milk and fish. Fewer results were submitted for PCB in fly ash (17%), and OCP in milk (<30%).

Overall, it can be concluded that POPs laboratories seem to be specialized on few matrices and a relatively small spectrum of POPs analytes.

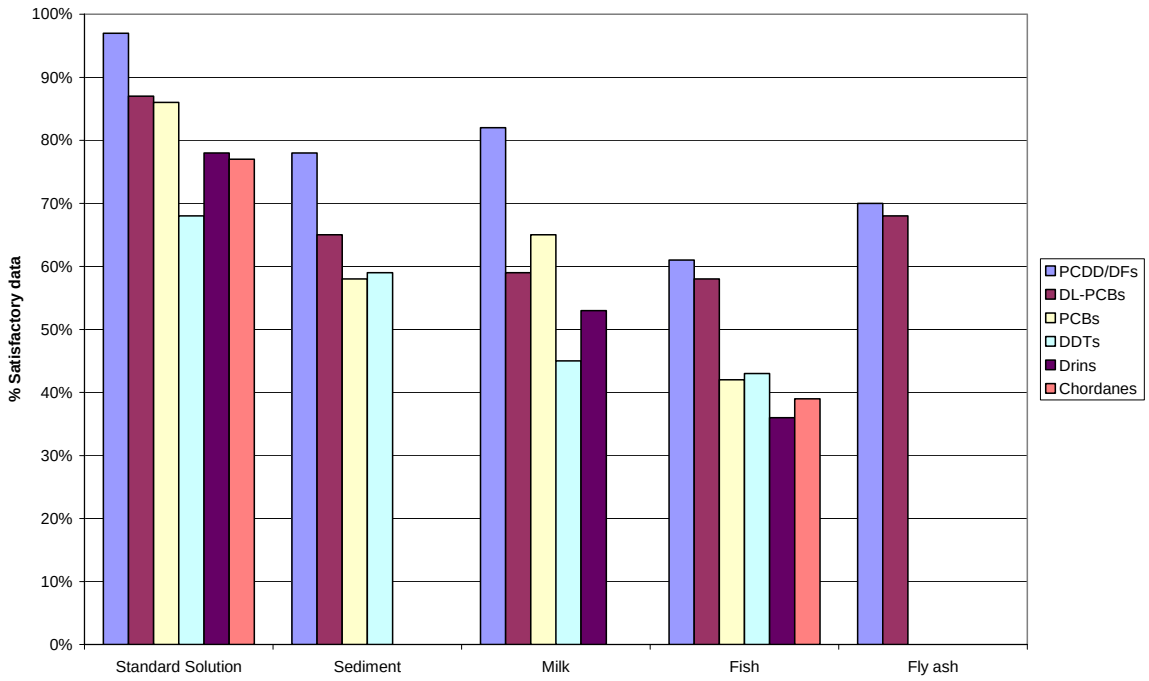


Figure 2 Percentage of laboratories with satisfactory z-scores (i.e. $z < \pm 2$) for OCP, PCB, PCDD/PCDF and dl-PCB in the test solution, sediment, milk, fish and fly ash.

The overall performance of labs measuring the test solution (certified standard solutions) was very good for the PCDD/PCDF (97% satisfactory z-scores), good for dl-PCB (87%) and indicator PCB (86%) and satisfactory for DDTs (68%), drins (78%) and chlordanes (77%) . The between-lab CV values for most of the POPs ranged from 6% to 13% for the individual PCDD/PCDF to 5% to 25% for OCP, indicating that factors associated with calibration (standards, calibration curves, storage of standard solutions, *etc.*) are satisfactory for most of the laboratories. This does not take away that several participants made large errors in this relatively simple matrix.

For the other test materials the between-lab CV values were larger and fewer satisfactory z-scores were obtained using the same criteria (z = 2.25%). For the fly ash only the PCDD/PCDF, dl-PCB and indicator PCB results were submitted. The results for the dl-POPs were generally good, but the results for the indicator PCB were not as good showing between-lab CV values of 25%-191% (Table 11). For the sediment sample the final data was too scattered to calculate a consensus value for the drins and the chlordanes. For the other compound classes the results for the sediment were 17%-54% (Between-lab CV) for PCDD/PCDF (Table 23), 31%-59% for marker PCB (Table 13) and 14%-358% for OCP (Table 3). As indicated the lipid normalised results for the fish and the milk samples varied more due to the variation in the lipid determination. Often around or less than 50% of the participating laboratories obtained satisfactory z-scores. Despite this the variation was reasonable for PCDD/PCDF TEQ for both samples: Between-lab CV 32%-38% in fish (Table 25) and 16%-26% in milk (Table 27)). It should however be noted that only a limited number of laboratories (n = 17) were able to analyze dioxins in the milk sample.

There was no clear indication of a “Horwitz trend” in the dataset, *i.e.*, lower concentrations inducing higher RSD values (Horwitz *et al*., 1980). Not even when PCDD, PCDF and dl-

PCB were removed, *i.e.* compounds analysed using labelled internal standards, any Horwitz trend was detected. On the contrary, there appeared to be a greater bias for herring tissue and milk with relatively high concentrations, than for sediment and fly ash. A similar trend was identified in a previous interlaboratory assessment analysing sediment, herring and a test solution in seven developing countries (de Boer *et al.*, 2008). Due to their relatively high lipid content milk and herring are more difficult to analyse.

The satisfatory performance of most laboratories for the test solution suggests that instrumental sensitivity is not the main source of error. It should be noted that far less data was submitted for other matrices than for the test solution probably due to the difficulties associated with real samples. None of the laboratories were able to submit data on all matrices. There is also a possibility that laboratories did not report data because those did not pass internal QA/QC measures.

All participating laboratories used in-house methods for sample preparation, clean-up, extraction and instrumental analysis. This included modified or adapted standard methods including for example EPA 1613 and EU 1613 for the dl-POP analysis, The participants were encouraged to use appropriate GC columns for the analyses, preferably dual-column sets. De Boer and Wells (2006) observed that in spite of a better availability of analytical standards and ^{13}C-labelled standards, many laboratories need a substantial period of time in order to establish a new analytical method. It is not unlikely that some of the laboratories had never analysed some of the matrices included in the present interlaboratory assessment before, and thus did not have sufficient time to adapt properly to the new methodology or, because of time constraints, chose to stick to methods they already were familiar with. In addition to the training provided to some of the laboratories world-wide, it will be essential to establish a routine in those laboratories of performing series of POP analyses on a regular basis.

4.2 Contaminant Group - Specific Performance

4.2.1 Organochlorine Pesticides

The individual results for the OCPs for the standard solution were satisfactory showing Between-lab CV values of 13%-22% for the drins, 5%-15% for the chlordanes and 11%-25% for the DDTs (Table 1). This is illustrated for the drins in Figure 3 in which the individual results from each laboratory are given in addition to the consensus value as calculated by the Cofino statistics and the UNEP criteria of 12.5% (z = 1) and 25% (z = 2).

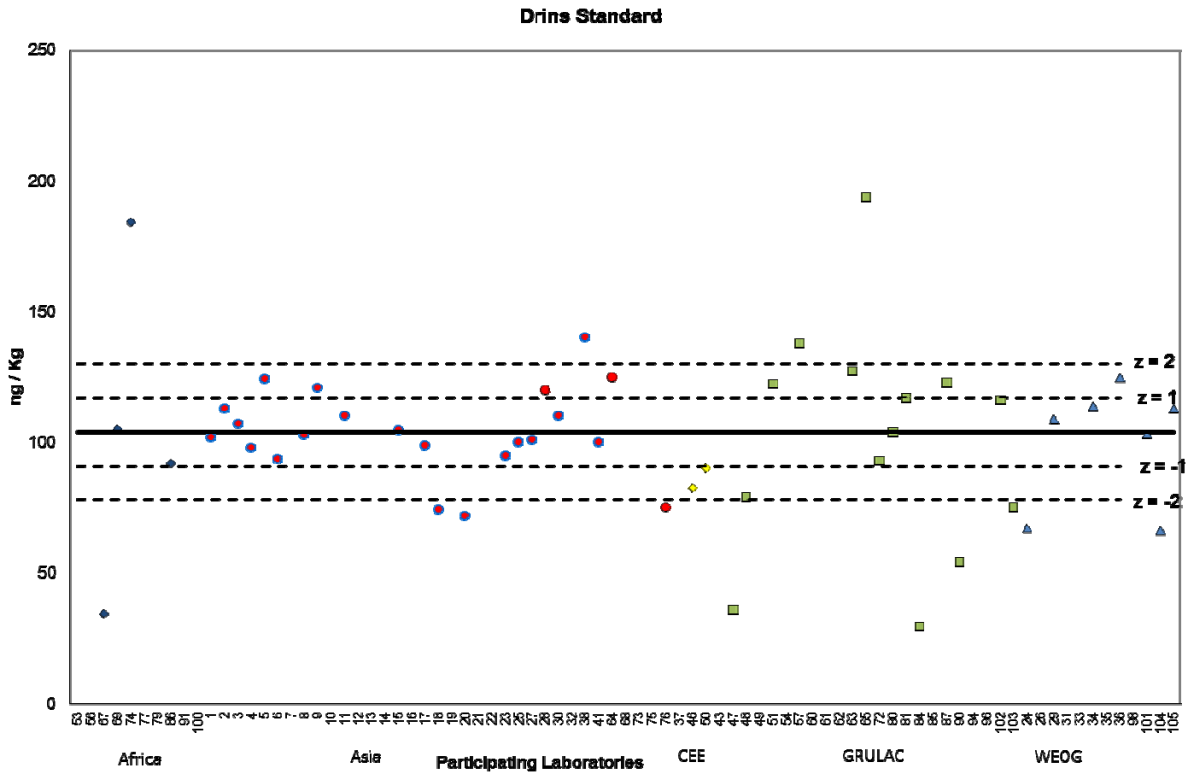


Figure 3 Results for sum of drins in the standard solution.
Laboratory code on the x-axis, concentration on the y-axis. The consensus value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ◇ symbols represent Africa, the red ○ symbols represent Asia, the yellow ◇ symbols represent CCE, the green □ symbols represent GRULAC and blue△ symbols represent WEOG.

The results for the other test materials showed a larger variation, sometimes more than 200%, and in some cases it was not possible to calculate a consensus value at all (most drins and chlordanes in sediment, drins and DDTs in the fish sample, chlordanes in milk). As an example, the results of the DDTs in fish are given in Figure 4 to show the large inter-laboratory variation. From this data no assigned value could be calculated and the median is used for comparison.

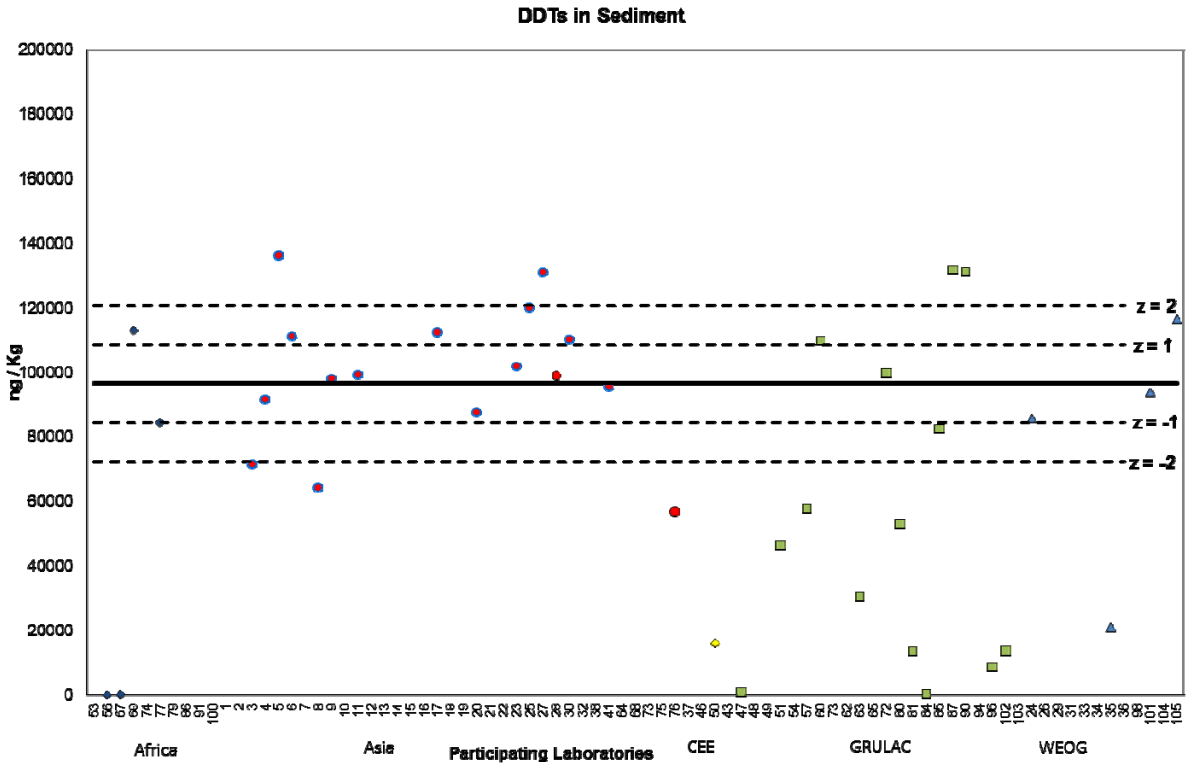


Figure 4 Results for sum of DDTs in the sediment sample.
Laboratory code on the x-axis, concentration on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ◇ symbols represent Africa, the red ○ symbols represent Asia, the yellow ◇ symbols represent CCE, the green □ symbols represent GRULAC and blue△ symbols represent WEOG.

The largest deviance from the assigned value was seen for the OCPs in milk and fish and often less than 50% of the data had a satisfactory z-score (see Tables 6 and 8). There are numerous challenges that might have obstructed the OCP analysis in particular, from decomposition in the injector (dirty liner) to interfering substances and co-elution in combination with non selective ECD detection (de Boer and Wells, 1997). Possibly, some laboratories may have used sulphuric acid to remove lipids; however, this may disintegrate some OCP such as dieldrin (de Boer and Wells, 1997).

OCPs like DDTs are easily degraded when the GC is not in the optimum condition (*i.e.*, dirty liner), resulting in inaccurate results. For indicator PCB, 69% of the labs showed an acceptable z-score. In the QUASIMEME interlaboratory studies, the general performance of laboratories analysing POPs in sediment was found to be lower for OCPs than PCB (de Boer and Wells, 1997). The authors noted that the vast majority of the participating laboratories were not able to the determine OCP levels with an acceptable accuracy. Even though this was fourteen years ago, it pinpoints some of the challenges encountered by several laboratories participating in the present assessment. The major problem with OCP analysis is in the GC/ECD analysis, which is in fact a compromise for a number of OCPs. The ECD is not specific, the baseline is rather noisy, separation of early eluting compounds is not very good, and internal standards may not compensate for all losses. The use of GC/MS, even low resolution MS, together with ^{13}C labelled standards would improve this performance substantially, as is shown for the analysis of PCDD/PCDFs, which are present at lower concentrations than the OCPs.

4.2.2 Polychlorinated Biphenyls

Also for the marker PCB the best results were obtained for the standard solution where the sum of marker PCB showed a Between-lab CV of 13%-19% (Table 9). As can be seen from Figure 5 the data contains three obvious outliers, and without removal of them by the model s the interlaboratory variation would have been much higher. The present value is good and in agreement with other studies.

The results for the other test materials show a larger variation: the Between-lab CV values for sediment were moderate with 31%-59% (Table 13, Figure 6), and the values for both fish and milk are relatively high (48%-113% and 26%-117% respectively, Tables 13 and 17). As discussed the lipid determination, which showed relatively high variation for both the milk (111%) and fish (32%) might contribute significantly to the overall RSD.

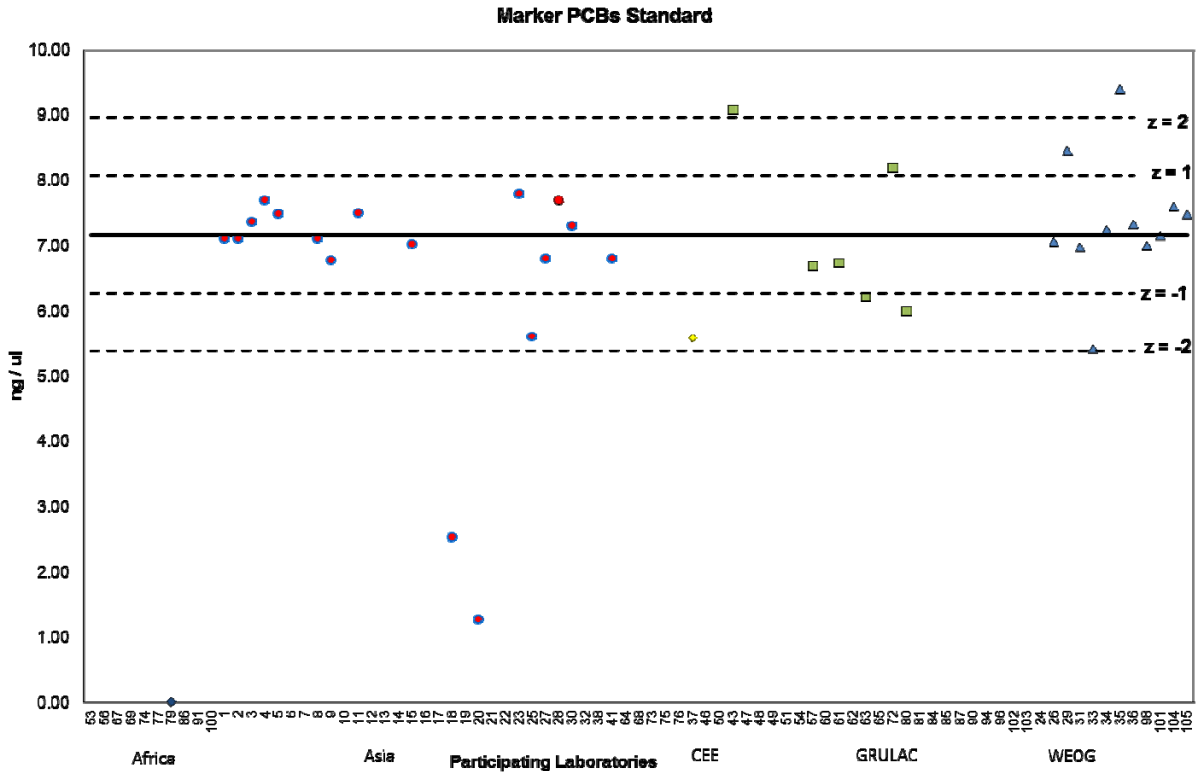


Figure 5 Results for the sum of marker PCB in the standard solution.
Laboratory code on the x-axis, concentration on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ◇ symbols represent Africa, the red ○ symbols represent Asia, the yellow ◇ symbols represent CCE, the green □ symbols represent GRULAC and blue△ symbols represent WEOG.

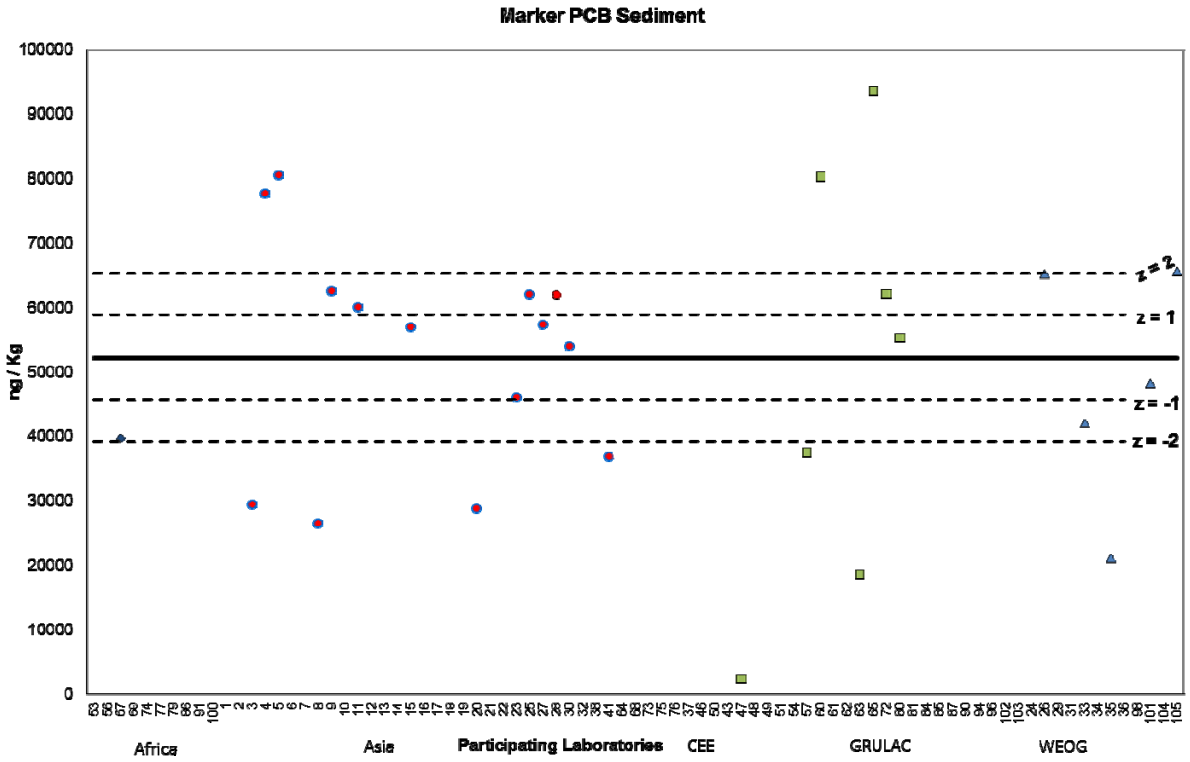


Figure 6 Results for the sum of marker PCB in the sediment sample. Laboratory code on the x-axis, concentration on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines. The blue ◇ symbols represent Africa, the red ○ symbols represent Asia, the yellow ◇ symbols represent CCE, the green □ symbols represent GRULAC and blue△ symbols represent WEOG.

4.2.3 Dioxin-like POPs

Better results were obtained for PCDD/PCDFs. The PCDD/PCDFs were present in lower concentrations (2-3 orders) compared to the PCB and OCPs. The use of high resolution GC/MS systems, the availability of a variety of ^{13}C labelled standards and well used standard methods improved the results.

The results for the standard solution was very good with an RSD of only 9% for the total TEQ (Table 19). The between-lab CV values for the individual PCDD/PCDFs were 6-12%. The PCDD/PCDF TEQ results were also good for the ash and sediment. The total TEQ between-lab CV values were 21%-26% for fly ash (Table 21) and 16%-19% for sediment (Table 23). The PCDD/PCDF total TEQ between-lab CV for fish was satisfactory (30%-31%) (Table 25).

The corresponding TEQ results for the dl-PCB showed somewhat higher between-lab CV of 12%-22% (Table 19). The results for the dl-PCB in fly ash were satisfactory (13%-18%, Table 21), but the same table shows that some large outliers were also present, resulting in an uncorrected RSD value of ca. 250%. The dl-PCB TEQ Between-lab CV value for sediment (12%-14%) is actually rather good (Table 23). In fish this value is 32%-38% (Table 25). Outliers are present in both cases. The results for milk sample are better again with 11%-17% between-lab CV values for dl-PCB TEQ.

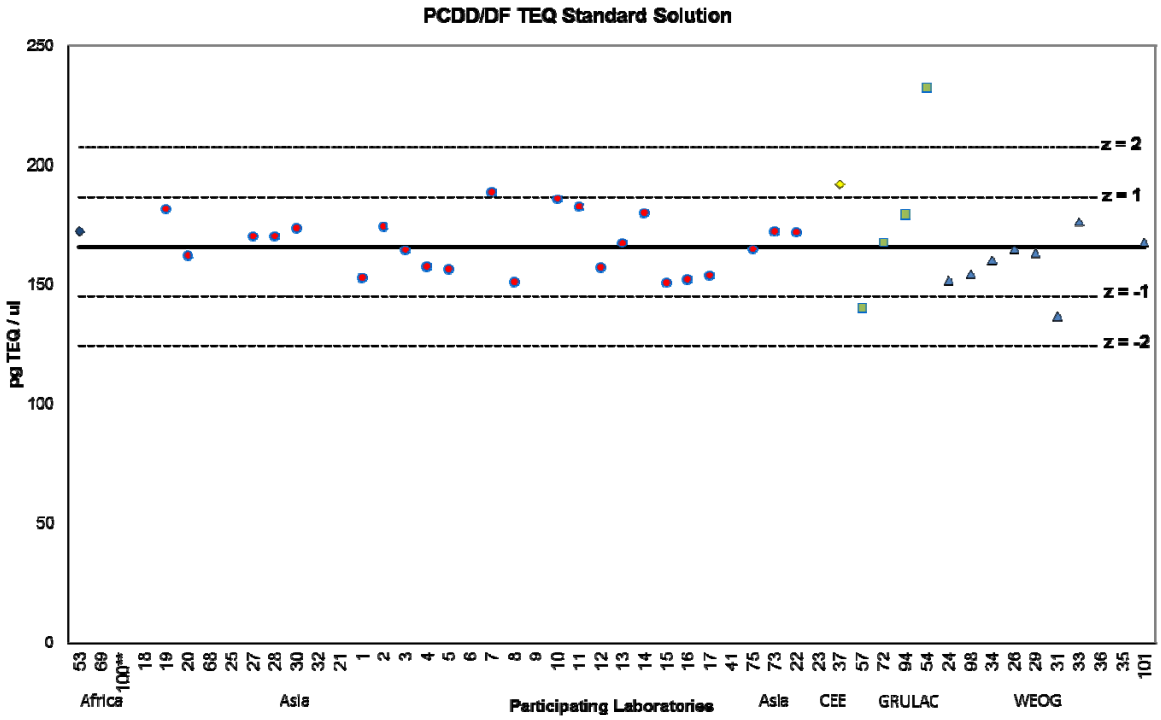


Figure 7 Results for the PCDD/PCDF TEQ in the standard solution.
Laboratory code on the x-axis, concentration on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines.

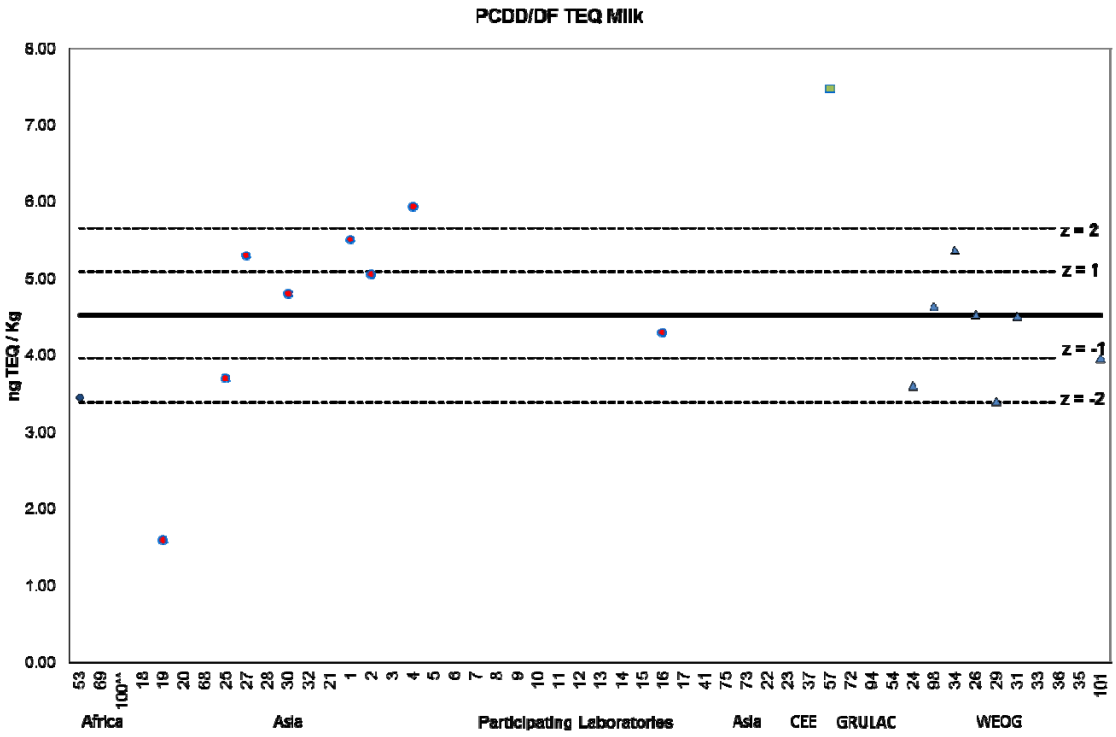


Figure 8 Results for the PCDD/PCDF TEQ in the milk sample.
Laboratory code on the x-axis, concentration on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines.

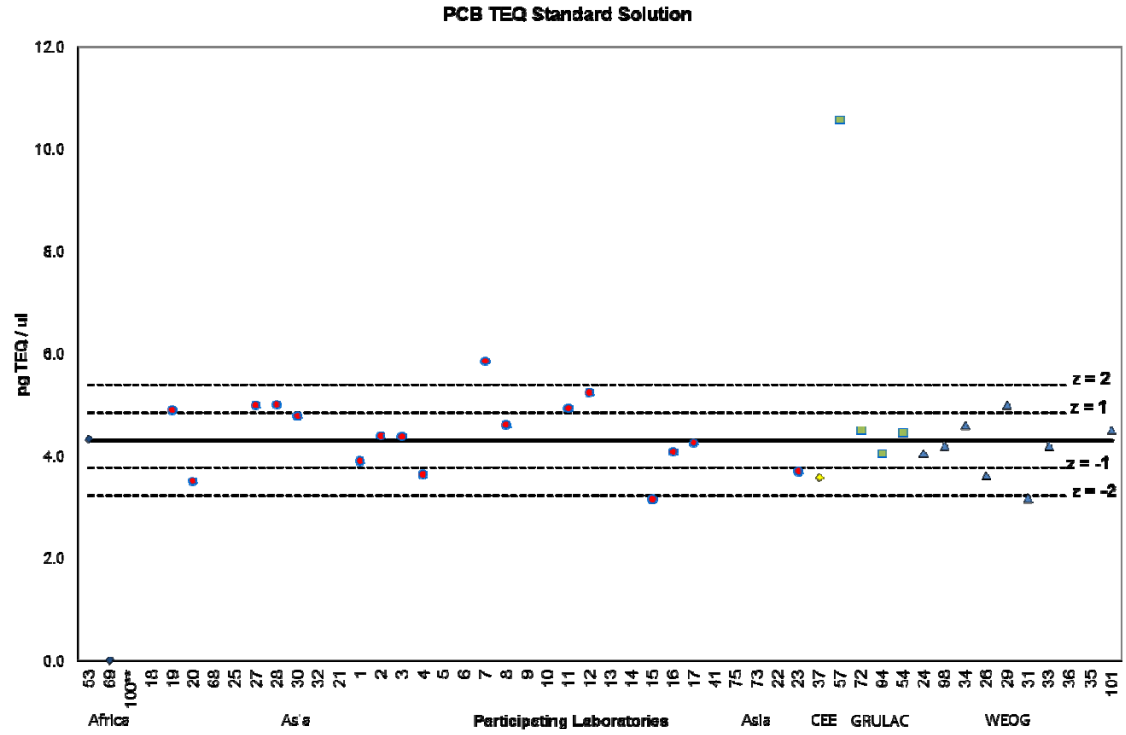


Figure 9 Results for the dl-PCB TEQ in the standard solution.
Laboratory code on the x-axis, concentration on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines.

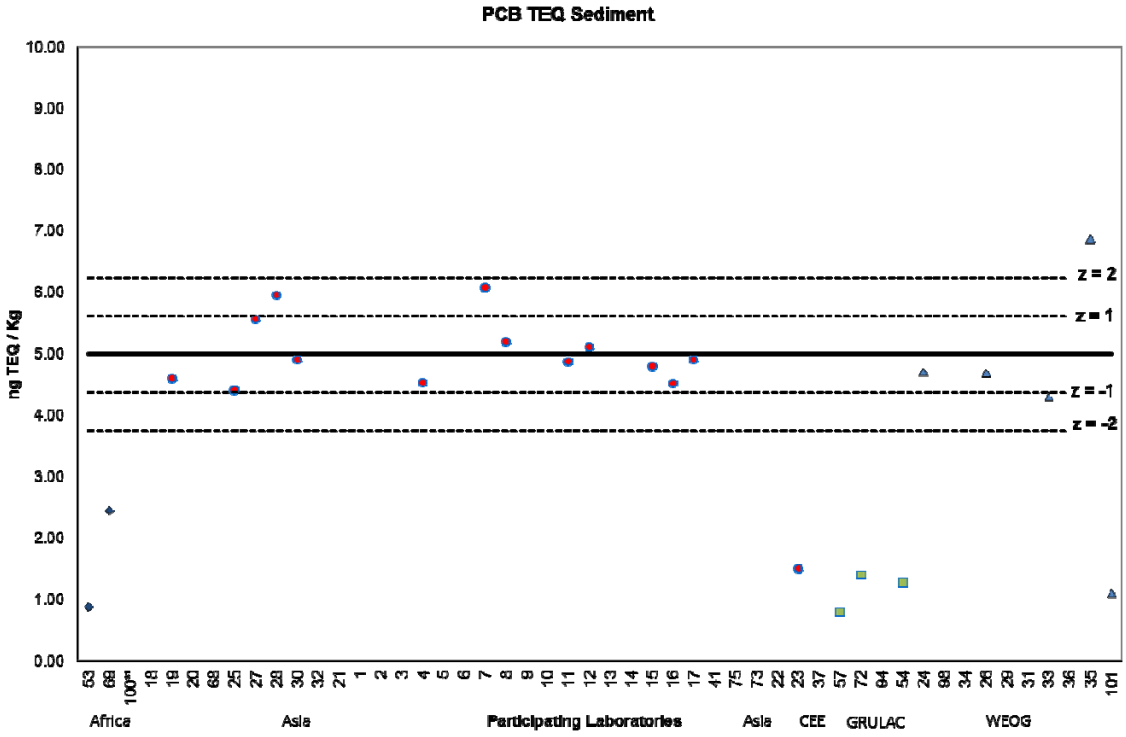


Figure 10 Results for the dl-PCB TEQ in the sediment sample.
Laboratory code on the x-axis, concentration on the y-axis. The assigned value is given by the straight line, z = ± 1 (12.5%) and z = ± 2 (25%) are given by the dotted lines.

The variation in the dl-POP data is in agreement with what is reported in the literature, *e.g.*, when more than 15 years of 'dioxin' QA/QC studies were evaluated to establish 'fit for purpose' RSDs (Van Bavel *et al.*, 2008). The RSD values for PCDD/PCDF and higher chlorinated PCB in milk are very good, but it should be taken into consideration that data was only submitted by a limited number of laboratories. For the fly ash the PCDD/PCDF RSD values were found acceptable. However, a substantial number of laboratories is still producing unacceptable results and further training and attention to QA/QC is needed to improve this.

4.3 Regional Performance

In the following section the performance per region (Africa, Asia, CEE, GRULAC and WEOG) are discussed with respect to the 'regional' RSD. Although such an evaluation gives valuable data on the analytical performance in each region, this data should be used with care because only a limited number of laboratories submitted data for some regions, sample types or target compounds. For example, most data for the dl-POPs was submitted by laboratories from Asia and the WEOG, while in the other regions only 1-5 laboratories submitted data. On top of that, for reasons of better illustration of differences between regions, uncorrected RSD values are used in these graphs.

4.3.1 Organochlorine Pesticides

The organochlorine pesticides show a fair distribution of the number of laboratories in the different regions. This is illustrated in Figure 11 for DDT and its metabolites for the standard solution. For the standard the results were good for WEOG, Asia and CEE (RSD < 35%), and reasonable for Africa (RSD > 45%) somewhat large for the GRULAC region (> 60%) when taking into account that for a z score = 2, only a RSD of 25% is allowed. Similar results were seen for the chlordanes, drins and other OCPs in the standards solution.

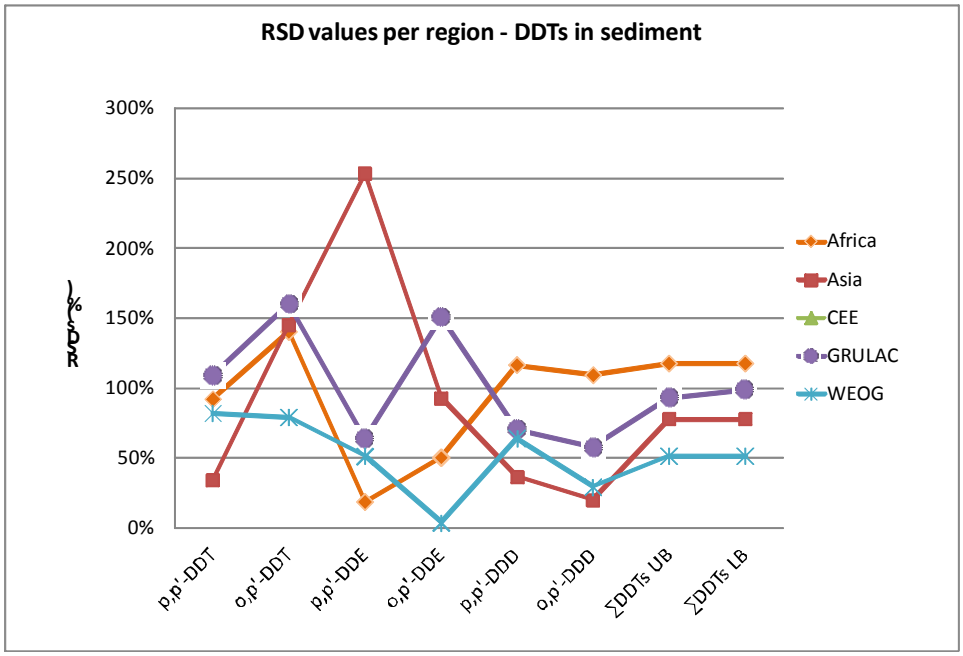


Figure 11 Regional variation in % RSD calculated from the raw data for the sediment sample of DDT and its metabolites.

For the samples the situation is worse as illustrated in Figure 11 for the DDTs in the sediment sample. Here several individual compounds showed RSD values > 100%. For the sum of DDTs (UB or LB) these RSDs were better and <100% for GRULAC, Asia and WEOG but still relatively high for Africa (RSD >100%). Again, similar trends were seen for the other OCPs, including the drins, chlordanes, mirex and HCB.

The results for the fish and milk showed larger differences between the different regions. This is illustrated in Figure 12, in which the results for the chlordanes in fish are shown. The results for the Asian region were very good, but the results for both Africa and the WEOG were relatively poor, and the results for the GRULAC region showed the largest variation (RSD > 200%). This might be due to calculation or unit errors but this has not been confirmed when the data was sent for control. For the CEE region only one laboratory submitted data.

For the milk relatively few results were submitted showing good results for some regions depending on which compounds were analysed. For several compounds (mirex, HCB, dieldrin and *p,p*'-DDE) the results were good for the WEOG and the Asian region, but again somewhat higher RSD values were found for Africa and GRULAC.

The scarcity of submitted results and the variance within the regions allow identification of needs for improvements. Targeted training can thus be directed towards filling these gaps.

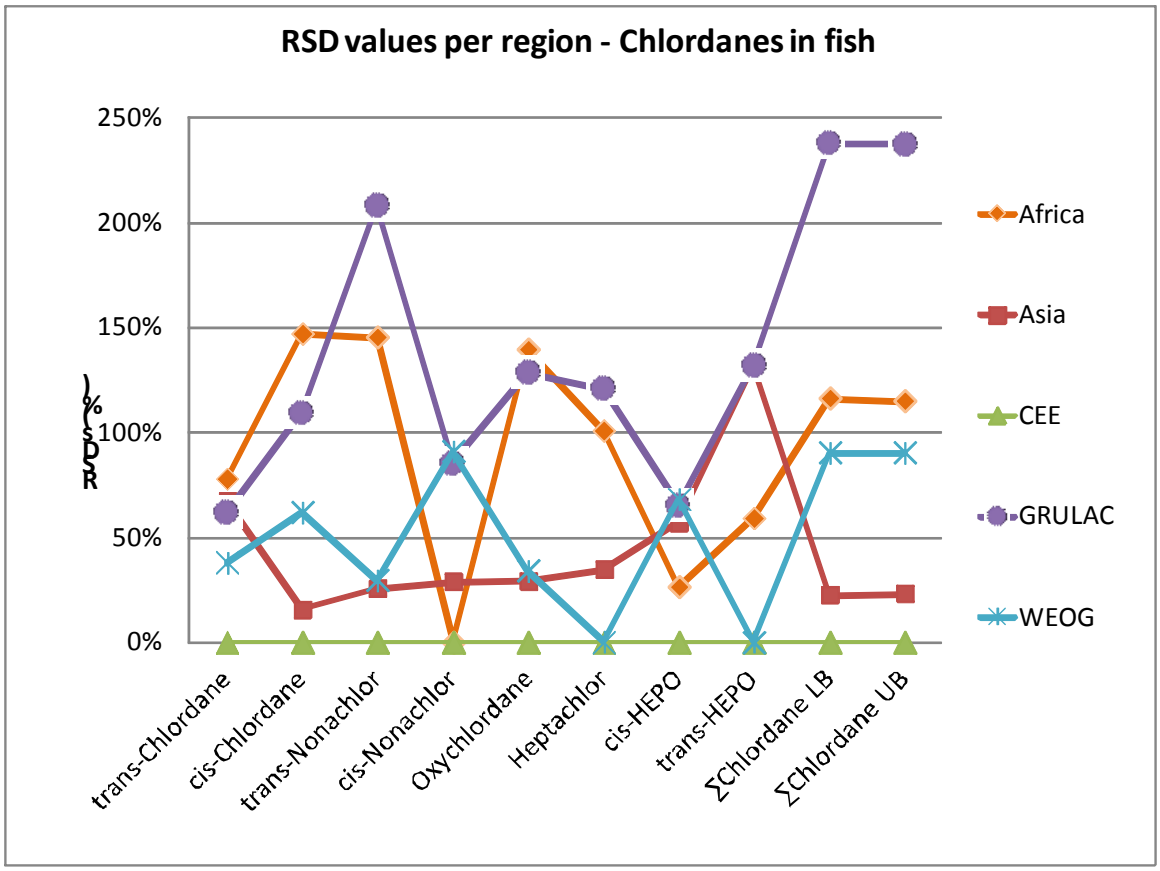


Figure 12 Regional variation in % RSD calculated from the raw data for chlordanes in the fish sample.
The CEE group consisted of only 1 lab, therefore no RSDs could be calculated.

4.3.2 Polychlorinated Biphenyls

The results for the marker PCB for the standard solution were good for all regions, with the exception of PCB 118, for which a large RSD was observed for the Asian region only, due to several extreme outliers for that region (Figure 13). The results for the sediment sample showed a similar trend and the RSD was also higher for PCB 118 for two regions (GRULAC, Africa). Overall the results for sediment were good for WEOG and Asia, showing RSDs < 40%.

The results for the marker PCB in fish showed somewhat larger RSDs for both WEOG and Asia (RSD around 50%) and CEE and Africa (around 100%). The results for GRULAC deviate somewhat, and while large individual variation is seen for some of the PCB (PCB 101, PCB 118, PCB 153, PCB 138, and PCB 180). On the other hand, the results expressed as Σ_7PCB for both UB and LB, are very good with RSDs below 50%.

For PCB in milk few results were submitted. A large variation is seen for especially the lower chlorinated PCB including PCB 28, PCB 52 and PCB 101. One of the reasons might be that just these congeners are present at relatively low concentrations in human samples compared to the more abundant congener such as PCB 153 and PCB 180 which show larger bioaccumulation.

The analysis of PCB congeners still is a challenge for many laboratories. However, in comparison with the RSDs for the OCPs, the results for PCB are better, especially when expressed as sum of 7 PCB congeners. Identification and quantification of PCB 118 seems to be a challenge.

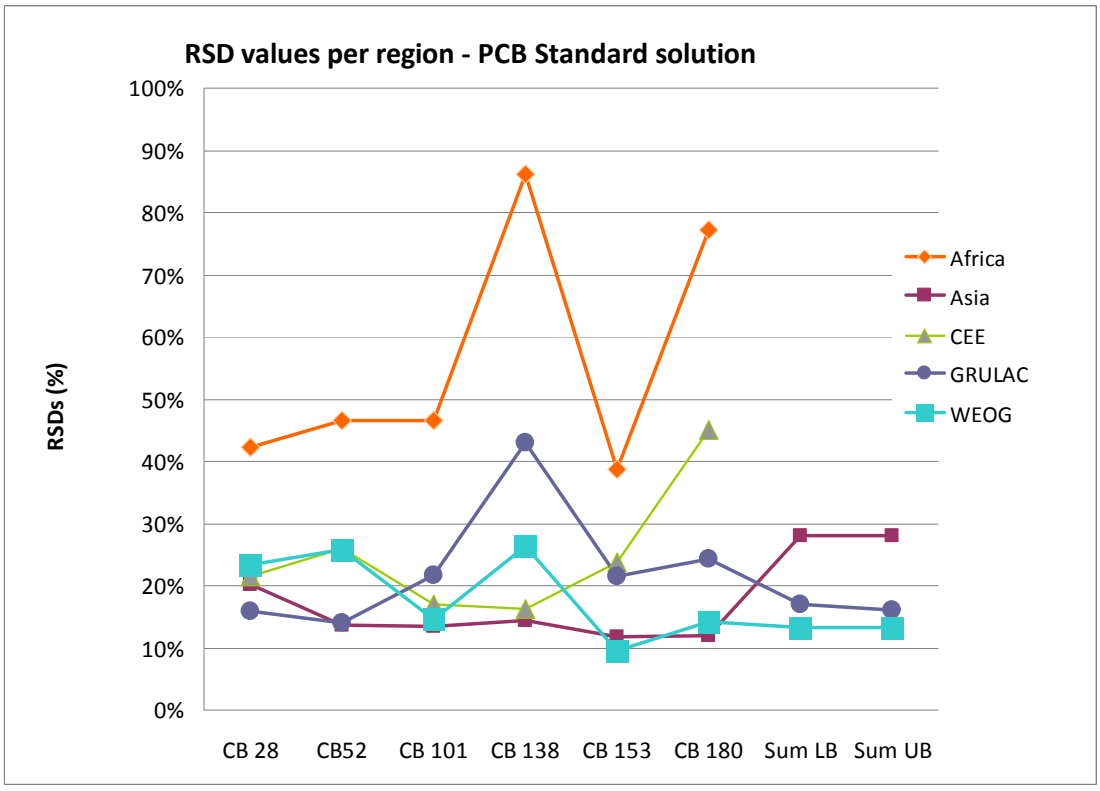


Figure 13 Regional variation in % RSD calculated from the raw data and the consensus value for PCB in the standard solution

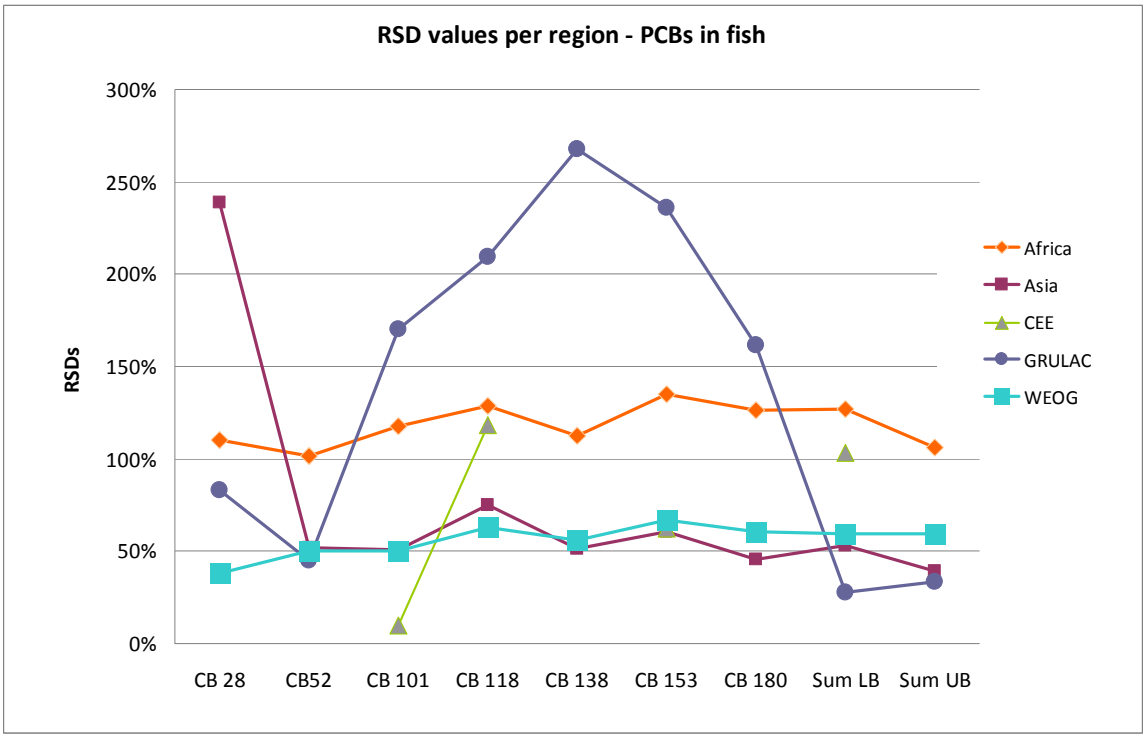


Figure 14 Regional variation in % RSD calculated from the raw data for PCB in the fish sample.

4.3.3 Dioxin-like POPs

The results for the dl-POPs were good, in particular for the standard solution. The results for the PCDD/PCDF were generally under 15% for all congeners for the participants from Asia and WEOG. The variation for GRULAC was somewhat higher but RSD values were still below 35% for nearly all congeners (Figure 15). No RSDs were calculated for the CEE and Africa because of too few results were submitted. The results for the dl-PCB showed a similar regional variation for the analysis of the standard solution, again with somewhat higher RSDs for the GRULAC region.

The results for the fly ash were good and in agreement with earlier studies showing RSDs around 30% for individual congeners and improved to less than 25% for the TEQ value. For the individual congeners 1,2,3,7,8,9-HxCDF showed a significantly higher RSD (> 100%), which might be due to a misidentification of this congener. The results for the dl-PCB was good for the WEOG and the GRULAC region but showed large RSDs for some of the PCB congeners in the Asian region.

For the sediment sample the results for the PCDD/PCDF were good, especially for the total TEQ (RSD < 25% for all regions), but the large RSD for 2,3,7,8-TeCDD indicated some problems, especially in the Asian region. Also here, larger variation and in many cases RSDs > 100% were seen for the Asian region, influencing also the total variation between all laboratories negatively because many of the reporting laboratories were located in this region.

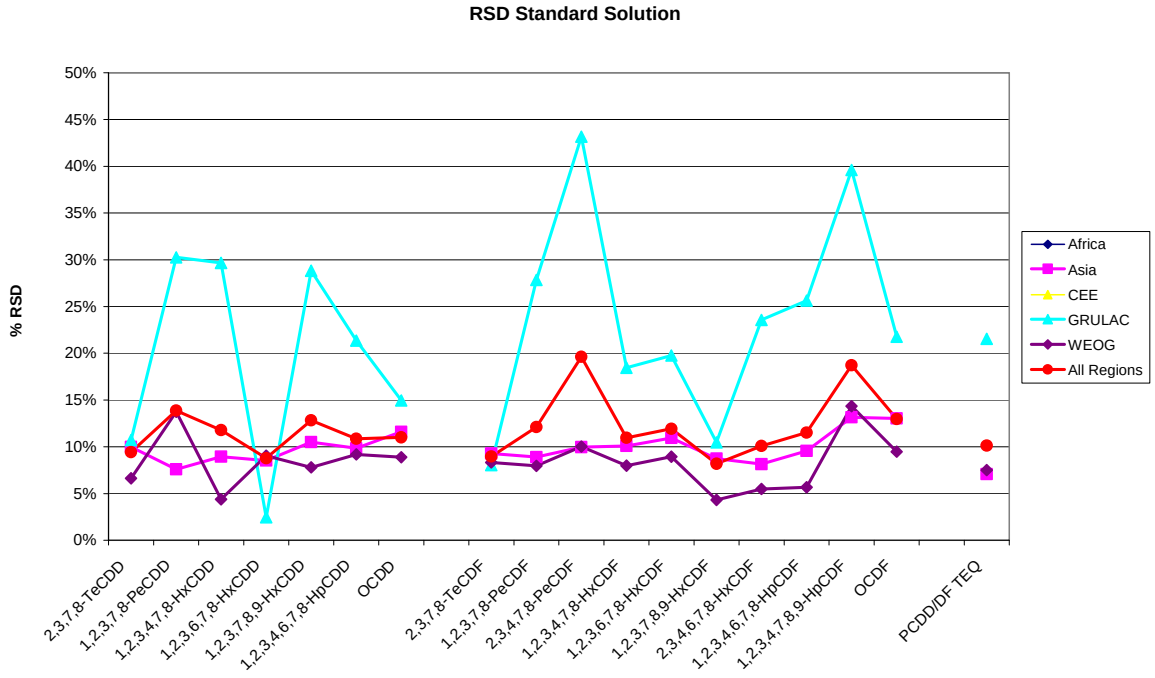


Figure 15 Regional variation in % RSD calculated from the raw data and the consensus value for PCDD/PCDF in the standard solution.

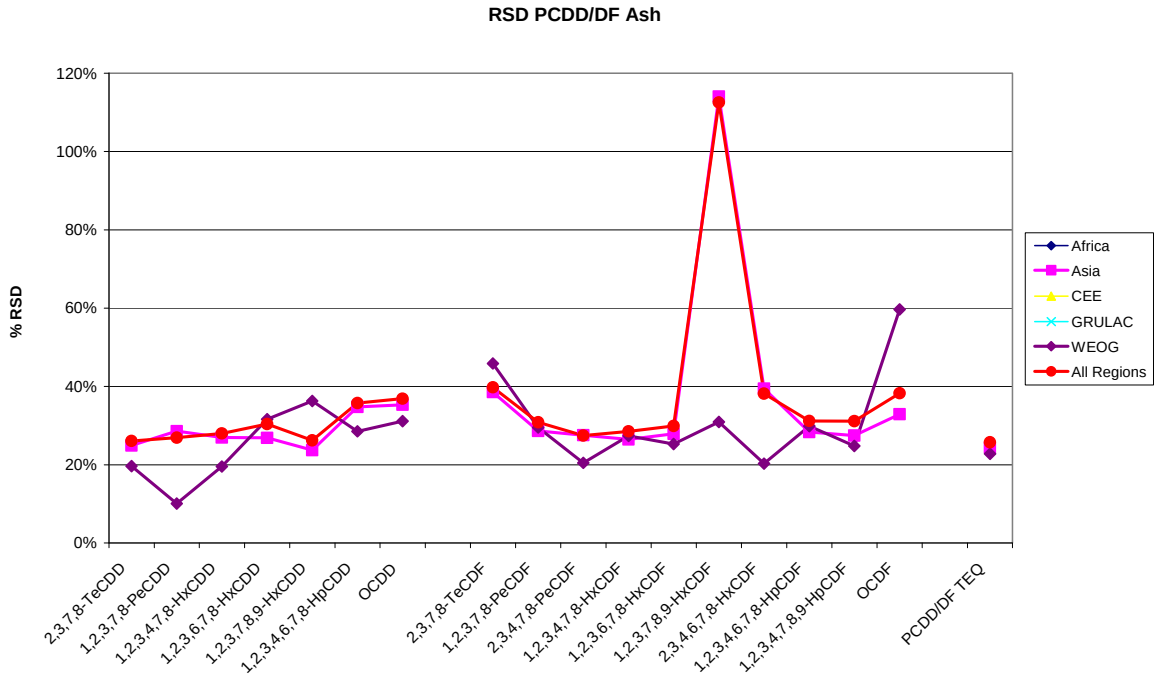


Figure 16 Regional variation in % RSD calculated from the raw data and the consensus value for PCDD/PCDF in the ash sample.

The data for PCDD/PCDF in the fish samples showed good agreement on the TEQ results (RSD < 50%). But, as can be seen from Figure 17, the individual congeners showed much larger variations with extreme values up to300% for OCDD. However, the TEF of OCDD is relatively small and this variation between the laboratories was not reflected in the total

TEQ. Also for this sample the dl-PCB data showed more variation with the best results for the WEOG region (RSD <40%) followed by GRULAC (RSD < 100%) and Africa and Asia (RSD < 140%).

For the milk sample the PCDD/PCDF TEQ concentration showed good agreement while the individual congeners varied sometimes quite a lot, especially the HpCDD and OCDD. The dl-PCB results showed large variations for two of the four dl-PCB congeners (PCB 77 and PCB 81) and two of the mono-ortho substituted PCB (PCB105 and PCB 123).

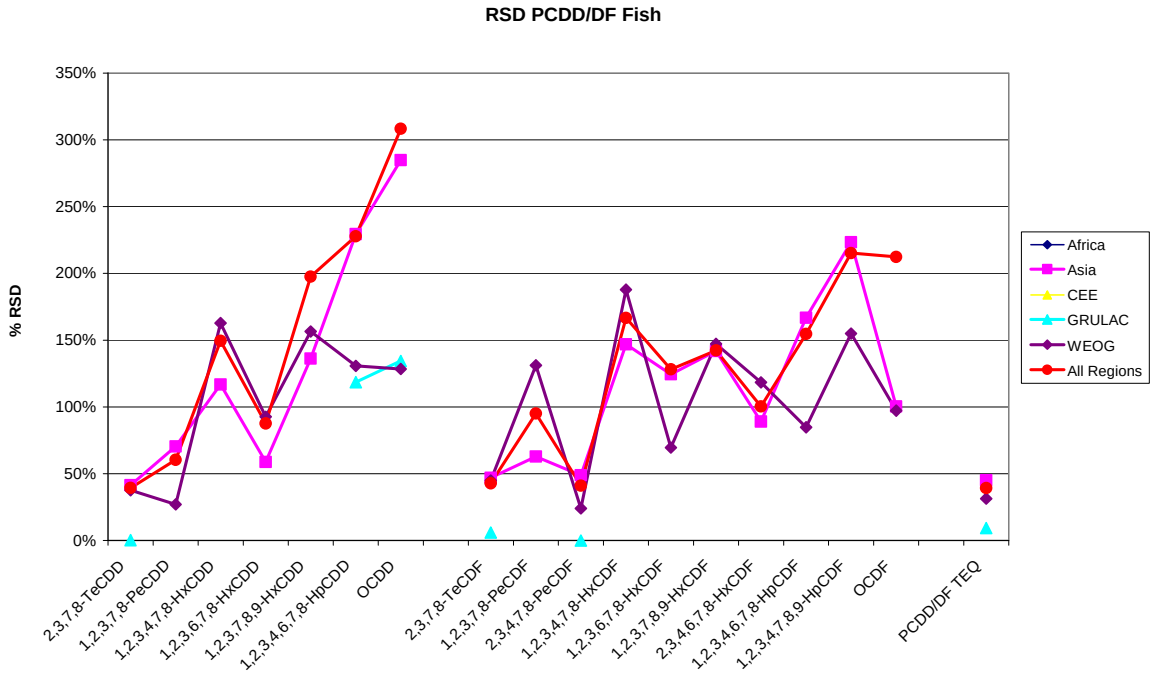


Figure 17 Regional variation in % RSD calculated from the raw data for PCDD/PCDF in the fish sample.

Overall, the performance of the dioxin laboratories was much better than for POPs laboratories analyzing basic POPs, *i.e.,* organochlorine pesticides (OCPs) or indicator PCB. Although the final target of RSD of 25 % could not yet been achieved for all laboratories and all matrices. The highest abundance of dioxin laboratories was found in Asia (dominated by the high number of dioxin laboratories in China) but also in other UN developing country regions, *i.e.,* GRULAC and Africa, laboratories could be identified that are willing to take on board the challenge of analyzing dl-POPs.

4.4 Comparison with Other Interlaboratory Studies

Analytical interlaboratory variability in POPs analysis is well documented (*e.g.* Mizikiewicz and Gibbs, 1992; de Boer and Wells, 1997; Holst and Müller, 2001, Rimkus *et al.*, 1993; Boekholt, 1993; de Boer *et al.*, 1996).

The laboratory performance of PCB in the test solution showed clear improvement (between-lab CV values 12%-19% and uncorrected RSDs 19%-36%, Table 9) compared to a previous interlaboratory assessment including seven participants (average uncorrected RSD = 57% except for PCB 101) (de Boer *et al.*, 2008), suggesting a better calibration of

equipment and instrumental analysis. For OCPs, the difference was slightly less, 22%-73% *vs.* 49% for uncorrected RSDs, respectively.

In sediment and fish, the results were in-line with the interlaboratory assessment of 2008. For the sediment, the laboratories participating in the present assessment had average uncorrected RSD values of 42% and 81%-241% for PCB and OCP, respectively, in comparison to 150% and 130% in the 2008 study (de Boer *et al.*, 2008). In fish the results corresponded even better, the present laboratories reporting average (uncorrected) RSD values of 81%-89% for the drins and the DDTs and 66% for PCB, in comparison to 65% and 90% in 2008 (de Boer *et al.*, 2008). The present results are slightly better than those of an interlaboratory study led by the International Atomic Energy Agency (IAEA), which reported RSD values between 30% and 150% for PCB and OCP in mussel homogenate (Villeneuve *et al.*, 2004). However, when compared to recent (mainly European) studies such as those of QUASIMEME, the present results are poorer (de Boer and Wells, 1997; and references herein). Also, comparing with some of the first interlaboratory studies on PCB and OCP in Europe, reporting CVs of 39% and 41% (both mean PCB) (Uthe *et al.*, 1988; Anon., 1993), the results presented here are weaker.

The results for PCDD/PCDF and dl-PCB were good and in agreement with and in some cases better than previously reported for this complex analysis (van Bavel *et al.*, 2008) except for the dl-PCB in the fly ash, fish and milk sample.

Considering this is the first Global Interlaboratory Assessment on POPs, including 103 laboratories and considering the status and difficult working conditions of many of the participating laboratories, the outcome is encouraging. The comparison with earlier studies in Europe shows that there is still a gap to bridge, but this was the *first* worldwide assessment and many of the participants performed for the first time in such a study.

5. CONCLUSIONS AND RECOMMENDATIONS

An overall reasonable-to-good performance on the test solution indicates that calibration and instrumental analysis is satisfactory at most laboratories. A number of laboratories struggled with the analysis of 'real' matrices such as sediment, fish and human milk. To be able to reach the overall goal of an analytical variation of only 25% between the participating laboratories (z = 2) to be able to establish and assess a 50% reduction over a ten year period, the analytical capacity and quality needs to be improved in several regions (GRULAC, CEE and Africa).

Poor performance was rather related to a variety of reasons than to one or two specific parts of the analysis. Laboratories were sometimes biased for certain samples only, sometimes for one or two contaminant groups and sometimes for all contaminants. Specific contaminants from the OCP group (*e.g.,* dieldrin and endrin) are vulnerable to degradation during extraction and clean-up as well as a dirty GC system. In addition, ECD detection is commonly used for detection of OCPs and because of interferences, inaccurate results can easily be obtained. It is assumed that application of GC/MS systems would substantially improve the OCP results.

In general, the performance of WEOG laboratories and Asian laboratories for the dl-POPs was somewhat better, although occasional outliers were observed also for this group.

None of the 103 participating laboratories was able to carry out all analyses that were offered in this assessment. This shows that none of the laboratories has the disposition of methods for all Stockholm Convention POPs for all samples types.

With respect to logistics, the overall delivery of the samples by an international carrier went well except for some minor hold up of some of the samples at customs in some countries.

Several regions and countries were under represented concerning the analysis of several of the compound classes or sample types. In the case of dioxin-like POPs in Asia, in particular China was overrepresented, while within GRULAC limited capacity was available and for Africa and CEE only one laboratory could do a full dioxin analysis.

The results of this assessment emphasise the need for **all** laboratories to pay more attention to quality assurance (QA) and method development. 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 estimated that several rounds of the present assessment will be needed to obtain a reasonable-to-good comparability of POP laboratories world-wide. Frequent discussions in workshops, mutual exchange programmes, e.g. per continent and provision of training and information on methods and QA/QC will be essential to make the desired progress.

Recommendations

Based on the results in this assessment, a series of recommendations can be made:

1. Follow-up through subsequent interlaboratory studies is needed to monitor and improve the overall level of performance of POPs analysis of the participating laboratories.
2. To centralise the submission of the results in order to minimise transcription errors and facilitate efficient submission of the results.
3. More laboratories should receive training, either in their own laboratory or in an expert laboratory or in a combination of these two options, preferably for a substantial period, in order to learn all details of the POP analysis and build up experience in this type of analysis.
4. Participating laboratories should maintain and improve the level of expertise in their laboratory by ongoing and frequent POPs analysis.
5. Laboratories analysing OCPs are encouraged to use GC-MS and ^{13}C labelled standards to improve their analysis
6. Participating laboratories are encouraged to train their own technicians by repeatedly analyzing certified reference materials and internal laboratory reference materials.
7. A second round of this interlaboratory assessment should also include an air sample or an extract from an air filter, as ambient air is one of the target matrices of the Stockholm Convention's Global Monitoring Programme.
8. Interactive workshops – through Webinars or on-site with the participating laboratories – should be organized to improve understanding and interpretation of the results and to dissimilate the lessons learned.
9. The next interlaboratory intercomparison assessment should include the newly listed POPs, such as HCHs, polybrominated diphenylethers, perfluorinated octylsulphonate, endosulfan, chlordecone and hexabrominated biphenyl.
10. In particular for the PBDEs and PFOS additional information and, if possible, training should be provided to the participating laboratories.

6. ACKNOWLEDGEMENTS

Cambridge Isotope Laboratories is gratefully acknowledged for providing the OCP standard solution, Wellington Laboratories for providing the PCB, PCDD/PCDF and dl-PCB solutions, and WEPAL for delivering one of the test materials. Prof. Wim Cofino is gratefully acknowledged for performing the statistical analysis. Dr. Eric Reiner is acknowledged for providing the fish material. The authors thank Dr. Heidi Fiedler of UNEP for supervising this project and for her valuable comments on this report.

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8. APPENDICES

Appendix I - List of Participants

Appendix II – Original Data

Appendix III – z-Scores

Appendix IV – Statistical Evaluation

Appendix V – Graphs z-Scores

Appendix VI – Regional z-Scores

Please note: Appendices II to VI are electronically available from the UNEP Chemicals Branch's WebSite.

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