# Training Workshop on Laboratory Methods and Procedures for Persistent Organic Pollutants in Biological Tissues

19-20 January 2009

Reef Yucatan Hotel, Merida Mexico

## Caribbean Coastal Pollution Project (CCPP)

***Assessment, Monitoring and Management of Persistent Organic Pollutants (POPs) and Persistent Toxic Substances (PTS) in the Coastal Ecosystems of the Wider Caribbean Region***

## Summary Report

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UNITED NATIONS UNIVERSITY

UNU-INWEH

Integrating Watershed and Coastal Areas Management in Caribbean Small Island Developing States

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# I. INTRODUCTION

## Rationale

The Caribbean Coastal Pollution Project (CCPP) aims at the assessment, monitoring and management of Persistent Organic Pollutants (POPs) and Persistent Toxic Substances (PTS) in the Coastal Ecosystems of the Wider Caribbean Region (WCR) and commenced in September 2007. It is being funded by the World Bank through the Canada Persistent Organic Pollutants Fund from the Canadian International Development Agency (CIDA) as well as by UNU-INWEH.

The purpose of CCPP is to build a network among environmental managers, analytical laboratories, and other appropriate governmental agencies in countries of the WCR that will be key in measuring, evaluating and then reducing pollution from POPs and other PTS in coastal marine environments. These reductions can be achieved through changes to behaviour that stem releases of pollutants into the environment in upstream agriculture and industry.

## Capacity Building

One of the aims of the project is to evaluate laboratory capacity by visiting existing lab facilities and performing cross-calibration trials, implementing advanced training of laboratory personnel through training workshops and secondments of staff to Canadian labs, and also providing for instrumentation upgrades.

During May-June 2008, questionnaires were sent to all the participating laboratories to assess their current capacity in terms of staff, infrastructure, laboratory equipment and capacity needs to sample and analyze POPs and other PTS. The responses from these questionnaires were synthesized by an expert team and used as preliminary information for an evaluation report. The experts conducted ten laboratory visits during June-August 2008 and a Final Laboratory Evaluation Report was submitted in October 2008.

Specific training needs were identified during the laboratory evaluation exercise and included sample preparation and analysis, GC calibration, examples of different extraction and clean-up methods, risk assessment, instrument detection limits and method quantization limits and quality assurance and control. This training was the first training held in context of the CCPP. All PowerPoint presentations can be found as PDF files at: http://www.inweh.unu.edu/inweh/Coastal/POPs%20Monitoring/Merida/Presentations/1_Training.pdf or can be requested at: contact@inweh.unu.edu.

## Objectives of the course

The objective of providing training is to build the understanding and capacity of laboratory staff in our eight project countries in: 1) analytical and extraction methods for POPs in biological tissues; and 2) methods and procedures for laboratory quality control/quality assurance programs.

## II. TRAINER AND PARTICIPANTS

### About the Trainers

Four trainers with extensive experience in environmental POPs issues conducted the training:

- Dr. Chris Metcalfe, Trent University
- Dr. Ken Drouillard, University of Windsor
- Dr. Gerardo Gold, CINVESTAV Unidad Merida, Mexico
- Ms. Nargis Ismail, University of Windsor

### Selection of Participants

Invitees to the training workshops preferably have laboratory supervisory responsibilities and the ability to pass along lessons learned to personnel under their supervision.

## III. TRAINING COURSE ACTIVITES

Training was held at the Reef Yucatan Hotel in Merida, Mexico, from 19-20 January 2009 and the workshop programme is given in Annex 1 of this report. A total of 15 participants from the eight project countries attended (the list of participants is given in Annex 2).

### Course handouts

Course handouts comprised electronic versions of the presentations, as well as the following documents:

- Canadian Association for Laboratory Accreditation (Inc): T27 – CALA 17025:2005 Handbook - Revision 2.3 – December 2007
- GLIER - Analytical laboratories - Quality Manual
- GLIER - Quality System Procedures
- Standard Operating Procedures - GLIER Organic laboratory
- Analytical methods for PCBs and organochlorine pesticides in environmental monitoring and surveillance: a critical appraisal

These handouts are available at
http://www.inweh.unu.edu/inweh/Coastal/POPs%20Monitoring/MeridaMeetings.htm
or can be requested from contact@inweh.unu.edu.

During the morning of day 2 of training a practical laboratory exercise was organized at the CINVESTAV laboratory, Unidad Merida campus, Merida. It gave participants an opportunity to visit the laboratory and campus, and to see some hands on demonstrations of different extraction techniques, and practice some of these techniques. It also gave them an opportunity to ask the

trainers specific questions and in exchange discuss different laboratory techniques used in their own laboratories, discuss problems etc.

## IV. PROCEEDINGS OF THE COURSE

The final programme of the training workshop is given in Annex 1 of this report. The following section provides a short summary of the workshop proceedings.

**Day 1 - 19 January 2009**

The participants registered at 8.30 am and the training was opened by Ms. Hanneke Van Lavieren at 9 am.

Dr. Drouillard followed this with a session on Standard Operating Procedures for POPs and gave an overview of the Analytical Methods for the Stockholm Convention Persistent Organic Pollutants (POPs). It was mentioned that UNEP has no consensus SOP in place. Sample storage and handling issues were discussed, as well as storage (temporary or long term) and sample preparation. GC MSD and GC ECD methods were discussed including analyte extraction from blood, milk- liquid and lipid contents: liver, muscle, adipose fin, and carcass. This session also covered examples of different extraction and clean-up methods, the importance of clean up and good instrument maintenance and instrument detectors and quality assurance.

This was followed by a session with Dr. Metcalfe on C*hromatographic Techniques, Limits of Detection and Calibration*. Subjects covered included partitioning – multiple partitioning of multiple solutes, separation of analytes, elution Chromatography, stationary phase, clean ups, size exclusion chromatography, analytical chromatography, and gas Chromatography.

Dr. Drouillard then conducted a session on *Quality Systems Documentation* and its function and why it is needed, elements of a Quality System Manual and Accessory Documents and the GLIER QA Manual Example. He discussed what is needed for accreditation, audits, technical requirements, Quality system Procedure, lab personnel, data reporting and storing, lab equipment (QA and QC), calibration, and lab monitoring guidelines.

Dr. Metcalfe followed with a session on *instrument detection limits, and methods detection limits*.

After lunch, Nargis Ismail focussed on *How to validate your method*. This covered approaches to method validation, validation protocol, frequency of method validation, machine calibration, performance parameters and troubleshooting.

Dr. Drouillard next presented *Quality Control Charts* and *Evaluation of Data Integrity*. This included procedures for record keeping, Westgard Rules, the issuance of non-compliance reports, control charts, database needs and validation, and database entry.

This day concluded with a short discussion session.

**Day 2  20 January 2009**

A practical laboratory and field day was organized from 9 am to 12 pm.

This included a laboratory demonstration and exercise on POPs extraction by cold column and micro-methods, POPs cleanup by florisil and/or by silica as well as preparation and extraction of a fish sample.

After lunch, Dr. Drouillard presented *Data Reporting &Data Management* which included official data reporting, data management, data back ups, data storage, QA Assessment and Sign-off of data quality and data reports.

This was followed by Nargis Ismail who presented *GC-Methods and instrument maintenance*. This included an overview of documented Methods for POPs (OCs/PCBs) and basic maintenance and troubleshooting, lab ware and sample collection, sample homogenation before extraction, soxhlet extraction, column extraction, extraction scheme, GPC columns, and bio beeds.

Dr. Drouillard then presented *clean-up methods of POPs from Biological Samples*, including Gel Permeation Chromatography GPC – Maintenance and Calibration Florisil Chromatography, and Silica Chromatography.

# V. ANNEXES

## ANNEX 1

### Schedule for POPs Training Workshop for Laboratory Managers - POPs in Biological Tissues

**Day 1 Monday Jan 19**

| | |
|---|---|
| **9:00 – 9:45 am** | Overview of SOPs for POPs in tissues (GC-ECD) **(Drouillard)**<br>-Examples of different extraction and clean-up methods<br>e.g. EPA and other accredited methods are reviewed |
| **9:45 – 10:15 am** | Introduction to chromatographic techniques (**Metcalfe**) |
| **10:15 – 10:30 am** | The Quality Assurance Manual (**Drouillard**)<br>-Components of a QA manual<br>-GLIER manual as an example |
| **10:30 – 10:45 am** | - BREAK |
| **10:45 – 11:30 am** | -Instrument Detection Limits and Method Quantitation Limits (**Metcalfe**)<br>-Overview of approaches to determining instrument detection and method quantitation limits with specific reference to gas chromatography and POPs |
| **11:30-12:00 pm** | How to Validate Your Method (**Ismail**)<br>-Approaches to method validation, Frequency and Troubleshooting |
| **12:00 – 1:00 pm** | Quality Control Charts and Evaluation of Data Integrity (**Drouillard)**<br>-Record keeping, Westgard Rules, Issuing Non-compliance reports |
| **1:00 – 2:00 pm** | LUNCH |
| **2:00 - 2:45 pm** | POPs extraction methods (**Ismail/Gold/Metcalfe**)<br>-Cold Column, Micro-Cold Column, Sonication, Soxhlet, ASE, Other<br>-Where available overview of literature comparisons across methods<br>-Note for Satellite labs – attention must be placed on infrastructure needs (e.g. ventilation, solvent cabinets etc.), equipment, supplies<br>-Appropriate spiking internal standards, blanks, CRMs etc. |
| **2:45 – 3:00 pm** | - BREAK |
| **3:00 – 4:30 pm** | -Clean-up of POPs from Biological Samples (**Drouillard**)<br>-Florisil/Alumina/Silica Gel/GPC |

**4:30 - 5:00 pm** Discussion

**Day 2 Tuesday Jan 20**

**9:00 – 9:30 am** Tour of CINVESTAV Laboratory

**9:30 – 10:30 am** Laboratory Exercise (**Ismail/Gold**)
-POPs extraction by cold column and micro- methods
-Extraction of CRMs according to SOP (3 stations?)

**10:30 – 10:45 am** - BREAK

**10:45 – 12:00 pm** Laboratory Exercise (**Ismail/Gold**)
-POPs cleanup by florisil and/or by silica

**1:00 – 2:45 pm** Lunch and Return to Conference Room

**2:45 – 3:30 pm** GC-Methods and instrument maintenance (**Ismail**)
-Documented methods for POPs (OC/PCBs)
-Basic maintenance and trouble shooting

**2:45 – 3:30 pm** Data base management/QA Summary/Data Report (**Drouillard**)
-Data storage and back-ups
-The QA Assessment and Sign-off of data quality
-The Data Report

**3:30 – 3.45 pm** Break

**3.45 – 4.45 pm** GC calibration techniques (**Metcalfe**)

**4.45 - 5.30 pm** General discussion and evaluation of training.

# ANNEX 2

Participants List

| # | Participant | Organization | Email |
|---|---|---|---|
| **Belize** | | | |
| 1 | Isaias Majil | Fisheries Department, Ministry of Agriculture, Fisheries & Cooperatives<br>P.O. Box 148, Princess Margaret Drive<br>Belize City, Belize<br>Tel: 501-223 2623 ext. 24 | isaias.majil@gmail.com |
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| **Guatemala** | | | |
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| **Jamaica** | | | |

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| **Coordinators** | | | |
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| 17 | Ken Drouillard | Head Organic Analytical Laboratory<br>Associate Professor Biological Sciences<br>Great Lakes Institute for Environmental Research<br>University of Windsor<br>401 Sunset Ave.<br>Windsor, ON, N9B 3P4, Canada<br>Tel: (519) 253-3000 ext. 4744<br>Fax: (519) 971-3616 | kgd@uwindsor.ca |
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## ANNEX 3

## SUMMARY REPORT OF EVALUATION QUESTIONNAIRES

**The Most Useful Parts of the Course:**

Through the review of the evaluation questionnaires, it was apparent that most found the laboratory visit with hands on exercises and the lectures and guidelines that dealt with practical aspects of sample preparation and analysis the most useful parts of the course. Respondents also highlighted that the presentations on control charts, westgard rules and detection limits were useful. Some found the session on spiking internal standards, and elution of sample extract for PCB and organochlorine most useful and the session on quality system documentation manual and procedures was also found to be useful by a few respondents.

Moreover, respondents highlighted that during the workshop they were able to network, exchange information and discuss issues amongst each other which was found useful.

**The Least Useful Parts of the Course:**

Most respondents said that the entire course was useful. It was felt by some that there was some duplication and others found that sessions were too short and did not go into enough depth of the subjects. Some suggested the use of practical exercises to apply the new techniques that were presented.

A few mentioned that the accreditation requirements and data reporting sessions were not so useful mostly because this is not part of the work of the respondents.

**Difficulties in Applying:**

Regarding the application of this workshop, there was some variance in response – most mentioned however that they would have no difficulties in applying what learnt. Some indicated they would have some difficulty in applying validation and calibration techniques.

**The Overall Feelings about the Course:**

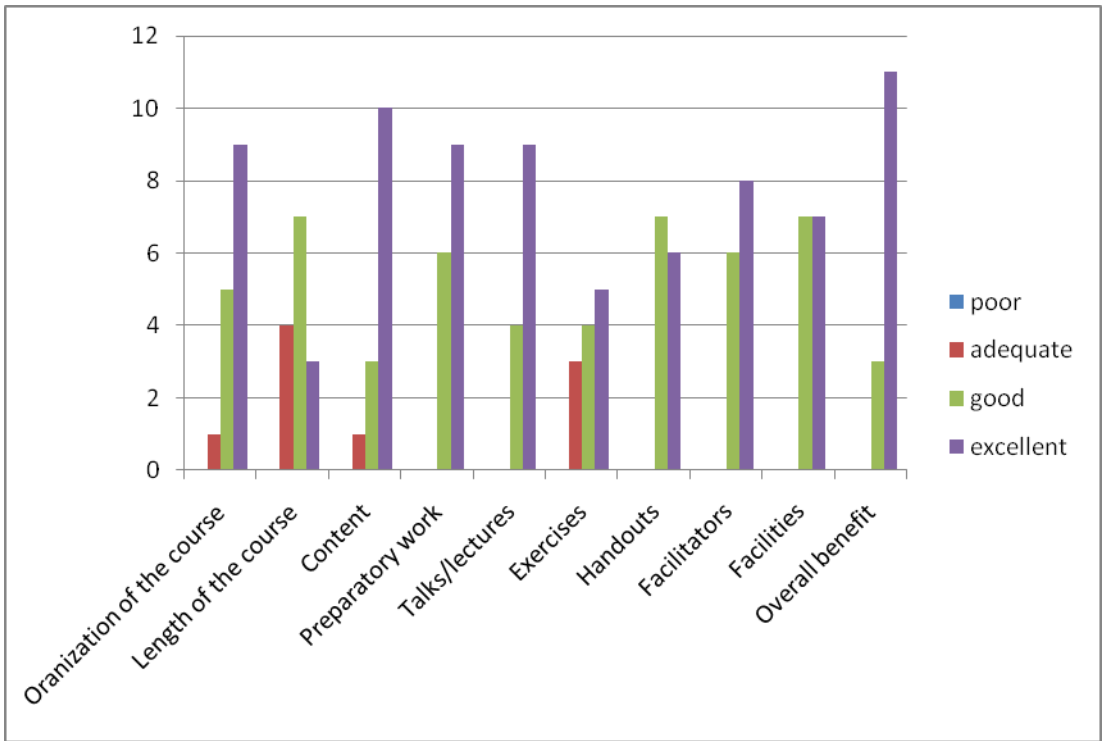
It was stated by almost all of the participants that their overall feeling about the course was well programmed, well prepared, useful, and very productive. It was mentioned that it was useful to discuss the different perspectives and examples from the countries amongst themselves and share knowledge and discuss experiences and needs.

A few participants mentioned the need for more hands on exercises and longer visit to the laboratory. Some indicated the sessions and overall course was too short. Most were happy to attend the course, learnt a lot and looked forward to applying it in their work.

**Other Comments:**

In addition to the above comments, the need for more exercises after each session would have been useful. Some mentioned that a few of the power points used too small fonts. Most indicated they would need more practical lab training.

**Summary Chart of Feedback in Section 2 of Evaluation Questionnaire**




**Suggestions for training workshops on related issues:**

- Data interpretation
- Hands on laboratory training in sample preparation and analysis procedures
- Distribution of POPs
- Use of biological markers e.g. Oysters
- Sample extraction, GC methods