# Training Report

## UN Environment Capacity Building for POPs Analysis

## for Laboratory Personnel

## of the Institute of Chemistry and Chemical Technology of the Mongolian Academy of Sciences

## 6 - 14 February 2017

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## Summary

The training of theory and hands-on training of POPs analysis was very helpful for the staff of the Institute of Chemistry and Chemical Technology (ICCT) of the Mongolian Academy of Sciences. Theoretical aspects of the analysis of POPs were presented and discussed at the first two days of the training session. Subsequently, five full working days were spent on the practical training. Environmental samples, including a PUF, fish homogenate and blanks were Soxhlet-extracted, cleaned-up, fractionated, and POPs were identified and quantified using GC-μECD during the training. Although current conditions are basic, this ICCT laboratory has a high potential for serving as a reliable POPs laboratory for the GMP in the near future.

## Introduction

This type of training as part of a capacity building project for the Global Monitoring Program (GMP) of the Stockholm Convention was given for the first time in Mongolia. Until now the Institute of Chemistry and Chemical Technology (ICCT) of the Mongolian Academy of Sciences had not carried out any work for the GMP. However, the staff was highly motivated to start these activities and responsible authorities seem to be willing to encourage such monitoring from the near future onwards. Based on a completed questionnaire and regular contacts by email and skype between the Vrije Universiteit and Dr. Enkthuul Suranjev, an inventory was made from which a list of articles was composed to be included in the procurement. This procurement included the delivery of consumables such as two GC columns, spare parts, glassware, chemicals and analytical standards to ICCT. Several laboratories are located in the building of ICCT and unfortunately no lab is equipped for the sample preparation of POPs analysis. The laboratory conditions were rather basic, but plans were there for renewal and further extension of instruments and other relevant items. This training programme was therefore focused on the basic principles of the PCB and OCP analysis, including theoretical concepts, with a focus on practical implication of the theory, and quality assurance and quality control procedures and tools, followed by hands on training and demonstrations of extraction, clean up and gas chromatographic (GC) analysis.

## The Training

The on-site training took place between 6 and 14 February 2017. The first two days were exclusively used for theoretical training, consisting of lectures given by Prof. Dr. Jacob de Boer. There were 11 participants, including a few participants from other laboratories in Ulaanbataar (Institutes of Plant Protection and Veterinary Medicine), but one extra presentation on publication of scientific results attracted 25 participants.

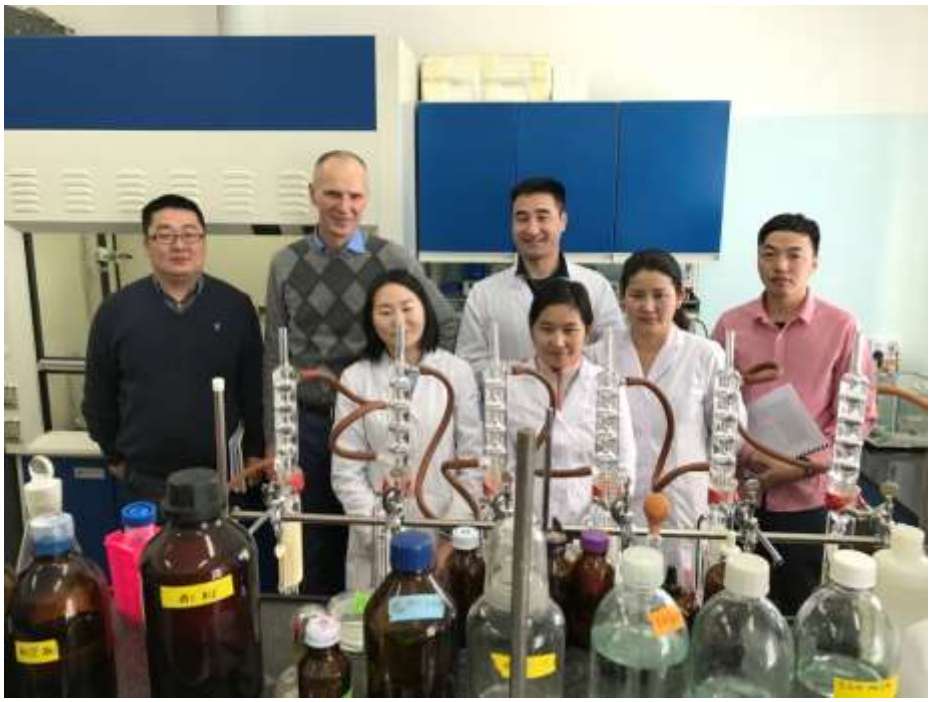
The following topics were covered: the relevance of POPs monitoring and the context of the UN Environment Global Monitoring Program, sampling and sample storage, extraction and clean up, GC analysis and QA/QC. One presentation on the national Samples study (Mirro study) was given describing the procedures, sample matrix selection, how to avoid contamination during sample preparation, and logistics for transport of samples to Europe. Attention was also spent to new POPs and their analysis. Proficiency testing and interlaboratory studies were also handled during the lecture, including the use of certified reference materials and certified analytical standards and addresses where to obtain those.

The course participants were actively participating, asking several questions. Printed manuals with procedure descriptions were given for use by the laboratory staff (Annex 2), as well as DVDs dedicated to the analytical research conducted and coordinated at the Vrije Universiteit Amsterdam.

The hands-on training in the laboratory in which the staff was trained in extraction and clean-up of test materials with a focus on air and biota samples, and analysis by GC-µECD, was attended by six participants. For reasons of limited space in the lab, more participants could not be included, as was agreed beforehand This part of the training was given by VU Senior Technician Jacco Koekkoek BSc. The sample preparation was carried out in a physical chemistry lab during the training. The lab is equipped besides several small instruments with three work benches and two fume hoods. The work benches were full with jars and bottles filled with different solutions. Important to mention is that all over the benches etc. lays a small film of sand and dust. This is mainly remnant of sand storms which occurs frequently during spring. New Soxhlet glassware was bought together with a heater. The glass columns for the clean-up and fractions of the extracts were provided by the Vrije Universiteit.

The GC (7890A, Agilent) is placed a separate room not equipped with an exhaust for the discharge of the gasses from the column and µECD detector and hot air from the blow out

from the GC oven. The gasses are provide by a hydrogen and nitrogen generator. Unfortunately, the applied procedure is to switch off the GC and generators after the measurements and keep the column installed in the GC. By this procedure the column is exposed to moisture and oxygen which erodes the stationary phase what will have a strong negatively influence on the chromatographic performance.



The hands-on training consisted of showing all steps necessary for the analysis of POPs in environmental samples. The trained method is comprehensively described in a manual what was given to the people of ICCT in a hard and digital copy.

Sample preparation

During the training five samples and a blank were analyzed. The five samples were:

- Soil sample originated from the industrial area in Ulaanbaatar
- Soil sample originated from Khovd province, Western Mongolia
- Sediment used in inter lab study UNEP 2016
- Collected sand and dust in the lab of the sample preparation
- Enriched passive air filter (PUF)
- A blank

The soil samples could not be dried prior the analysis because the lack of time. The water content was estimated as lower than 5% and it was estimated that this amount would not influence the efficiency of the extraction. The soil samples were manually homogenized prior the sample intake. The passive air filter was cut in small pieces.

At the start of the training the ordered control standard (PCB-112) were not available due to a late delivery. Instead a mixture of PCB112, PCB155 and PCB198 - 5 µg/ml (CIL, EC-5460) provided by the Vrije Universiteit was used.

The glassware was washed with soap and water and rinsed with acetone prior the extraction.

The samples were extracted for 16 hours. The obtained raw extracts were cleaned by applying the standard method of deactivated (8%) alumina and pentane. The non-polar compounds were separated from the more polar pesticides by applying deactivated silica gel (1.8%), hexane and a mixture of hexane and acetone. The latter solvent was used as an alternative for di-ethylether what was not available.

The evaporation of the large volumes was done by a rotary evaporator and unfortunately this was also necessary for the small volumes. ICCT doesn't have equipment to evaporate small volumes with a gently nitrogen stream.

The sample extracts were ready for measurement on the third day of the practical part of the training.

On the fourth day the additional clean-up method of acidified silica gel and a mixture of hexane and dichloromethane was trained.

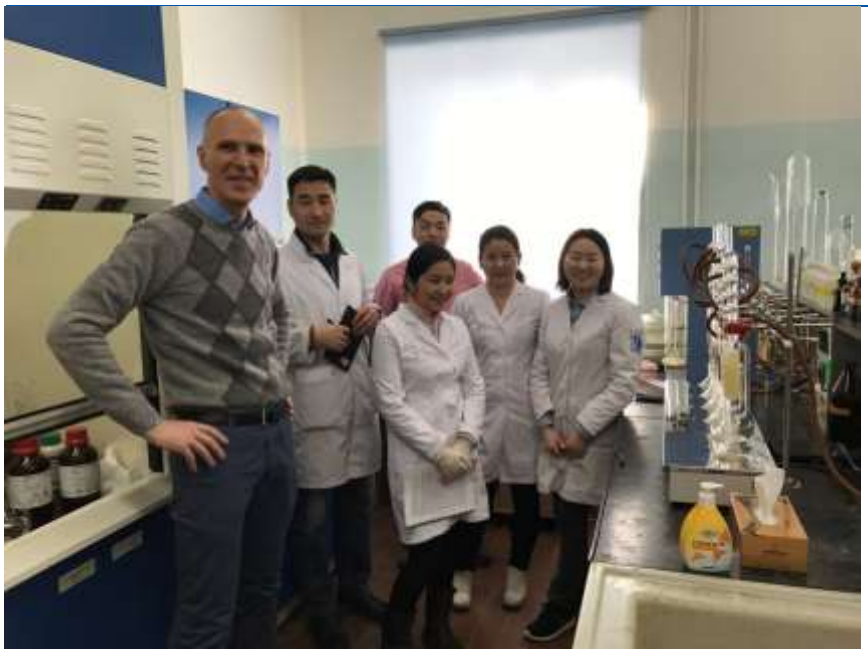
Measurements

Prior to the training the provided column Restek Rxi-5ms column (60m * 0.25mm * 0.25µm) was installed in the GC. The injection liner, septum and ECD liner were replaced.
The calibration standards were prepared from the standard solutions – OCP, ES-5467-A and PCB, EC-5495 both CIL – in the range of 0.5 – 100 ng/g.
The sensitivity and peak shape of all the compounds were good.
On the fourth day, the extracts of the soil samples were diluted and re-measured.

Data processing

The GC is controlled by the Agilent software Chem station. It should be possible also to use this software for the calculating of the concentrations in the measured extracts. But unfortunately the short time available did not allow to find a working data processing method.

As an alternative a data processing method was set up by using Mass Hunter software. The software used for the processing of the data is not useful in the future. The best option is look for alternatives. There are enough different software available who can handle data files which generated by Chem Station.



ICCT has the ambition to set up a lab for the analyzing of dioxins. The best option is to develop a POPs laboratory first and separate the dioxin lab from the other labs.

Also, it is important to start with the development of a basic quality system by

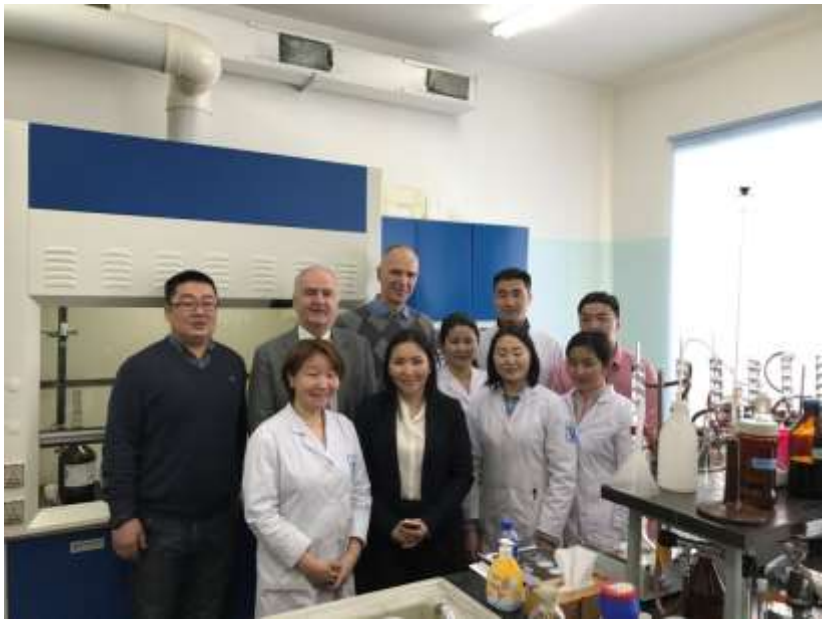
- Analyzing on regular base of blanks and control sample and archived the results in a Shewart control chart
- Checking the correctness of the balance by weighing a mass standard (before use)
- Checking the correctness of the pipette by weighing the dispense with water (before use)
- Determinate the mass of every addition by the preparation of all the standards
- Clean the lab on regular base and every day during the sample prep

**Conclusions and recommendations**

The laboratory is still of a very basic quality. Numerous improvements need to be made. These are not necessarily very expensive. It will be essential to make the laboratory dust-free and keep it like that. Unnecessary items, glassware and chemicals should be removed from the laboratory and packing materials should also be taken away. To reduce dust levels, new deliveries of chemicals and glassware need to be unpacked at a different place. More and clean glassware need to be bought and solvents should be regularly available. The GC needs to be kept under gas and power continuously to avoid deterioration of the columns used. Most important is, that regular analyses of POPs are carried out so that the staff can build up routine. Some more training at a later stage, e.g. by a traineeship in a reference lab, may be useful.

However, the head of the laboratory and the staff are highly motivated to make the lab work for the GMP. The lab head received her training (PhD) in Germany. She also made the effort to attend the UNEP Interlab Study Workskop in Beijing, China in March 2017 to learn more about POPs analysis. They also have an active sampler and would be willing to work with that, if appropriate, for the GMP and the capacity building project.

In summary, this is a laboratory with potential. On the longer term this lab could serve as a very useful POP laboratory for the GMP in Asia.



## Annex 1 LIST OF PARTICIPANTS

| Name, Surname | Affiliation |
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## Annex 2. Training Manual

The manual is attached as a separate file.