Training Report

UN Environment Capacity Building for POPs Analysis

For Tanzanian Laboratory Personnel

at the Government Chemist Laboratory Agency

Dar es Salaam, Tanzania

12 July – 20 July 2018



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10 January 2019

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Summary

The training of theory and practice of POPs analysis was very helpful for the laboratory personnel of the Government Chemist Laboratory Agency (GCLA) stationed in Dar es Salaam. Theoretical aspects of the analysis of POPs served as an introduction to the activities that took place in the practical training that followed. Environmental samples, including a fish homogenate and honey were Soxhlet-extracted, cleaned-up, fractionated and analysed for PCB/OCB by GC-ECD. In additionan, sediment and water samples were extracted by Solid Phase Extraction (SPE) for PFOS analysis.

Introduction

The GCLA stationed in Dar es Salaam is responsible for Stockholm POPs measurements in the UN Environment Global Monitoring Programme. This training program was a course in both PCB and OCP analysis as well as PFOS analysis for personnel of GCLA (see Annex 1 for participants list). The training was 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 on-site training took place between 12 and 20 July 2018. The first two days were used for theoretical training, consisting of lectures given by Dr. Sicco Brandsma. Martin van Velzen participated in the lectures and thus creating a lively discussion with the participants.



The following topics were covered: the relevance of POPs monitoring and the context of the UN Environment Global Monitoring Program, sampling and sample storage, extraction and clean-up, GC analysis and QA/QC. The context of industrial chemical pollution and the regulations in place needed to address them were sketched, with several examples of the

sources, exposure pathways, persistence, bioaccumulation, and toxicity of industrial chemicals, with special emphasis on Stockholm POPs and potential POP candidates such as chlorinated paraffins. The analytical scheme was explained to the trainees to prepare them for the hands-on training that followed. Details regarding the solvent extraction, clean-up and fractionation steps and following that analysis by GC were explained for the matrices of interest. A presentation was given on QA/QC of laboratory analysis reviewing the principles of QA/QC, QA/QC tools and practice using examples relevant to POPs analysis. Sampling QA/QC, study design guidelines and proficiency testing and interlaboratory studies were also handled during the lecture.

The course participants were actively participating, asking questions and sharing their own knowledge, experiences and opinions with the group.

The following days, until July 20th, consisted of hands-on training in the laboratory in which the staff was trained in extraction and clean-up of test materials with a focus on biota samples, and analysis by GC-ECD. Besides the PCB/OCB analysis also a training was given for the extraction/cleanup and measurement of PFOS in water and sediments. 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 consisted of demonstrating all steps necessary for the analysis of POPs in environmental samples. This was done by taking two types of sample matrices: honey and fish (Red Snapper), and a blank sample. All samples were extracted and cleaned by the methods described in the training manual (Annex 2). In short, the samples were Soxhlet extracted and subsequently cleaned with alumina (deactivated with 8% water) and fractionated with Silica (deactivated with 1.5% water). After that the final extracts were ready for measuring on GC-ECD. A calibration curve of OCPs was prepared by the trainees in order to quantify the samples. Due to unforeseen connection problems with the autoinjector and software of the GC-ECD, we were not able to measure the samples during the training period. However, the samples were stored to analyze on a later date. For the PFOS analysis we performed two types of extraction/cleanup methods: for water and sediment samples according to the schemes in the training manual. Since this is the first time perfluorinated compounds would be analyzed in this lab, we used three blank samples and three drinking water samples. For the same reason, also for the sediments we used three blanks and three sediment samples. The trainees also prepared PFOS stock solutions and a calibration curve for quantification of the samples. Unfortunately, in the week before we arrived the LC-Orbitrap that was going to be used for measuring the PFOS samples was not working properly. We had contact with a service engineer by remote desktop connection but he couldn't solve the problem. We stored the calibration curve and the final sample extracts in the fridge, so those could be measured when the LC-Orbitrap would be repaired.



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. Because we were not able to analyze the samples prepared during this course a "handmade" dataset to do calculations was provided to the trainees. In a group session we constructed calibration curves in Excel and calculated the "samples" taking all QA/QC aspects in account.

Conclusions and recommendations

In total, 8 certificates of course completion were given at the closure of the training. The trainers received positive feedback on the training. The practical part of the training was valuable to the participants who practiced techniques hands-on and learned some skills regarding GC and LC maintenance. A large amount of practical and theoretical knowledge was transferred to a motivated, young and well-educated group of participants.

The following recommendations can be given.

- 1. Make permanent setups in the laboratory for extraction (Soxhlet) and evaporation instead of taking it apart after use.
- 2. Try to make the laboratories as much dust-free as possible because dust can cause problems with blank values.
- 3. Start analysing a series of blanks to build up knowledge about the background in the lab (construct a Shewart chart).
- 4. Take advantage of the knowledge that can be found in literature to develop new methods.



Annex 1. Participants in the laboratory training

Name	Organisation
Emmanuel Gwae	GCLA
Shimo Peter	GCLA
Kagera Ng'weshimi	GCLA
Francis Kway	GCLA
Elias Mulima	GCLA
Gabriel Gabriel	GCLA
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Annex 2. Training Manual for Tanzanian laboratory personnel

The manual is attached as a separate file.