Training Report

UN Environment Capacity Building for POPs Analysis

For Philippine Laboratory Personnel

at the Environmental Management Bureau, Quezon City, Philippines

4 and 5 December 2017 and 13 August – 17 August 2018

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Summary

The training of theory and practice of POPs analysis was very useful for the laboratory personnel of the Environmental Research and Laboratory Services Division (ERLSD) that is part of the Environmental Management Bureau (EMB) stationed in Quezon City and to some representatives of the other departments within the EMB. Due to private circumstances of one of the trainers the theory and practical training were done separately. Theoretical aspects of the analysis of POPs served as an introduction and were given in December 2017. The practical part took place in August 2018. Environmental samples, fish homogenate and a sediment were both Soxhlet-extracted and ASE extracted, cleaned-up and fractionated for PCB/OCP analysis. The extracts were measured on the GC-ECD. Both samples were also extracted and cleaned for the analysis of PFAS together with water samples (drinking water and water from a septic tank). These couldn't be analyzed due to a problem with the LC-MS.

Introduction

The EMB stationed in Quezon City is responsible for Stockholm POPs measurements in the UN Environment Global Monitoring Programme. This training program was a course in both PCB/OCP analysis and PFAS analysis (see Annex 1 for participants list). The training is intended to assist the laboratory in the POPs analysis work necessary for the mirror analyses, interlaboratory study, and tasks in the Global Monitoring Network of the UN Stockholm Convention on Persistent Organic Pollutants.

The Training

The first two days of theoretical training, consisting of lectures, were given by Prof. Jacob de Boer. These presentations took place on December 4 and 5 2017 (See Programme Theoretical Training in Annex).

After an introduction in the reasons of monitoring of POPs and the context of the UN Environment Global Monitoring Program, several lectures were given on the various steps of the analytical method for ndl-POPs, in particular OCPs and PCBs. These included sampling and sample storage, extraction and clean up, GC analysis and QA/QC. To prepare the trainees for the hands-on training that followed, QA/QC in particular was extensively discussed, with emphasis on the preparation of quality charts and use of reference materials. A more philosophical lecture was included on the reasons of making mistakes in a laboratory. After each presentation time was included for discussion and questions. At the second day the results of last interlaboratory study were discussed. Part of the second day was devoted to new POPs such as PFASs and specific problems around the more difficult to analyse POPs such as toxaphene and mirex. Due to time constraints the newest POPs such as PCNs and CPs could not be discussed.

The hands-on training was scheduled for August 13 until August 17 (2018). The staff was trained in extraction and clean-up of test materials for PCBs and OCPs with a focus on sediment and biota samples, and analysis by GC-ECD. This part of the training was given by VU Senior Technician Mr. Martin van Velzen. Printed manuals with procedure descriptions were given for use by the laboratory staff (Annex 2).

The hands-on training focused on all steps necessary for the analysis of POPs in environmental samples. This was done by taking a blank sample and two types of sample matrices: a fish (*Tilapia*) and a sediment sample. All samples were extracted and cleaned by the methods described in the training manual (see Annex 2). In short, the samples were both Soxhlet extracted and ASE extracted and subsequently cleaned with Alumina (deactivated with 8% water) and fractionated with Silica (deactivated with 1.5% water). For the sediment sample, sulphur was removed using activated copper powder. For the fish sample also the total fat content was calculated. The final extracts were analyzed on the GC-ECD with a dual column system (HP-5 and DB-608, 30m x 0.25 mm x 0.25 µm). A calibration curve of a selected set of PCBs was prepared by the trainees in order to quantify the samples. In both fish and sediment sample no quantifiable amounts of PCBs were present. The internal standards (PCB 112, 155 and 198) added to the samples and the blank were found with a recovery around ca. 100% for all samples. Besides the PCB/OCP analysis also all steps needed for the analysis of PFAS compounds were shown. For this six samples were taken: a blank MilliQ water sample, a drinking water sample, a water sample from the local septic tank and the fish and sediment sample (in duplicate) which were also used for PCB/OCP analysis. The fish and sediment samples were pretreated (see schedule in Annex 2) and all samples were extracted and cleaned using solid phase extraction (SPE). For the analysis a new internal standard mix was prepared consisting of labeled PFAS compounds. Due to computer issues of the LC-MS system it was not possible to measure the samples during the training.

During the training emphasis was put on working clean and precise. The last day a lecture was given about the procedures necessary to perform a correct calculation of the results (QA/QC). Because no quantifiable amounts of PCBs were found in the samples a “handmade” dataset to do calculations was provided to the trainees. In a group session the

trainees constructed calibration curves in Excel and calculated the “samples” taking all QA/QC aspects into account.

During the closing session of both the theoretical and the practical training certificates of course completion were given to the participants (Annex 4). The trainers received positive feedback on the training.

Conclusions and recommendations

This laboratory is in a phase of building up its capabilities. A new dioxin laboratory funded by the government is for example planned. The room for that laboratory was already built and prepared. A GC/triple quad MS was available as well as several LC/MS instruments. The entire staff showed a high motivation to reach a higher level of analytical quality for POPs. Experience was already present with the technician in trace metal, phenol and VOC analysis. The PUF sampling started at 1 January 2018. The laboratory also had a passive high volume air sampler from Japan (see photo).

The practical part of the training was valuable to the participants who practiced techniques hands-on and learned some skills regarding sample extraction/cleanup and standard preparations for both PCB/OCP and PFAS analysis and optimizing the GC-ECD program.
Still, several difficulties need to be solved. The suction capacity of the fume hoods is too low. There are administrative issues with ordering certified standards for the various POPs which cause serious delays in the work. It seems useful to offer a training in an external reference laboratory for at least one staff member. However, there is potential is this laboratory to grow into its role as POPs laboratory for the Global Monitoring Plan of the Stockholm Convention.

Annex 1. Participants in the laboratory training

Name	Division
Ma Fatima Anneglo R. Molina	Environmental Research Laboratory and Services Division
Ellaine Gellie S. Nicdao	Environmental Research Laboratory and Services Division
Maria Veronica C. Eulogio	Department of Health - East Avenue Medical Center
Noemi Ruth Q. Infante	Environmental Research Laboratory and Services Division
Renz Jonnar D. Subida	Environmental Research Laboratory and Services Division
Roberto L. Co	Environmental Research Laboratory and Services Division
Roger C. Evangelista Jr.	Environmental Research Laboratory and Services Division
Rosemarie G. Hibo	Environmental Research Laboratory and Services Division
Sammy L. Aytona	Environmental Research Laboratory and Services Division
Benzon Karl T. Bongar	Environmental Research Laboratory and Services Division
Stephen C. Yecpot	Environmental Quality Management Division – Chemical Management Section

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Annex 2. Training Manual for the Philippine laboratory staff

This manual is attached as a separate file.

Annex 3. Laboratory Training on POPs Analysis Continuing regional Support for the POPs Global Monitoring Plan (GMP) under the Stockholm Convention in the Africa Region Project (Phase II), Quezon City, 4-5 December 2017

International Expert: Professor Jacob de Boer

Training Program Theory and Background:

4 December

9.00	Visit Prof. de Boer to laboratories
10.00	Introduction (trainer, participants)
10.15	Why we need to monitor
10.45	Break
11.15	Analysis of PCBs and OCPs by Gas Chromatograph
12.15	Discussion
12.30	Lunch
13.30	Extraction and Clean-up
14.15	QA/QC
15.15	Break
15.45	Why do we make mistakes?
16.30	Discussion
17.00	Closure

5 December

9.00	Sampling
9.45	Lessons from the last interlab study
10.30	Break
11.00	Perfluor alkyl substances
11.30	New POPs
12.00	Lunch
13.00	Toxaphene, Mirex, Kepone
13.30	Lipids
14.00	National samples and PUFs
14.45	Break
15.15	Final discussion
16.00	Closure

Annex 4. Certificate Theoretical Training

ENVIRONMENTAL MANAGEMENT BUREAU
DEPARTMENT OF ENVIRONMENT AND NATURAL RESOURCES

awards this

CERTIFICATE OF COMPLETION

to

EMMANUELITA D. MENDOZA

for having successfully completed the

TRAINING ON ANALYSIS OF POPs

held at the ERLSD Laboratory, Visayas Avenue, Quezon City
on 04-05 December 2017

PROF. DR. JACOB DE BOER
UNEP International Expert on POPs analyses

ENGR. METODIO U. TURBELLA
Director