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United Nations Environment Programme Capacity Building Pilot Project—Training and interlaboratory study on persistent organic pollutant analysis under the Stockholm Convention

J. de Boer^{a,*}, H. Leslie^a, S.P.J. van Leeuwen^a, J.-W. Wegener^a, B. van Bavel^b, G. Lindström^b, N. Lahoutifard^c, H. Fiedler^d

^a VU University, Institute for Environmental Studies (IVM), De Boelelaan 1087, 1081 HV Amsterdam, The Netherlands

^b Örebro University, Department of MTM, Örebro, Sweden

^c SGE Europe Ltd., 12 Avenue du Québec, BP98, 91943 Courtaboeuf, France

^d UNEP Chemicals, 11-13 chemin des Anémones, CH-1219 Châtelaine, Switzerland

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ABSTRACT

Within the framework of a United Nations Environment Programme (UNEP) Capacity Building Project for training of laboratory staff in developing countries on persistent organic pollutant (POP) analysis, an interlaboratory study was organised following an initial evaluation of the performance of laboratories (reality check) and a series of training sessions. The target compounds were polychlorinated biphenyls (PCB) and organochlorine pesticides (OCP). Seven laboratories from five countries (Ecuador, Uruguay, Kenya, Moldova, and Fiji) participated. Most of the laboratories had no experience in determining PCBs. Although chromatograms improved considerably after the training and installation of new gas chromatographic (GC) columns at participating laboratories, the level of performance in the interlaboratory study was essentially on par with the moderate performance level achieved by European POP laboratories in the 1980s. Only some individual results were within $\pm 20\%$ of the target values. The relative standard deviations (R.S.D.s) in POP concentrations determined by laboratories in a sediment sample were $>200\%$ in a number of cases. The results for a certified herring sample were better with at least some R.S.D. values below 50% and most below 100%. Clean up was as one of the main sources of error. After inspection it was ascertained that training of laboratory staff and investments in simple consumables such as glassware and GC columns would help to improve the quality of the analysis more than major investments in expensive instrumentation. Creating an effective network of POP laboratories at different continents together with a series of interlaboratory studies and workshops is suggested to improve the measurements of POPs in these countries.

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* Corresponding author. Tel.: +31 20 5989530; fax: +31 20 5989553.

E-mail address: jacob.de.boer@ivm.vu.nl (J. de Boer).

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1. Introduction

The Stockholm Convention is a global treaty aimed at protecting human health and the environment from persistent organic pollutants (POPs). Based on their global distribution, persistence, bioaccumulation, and toxic effects to humans and wildlife, polychlorinated biphenyls (PCB), polychlorinated dibenzo-*p*-dioxins and dibenzofurans (PCDD and PCDF), aldrin, dieldrin, DDT, endrin, chlordane, hexachlorobenzene (HCB), mirex, toxaphene, and heptachlor have officially been identified as the initial twelve POPs in this convention [1]. Although in the western world concentrations of most of these POPs are slowly decreasing [2–4], and, monitoring efforts may be justifiably reduced, many developing countries and countries with economies in transition are now developing analytical methods and monitoring programmes for these contaminants in food, humans and the environment. This is necessary because the Stockholm Convention has called for the establishment of a Global Monitoring Plan (GMP) for POP analysis. Moreover, some of the Stockholm Convention organochlorine pesticides (OCPs) are still in use in some of those countries. For example, after 30 years of gradually reduced use of DDT, an increasing number of African nations is using this insecticide again for indoor spraying against mosquitoes to cut malaria death toll [5]. These countries also need to analyze POPs in food for export purposes. Because of the requirement of countries to monitor POP levels, the United Nations Environment Programme (UNEP) initiated a Capacity Building Pilot Project for POP analysis training of laboratory staff from developing countries [6]. This project was comprised of a selection of countries and laboratories, laboratory inspections, a pre-study/reality check on the quality level of the laboratories, on-site training of laboratory staff, an interlaboratory study, a workshop for evaluation of results and further training at the laboratories of the Örebro University, Sweden and VU University, Amsterdam, The Netherlands. Training in analysis of PCDDs, PCDFs, and dioxin-like PCBs, a separate part of the overall project, was covered by Örebro University where the staff has over 10 years experience in organising interlaboratory studies for PCDDs, PCDFs, dioxin-like PCBs, and other POPs [7]. The training in analysis of PCBs and OCPs in environmental and food samples, on which we report in this paper, was provided by the Institute for Environmental Studies (IVM) of the VU University. The staff of IVM has over 25 years experience in organising interlaboratory studies and training activities in POPs analysis [8–11]. Seven laboratories from five countries representing three continents (South-America, Africa, and Europe), and the Pacific were selected for participation in this project (Table 1). The pre-study, a 'national' sample comparison, was organised in October/November 2006, after the inspection of the laboratories and before the on-site training sessions. It comprised shadow analyses by the expert laboratories of 'national' samples. These samples had been selected and analysed by the participating laboratories in their own countries. The second exercise was organised in December 2006/January 2007, after the on-site training sessions, and comprised a full interlaboratory study consisting of a test solution, a sediment sample and a herring tissue. In a global activity under the GMP it was

Table 1 – Participating laboratories

Ecotoxicology Laboratory, Ecuadorian Atomic Energy Commission, Quito, Ecuador
Laboratorios de Plaguicidas del Service Equatoriano de Sanidad Agropecuaria del Ministerio de Agricultura y Ganadería, Quito, Ecuador
Institute of Applied Sciences, University of the South Pacific, Suva, Fiji Islands
Department of Chemistry, University of Nairobi, Nairobi, Kenya
Centre on Soil Quality Monitoring, Chisinau, Moldova
National Scientific and Applied Centre for Preventive Medicine, Chisinau, Moldova
Technological Laboratory of Uruguay, Montevideo, Uruguay

agreed that performance-based criteria be applied for controlling quality of analysis rather than prescribing a single specific method. The studies led by IVM intended to cover all twelve POPs, but as the laboratories had no experience at all with the analysis of mirex, toxaphene, and chlordanes, these compounds were excluded from the study. Consequently, the study focused on PCBs (individual chlorobiphenyls with an emphasis on the seven indicator PCBs), DDT and its metabolites, HCB and dieldrin.

2. Methods and materials

2.1. Selection of laboratories

A number of criteria and conditions were applied to the selection of the laboratories in this project, which was subject to a maximum number of participants. The willingness of the authorities and the laboratory staff in the candidate countries to spend time on such a project was a key criterion. It was important to select different laboratories in the project: different in size, different in available equipment, and representing different continents. This would provide information to fulfil one of the main objectives of this pilot project: to determine if a training programme in combination with interlaboratory studies could be successful and be applied on a larger scale later in the GMP. Therefore, three laboratories (one from China and two from Vietnam) were selected for the dioxin program in the project because they are set up to perform the analysis of dioxins. Another group of POP laboratories (without dioxin analysis capacity) were selected (Table 1) and this group's results are discussed in this paper.

2.2. Laboratory inspection

An extensive questionnaire was completed prior to the start of the inspection. This enabled the inspection teams to collect general information in advance on the type of samples analysed in the laboratory, safety issues, type of instruments used, type of gas chromatographic (GC) columns used and dimensions, extraction and clean up methods, quality assurance/quality control (QA/QC) issues, education of staff, etc. The inspection and training of the laboratories were always carried out by teams of at least two persons. The qualifications of these teams are given in Table 2. All

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