

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**

26-28 February 2010

Final Workshop of First Worldwide UNEP Intercalibration Study on POPs — Asia Region
Hong Kong, 26-28 February 2010



**UNEP/DTIE
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Listing of presentations (accessible at <http://www.chem.unep.ch/Pops/GMP/Asia/default.htm>)

- 0A: UNEP – Context and Elements of the Project
- 0B: Bert van Bavel: International intercalibration studies as a QA/QC tool
- 0C: National Institute of Nutrition and Food Safety - China CDC
- 01: CDC-China
- 02: CPCB-India
- 03: JESC-Japan
- 04: RCEES-China
- 05: DICP-China
- 06: GIG-China
- 07: USP-Fiji
- 08: HKGL
- 09: IERM-China
- 10: ITPC-China
- 11: JSEM-China
- 12: NEMC-China
- 13: NIST_CSIR-India
- 14: CNEAC-China
- 15: VRTC-Vietnam
- 16: S-APM-China
- 17: Baosteel-China
- 18: SCIES-China
- 19: THU-China
- 20: CETASD-Vietnam
- 21: ZEMJ-China
- 22: ZEMC-China
- 23: Shimadzu-Japan
- B1: IVM MTM Detailed presentation of results
- C2: IVM VU Amsterdam_New POPs
- C3: Shimadzu-Japan_Analysis of New POPs
- C4: MTM Örebro University_Methods for PFC

Final Results Workshop of First Worldwide UNEP Intercalibration Study on POPs – Asia Region

1 OPENING OF THE MEETING

The workshop was held at Regal Riverside Hotel Shatin, Hong Kong SAR, China, from 26 to 28 February 2010. The opening session took place in the evening of 26 February 2010 and was chaired by Dr. Zongwei Cai, Chemistry Department of Hong Kong Baptist University, the organizer of the workshop. The workshop participants were welcomed by Professor Rick Wong, Dean, Faculty of Sciences, Hong Kong Baptist University, who highlighted the long-standing cooperation between his university and UNEP Chemicals on POPs issues, when the Asia regional report of the UNEP-GEF project “Regionally Based Assessment of Persistent Toxic Substances” was coordinated by his university.

Dr. Heidelore Fiedler, UNEP Chemicals, welcomed the participants on behalf of the United Nations Environment Programme (UNEP) and as project manager for this project. She thanked the Norwegian government for funding this project and Hong Kong Baptist University for convening this workshop. She was very pleased to see that the objectives of the project have been achieved and that with this Final Workshop, the results of the UNEP Worldwide Intercalibration Study for POPs could be presented and discussed. She expected that important conclusion could be drawn from this project. She referred to the project’s webpage at <http://www.chem.unep.ch/Pops/GMP/Asia/default.htm> where the report of the inception workshop and its presentation can be accessed and also this workshop report will be placed.

Participants introduced themselves and their relation to POPs analysis or environmental monitoring. The list of participants is attached as Annex I. University Sains Malaysia, Malaysia, although was invited, was not able to attend.

The workshop agenda included presentations of all 22 laboratories present as well as summarizing presentations by the two expert laboratories. Day 3 discussed the issue and challenges posed by the nine new POPs that were adopted at Stockholm COP4. The workshop programme is shown in Annex 2. The program proceeded accordingly.

2 INTRODUCTORY PRESENTATIONS

This session was chaired by Professor Zongwei Cai, Hong Kong Baptist University.

2.1 Objectives of the UNEP Workshop

Dr. Heidelore Fiedler updated on the UNEP activities with respect to POPs analysis such as four UNEP/GEF and two UNEP SAICM QSP projects that started at the end of 2009. These projects started in 2009 and are expected to deliver main outcomes in 2011. Information can be accessed at the following WebPage <http://www.chem.unep.ch/Pops/GMP/default.htm>. The laboratories from the developing countries involved in these projects will also take part in the UNEP Worldwide Intercalibration Study on Persistent Organic Pollutants and their results will complete the results from the Asian region. The presentation can be viewed in the Annex as Presentation 0A to this report.

2.2 Overview on the Intercalibration Data for Dioxins and dl-PCB

Dr. Bert van Bavel, MTM Centre, Örebro University, Sweden, presented the results from this intercalibration study in relation to dioxin-like POPs, namely PCDD/PCDF and dl-PCB. He reported that in total 37 laboratories from Asia, Europe and North-America participated in the study. Of these, 34 laboratories submitted data on the test solution, 30 on the sediment, 20 on human milk, 23 on fish and 24 on fly ash.

The results for the standard solution were very good with an RSD of only 8 % for the total TEQ. The PCDD/PCDF results were also good for both the ash and sediment samples based on the total TEQ showing a RSD of 24 % for both matrices. As an example for fly ash, the RSD were very good for the individual congeners; with the exception of 1,2,3,7,8,9-Cl₆DF where a RSD of 112% was obtained. When one extreme outlier was removed; there is still a RSD of 61%. Dioxin-like PCB in fly ash showed very large variation; when 2 outliers removed, the RSD was reduced to 24% (n=16). The PCDD/PCDF results for the total TEQ for the fish sample was satisfactory (33 %) taking all entries into account, after removing one extreme outlier this RSD improved to 18 % for the remaining 12 laboratories, which is exceptional good for this complex analysis. The same is applicable on the milk sample where the RSD for the total improved from 53 % for all 12 participating laboratories to 13 % after removing two outliers. This is all in agreement or in some cases better than reported in the literature when more than 15 years of 'dioxin' QA/QC studies were evaluated to establish 'fit for purpose' RSDs. The RSD values for PCDD/PCDF and higher chlorinated PCB in milk were far better. In fly ash the PCDD/PCDF RSD values were found acceptable, whereas the individual dl-PCB showed larger variation due to some extreme outliers (RSD > 300).

For dioxin-like compounds, less laboratories participated in fish (n=15) and even less in mothers' milk (n=12).

He highlighted that the performance error was set to 12.5%, which approach was taken from QUASIMEME. Setting the error to 12.5% rather than deriving an error from the dataset brings the major benefit that the error is independent of the dataset and will not be influenced by e.g. outlying values. The error of 12.5% is chosen so as to reflect the performance level

that should be reasonably achievable by laboratories for a specific analyte (group) and matrix combination.

The presentation can be viewed in the Annex as Presentation 0B to this report.

2.3 Overview on the intercalibration data of basic POPs

Prof. dr. Jacob de Boer, IVM VU Amsterdam, started by stating that interlaboratory studies are a complete blind test and therefore preferred towards analysis of certified referenced materials.

Results for the OCPs and PCB can be summarized that the overall good performance on test solution was that 67% of the participating laboratories stayed within 10% from the nominal concentration; 98% of the labs were within 20% from nominal concentration. Variable results were found for some matrix/POP combinations: Sediment, fish, human milk. For these matrices the minimum levels were acceptable but – with the exception for sediments – the maximum concentrations were too high. The good performance of most laboratories for the test solution suggests that instrumental sensitivity did not cause problems; rather, extraction, clean-up and resolution maybe the main sources of error. Overall, the performance for OCPs was poorer than for dioxin-like POPs: The largest deviance from the assigned value was seen for OCP; on average only 62 % of the data had a satisfactory z-score, as compared to 79 % of dl-PCB and 82 % for PCDD/PCDF.

He observed that some laboratories have treated the OCP extracts with sulfuric acid and therefore, the drins were destroyed and could not be detected (Caution: HCHs would also be destroyed).

The presentation can be viewed in the Annex as Presentation 0C to this report.

3 REPORT ON EXPERIENCES FROM PARTICIPATED LABORATORIES

The session was chaired by Professor Minghui Zheng, RCEES.

In summary: One laboratory (lab 20) had a consistently poor performance for all three contaminant groups. Four laboratories (labs 18, 23, 35 and 36) had a low performance for two of three contaminant groups, and ten laboratories (3, 4, 5, 8, 15, 19, 25, 31, 33 and 37) scored low on one of the contaminant groups.

However, the most important objective of the workshop is not necessary the result *per se* but what we can learn from this study.

Dr. Zongwei Cai's laboratory, HKBU, participated in the intercalibration study; but he was too busy with the workshop preparations that no time was left to prepare a presentation. He revealed his number for consultation.

3.1 Monitoring and Control of Contaminants and Residues, National Institute of Nutrition and Food Safety - China CDC

Dr. Yongning Wu on behalf of the two laboratories at CDC presented and discussed the results. In this intercalibration study CDC analyzed food and biological samples for PCDD/PCDF, dl-PCB, and POPs pesticides; the methods differed a bit between the two labs. Noteworthy is that one of the laboratories used HRMS for all analytes whereas the other did the detection with LRMS. He detailed the extraction and clean-up methods and reported on the differences by using different columns and between LRMS and HRMS detection. As expected, the HRMS method resulted in lower concentrations that could be quantified. He emphasized that international intercalibration studies are excellent tools for internal control and to identify deficiencies in the lab. The CDC laboratories are committed to participate in future proficiency tests including the new Stockholm POPs.

The presentation can be viewed in the Annex as Presentation 01 to this report.

3.2 Central Pollution Control Board, Ministry of Environment & Forest, India

Dr. Sharma detailed the methods used by his laboratory such as use of accelerated solvent extraction and HRMS for detection (at resolution of $>60,000$). The laboratory analyzed PCDD/PCDF in ash, sediment, standards 1 and 2 with HRGC/HRMS; indicator PCB and OCPs were analyzed with GC-ECD. The lab had reasonably good results for PCDDs/PCDFs in standard 1A as well as ash, while for sediments clean up needs review. He explained that his laboratory plans to move away from ECD detection and upgrade for GC-MS for marker PCB. The lack of calibration standards for individual marker PCB as well as internal dl-PCB standards was identified as major weakness. The laboratory participated for the first time in an intercalibration study with all POPs and the needs for improvement were identified especially for dl-PCB and indicator marker PCB.

The presentation can be viewed in the Annex as Presentation 02 to this report.

3.3 Japan Environmental Sanitation Center, Japan

Mr. Koichiro Matsumoto reported that his laboratory participated in all matrices. Typically; all standards were added before extraction, recovery standards for all groups of POPs, ash was pre-treated with HCl; Soxhlet or liquid-liquid extraction were applied. All compounds were measured by HRGC/HRMS; BPX Dioxin I and BPX Dioxin 2 for dl-POPs. 6-point calibration curves were established for quantification. Clean-up for sediment was identified as most difficult. Some problems were reported with heptachlor; recovery of DDTs was not good because it degraded in GC injector.

The presentation can be viewed in the Annex as Presentation 03 to this report.

3.4 Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, China

Dr. Gao presented the results for RCEES. The laboratory participated with all matrices. The ash sample – ofr PCDD/PCDF analysis - was pre-treated with HCl. Clean-up included multi-layer silica column (self-made); OCPs did undergo ultrasonic extraction and clean-up by GPC. Methods used included EPA 1613 for PCDD/PCDF (Wellington standard - LCS), dl-PCB EPA 16668A for dl-PCB (Wellington standard); and EC-5349 and EC5340 for OCP analysis (CIL standards added). Very good results were obtained for PCDD/PCDF and dl-PCB in ash, very good results in sediment and ash; for fish PCB 118 was much lower than the median; in milk occurred some problems; for OCPs good results in all matrices. Noteworthy is that OCPs were analyzed by HRGC/HRMS.

The presentation can be viewed in the Annex as Presentation 04 to this report.

3.5 Dalian Institute of Chemical Physics, Chinese Academy of Sciences, China

Dr. Jiping Chen reported that his laboratory analyzed PCDD/PCDF and dl-PCB in standard solution, sediment, and fish. For fish, the data are higher than the mean for PCDD/PCDF, some of the datapoints were out of the satisfaction range. There might have occurred some systematic error; especially since the profile is similar.

The presentation can be viewed in the Annex as Presentation 05 to this report.

3.6 Guangzhou Institute of Geochemistry, Chinese Academy of Sciences, China

Ms. Jianfang Hu presented the results for her laboratory, which submitted results for PCDD/PCDF on sediment, fly ash, and standard solution 3. Challenges did occur for clean-up procedure; time was too long and the material consumption is too high. AlOx column; Florisil cleanup modified.

The presentation can be viewed in the Annex as Presentation 06 to this report.

3.7 Institute of Applied Sciences, University of the South Pacific, Fiji

Waisea Votadroka, USP, stated that his university is a regional institution to cover 12 Pacific Island countries. POPs sub-unit is part of the Food and Water units; a Tier 3 laboratory that has GC/ECD systems. Draw-back include that the POPs laboratory has to share fume hood with other analysts, *i.e.*, can be used for POPs only when the other analysis are not using it. The POPs unit has own glassware but specific thinks like Soxhlet or Rotavap are shared with other Food/Water and organics units. First time, the laboratory has analyzed human breast milk. The laboratory obtained satisfactory results for PCB and OCP solutions. Further assistance is needed for fish, sediment, and human milk. Cross-contamination sources most likely might have caused problems. The POPs unit looks forward to a new room in the next

weeks; this was granted together with a new Rotorvap, which was granted upon recommendation from Dr. de Boer after an inspection tour in September 2009. Further future improvements include the acquisition of a Micro-ECD. The laboratory is confident to benefit from this workshop and future training. He proposed to have some little blank tests with other experienced labs in the Asia region and he hopes for further method development support from more experienced labs.

The presentation can be viewed in the Annex as Presentation 07 to this report.

3.8 Hong Kong Government Lab., Hong Kong SAR

Dr. Tony CH Lam reported that his laboratory participated in the analyses of dl-POPs in standard solution, sediment, and fly ash and OCP for standard solution, sediment, and fish. He briefed the meeting that that, DB-XLB was more suitable than DB-1701 for *cis*-nonachlor GC analysis. Also, using helium as carrier gas had given better separation than nitrogen. He also remarked that clean-up of UNEP sediment was critical and sufficient time was required to optimize the clean-up process. It was interesting to find that clean-up for op'-DDT analysis in the UNEP sediment resulted in some greenish residual left after Florisil and GPC clean-up. As such additional clean-up with aminopropyl column was required to remove those greenish residual. For the dl-PCB in the sediment analysis, the congeners were found to elute earlier than the corresponding instrument calibration standards for PCB 77 and PCB 81. After GPC clean-up, the retention times of the PCB 77 and PCB 81 of the samples matched with those of standards. Further, co-elution of PCB 123 and PCB 118 had been observed under DB-5MS column. The problem was solved satisfactorily by using HT-8 column.

The presentation can be viewed in the Annex as Presentation 08 to this report.

3.9 Institute for Environmental Reference Materials, China

Mr. Fang Liping, MEP/IERM, reported that his laboratories mainly used Canadian methods for the analysis of POPs. His laboratory used HRGC/LRMS for the analysis of PCB₇. Soxhlet extraction was used for the sediment. Overall, the experiences gained in this project were very I27 Jul 2009 PCB standard solution, 10 Aug 2010, 75 g sediment sample, PCB₇ done with isotope dilution and according to Canadian standard, Soxhlet extraction for clean-up. Thank for experiences gained in this project.

The presentation can be viewed in the Annex as Presentation 09 to this report.

3.10 Institute for Thermal Power Engineering of Zhejiang University, China

In general, the results are good, but some congeners in the flyash sample were not so good. One column was used. Some results were lower than the median value; HCl digestion was not used; PCB 105, 77 and 118 were higher than the mean or median value. Which column is best for dioxin and dl-PCB analysis.

The presentation can be viewed in the Annex as Presentation 10 to this report.

3.11 Jiangsu Environmental Monitoring Center, China

JSEMC, Hui Wang (Ms), participated in OCPs in sediment. Degradation of DDT on the column; some problems identified; checks necessary.

The presentation can be viewed in the Annex as Presentation 11 to this report.

3.12 Ningbo Environmental Monitoring Center, China

They have GC/ECD, GC/LRMS, and GC/HRMS. They analyzed PCDD/PCDF in flyash, standard, marker PCB and OCP in standard. Fly ash was pretreated with HCl. Fly ash the results were satisfactory. dl-PCB in fly ash was satisfactory. Dioxin laboratory is new and lacks experiences; therefore, only standard solution and fly ash were analyzed; hope to have more chances in the future to exchange with other laboratories. For PCB, they used PCB 209 as a surrogate. Results for marker PCB were not very good; twice lower than the median of the UNEP report; the recovery of the surrogate is 83.3% and they think that there is no problem with the clean-up but maybe with the ASE extraction; in the OCP were some problems, maybe the GC-ECD sensitivity is too low; the results did not appear in the report because they reported ND instead of <DL.

The presentation can be viewed in the Annex as Presentation 12 to this report.

3.13 National Institute for Interdisciplinary Science and Technology, India

Dr Anbu Munusamy reported that his institution has a small laboratory working on POPs from 2004 onwards; still not yet received the expertise. Received standard solutions, ash, sediment, fish, and mothers' milk; they have LRMS; they used Soxhlet extraction and LL for mothers' milk; SP2331 GC column used. For milk and fish they got very low values; considerably good results. They may have lost analytes during acid clean-up. They would like to train themselves to analyzing PCDD/PCDF in air, water, land, product and residue although they only have LRMS. From this training they understand that they need to have modern sample preparation techniques (extraction and acid treatment and clean-up), lack of instrumentation (HRMS needed), and needs for refining techniques (training from skilled laboratories).

The presentation can be viewed in the Annex as Presentation 13 to this report.

3.14 National Research Center for Environmental Analysis and Measurements, China

Ms Yue Ren, CNEAC; samples received were solution, fly ash, and sediment for dl-POPs and basic POPs. Analysis was done from 11 Sep to 30 Sep 2009 and dl-POPs in October 2009. Fly ash was pre-treated with HCl, DB5 column used. Each target compound was acceptable for the flyash. Duplicate analysis was done for PCD/PCDF; recovery is acceptable but not very well. For OCPs and PCB₇, LRMS was used.

The presentation can be viewed in the Annex as Presentation 14 to this report.

3.15 Research Centre for Environmental Technology and Sustainable Development, Vietnam

Dr. Duong Hong Anh, CETASD, target groups and matrices were OCPs, PCB, dl-PCB in sediment, fish, ash matrix; not analyzed were trans-nonchlor, cis-nonachlor, PCB 105. After Florisil column, acidic washing was applied to remove lipids and pigments (3x1 mL H₂SO₄ (98%) and 3x1 mL H₂O, dehydration with Na₂SO₄. phenanthrene and chrysene d12 was used for calibration. Identification and quantification was done with LRMS (Shimadzu). For test solutions z—scores were OCPs<2, PCB<2, dl-PCB ; but for the real samples: OCPs >6. For the fish sample there was a mistake in the calculation of the unit (ng/g dry fish sample and not ng/g lipid), column 60 m Rtx-PCB column was used. The study is a very good lesson for the lab; although they analyze OCPs for more than 10 years; performance of their in-house method needs to be improved. For PCDD/PCDF and dl-PCB they should adopt for suitable method.

The presentation can be viewed in the Annex as Presentation 15 to this report.

3.16 Shanghai Academy of Public Measurement, China

Ash, sediment, fish, and solution were analyzed. Recoveries are not too good; they only used one column for PCDD/PCDF, coelution for, *e.g.*, Cl₆DF? PCB 105 and PCB 118 gave problems; overloading for PCB 118? PCB concentrations were higher than those from most of the labs; the PCDD/PCDF were in good agreement. Since the results for PCDD/PCDF were all higher than the median, there may be cross—contamination in the lab from the fly ash sample; further consideration may be the use of only one GC column. Standard curves are a little flawed and this may have caused the bias. Still analyzing their data, hope to apply more advanced techniques in clean-up, expand the scope in POPs analysis.

The presentation can be viewed in the Annex as Presentation 16 to this report.

3.17 Shanghai Baosteel Group, China

Ms. Yongmei Yu from R&D Center; only flyash was analyzed for PCDD/PCDF; laboratory is quite new. Sample received 1 Aug 2009 and stored in refrigerator; analyzed on Sep xx. Presented each step of the analytical procedure to invite comments (since only one matrix analyzed). Wellington standard added. Results are a little higher than the medium value.

The presentation can be viewed in the Annex as Presentation 17 to this report.

3.18 South China Institute of Environmental Sciences, China

15 Aug 2009 samples were received, standards, fish, fly ash and sediment; submitted were data on PCDD/PCDF and dl-PCB. EPA 1613 was followed (in all steps). Results for dl-PCB were not submitted because a mistake was made when adding the OCB standard. For each sample, duplicate analysis was performed. Recoveries do not meet the requirement. The amount of each sample should be larger, especially for fish.

The presentation can be viewed in the Annex as Presentation 18 to this report.

3.19 Tsinghua University, China

Dr. Wu Changmin, samples were received in August/September 2009, and the analysis were finalized in October 2010. Organic solvent from USA, materials from Japan, standards from CIL and Wellington. For ash, HCl pretreatment was applied. H₂SO₄ not used for drins and heptachlor; instead (TBA)₂SO₃ treatment used for drins and heptachlor.. Instruments are HRGC Agilent and JEOL JMS 800D, columns BPX-DXN. Lab is accredited by China CMA; for their lab control RRF of up to 20% is acceptable. For the pesticides the recovery rate was not so good whereas there was no problem with the dl-POPs. Dual measurements were done for flyash and sediment. Typically, one duplicate is measured every batch of 10 samples. Most of the results are satisfactory; some problems with OCPs (heptachlor, oxychlordan, chlordan, DDD, DDT); also high detection limits for DDD, DDT, DDE. First time participated in intercalibration study; hope to future participation; suggest number of results to be reported.

The presentation can be viewed in the Annex as Presentation 19 to this report.

3.20 Vietnam – Russian Tropical Centre, Vietnam

Mr. Trinh Khac Sau informed that in 2012 the new building and new investment project for the laboratory will be finalized, but already in 2010 new equipment will arrive. In house methods were used based on US.EPA 8280A. Standards for dl-PCB arrived only in December 2009 from Wellington; too late for use in this study. They analyzed standard solution, fish, sediment, and fly ash. Z-scores of TEQ_{PCDD/PCDF} for standard solution, fish and fly ash were <1, sediment was <2. In fish, 7 congeners could not be quantified because of LRMS; recalculate sediment sample. Difficulty at VRCT lies with the limitation due to instrumentation for the biological matrices. Need training courses for the HRMS, need training in the core media (air, human milk, blood, stack emissions). Encourage participation in the intercalibration studies. Method building for the new POPs.

The presentation can be viewed in the Annex as Presentation 20 to this report.

3.21 Zhejiang Environmental Monitoring Center, China

Samples received were ash, milk, sediment (freeze-drying of the milk resulted in only 7.6 g of sample). Finally they only reported PCDD/PCDF, dl-PCB, and indicator PCB; milk and fish were not reported – this was the first time that the laboratory participated in such a study. The sample amount was too small to do the analysis. GC-ECD analysis: avoid the loss of some marker PCB (PCB 28, 52) and OCP. Double analysis and two columns used for dl-POPs. The marker PCB were much lower than the median; the results of the QA/QC sample was very low. For some dl-PCB the z-score was greater than 2. More training will be needed to analyze fish and milk.

The presentation can be viewed in the Annex as Presentation 21 to this report.

3.22 Shimadzu Techno-Research Inc., Japan

Dr. Takuma Takasuga detailed the results and experiences of his laboratory. His lab was one of the few laboratories that used HRMS detection for all analytes. Further, he mentioned that PCB 118 and PCB 28 were a bit further away from the assigned value. For the OCPs the concentrations were a little higher than assigned values, pp'-DDT a bit lower than assigned. Samples are Soxhlet extracted, flyash with pretreatment, liquid-liquid extraction was used for the milk sample. OCPs were separated on DB-17HT. All analytes were reported in all matrices. ¹³C₁₂ recovery – aldrin in HV samples very poor. CIL has new ¹³C₁₂ labelled mixture of OCPs. DB-5MS does not have same quality of separation for OCPs. Unstable POPs affect variation of sensitivity in GC-HRMS analysis: DDT, DDD, Endrin, Heptachlor. Strongly recommended use of ¹³C internal standards

Basically, Florisil clean-up applied. Cleanup is most important for HRMS analysis. DMSO/hexane. partitioning is effective to remove lipid, mineral oil and aliphatic hydrocarbons. Very high concentrations of major PCB, DDE, pp-DDD saturated peak. Sediment sample had large differences between concentrations of lowest and highest congeners, e.g., *mono-ortho* PCB#118 > *non-ortho* PCB#169 factor of 1,500; OCDD>TCDD = 10,000.

The presentation can be viewed in the Annex as Presentation 22 to this report.

4 DETAILED DISCUSSION OF THE RESULTS

First, it is worth to mention that participating laboratories were not restricted in the methodology used for the analysis of the target compounds in this first UNEP intercalibration study. The use of capillary GC was considered mandatory to achieve the separation needed for an accurate determination of the analytes. The laboratories used their own extraction and clean up protocols, spiking schemes, standards and internal QA/QC.

4.1 Presentations of the Intercalibration Results

The coordinators of the UNEP Intercalibration Study, Dr. Bert van Bavel and Dr. Jacob de Boer presented some detailed observations from the intercalibration study and asked for feedback. These include the following:

Typo in the report OCP concentrations were in µg and not ng.

Outlier removal ⇒ maximum RSD will go down. How to do? In Quasimeme, z-scores >6 are removed. After this morning presentations, the number of HRMS used in OCPs and PCB analysis have used GC-ECD or LRMS for these analytes.

Methods for PCBs (in sediment): ECD was used by labs 18 and 20. LRMS was used by labs 5 and 9. and HRMS was used by labs 4, 7, 8, 11, 12, 15, 24, 25, 26, 27, 28, 30, 33 and 35.

Methods for OCPs (in sediment): ECD was used by labs 6, 17 and 18. LRMS was used by labs 5, 9, 20 and 28 and HRMS was used by labs 4, 11, 12, 24, 25, 27, 30 and 35.

In most of the samples, lower PCB have more problems than higher chlorinated PCB. TCDD as all congeners in the standard solution works very well. There is no disagreement at all between the laboratories for PCDD/PCDF.

Some laboratories did not correctly calculate the TEQ (by using different TEF schemes). These have been corrected for all laboratories where necessary.

Human milk: problem was with PCB81 where no consensus value could be assigned. The concentration was very low and there is a lot of black (=disagreement between labs). However, the contribution to the TEQ is low.

Sediment PCB fraction CP Sil8 (or DB5) column; PCB138 (includes PCB 163, which makes about 15% of the peak). PCB153 in sediment caused some problems: Labs 35, 8, 3 reported too low, Lab 20 was too high. More problems were for PCB28 and PCB52.

It was observed that two laboratories found too high concentrations for 2,3,7,8-TCDD; maybe due to the fact that these were LRMS laboratories.

DDTs in sediment, op-DDT concentration very low and therefore problems (a general problem is the high difference in concentrations; recommendation: run the sample twice, once for low peaks and once for high peaks).

Among the OCPs, pp'-DDT caused problems for two laboratories; it is assumed that there is something wrong in the calibration curve (Labs 18 and 35). With respect to the drins, the results were very diverse and almost no agreement between laboratories. Dieldrin is always difficult and therefore, this result is not surprising. However, it should be noted that sulfuric acid treatment cannot be applied because it degrades the analyte. DMSO treatment is a recommended alternative.

Problems with the fish results occurred when labs reported on dry weight basis and not on lipid base – for two labs, the concentrations are too low.

Dieldrin in fish: there are several outliers; interferences, how to clean the second fractions.

pp'-DDE caused some problems at some labs, especially since the concentrations are high; bimodal distribution: one group too high, one group too low; the HRMS are one in each group. Further, some misreporting because of dry weight vs. lipid.

PCB153 in fly ash; outliers. The expertise of these labs is with PCDD/PCDF and therefore, the variation was too large, therefore, no value assigned.

H₂SO₄ was used by labs 11, 30, 35; KOH by 25 and 30; freeze-dried: 1, 2, 4, 24 (danger of cross-contamination, evaporation of volatile compounds). A possible solution is to cover the sample with a steel plate and make a hole into it to allow the water to evaporate but to protect the sample from cross-contamination.

A bit surprising was the high variation for PCB118 in flyash and sediment; lipid weight for fish and milk (mistakes in reporting); reporting methods for pesticides. Problem related to PCB105 and PCB52 often due to coelution (known). Cl₄DF known problem with coelution with DB-5MS column, polar and apolar column needed to separate it from other coeluting congeners (⇒ DB5 gives too high concentrations). 1,2,3,7,8,9-Cl₆DF sometimes writing mistakes because of sequence. DDTs degradation in the column is known; same for aldrin

and other drins. Calibration curves for high res are available from CIL; sometimes the RRF are okay, sometimes they are not within 15% of variability. More care on lipid determination.

Materials sent out should be larger to allow double or triple determination and on addition, the material can be used for further check.

4.2 Discussion on Problems, Difficulties, and Challenges

In the discussions, it was concluded that it would be useful to run the lipid determination also through the statistics.

With respect to terminology, it was clarified that “duplicate analysis” means that all steps starting from extraction to final identification was done twice (and not only two injections on the GC column were made).

Lively discussion was around the “UNEP criterion” to set 12.5% as acceptance criteria. Finally, it was concluded that more variation between laboratories could not be allowed since otherwise the Stockholm criteria – to monitor 50% decrease in concentrations over a 10 year period – could not be met.

Dr. Wu, CDC China, and supported by others highlighted that the IOMC logo would have more weight than just a UNEP logo and therefore, it should be attempted to have the final report with the IOMC logo. With such status, the successful participants in the intercalibration study could use the results for accreditation.

Environmental analysis vs. food analysis: best is to buy a second instrument and labs; cross contamination is too high.

If RRF is beyond the QA/QC criteria; resolve this problem first before starting analysis; try to find the problem; if not, then sample analysis does not make any sense.

Bert: Working on ion mobility and new technologies (← Waters). Looking into atmospheric pressure techniques → results for dieldrin look very good. May take 10 years.

With respect to time allowed for analysis of the test samples, it was found that 6-8 weeks were adequate although some laboratories submitted late. Some laboratories had difficulties with the amount of test sample supplied and requested to have more.

5 FINALIZATION OF THE REPORT - SUMMARY

It is also worth noting that the qualifying parameters were sums of several congeners or isomers within one group of POPs. Whereas the assessment is available for each individual congener, the participating certificate will refer to the sum parameters. More explicitly:

The qualifying values for the POPs are:

- for PCDD/DF: total TEQ (PCDD/PCDF only, using the WHO₁₉₉₈-TEFs)
- for dioxin-like PCB: total TEQ (dl-PCB only, using the 1998 WHO₁₉₉₈-TEFs)

- for PCB: sum of 7 indicator PCB
- all POPs pesticides with more than one substance (chlordane, DDT, heptachlor, and toxaphene): sum parameter of all parent compounds and transformation products on mass basis.

The schedule for this intercalibration study was/is as follows:

Jan-May 2009:	Preparation of test samples and standard solutions
Apr 2009:	Inception workshop in Beijing
Until 31 May 2009:	Registration to intercalibration study from other regions
July 2009:	Shipment of the intercalibration samples
Oct-Nov 2009:	Deadline to report results
Dec 2009-Jan 2010:	Evaluation of results and initial exchange with participating laboratories
Feb 2010:	Final results workshop in Hong Kong
Mar 2010:	Allow 2 weeks to comment back on correct information from the labs
May 2010:	Preparation of report from final workshop. Workshop report will be prepared as was for the inception WS; presentations in pdf will be placed on the UNEP WebPage
May-Jun 2010:	Preparation of final report of intercalibration study. UNEP will have the report circulated to obtain IOMC logo
Apr-May 2010:	Issue of the UNEP certificates (for TEQ and sum parameters as agreed in Beijing)
Jun 30, 2010:	Termination of the project; publication of the report at the UNEP Chemicals WebSite

6 OUTLOOK

The last day of the workshop concentrated on the new POPs and next steps in intercalibration. Four formal presentations were held followed by a general discussion.

6.1 New POPs

6.1.1 Emerging POPs – Status in China and Challenges

Dr. Guibin Jiang, RCEES, reported that nine new POPs were listed in the Stockholm Convention's annexes in May 2009 and that three more POPs are under review. According to Dr. Jiang, short-chained chlorinated paraffins (SCCP) are the biggest challenge. Most concern is with toxicity; restriction of these chemicals should be determined by the balance of the environmental impact and socioeconomic impact. Since all these compounds were proposed by developed countries, they are challenges for developing countries. For developing countries, schools, drinking water, *etc.* are first priority and typically chemicals managements are found further down the national priorities. As to the situation of the new

POPs in China: BFR production in China since 30 years, 70 kinds of BFR come from China. PFOS production has increased from 50 tons p.a. to 200 tons p.a., with half of it exported. HBCD is produced in 30 factories in Shandong, Jiangsu, *etc.* provinces. For SCCP, there are 140-160 plants, there is a huge market domestically and some are for export. NSFC is the principal funding organization for POPs in China, MOST support projects including fundamental research (program 863), MEP – investigation of occurrences of POPs (monitoring, standards, regulation, international affairs), CAS – attention to regional and global scale of POPs pollution problems (including e-waste issue in Guangdong province).

With respect to analytical capacity in his institution: RCEES analyzes all 12 POPs with HRGC/MS; for toxaphene method development is underway. PBDE fall into the same category (BDE 209 unstable on column). Use 2-D HPLC and HRGC/MS for 46 congeners. PFOS with HPLC with MS/MS; high sensitivity, interferences (PE). Separation of α -, β -, and γ -HBCD and TBC. SCCP: a GC-MS/MS method has been developed GC-EI-MS/MS (technique cannot differentiate MCCPs from SCCPs). Dual directions: reduce environmental pollution but also develop analytical techniques to determine the compounds accurately. Paper on e-waste on D2009 CD ROM (Bo YUAN *et al.*). In general, PBDE pollution is low in China; however, there are several hotspots in China; found in remote areas in Tibet (\leftarrow long-range transport); PFOS in blood is high (China is the highest); workers in Wuhan have same blood concentration like exposed workers at 3M. .

6.1.2 New POPs

Dr. Jacob de Boer, IVM VU Amsterdam, mentioned that among the PBB, the most relevant congener is the PBB153; this was the main congener in the technical mixture. DecaBDE analysis is difficult to analyze because of instability (remains too long in injector and breaks down). Separation can be done by single column GC combined with LRMS or HRMS (NCI, ECNI; with ECD there may be many interferences also from chlorinated compounds). HBCD maybe present in the sample (same degradation products can easily interfere with target PBDEs). PBDE separation by multi-dimensional GC (GCxGC-ECD); also “traditional POPs”, *e.g.*, toxaphene can be separated by 2D GC techniques. Open question which CPs to analyze. Proposal: not too go with technical mixtures because they will not stay in the environment; rather select some indicator congeners. Also for HCHs, a lot of experience but general poor performance; GC/MS with labeled standards is preferred. QUASIMEME workshop in Ostend, Belgium, 17-18 March 2010.

6.1.3 Analysis of New POPs, POPs candidates

Dr. Takumi Takasuga, Shimadzu, Japan, introduced a new POPs and POPs candidates trial: PeCBz, PBDEs, HBB use HRGC-HRMS(EI). Endosulfan, SCCP use HRGC-HRMS(NCI). PFOS/PFOA use LC-MS/MS. PFOSF use HRGC-LRMS. PeCBz Large problem in air sampling; low volume sampling is preferred. Photodegradation for some of the PBDE; PFOSF unstable in aqueous solution to form PFOS. PFOSF need special GC column because same GC Rt. with general solvent in normal GC column, same accurate masses with PFK (169, 219, 331); SCCPs complex mixture lack of ¹³C surrogate and limitation for congener separation. HV samplers with PUF and insert active carbon disc. Chlordecone all retained in QFF and nothing found in PUF or ACF; SCCP and endosulfane will go into PUF1. Clean-up scheme: Chlordecone needs different treatment (ACN/hex part) because too late elution in

column chromatography. Clean-up does not use H₂SO₄ or strong alkali except for PCB, PBDE and HBB; therefore, no degradation of certain compounds. HexaBB and PBDE: use narrow GC column (0.1 μm). BFR often poor linearity need ¹³C surrogate. Some BFRs also with LC-MS/MS. SCCPs better with NCI techniques. Endosulfane POPs candidates and chlordane in overlap/coelute HRMS(NCI).

6.1.4 New POPs , esp. Analysis and Sampling

Dr. Bert van Bavel, MTM Centre, Örebro University, Sweden, informed about research underway such as that the Swedish cross-country ski team uses PFC-containing waxes → source of environmental contamination and workers exposure. Producers are 3M, DuPont, Scotchgard (Teflon) Stainmaster carpet, etc. PFCs in humans; levels are 10-50 ng/mL of blood ← occupational exposure. Food exposure: fish/seafood (52%), meat-13%, vegetables 8%, dairy (20%) but have a very different behavior. Compounds preferentially found in human blood (and not human milk) and in water. PFOA also found in potatoes (1 ng/g); UK study. Clean-up for PFOS includes ion-pair extraction, different types of solid-phase extraction, dispersive active carbon clean-up; analysis and detection: LC-ESI-MS/MS (triple quad), (ion trap), LC-ESI-MS (single quad). Important measure two transitions rather than only one. PFOS: note there are one linear and 11 branched isomers. ISO 25101 from 2009-03-01 at ISO homepage – water standard. US found very high concentrations in chicken eggs and potatoes (Jessica Reiner); Örebro did not find any PFOS in Swedish eggs – paper is amended, they did not run two transitions and measured ; same for the UK diet study (re-published at DIOXIN2009; they did not find anything in the same potatoes).

2nd Worldwide QA/QC study on PFC: human serum A and B, standard solution. For serum RSD = 12% (n=14) and RSD = 16; in the standard solution RSD = 32%, n=11.

6.2 Discussion on Future Collaborations and QA/QC

The final session was chaired by Dr. Heidi Fiedler, UNEP, and addressed some thoughts on future collaboration and QA/QC issues. In order to stimulate the discussion, a thoughtstarter was developed.

6.2.1 New POPs to be recommended for monitoring purposes

HexaBB – which are the congeners of interest to be analyzed?

Although for the hexachlorinated analogues five congeners are listed as the target analytes (two for indicator PCB: 2,2',3,4,4',5'-hexachlorobiphenyl-PCB 138 and 2,2',4,4',5,5'-hexachlorobiphenyl-PCB 153; and four dioxin-like congeners: 3,3',4,4',5,5'-hexaCB-PCB 169, 2,3,3',4,4',5-hexaCB-PCB 156, 2,3,3',4,4',5'-hexaCB-PCB 157, and 2,3',4,4',5,5'-hexaCB-PCB 167), only PBB 153 has occurred in commercial HexaBB, therefore **it is suggested to only analyze PBB 153 for Stockholm Convention monitoring purposes.**

Polybrominated diphenyl ethers (penta- and octa BDE)

BDE #47 and #99, BDE #153, BDE#154, BDE#183

It was noted that BDE #175 might be a mistake, since this congener was not identified in commercial mixtures (see information on labeled standards by Wellington Laboratories).

PFOS, its salts, and PFOSF

Perfluorooctane sulfonic acid (CAS No: 1763-23-1), its salts, and perfluorooctane sulfonyl fluoride (CAS No: 307-35-7)

For example:

potassium perfluorooctane sulfonate (CAS no. 2795-39-3);
lithium perfluorooctane sulfonate (CAS no. 29457-72-5);
ammonium perfluorooctanesulfonate (CAS no. 29081-56-9);
diethanolammonium perfluorooctane sulfonate (CAS no. 70225-14-8);
tetraethylammonium perfluorooctane sulfonate (CAS no. 56773-42-3);
didecyldimethylammonium perfluorooctane sulfonate (CAS no. 251099-16-8)

The above listed compounds are listed as examples in the report of the Fourth meeting of the Conference of the Parties to the Stockholm Convention. It was noted that these are all linear PFOS; however, in commercial mixtures and in the environment branched congeners occur as well. Further, the listing of perfluorooctane sulfonyl fluoride (PFOSF) is important for the evaluation of production, but this compound does not occur in the environment or humans since it is transformed rapidly to PFOS.

Note that consensus has been reached to report on anion basis on the latest QA/QC study. It is not relevant to report PFOS on the different salt values.

Core matrices as approved by the Conference of the parties for the 12 initial POPs:

Ambient air: passive air samplers (PAS), few high-volume samplers (HV)
Mothers' milk: main activity
Human blood: few programs

In air only the telomer alcohols of the perfluorinated compounds are found, but PFOS is found in corresponding lake water. Therefore, water is the preferred sample matrix where the stable end product ends up. There is already data on water available, very limited on air, levels below LOD, it is not likely that that PFOS, as the salt, ends up in the air. This compound is soluble in water. It was suggested to propose steady water bodies such as lakes and oceans as a core matrix for some of the new POPs. In the literature, there were papers published where PFOS was monitored using passive samples (see Abad *et al.* from CSIC).

So far, mothers' milk was most used in the human monitoring programs, with blood there were some practical problems such as lower concentrations, handling in a chemical laboratory, customs. Since the concentrations are higher, the preferred matrix for PFOS would be blood, despite the problems with the practical handling.

6.2.2 Cooperation:

1. through IGO (Intergovernmental organization)

UNEP mandate: global guidelines, standards developing – preferentially at regional basis

International financing, especially for developing countries exist through the following mechanisms:

a) GEF projects: 1:1 cofinance minimum and up to 1:3 for investment projects; endorsement from GEF focal point is mandatory; recipient country must be Party to the Stockholm Convention

b) SAICM QSP (IGO not required): up to 250,000 USD; eligible are developing countries through different formats

1 single country

1 regional/multi-country

1 NGO (it should be noted that typically, more country-projects are approved than NGO-led projects)

c) Bilateral donor: the large donors are developed countries and bilateral projects/programs are supported

2. South-south Cooperation

3. Developing country – Academic institution

4. Developing country - Industry

6.2.3 Project suggestion

Identification of a network fro new POPs especially PFOS. CDC, MTM, IVM qualifying labs from different regions through existing QA/QC studies. See existing air sampling networks. Check capacity to analyse in Japan (Dr. Yamashita, Dr. Takasuga).

7 CLOSURE OF THE WORKSHOP

After exchange of the usual expressions of courtesy and thanks, the workshop was closed at on Sunday, 28 February 2010 at 12:30 hours.

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9 ANNEX 2: PROGRAM OF THE WORKSHOP

Final Workshop of the First Worldwide UNEP Intercalibration Study on POPs from Asian Region, Hong Kong, SAR, China, 26-28 February 2010

Program of the Workshop

Feb. 26, 2010 (Friday)		
<i>Regal Riverside Hotel Shatin</i>		
17:00-17:30	Registration	<i>The Forum, 1/F</i>
17:30-19:00	Welcome Dinner	<i>The Forum, 1/F</i>
19:00-19:20	Opening of the Workshop	Chair: Dr. Zongwei Cai
	Welcome	Prof. Rick Wong Dean, Faculty of Sciences, Hong Kong Baptist University
	Welcome	Dr. Heidelore Fiedler United Nations Environment Programme
19:20-19:30	Introduction of the participants	Dr. Zongwei Cai
19:30-20:00	Objectives of the UNEP Workshop	Dr. Heidelore Fiedler
20:00-20:45	Overview on the intercalibration data of dioxins and dl-PCBs	Dr. Bert van Bavel
20:45-21:30	Overview on the intercalibration data of other POPs	Dr. Jacob de Boer
Feb. 27, 2010 (Saturday)		
<i>Tai Po Room, 2/F</i>		
8:30-10:00	Report on experiences from participated laboratories (10 min each)	Chair: Dr. Minghui Zheng
	<ol style="list-style-type: none"> (1) Monitoring and Control of Contaminants and Residues, National Institute of Nutrition and Food Safety - China CDC (2) Central Pollution Control Board, Ministry of Environment & Forest, India (3) Japan Environmental Sanitation Center, Japan (4) Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, China (5) Dalian Institute of Chemical Physics, Chinese Academy Of Sciences, China (6) Guangzhou Institute of Geochemistry, Chinese Academy of Sciences, China (7) Institute of Applied Sciences, University of the South Pacific, Fiji (8) Hong Kong Government Lab., Hong Kong SAR (9) Institute for Environmental Reference Materials, 	

	China (10) Institute for Thermal Power Engineering of Zhejiang University, China (11) Jiangsu Environmental Monitoring Center, China	
10:00-10:20	<i>Coffee break</i>	
10:20-12:00	Report of individual participated laboratory (Cont.)	Chair: Dr. Yongning Wu
	(12) Ningbo Environmental Monitoring Center, China (13) National Institute for Interdisciplinary Science and Technology, India (14) National Research Center for Environmental Analysis and Measurements, China (15) Research Centre for Environmental Technology and Sustainable Development, Vietnam (16) Shanghai Academy of Public Measurement, China (17) Shanghai Baosteel Group, China (18) South China Institute of Environmental Sciences, China (19) Tsinghua University, China (20) Vietnam – Russian Tropical Centre, Vietnam (21) Zhejiang Environmental Monitoring Center, China (22) Shimadzu Techno-Research Inc., Japan	
12:00-13:30	<i>Lunch</i>	
13:30-15:30	Detailed presentations of the intercalibration results	Dr. Bert van Bavel, Dr. Jacob de Boer, Dr. Gunilla Lindström
15:30-16:00	<i>Coffee break</i>	
16:00-16:45	Discussion on problems, difficulties and challenges	Dr. Bert van Bavel, Dr. Jacob de Boer All
16:45-17:00	Finalization of the report - Summary	Dr. Heidelore Fiedler
18:00-21:00	Workshop Banquet <i>Peak Restaurant, Hong Kong Island</i>	
Feb. 28, 2010 (Sunday) <i>Tai Po Room, 2/F</i>		
9:00-10:30	New POPs	Dr. Guibin Jiang Dr. Takumi Takasuga Dr. Jacob de Boer Dr. Bert van Bavel
10:30-11:00	<i>Coffee break</i>	
11:00-12:00	Discussion on future collaborations and QA/QC	Dr. Heidelore Fiedler, Dr. Gunilla Lindström, Dr. Jacob de Boer
12:00-12:15	Concluding Remarks	Dr. Heidelore Fiedler
12:15-13:30	Lunch	
13:30	End of Workshop	