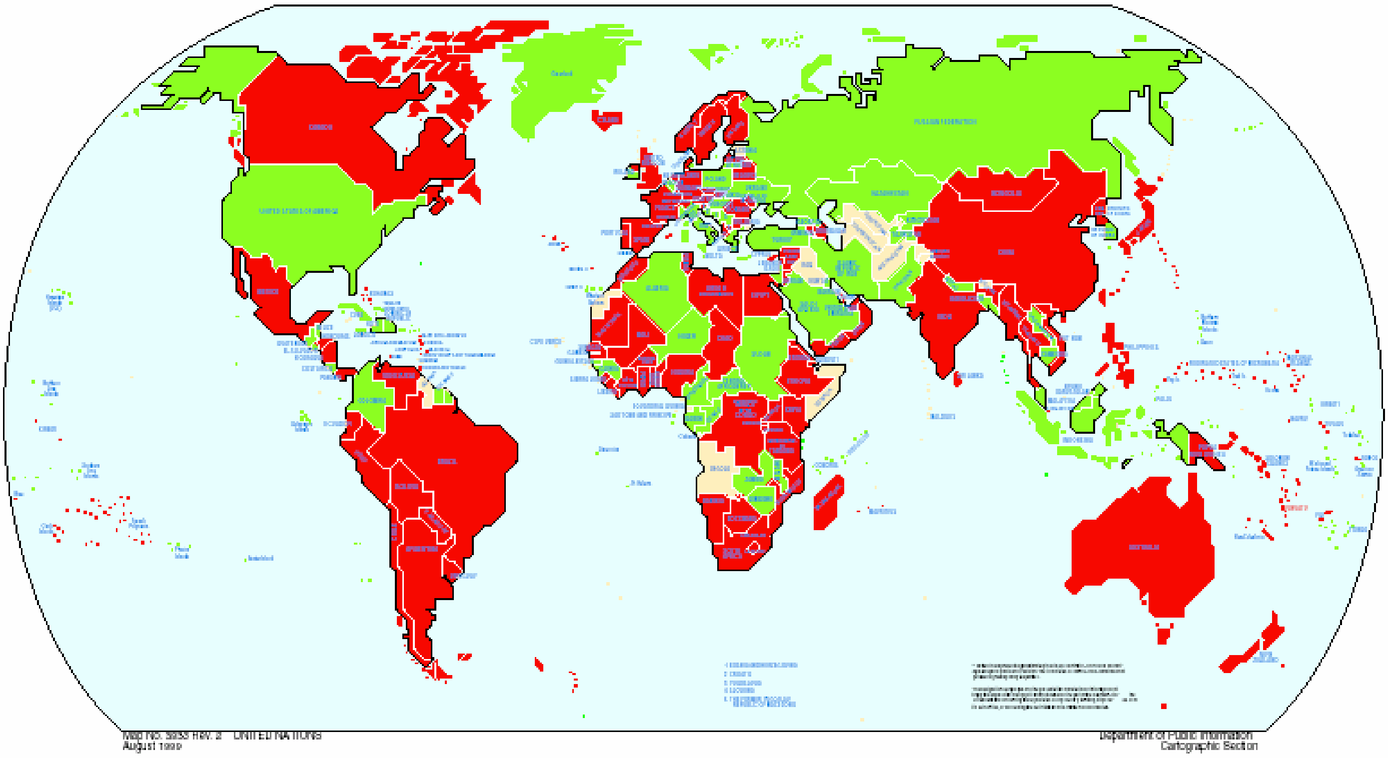
Analysis of Persistent Organic Pollutants in Developing Countries: Lessons Learned from Laboratory Projects



UNEP Chemicals

Geneva, February 2006

IOMC

INTER-ORGANIZATION PROGRAMME FOR THE SOUND MANAGEMENT OF CHEMICALS
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This publication has been prepared as a reference document for the UNEP/GEF Project "Assessment of Existing Capacity and Capacity Building Needs to Analyse Persistent Organic Pollutants (POPs) in Developing Countries". The project is financed by the Global Environment Facility and implemented by UNEP through UNEP Chemicals. Co-financing of the project is through the governments of Canada, Germany, and Japan.

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Title page: World map showing the 118 Parties and 151 signatories to the Stockholm Convention, status February 2006. Graphic courtesy of the Secretariat of the Stockholm Convention

This publication is produced within the framework of the Inter-Organization Programme for the Sound Management of Chemicals (IOMC)

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UNEP Chemicals is part of UNEP's Technology, Industry and Economics Division

UNEP CHEMICALS

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Abbreviations and Acronyms

CS_2	Carbon disulfide
DM	Deutsche Mark (German Mark, former German currency; conversion: 1.95583 DM = 1 €)
DOA	Department of Agriculture (Malaysia)
EBDC	Ethylene *bis*-dithiocarbamates
ECD	Electron capture detector
FRG	Federal Republic of Germany
GC	Gas chromatography
GDCh	Gesellschaft Deutscher Chemiker (German Chemical Society)
GTZ	German Agency for Technical Cooperation
HPLC	High performance liquid chromatography
IPM	Integrated pest management
KL	Kuala Lumpur
LOD	Limit of detection
LOQ	Limit of quantification
MGPP	Malaysian-German Pesticide Project
MRL	Maximum Residue Limit
M$	Malaysian Dollar
NIL	Residue value below limit of detection
NMC	N-Methylcarbamates
NPD	Nitrogen phosphorus detector
OC	Organochlorine compounds
OP	Organophosphates
p.a.	*pro analysi* (grade of purity for chemicals)
PCBs	Polychlorinated biphenyls
PCDD/PCDF	Polychlorinated dibenzo-*para*-dioxins and polychlorinated dibenzofurans
POPs	Persistent organic pollutants
QA	Quality assurance
QC	Quality control
RRT	Relative retention time
SIRIM	Standard Industrial Research Institute of Malaysia
SP	Synthetic pyrethroids
TEQ	Toxic equivalents (for the toxicity-weighted mass concentrations of mixtures of PCDD/PCDF and dioxin-like PCB)
UNEP	United Nations Environment Programme
UV	Ultra-violet
WHO	World Health Organization

1 INTRODUCTION

For the international scientific community it is generally acknowledged that due to human activities certain chemical compounds are distributed and found as residues in several abiotic and biotic materials. Due to their already known or at least assumed (eco)toxicological impact they should be monitored by trace analytical means in order to gain an overview regarding their factual qualitative and quantitative distribution. Only on the basis of such monitoring system with evaluable/evaluated and comparable data it is possible that endeavours undertaken by individual countries (national basis) lead to appropriate legal principles as to limiting and eliminating spreads of these undesired chemicals into the environment.

In the context of this report it will be dealt only with a special group of semivolatile chlorinated organic compounds, classified as POPs ("persistent organic pollutants").

This means that any other organic chemicals (*e.g.,* volatile organic chemicals = VOCs) and all inorganic chemicals, such as certain heavy metals (*e.g.,* Pb, Cd, Hg, Cu) and metalloids (*e.g,,* As, Se, Sb) and others (*e.g.,* CO_2, NO_x, Br_2, N_2O) are not considered here, notwithstanding their (eco)toxicological importance and the need of their analytical surveillance.

As to POPs it has to be differentiated between those compounds which have been and/or still are introduced into the environment with purpose as they were/are needed as biocides or/and agrochemicals (pesticides): DDT, dieldrin, endrin, HCB, heptachlor, toxaphene, or as industrial chemicals (PCB). The third sub-group comprises of the polychlorinated dibenzodioxins/furans (PCDD/PCDF) which are generated by combustion processes and are released into the environment without intention [for further details see Stockholm Convention on POPs (ANONYMOUS 2001) and ANONYMOUS (undated), ANONYMOUS (2005), HOSSEINPOUR & ROTTLER (1999), HOLOUBEK (2005), HERRMANN (2005)].

It is the duty of a sovereign country and with responsibility of the government to protect people from negative health impacts and the environment from deleterious interferences/exposures due to the application and emission of (potentially) toxic chemicals by establishing the necessary legal basis for their avoidance and/or judicious management/handling, use/trade and disposal, respectively, and the installation of adequate and effective institutions (laboratories) for the execution/transformation of these legal premises.

For agrochemicals (pesticides, biocides) it means the development of rules and regulations as to their registration, trade, distribution and application [*e.g.,* establishment of rules for good agricultural practice (GAP) and establishment of toxicologically approved maximum residue limits (MRL) for food and feedstuff, which must not be exceeded when reaching the trade/consumer level], *e.g.* for pesticides, plant protection products (ANONYMOUS 1991-2004), for biocides (ANONYMOUS 1998). Further it includes the proper disposal of obsolete pesticides (*e.g.,* GTZ 1999).

PCB – and to a certain extent also HCB – have been used worldwide as industrial chemicals for a multitude of technical applications for several decades. Further details may be explored *inter alia* from OEBERG (1996) and ANONYMOUS (1999). Due to their recalcitrance against degradation to environmentally harmless products and their bio-accumulation potential they pose a significant ecotoxicologic risk. Therefore, it is necessary to obtain sound knowledge as to the various emission sources and their extent (inventories) and to monitor their concentrations in selected, *i.e.,* ecologically representative materials.

Polychlorinated dibenzo-*para*-dioxins and polychlorinated dibenzofurans (PCDD/PCDF) comprise of highly toxic by-products of industrial processes involving chlorine. Sources of dioxins include paper and pulp mills, hazardous waste incineration, sludge from waste facilities, cement kilns that burn chemical waste, and the manufacturing of PVC plastics and some pesticides. As, up to now, their emission is obviously inevitable, a powerful legal framework must be elaborated on a national basis by which the effective and long term protection of man and the environment can be secured.

This implies the fixation of scientifically sound emission limits (TEQ for PCDD/PCDF in air, *e.g.,* of chimney stalks, stack emissions), the improvement of technical processes for the reduction of emissions (Best Available Technology = BAT), the establishment of monitoring programmes and highly qualified and well-equipped laboratories for the unambiguous detection and determination of the different dioxin/furan congeners in environmental samples.

As it lies in the nature of matter and experience legal prescriptions are of little value if and as long they are not substantiated by respective administrative rules and – which is even more important – by competent administrative and technical bodies (laboratories) which can effectively execute the abovementioned legal requirements.

The POPs comprise – with respect to the Stockholm Convention (2001) – twelve defined organic chemicals (for the time being) that are known for their widespread (transboundary) distribution *via* various transport means (air, water, biota, atmosphere) and/or longterm (eco)toxicological relevance – irrespective of their intended use. These chemicals have evoked international concern which resulted in an agreement laid down in the UNEP "Guidance for a Global Monitoring Programme for POPs" (ANONYMOUS 2004). Among other items this document contains basic needs and premises and suggestions for their national and transnational monitoring by analysing them in selected matrices.

2 Malaysian-German Pesticide Project

2.1 Introduction

During the last 30 years a great number of projects in developing countries had been realized where national and international donor agencies were involved. These projects covered almost all areas of human activities where deficiencies had become apparent and where the donors were asked for financial and/or technical input, or both.

Among these projects quite a number dealt with the implementation of analytical laboratories for the control and surveillance of pesticides/biocides in agriculture and other areas (*e.g.,* locust control, termite control) (Sievers *et al.* 1986).

The following remarks on the project described below shall elucidate its historic development, the experiences gained and the overall result. It was conceived to cover a broader range of sub-goals, therefore, it will be dealt mainly with the planning and implementation of a pesticide formulation and residue laboratory under the umbrella of the Malaysian Federal Ministry of Agriculture, Kuala Lumpur.

Due to increased demand of food for home consumption and for exportation in many developing countries special projects were made up, where the particular interest was stressed towards improvement of agricultural production with the help of fertilizers and plant protection products (pesticides). As pesticides had to be imported from a few industrialized countries (*e.g.,* USA, Great Britain, France, Japan, Germany), a particular responsibility laid with these countries which in turn were asked for technical and financial support to reduce the problems which were – to a certain extent – "co-imported" with these agrochemicals.

As a matter of self-understanding these countries had to develop effective strategies and build up special national agencies which became independent as executive bodies for transformation of the respective rules and regulations regarding the judicious use of these chemicals.

The German contribution for making up projects, specially for pesticide formulation control and residue analysis, since the foundation of the German Agency for Technical Cooperation (GTZ) in 1975, mounts up to a number of long-term projects in more than ten developing countries whereby it has to be noted that the extent of input (financial, technical, human power) varied broadly from country to country (Sievers *et al.*, 1986).

The following text passages are excerpted from the Project Completion Report of the Malaysian-German Pesticide Project (Ahmad and Walter-Echols, 1993).

2.2 Project Identification and Goal(s) of the MGPP

In 1978 the Government of Malaysia submitted a request for technical assistance to the Federal Republic of Germany to implement its Pesticides Act of 1974 and "to train local chemists on residue analysis and also to obtain foreign expertise and equipment in the establishment and operation of a new pesticide residue laboratory".

The following benefits were expected by Malaysia from this project:
- Safeguarding the population from pesticide residues detrimental to their health
- Protecting fish, birds and other beneficial organisms in the environment
- Ensuring that export of agricultural produce is not adversely affected
- Providing information on degradation rates of pesticides under local conditions
- Guiding farmers on the safe and effective use of pesticides.

As the result of an inspection visit to Malaysia of a German expert from the Pesticide Project of the GTZ it was recommended to the German Ministry of Economic Cooperation to support the Malaysian request. The subsequent German project offer included a proposal to use the planned laboratories in Malaysia for regional activities in the field of pesticide residue and formulation control within its technical cooperation programmes.

The Technical Cooperation Agreement between the two Governments to establish this project was signed in 1983. In this agreement the Government of the FRG resolved to assist the Department of Agriculture, Malaysia, in particular the Secretariat of the Pesticide Board (the national registration authority for pesticides) in strengthening and upgrading its capabilities and facilities for the control of pesticides under the Pesticides Act 1974. The German project contributions were to be provided through the above named GTZ, which prepared a project offer in December 1983, and – after its approval – recruited the experts for the project who arrived in Malaysia in September 1984. The project was called "Malaysian-German Pesticide Project" (MGPP) and was established as a cooperation between the Federal Department of Agriculture (DOA) in Kuala Lumpur and GTZ.

At the time of the project request the agricultural sector contributed approximately 50 % to the gross national product (GNP) and employed about half of the population. These figures had dropped to about 33 % of the working population and about 18 % of the GNP in 1990, being the third largest employer as a sector. The National Agricultural Policy aimed at improving farm and national income from agriculture.

This means that besides new land development, the productivity of existing systems were to be increased through a higher use of technical input (mainly fertilizer and pesticides) and/or shifting to crops with an increasing demand such as vegetables and fruits.

2.2.1 Pesticides in the 1980s

2.2.1.1 The Pesticide Market

In 1978 there were about 180 active ingredients of pesticides available in Malaysia. The use of pesticides steadily increased over the years, and in 1988 sales of agricultural pesticides amounted to Malaysian dollars (Ringgit) M$ 341 million. Since then, the agrochemical market had declined to M$ 276 million in 1992, while the public health market increased to M$ 145 million. In 1990 approximately 400 companies had registered about 1,500 types of pesticides comprising 275 active ingredients. Approximately 50 % of the pesticides were imported. Due to an estimated 8,000 pesticide dealers the products were available all over the country. Approximately 200 stores were run by the Government. One of the problems that needed to be addressed were the use of unsuitable, adulterated or spoiled products that had been occasionally sold in the market.

2.2.1.2 *Use of Pesticides*

Pesticides were mainly used on rubber, oil palm, cocoa and paddy. About 75 % of the total pesticides were herbicides which were used for weed control in rubber and oil palm estates. Insecticides were applied as a matter of routine on vegetables, tobacco and paddy and, according to pest occurrence, on pepper, cocoa and oil palm. Fungicides were mainly used on highland vegetables as well as in fruit, pepper and tobacco. Rodenticides were used in oil palm estates and paddy fields.

Besides the big estates, smallholders, specially vegetable farmers, increasingly intensified their pesticide input. Often insecticides and fungicides were applied regularly once a week or – during the raining season – as often as every three days.

2.2.1.3 *Pesticide Residues*

Due to the intensive pesticide use in vegetables contamination – particularly with methamidophos and dithiocarbamate fungicides – was often found to exceed the maximum residue limits (MRL). Consumers became more concerned about food contamination and consumer associations publicised the problems arising from an improper use of pesticides.

High residue levels became a problem to vegetable dealers, especially if they wanted to export their products. Importing countries expanded their import screening and rejected goods which exceeded the permitted/permissible residue concentrations. In early 1987 Singapore rejected vegetable imports from Malaysia due to high dithiocarbamate residues. During two weeks vegetables to the amount of M$ 3 million were destroyed. Pepper had been rejected by Hungary and cocoa by certain US importers. Brunei which imported vegetables from Sabah and Sarawak had also set up an import control in 1988 and rejected vegetables from these two states in 1993.

2.2.1.4 *Pesticide Poisoning*

Pesticide poisoning was a serious problem, especially among the Indian estate workers. Out of about 5,000 human poisoning cases between 1979 and 1988, about 2,000 involved pesticides. Many of the poisonings were suicides using paraquat-containing herbicidal products. Reasons for poisoning were the easy availability of pesticides and their improper use. Application was usually carried out by so-called knapsack sprayers and usually no protective clothes were worn.

2.2.1.5 *Pesticide Legislation*

Under the Poison Ordinance of 1952 eight organophosphate insecticides and the organochlorine pesticide endrin were banned due to their high mammalian toxicity. Later, the control of pesticides in Malaysia came under the purview of the Pesticides Act 1974. The Department of Agriculture (DOA) was then entrusted with the responsibility of implementing the provisions of this Act. The Pesticide Section was created in 1971 under the Crop Protection Branch of the DOA, became the secretariat to the Pesticides Board and became responsible for the implementation of the Act.

Applications for the registration of pesticides were evaluated by the Technical Committee which had been formed in 1983 by the Pesticides Board and consisted of representatives of ministries and other national research bodies.

In order to implement the Pesticides Act effectively, rules and regulations were drafted under the relevant sections of the Act. By 1993 four sets were gazetted:

1. Pesticide (Registration) Rules 1976
2. Pesticides (Importation for Educational and Research Purposes) Rules 1981
3. Pesticides (Labelling) Regulations 1984
4. Pesticides (Licensing for Sale and Storage for Sale) Rules 1988.

2.2.1.6 Control of Pesticide Residues in Food

Part IV of the Pesticide Act 1974 provided for the control of pesticide residues in food. With the passage of the Food Act 1983 and the Food Regulations 1985 the control of pesticide residues in food became the responsability of the Ministry of Health.

According to the Food Regulations all products for the local market had to satisfy certain requirements concerning quality (*e.g.,* water content, protein content), package, labelling and durability, additives and preservatives and pesticide residues.

Schedule 16 of the Food Regulations specified the MRL permitted in food.

2.2.2 Implementation of the Project

2.2.2.1 Project Goal

The final goal reads: Damage to man and the environment caused by pesticides is minimized. Even though the legal control of pesticide residues in food became the responsibility of the Ministry of Health, it remained with the responsibility of the DOA to encourage farmers to use the correct production methods and that agricultural produce to be free from excessive pesticide residues.

2.2.2.2 Project Concept and Design

Project activities were planned according to the logical framework of Target-oriented Project Planning (TOPP) methodology which stipulated the formulation of the results to be achieved, the activities to be executed, the indicators to determine result achievement and basic assumptions to be considered.

The project as whole was split into five phases (1984-1993), of which the first three phases (1984-1990) will be focussed in this paper.

The objective of the first phase (1984-1986) had been to strengthen the facilities for the implementation of the Pesticides Act 1974 (“Establishment Phase”). The project infrastructure was established and activities concentrated on strengthening the existing Formulation Control Unit and establishing the Residue Control Unit and its laboratory. In a first step purchase, installation and set-up for analytical equipment were executed and complemented with adequate training programmes.

In the course of the first joint evaluation of the project (November 1985) the evaluation committee recommended that the MGPP be extended for more two years.

The second phase (1986-1988) was aimed at implementing the Pesticides Act 1974 ("First Implementation Phase") and the objective was to strengthen pesticide control. The plan of this phase took into account the commitment of the Pesticide Section to service some provisions of the Food Regulations 1985 (analyses and surveillance of pesticide levels in food, definition of "NIL" value, and assistance in defining the appropriate residue levels of certain pesticides in food items).

Purchase of laboratory equipment was pursued and the formulation and residue laboratories were nearly fully equipped by the end of the 2nd phase. Activities of the first phase were intensified. The number of samples analysed increased and new analytical methods were adopted in both laboratories. Supporting programmes were initiated, such as a system for data management, for dissemination of pesticide data for extension purposes and for disposal of unwanted pesticides and laboratory wastes.

The third phase (1988-1990) started after the second joint evaluation in 1987. Besides Peninsular Malaysia the two Federal States Sabah and Sarawak (both Borneo) were included in the project and it was recommended to set up a regional residue laboratory in each state.

A new goal was formulated "Pesticides are used more judiciously" and two objectives were stated:

1. "Pesticide control is strengthened"
2. "Extension programmes on pesticides are strengthened".

The main activities concentrated on residue analysis, toxicology and the establishment of a computerized data management system for pesticide registration. The Residue Control Unit with its residue laboratory was fully operational at the end of this phase.

From Sabah and Sarawak vegetable samples were analysed in the residue laboratory in Kuala Lumpur (KL) and analysts from Sabah and Sarawak were trained in pesticide analysis. Two regional residue laboratories, one in Sabah and one in Sarawak, were initiated and equipped with basic instruments and supplies.

The fourth phase (1991-1992) finalised the project activities with the Pesticide Section in KL and concentrated on the establishment of the new residue laboratories in Sabah and Sarawak.

The fifth and final phase covered most of the pilot implementation of the pesticide extension programmes and the upgrading of the new pesticide laboratories in Sabah and Sarawak.

Project field activities ended in August 1993 and were followed by a joint review of the project which included a planning workshop for the continuation of the project-initiated activities after the termination of the German support.

2.3 Sustainability and Continuation of Project Activities

The end-of-project review found that Malaysian counterparts valued MGPP's achievements and made plans for sustaining both major project thrusts – pesticide laboratories and participatory farmer training – after the project had terminated.

2.3.1 Pesticide Laboratories

The laboratories, namely the pesticides formulation laboratory and the pesticides residue laboratory, which had been the main thrust of the project were well realised and proved to be sustainable. They were completely institutionalised, staffed with appropriately trained officers, and poised to continue to function to a high standard with adequate Malaysian government support. The review recommended that the laboratories would continue monitoring pesticide use and residues, functioning as centres of specialised training, playing watchdog for pesticide use problems at farm level, and communicating data in useful formats to extension officers, researchers and policy makers. In addition the DOA wished to consider assuming responsibility for monitoring environmental pollution caused by agricultural pesticide use and/or a certification programme for agricultural products.

Several demands were formulated to improve on long run the already existent effectiveness and efficiency of the laboratories, *inter alia*:

- to increase staff to make fuller use of the analytical equipment
- to upgrade constantly technical expertise of staff
- to extend residue monitoring to additional crops and pesticide active ingredients
- upgrading of facilities in some laboratories and maintenance of a linkage to GTZ, perhaps through GTZ's Pesticide Service Project, for continuing technical assistance
- better-targeted and supervised sampling for residue monitoring
- faster reporting in more appropriate formats and increased cooperation between the Agriculture, Health, Environment and Chemistry Departments
- joint Agriculture/Health sampling workshops to promote effective residue monitoring and enforcement.

2.3.2 Malaysian Contributions

Personnel

The MGPP was established as an activity of the Department of Agriculture (DOA) and the Malaysian project staff were employees of the DOA. Depending on the tasks required in each project phase the local counterparts to the German experts varied from phase to phase.

The Residue Unit of the Pesticide Section in KL was staffed with one chief chemist, two junior chemists and five lab assistants. All three chemists and four of the lab assistants attended specific training courses locally and overseas.

Equipment

At the beginning of the project the DOA made available the already existing laboratories in Kuala Lumpur (including certain basic laboratory equipment) as well as office furniture and equipment to the value of approximately M$ 520,000 (approx. equivalent to 350,000 DM).

Laboratory equipment was purchased for the laboratories in Sabah (approx. M$ 40,000 for equipment and materials) and Sarawak (approx. M$ 50,000 for equipment and renovations) (together M$ 90,000, approx. equivalent to 60,000 DM).

It should be mentioned that a number of decisions as to selection, purchasing and installation of sophisticated lab equipment and instruments which were considered for the pesticide residue laboratories had been made in close cooperation with the Formulation Control Unit (pesticide formulation laboratory).

In the laboratory of the Residue Unit, Kuala Lumpur, some basic equipment was available at the beginning of the project (gas chromatograph, laboratory drying oven, vacuum rotary evaporator, laboratory vacuum pump, analytical balance, refrigerator, freezer, household blender, laboratory mill and basic lab glassware). In the following years DOA purchased further chromatographs (capillary GC and HPLC) for the residue laboratory at a cost of M$ 450,000.

The equipment of the residue laboratory was planned following a list provided by the GTZ Residue Project (Darmstadt, FRG). Major equipment which required repair and spare parts were ordered through local agents, whenever possible, to ensure that proper follow-up service would be available.

Facilities

The buildings and the costs for construction and renovations were fully borne by the Malaysian partner. The building for the Pesticide Section (containing the formulation and residue laboratories) was available from the beginning of the project. During implementation several minor modifications were undertaken.

The facilities of the Residue Unit in the Pesticide Section in Kuala Lumpur comprised of the following: sample store/freezer room, sample preparation room, washing-up room, main laboratory for sample homogenization, extraction, cleanup *etc.*, gas-chromatography room, office. Basic lab furniture (benches, cupboards), water supply, fume hoods and basic air-conditioning were already installed.

Some rooms in the Agricultural Research Centres in Sabah and Sarawak were renovated so that they could adequately accommodate the residue laboratories.
The estimated total value of the Malaysian contribution summed up to M$ 1,12 million (approx. equivalent to 750,000 DM).

Running costs

The DOA met the operating and maintenance costs of the project from its regular budget. This included the service contracts with the instrument manufacturers, costs for maintenance of the laboratory equipment at the facility in Kuala Lumpur, vehicle operation and maintenance, chemicals, technical gases, glassware, spare parts, training materials, office supplies, telecommunication *etc.*

German contributions

In the five Project Agreements (1983, 1986, 1988, 1991, 1993) the Government of the Federal Republic of Germany (FRG) offered contributions for the MGPP, totalling in about 10.7 million Deutsche Mark (DM) which covered:

– - Personnel (long-term and short-term consultants, scientists for special studies, local staff)
– - Materials
– - Administration and operating expenses
– - Counterpart training.

Major technical equipment to the value of approx. M$ 546,000 (equivalent to approx. 364,000 DM) was purchased by GTZ (gaschromatographs with ECD and NPD, integrators, auto-injectors, refrigerators/freezers, vacuum rotary evaporators, laboratory dishwasher, high performance liquid chromatograph with post-column reactor).

2.3.3 Activities and Results

Lectures and on-the-job training

All members of the technical staff were constantly supervised and trained on several topics of residue analysis, *inter alia*:

Sampling

- Preparation and storage of samples
- General procedures in the laboratory including quality control
- Extraction
- Cleanup (adsorption and gel-permeation chromatography)
- Operation and maintenance of gas chromatographs
- Gaschromatographic methods for pesticide residue analysis
- Interpretation of chromatograms
- Calculation and interpretation of results including descriptive statistics
- Derivatisation techniques.

Lectures were held on special topics, which were open to the laboratory staff of the whole Pesticide Section, *inter alia*:

- Principles and applications of chromatographic methods, incl. gas chromatography
- Good laboratory practice
- Evaluation of chromatographic results
- Multi-residue analytical methods.

Establishment of standard residue analysis methods

It was the aim to implement practicable technical instructions (standard operating procedures) on the basis of already published and approved methods for the analysis of residues of as many as possible active ingredients in the different matrices (plant materials, soil, water). These methods had to comply with the national MRL List of pesticide residues in food. For 21 organophosphates (OP) (GC/NPD), 17 organochloro compounds (OC) (GC/ECD), 16 N-methylcarbamates (NMC) (GC/NPD and HPLC/post-column derivatisation/FluD), four synthetic pyrethroids (SP) (GC/ECD) and three herbicidal aromatic acids (GC/ECD, after methylation with diazomethane) multi-methods were implemented for various kinds of vegetables. For some active ingredients which were of great importance and could/can only be analysed individually appropriate methods were implemented: paraquat/vegetables, dithiocarbamate fungicides/vegetables (headspace GC/ECD, as CS_2), methylbromide/rice (GC/ECD), carbofuran and metabolites/fruits (HPLC/UV), carbendazim and benomyl/fruits (HPLC/UV). The above methods were only accepted and implemented after thorough successful validation of the complete procedure.

Quality assurance programme and quality control

During the initial phase of the project, the laboratory staff were constantly instructed and supervised by the German experts, and all chromatograms obtained, their interpretation, and the calculated results were individually checked and verified. Only after the staff members were individually and adequately trained and had acquired considerable experience they were allowed to carry out the analyses independently, but the results they obtained were still checked by the chemists in charge. In cases where high concentrations of pesticide residues were found in the samples or where other obscurities were noted, the analyses were repeated and only after reconfirmation to ensure that, *e.g.,* no cross contamination had occurred, the result was approved.

When the laboratory first started to analyse samples of unknown residue concentrations, duplicate samples were sent to a laboratory in Germany for confirmation.

For internal quality control, recovery tests with spiked blank samples at various concentration levels of the analytes in question were carried out at regular intervals.

Apart from internal quality control measures, the laboratory participated in international ring tests (round-robin studies), organised by the German Chemical Society (GDCh = Gesellschaft Deutscher Chemiker, Working Group "Pesticides") and the GTZ Pesticide Service Project.

From 1986-1992 the Residue Unit participated successfully in five ring tests (pesticides in urine, carrot powder, cocoa butter, vegetable oil, vegetable extract).

Additional quality assurance measures were undertaken in form of cross-checks between the laboratory in KL and the two regional laboratories in Sabah and Sarawak, which turned out to be of very high informative value, so that it was planned to establish this instrument to be realized on a regular basis (twice a year).

Monitoring of pesticides in food

A preliminary study was carried out in 1985 to obtain information on the extent of pesticide contamination in vegetables in various parts of Malaysia, whereby also experiences as to sampling, sample reduction, transport, storage and documentation were gained. These and samples from further collection actions in other parts of the country were analysed using multi-residue methods for organophosphates, organochlorines, and synthetic pyrethroids.
Great emphasis was placed on the analysis of ethylene bis-dithiocarbamate (EBDC) fungicides (*e.g.,* mancozeb, maneb) in Malaysian vegetables after the Singapore government reported high EBDC residues in selected produce. Batches with excessive residue concentrations were confiscated and destroyed by the Singaporean authorities thus causing great financial losses to the farmers. Measures taken by the DOA to rectify the situation included the analysis of samples prior to export and dialogues with the farmers to advise them

against overspraying of EBDC fungicides shortly before harvest. From 1984 until 1992 the Residue Unit analysed 4,760 samples (1,783 on OP, 731 on OC, 479 on SP, 2,893 on EBDC, 276 on other active ingredients).

2.4 Summary

The residue laboratory of the Pesticide Section in Kuala Lumpur was established during the first three phases of the MGPP (1.9.1984-31.8.1990) and was fully operational by the end of the third phase.

The laboratory was completely equipped with modern analytical instruments by GTZ, but was increased and upgraded parallel and lateron by the counterparts. Besides on-the-job training by long-term and short-term consultants, all three chemists and four of the five lab assistants attended specific training courses overseas and locally.

After six years of project implementation standard methods to determine pesticide residues in food (mainly vegetables) were available for about 70 active ingredients. Residues in food could be determined for almost all pesticides classified as ‘extremely’ or ‘highly hazardous’ by WHO and registered in Malaysia. Regular participation in international ring analyses (round-robin studies) showed that good quality analytical results were obtained by this laboratory. To further ensure good laboratory (analytical) practice, an internal quality assurance programme for the laboratory was established maintaining the principle of traceability (although at that time this technical term was not yet “invented”).

The main activity of the residue laboratory was the monitoring of pesticide residues in food, especially vegetables. A preliminary study showed that problematic pesticides were mostly insecticides of the OP group and EBDC fungicides. With emphasis on those two groups, monitoring of residues in vegetables were carried out on a routine basis. In general, the problem of high EBDC residues appeared to be mainly restricted to leafy vegetables produced in the highlands, while high organophosphate residues were found predominantly in the lowland vegetables. To determine the degradation behaviour of pesticides under Malaysian climatic and production conditions trials were carried out with 12 pesticides on different vegetables at two locations (highlands, lowlands).

Apart from the regular monitoring of residues in various agricultural produce, the Residue Unit of the Pesticide Section in KL also carried out specific projects in cooperation with other government agencies to study the dissipation behaviour of pesticides applied in the fields and other aspects of pesticides affecting the food crops and the environment.

Much time was spent on the development of analytical methods to expand the capabilities of the laboratory. On request of other government departments and international organisations, trainings were given at the laboratory to share the knowledge and experience of the laboratory staff.

Due to the great distance between Peninsular Malaysia (KL) and the two East Malaysian Federal States Sabah and Sarawak, it was decided that separate pesticide residue laboratories be established in the two states for more effective monitoring of pesticide residues in crops grown locally. In the third phase of the MGPP activities were extended to Sabah and Sarawak. During that period of time the laboratory staff in KL – together with the GTZ experts – supported the East Malaysian counterparts in providing advice and assistance in the planning of their laboratories. Samples taken from the two states were sent for analysis to the Residue Unit in KL and the staff was given additional training to get prepared for their work. Support for the East Malaysian residue laboratories continued until the staff was able to analyse the samples in 1990 independently. Continued support was given lateron on an ad-hoc basis whenever needed.

Up to 1993 the residue laboratory in the Pesticide Section in Kuala Lumpur was continuously upgraded so that it became one of the best equipped pesticide residue laboratories in Malaysia. The laboratory conducted method testing and development schemes in order to keep abreast with the dynamic pesticide industry.

As the two residue laboratories in Sabah and Sarawak had almost the same history of origin, it was decided not to present at length the respective details as of the residue laboratory in Kuala Lumpur.

From the time of initiation the three laboratories had undergone changes in their sampling and reporting which can be summarized as a three-step development:

Level 1:
- Extension to inform farmers of laboratory results
- Random field sampling

Level 2:
- Identification of problem crops for research/extension
- Direct sampling (4-6 crops, incl. markets)
- Summary (annual) reports for management and other departments

Level 3:
- Cooperation/complementation of work with other departments/ministries
- Trend/special reports suitable for policy decisions
- Investigative sampling by lab staff (progress monitoring; tracing problems; environmental sampling).

The three DOA pesticide residue laboratories had a theoretical instrumental capacity of almost 20,000 samples per year under optimal staffing and budget at the end of 1993. The actual numbers, however, were lower, since many factors had to be taken into consideration, such as that some equipment could only be used for specialised tasks, their age and the number of staff. In 1992 the sample output on the average exceeded 50 % of the theoretical instrument capacity which could be deemed an acceptable result and also as an indicator of the efficient use of the instruments.

The residue data provided by these laboratories were aimed to be – and were – used by the Pesticides Board, the Federal DOA and the State Departments of Agriculture in Sabah and Sarawak, resp., for further action (*e.g.,* the withdrawal of the use of methamidophos-containing products on leafy vegetables after respective residue results were delivered by the residue laboratory, valuable information to the authorities after vegetable exports were rejected by Singapore in 1987 and Brunei in 1993).

In addition the laboratories served as specialised training centres for local staff in residue and formulation analysis and for the development of new methods in an effort to further upgrade knowledge, skill and competency in pesticide control and management. The scope of analysis had been increasingly broadened to cover more types of active ingredients of pesticide products with the introduction of new analytical methods in all three laboratories.

The pesticide residue laboratories greatly strengthened the position of the DOA in its efforts for better pesticide control and collaboration with other departments. Close cooperation between the DOA and the Ministry of Health were established at federal level and in the project regions, and continuing cross-checks between the DOA and the Chemistry Laboratories enhanced the consistency and reliability of residue analysis.

2.5 Costs and Benefits

The project evaluation 1987 concluded that it is virtually impossible to assess the cost-benefit ratio for this type of project. The benefits from health risk prevention are difficult to assess in economic terms. In the absence of reliable statistics on pesticide-related health effects in Malaysia the benefits would have to be based on speculations only.

It is not so much the question whether the laboratories and extension activities are economically justified since it is a government's moral obligation to protect its citizens from harmful effects of pesticide residues in food. The question is rather to what extent residue analyses are necessary to secure the health, quality and trade objectives, and how can pesticide residues be effectively reduced at minimum cost.

Residue control is based on a tripartite relationship between monitoring, education and enforcement. It is, therefore, not so important how economically one component operates, but how well the whole system functions. Maximum cost-benefit can only be obtained when all three components work optimally together. When one component fails, the cost-benefit ratio is adversely affected.

Pesticide laboratories whose data are neither used for farmer education or enforcement purposes are a very expensive luxury. Pesticide safety and residue control does not happen in the laboratories. Even if a laboratory works cost-effective and its expenses are only a fraction of the total crop value or export earnings, the expenditures would not be economically justified when data are not used. A cost-benefit analysis of a laboratory must therefore always include an analysis to what extent the expenditures are balanced with those for extension and enforcement.

2.6 Recommendations for Future Activities to Sustain Project Efforts

Recommendation 1

Close collaboration between the Pesticides Board and Agriculture, Health, Chemistry and Environment should be maintained on an operational level, both within and between States, to facilitate effective pesticide management in Malaysia in accordance with the Pesticide Act (agriculture), the Food Act (Health) and the Environmental Act (Environment).

Recommendation 2

In line with Malaysian government policy to promote integrated pest management (IPM), crop protection training initiated under the MGPP should be broadened to IPM, while not losing sight of the project's original concern for judicious pesticide use. This is the best way to meet farmer's expressed need for training on effective and economic pest control while minimising pesticide residue and health problems.

Recommendation 3

In the long run DOA residue laboratories should continue to:

- Enable the implementation of the Pesticide Act 1974 through high-quality post-registration monitoring of pesticide use and of residues in food;
- Serve as centres for specialised training in pesticide residue analysis and method development;
- Provide an early-warning system for pesticide residue problems at farm level through systematic monitoring, and
- Summarise and communicate pesticide residue data in appropriate formats in support of extension and research programmes and agricultural policy decision making.

Recommendation 4

The DOA may wish to consider applying its superior knowledge of pesticide use patterns and pesticide residue monitoring capacity to monitor environmental pollution caused by agricultural pesticide use, and/or to a certification programme for agricultural products. The priority and feasibility of these additional activities would have to be considered very carefully, however, because of the additional investment of resources that may be required, as well as legal, logistical, and other complications connected with certification.

Recommendation 5

Initiatives taken to procure accreditation from the Standard Industrial Research Institute of Malaysia (SIRIM) for the DOA residue laboratories are a valuable contribution toward maintaining the quality of analyses and should be further pursued.

Recommendation 6

In order to sustain and enhance the pesticide residue laboratory programmes:

- The number of staff should be increased to make fuller use of the analytical equipment and the technical expertise of staff should be upgraded continuously;
- Upgrading of facilities could be considered for some labs, depending on activities undertaken, and
- Pesticide residue monitoring should be extended to additional crops and pesticide active ingredients (*e.g.,* additional fungicides and carbamate insecticides, as well as newer pesticides, such as insect growth regulators).
- A flexible technical backstopping linkage with GTZ should be maintained with regard to information, international inter-laboratory cross-checks and, perhaps, further sponsorship of training in new technologies and analyses. This could be possible *via* GTZ' Pesticide Service Project in the immediate future.

Recommendation 7

Residue laboratory staff should participate in sampling at times so that they are better informed and motivated, and can ensure that proper procedures are followed.

Recommendation 8

Residue sampling should be designed – perhaps with a statistian's input on sequential sampling – to obtain enough samples to produce conclusive results for each crop within a reasonable time frame to indicate whether or not residues are a significant problem; the easiest way to obtain a random monitoring sample is in the market, while tracing back an identified problem to its source may require field samples.

Recommendation 9

Joint Agriculture/Health residue sampling workshops should be held at state level to train officers in the proper design and targeting of coordinating sampling programmes.

Recommendation 10

Appropriate summary reports should be prepared regularly by all laboratories for management and other departments, as well as special reports on trends or key topics that can be used as a basis for making policy decisions.

Recommendation 11

Residue analysis results should be used not so much to warn a few individual farmers who have misused pesticides, but rather to guide the DOA in targeting problematic crops/chemicals/regions for investigative follow-up sampling and general extension training, and to evaluate the impact of extension efforts.

Recommendation 12

With regard to pesticide information systems,

- steps taken by the DOA to continue the MGPP project's efforts to establish a pesticide information system that can produce printed informational materials for extension and advisory services are commendable; and
- DOA's extension-oriented pesticide information system should allow for integration into a larger, user-friendly system that assures networking between Agriculture, Chemistry, Health, Environment and Food laboratories and programmes.

3 QUALITY CONTROL IN ORGANIC TRACE ANALYSIS

This chapter summaries the lecture given at the UNEP workshops in Montevideo, Uruguay, and Beijing, China, in September and December 2005, respectively.

- Any human activity incurs the chance of making mistakes and/or committing errors.
- In chemical trace analysis we are confronted with two principal sorts of errors:
  - Identification of analyte (chemical compound)
  - Quantification of analyte (amount, concentration)

For the generation of correct analytical results certain basic human qualities are indispensable:

- intelligence
- scrutiny
- sincerity
- fortitude
- (self-)consciousness
- endurance
- discipline

The risk to produce erroneous results/data is associated with technical, material and methodological reasons:

- Lowering of limit of detection (LOD)/Limit of quantification (LOQ) of analyte to detect/determine
- Recalcitrance of the analyte/matrix
- Complexity of the matrix
- Increasing number of analytes in the same matrix
- Malfunctioning of analytical instruments

Further reasons:

- Time (hurry)
- Budgetary limitations
- Organisational handicaps
- Competitive interests (demands and claims)

Definition of quality assurance (QA)

An integrated system of management activities involves

- planning, - implementation, - assessment, - reporting and - quality improvement

to ensure that a process, item, or service is of the type and quality needed and expected by the client.

Definition of quality control (QC)

The overall system of technical activities that measures the attributes and performance of a process or service against defined standards **to verify** that they meet the stated requirements established by the costumer; operational techniques and activities that are used **to fulfill** the requirements of quality. The system of activities and checks used **to ensure** that measurement systems are maintained within prescribed limits, providing protection against „out of control“ conditions and ensuring the results are of acceptable quality.

Internal QC

Samples
- Handling, treatment, storage, reduction, disposal

Reagents and chemicals
- Purity (p. a. and better, e.g., residue grade)
- Storage
- Handling
- Disposal

Reference substances (and materials)
- Acquisition (certified purity degree)
- Documentation from receipt to disposal („life cycle“)
- Handling and storage
- Confirmation of identity
- Disposal (or re-analysis).

Validation steps of a residue analytical method
- Step 1: Knowledge of physico-chemical and environmental properties of target analyte
- Step 2: Search for the best suitable separation and detection principle and techniques
- Step 3: Aquisition of high purity reference/calibration standard substance (> 95 %)
- Step 4: Determination of the lowest detectable amount of target analyte (detector sensitivity)
- Step 5: Determination of the linear-dynamic range of the detector for target analyte
- Step 6: Selection of appropriate reference standard, as internal standard or/and for Relative Retention Time (RRT) determination
- Step 7: Confirmation analysis of analyte (incl. reference standard)
- Step 8: Reagent blank
- Step 9: Reagent blank spiked with analyte and reference standard
- Step 10: Blank sample [without analyte] with reference standard
- Step 11: Blank sample spiked with definite amount of analyte (recoveries)
- Step 12: Analysis of Certified Reference Material (CRM) (if available)
- Step 13: Analysis of unknown sample(s)
- Step 14: Confirmation analysis
- Step 15: Calculation of results and statistical check
- Step 16: Archiving of all raw data.

External QA and QC (Proficiency tests)
- Re-analysis of known samples by other (befriended) laboratory(ies) (between-laboratory studies)
- Interlaboratory proficiency studies – ring tests or round robin studies (RRS)

Round robin studies (RRS)

Definition

A method validation study involving a pre-determined number of laboratories or analysts, all analysing the same sample(s) by an appropriate method. All results of a RRS are compared and used to develop summary statistics, such as interlaboratory precision and method bias or recovery efficiency.

The purpose of a RRS is to assess the capabilities of analytical laboratories to determine residues of definite environmental contaminants in definite samples.

Prerequisites („Musts“)
- Laboratory provided with modern functioning infrastructure (state of the art)
- Laboratory fully equipped with adequate instrumentation
- Staff sufficiently qualified and motivated
- Staff familiar and experienced with adequate analytical methods and modern technical equipment
- Head of institution/laboratory convinced of the need of external QC
- Sufficient budget secured
- Sufficient time for analyses provided
- No hurry while carrying out RRS analyses
- Average time budget for QC (which can be > 30 %)
- Reliable institutions existent for organising RRS (incl. sample shipment, evaluation of results, further support)
- Warranty of anonymity of participating laboratories
- Analytical method to be decided by the laboratory
- Responsible superior must not impose a negative image on the analyst(s) in case of false or/and deviating or missing results
- Sober and objective analysis of all deviations reported in the official RRS document necessary (no blame to anyone involved in the RRS!)
- Enough time provided for repetitive analyses to track down errors and deviations.

Reporting of RRS results
- Total number of participating laboratories
- Number of results
- Number of not reported data
- Evaluation procedure and calculation incl. statistics
- Number of valid results
- Number of outliers
- Recommendations

Reporting of RRS results (Statistics)
- Minimum value
- Maximum value
- Arithmetic mean value
- Median (50-percentile)
- Z-score (standard score)

Z-score Definition (see Figure 1)

Z-scores are a special application of the transformation rules. The z-score for an item indicates how far and in what direction, that item deviates from its distribution's mean, expressed in units of its distribution's standard deviation.

The mathematics of the z-score transformation are such that if every item in a distribution is converted to its z-score, the transformed scores will necessarily have a mean of zero and a standard deviation of one.

Z scores are sometimes called "standard scores". The z-score transformation is especially useful when seeking to compare the relative standings of items from distributions with different means and/or different standard deviations.

Z scores are especially informative when the distribution to which they refer, is normal. In every normal distribution, the distance between the mean and a given z score cuts off a fixed proportion of the total area under the curve. Statisticians have provided us with tables indicating the value of these proportions for each possible z-score (HOFFMAN 2005).

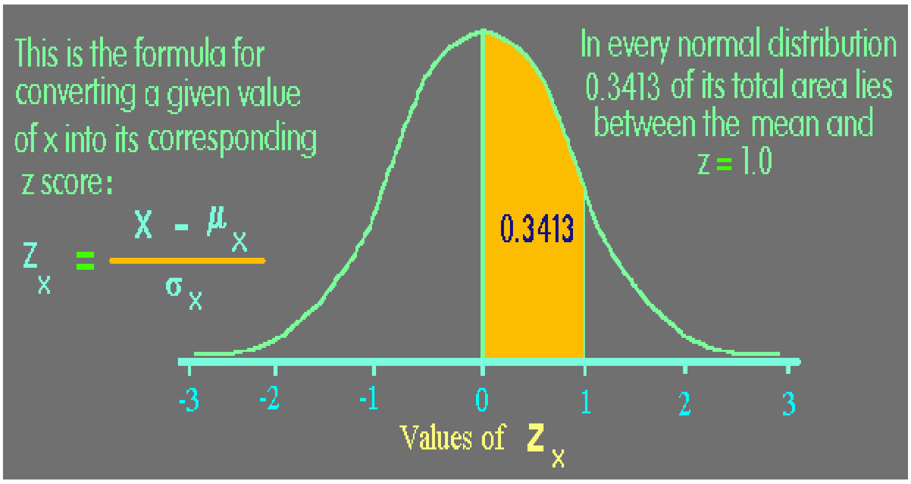

Figure 1: Z score

Reasons for major deviations from nominal data (examples)

- Analytes erroneously not reported (false negative)
- Analytes erroneously reported (false positive)
- Analytes not correctly quantified (outliers)

Other reasons for erroneous or not reported results (examples)

- Reference substances not available
- Inadequate storage conditions and time
- Separation problems (chromatographic system)
- Detector problems (sensitivity loss)
- Calibration errors (wrong standard solutions or/and dilutions, defect dilutors, pipettes)
- Confirmation failed or not done
- Limited capacities and/or time
- Personnel limitations

Conclusions

- RRS are an excellent means of quality control to demonstrate the capabilities and deficiencies of a trace analytical laboratory
- Participation in RRS helps to improve the self-consciousness and to strengthen the preparedness to self-criticism of the analysts
- RRS results can be taken as a classification mark in comparison with similar institutions (national and supranational)
- RRS may be considered as a suitable alternative to accreditation or/and certification schemes

- Analytical data of RRS-participating laboratories may facilitate acceptance by other institutions
- In the future RRS will become a standard tool due to increasing requirements as to quality and better comparability of environmental data.

4 PREREQUISITES OF A POPS LABORATORY

4.1 Criteria for Constitution of a POPs Laboratory that Generates Reliable Results that Are Accepted Worldwide

The following remarks may serve as a baseline document on analysis of past experiences and lessons learned from projects where laboratories have been planned and established with the assistance and through either funding by (foreign) donor agencies or by national/local governments. This document contains information on what has worked and what did not work in these projects.

The approach to POPs analysis can be assumed under the general scope of (ultra)trace analysis ranging from mg/kg (biocidal POPs in soil, waste) to ng/kg (PCDD/PCDF in soil, biota). With respect to PCB in dielectric fluids their contents may range from 0.1 % to <50 mg/kg. In sediments of harbours PCB can accumulate up to approx. 200 mg/kg dry matter (ANONYMOUS 1999).

Technical criteria (infrastructure, building, facilities)

As residue analysis differs significantly from macro analysis, a POP laboratory, which is primarily dedicated to (ultra)trace analysis, should be located apart from potential contamination sources. This is particularly important with respect to PCB that are found ubiquitously and are widely spread. Therefore, the interior construction and decoration materials should be approved before use by the later user/occupant of the building in order to avoid permanent (creeping) contamination due to constant evaporation and spoiling of the lab atmosphere. Particular attention should be given to construction and decorum materials such as paints for walls and ceilings, for wooden elements such as doors, window frames, furniture, for radiators and tubings (water, technical gases, sewage water, *etc.*), tile seams, insulation materials (electric cables, *etc.*).

As to installation of pipes for technical gases (*e.g.,* for gas chromatographic apparatus) utmost care must be secured in selecting the appropriate materials (oil-free tubing and fittings, high quality gauges and valves) and special construction techniques (welding with shielding gas, hard soldering), which should be carried out exclusively by special companies with the necessary equipment and experienced staff.

The tenderer (future responsible user) should describe the necessary criteria in the tender. They should be certified in writing before start of construction by the main contractor. In the course of construction of the building the future user should assure himself of the implementation of the contractual details by frequent visits of the construction site.

In case a special POPs laboratory cannot be set up and an already existent laboratory must take over this task, a well-defined area (*i.e.,* number of rooms) with sufficient space must be reserved and prepared accordingly.

The laboratory should be equipped with sufficient safety devices, fume hoods, effective air circulation, with smoke detectors in each room, intrusion protection, and protection devices against water damage.

The laboratory atmosphere should be physiologically admissible and must not influence the quality of chemicals, reagents and measurements. The room temperature should be constant at 20 °C (± 2 °C), and the relative humidity should be set at ≤ 60 %.

To fulfil these requirements the laboratory has to be equipped with controllable heating devices (central heating) at sites where temperatures can fluctuate beyond the set-point (*e.g.,* in countries with temperate climate). Air-conditioning devices in the cleanup room and measuring room (split air-conditioner devices) should be generally installed (not only in tropical and subtropical countries, but also in other climatic zones) because of large daily temperature differences even in the summer season. In the measuring room where several GCs and computers normally work over day and night the average temperature in the summer season can easily climb up to 35 °C and higher, if not controlled by effective air-conditioning.

Where the complete building is equipped with a central air-condition device, the separate installation of individual air-conditioning is – though causing higher initial costs – still economic, as its continuous operation can be maintained while the central air-conditioning will be switched off (*e.g.,* over night and weekends, during public holidays).

For the reduction of solar radiation heat in the rooms adjustable sun-blinds should be installed in front of the windows (either outside or inside the lab).

Energy supply

Adequate electricity supply is necessarily required for the reliable functioning of the technical and analytical instruments. There should be sufficient electric circuits, including three-phase current, installed in the respective rooms. The electric power supply and installation should be at a voltage within the designated range (230 V, 380 V) and protected from mains spikes and earth leakages. If good (reliable) electricity supply cannot be guaranteed (*e.g.,* due to lightning, installation faults, overloading, corrosion), local transformers and/or regulated power supplies (uninterruptible power supply, UPS) should be installed at least for the smooth running of sensitive instruments (chromatographs and electronic data processing devices).

Gas supply

For the control of gas leaks (*e.g.,* hydrogen as GC carrier gas and burning gas for NPD and FID) the GC room should be equipped with a hydrogen detector (desirably battery-supplied).

Water supply

Drinking water will be normally supplied *via* the municipal water distribution enterprise and will be regularly checked as to chemical and sanitary quality according to relevant legal regulations. Therefore, many requirements as to the water quality for laboratory uses are normally fulfilled.

For special laboratory uses water purification devices (*e.g.,* reverse osmosis, de-ionization, or distillation) should be provided.

Although it may be considered as a matter of course, it is pointed out again: For liquid chromatographic use (HPLC) water should never be taken from the tap, but only be purchased from producers who can provide the necessary specifications (*e.g.,* HPLC grade) (see Reagents).

Sewage and waste disposal

Effective sewage collection (contaminated with lab chemicals) and disposal should be provided and so-called household sewage should never be mixed up with laboratory sewage.

A special disposal system must be implemented for bulk chemical wastes and used organic solvents, as the latter can be recycled. For this purpose specially labelled containers and an independent registration procedure should be implemented.

Technical security

The laboratory building or at least the laboratory rooms should provide means for restricted access either by visual control (*e.g.,* gate keeper) or electronic means (*e.g.,* biometric chips).

Effective means for fire prevention, red alarm and other unexpected events should be provided. The local fire brigade should be informed in detail about the "hot spots" of the laboratory [storage facilities for chemicals, solvents and other (highly) combustible materials, compressed technical gases].

A telephone hotline (emergency call) to the local fire brigade and to the nearest emergency transport or/and the local hospital should be installed.

Necessary rooms and facilities

The size of the laboratory and the number of rooms may differ depending on the requirements and needs.

The minimum number of rooms should comprise of the following:
- So-called wet lab: room(s) for sample preparation and cleanup
- Measuring room(s): room(s) with the measuring instrumentation (GC, HPLC, GC/MS AAS, spectrometry and peripheries, as instrument control and electronic data processing)
- Balance cabinet: with micro/analytical balance(s) and fume hood
- Storage rooms: for solvents and inflammable bulk chemicals, reagents and fine chemicals (separate one for each category)
- Separate storage facility for high purity analytical standards (e.g., calibration standards of pesticides, metabolites, POPs) (separated refrigerated storage at + 4 °C)
- Freezing facility (- 20 °C): central deep freeze cabinet or sufficient single freezers (separate ones for samples and thermolabile chemicals)
- Storage rooms for materials (*e.g.,* packaging materials), consumables, equipment and spare parts
- Sufficient office premises for all personnel
- Staff room with kitchenette (also to be used if no separate meeting room is available)
- Meeting room
- Lavatories and locker rooms
- First-aid room (rest room).

Equipment and instrumentation

Apart from the standard equipment of analytical laboratories there are special requirements as to the equipment (for sample homogenization, extraction, cleanup, purification/concentration) and analytixal instruments [capillary column gas chromatographs with electron-capture detector (ECD) and/or mass-spectrometric detector (GC/MSD) with the respective electronic tools for control, storage and calculation of data] which have to be fulfilled for the successful operation of a POPs laboratory (for details: ANONYMOUS (2004): "Guidance for a Global Monitoring Programme for Persistent Organic Pollutants").

Personnel

Depending on the basic concept and number of tasks to be covered by the laboratory the minimum requirements as to lab organisation, personnel and their particular qualifications (dependent on the individual position) are as follows:

- Head of the laboratory (lab manager) (one, full-time): academic, preferably chemist with main focus in organic chemistry and experienced in analytical chemistry, further qualifications in at least one foreign language (preferably English), interested in and open for establishing scientific contacts to other – national and international – institutions with respect to POPs analytical and associated questions; capable to maintain the laboratory's standards against political and other (non-scientific) arguments; having managerial/microeconomic capabilities for the sound and transparent financial planning (*i.e.,* cost effectiveness)
- Graduated chemist or chemical engineer (one, full-time), experienced in analytical chemistry, responsible for development of analytical methods and their introduction and optimization, supervision and training of lab staff, in-house repairs, and maintenance of technical devices and instruments (as far as possible and reasonable); deputy function.
- Laboratory assistants (minimum two, full-time)
- Worker (one, full-time) (room make-up, transport, local purchasing, *etc.*)

Health and safety precautions in the laboratory (Occupational health and safety)

Since this document cannot not cover all detailed information regarding health and safety precautions of laboratory personnel it has to be referred to general rules, which should be laid down under the relevant schemes of the responsible authorities in each country and should be accessed from the respective official sources on demand (*e.g.,* USA: http://www.osha.gov, Great Britain: http://www.hsl.gov.uk/about-us/index.htm, Germany: http://www.zchl.uni-sb.de/zchl/sicherheit/laborrichtlinie/).

4.2 Criteria for Sustainability of a POPs Laboratory

To learn more about the concept of setting-up a functioning POPs laboratory and maintain it in a sustainable manner it is referred to a comprehensive paper elaborated by SARGENT and MACKAY (1995). This compendium is worth reading as it covers almost all important aspects regarding prerequisites for the successful functioning of a trace laboratory, including POPs analysis (for further reading, see also PRICHARD *ET AL.* 1996).

From the experiences made with the set-up of laboratories in developing countries, the following conclusions and recommendations should be taken into account.

- Political stability of the countries is needed on long run.
- Economic stability in the countries will have a catalytic effect on the realization of the various technical elements, demanded by the Stockholm Convention.
- A legal basis for the establishment of a new POPs laboratory or the specification of an existing laboratory for the regular POPs analysis according to SC should be established which can withstand short-term political, organisational, administrational and/or financial mood changes.
- In case that due to certain shortfalls a country may not be in the position to fulfil certain SC requirements by own resources the cooperation with other countries' laboratories or/and laboratories of the private sector should be achieved on a contractual basis.
- Governments of countries, which are short of resources, but willing to work under the scheme of the SC should not hesitate to seek support (financial, technical, personal, informational) from national and international donors.
- A long-term financing system must be developed on a legal basis to warrant the stable establishment of competent and experienced institutions with well-trained and experienced scientific and technical personnel.
- Regular training of personnel (at all working levels) must be scheduled as a standard educational element.
- The employees/officers of a POPs laboratory need continuous support, which includes financial incentives (whenever possible) and other good contractual conditions, in order to keep them for long time at the laboratory. There is the permanent risk that skilled and experienced staff leaves the lab because of attractive alternatives from the private.
- Intra- and supranational exchange of information on POPs analysis and results, methodologies, technical hints and experiences in/among the laboratories (*e.g.,* workshops) should be established for accelerating spread of knowledge (and reducing the risk of duplication of work).
- As trace analysis needs high-sophisticated technical instrumentation not only the purchase of such equipment must be thoroughly planned and executed, but also the maintenance and short-term repair (service contracts) and short-term purchase of spare and wear parts and upgrades (software) of control and electronic data processing has to be secured.
- The budget regulations must be such flexible that in case of equipment average (*e.g.,* fire, water, intrusion) immediate remedy can be procured in order to avoid a longer standstill of the laboratory.
- The purchase of chemicals, reagents, certified reference materials, high-purity analytical reference substances (*e.g.,* pesticide standards) must be procured on a sound basis; short-term additional provision of finance must be possible.
- For all laboratory equipment and consumables (*e.g.,* glassware, spare and wear parts, instruments, data processing hard- and software) competent local vendors are needed.
- Any POPs laboratory has to establish a quality assurance programme with a sophisticated quality control system.
- The achievement of accreditation of a governmental POPs laboratory makes only sense, if it is there are enough qualified personnel available that understand the accreditation system principles and have an affirmative mental position to it.

- If accreditation of a POPs laboratory is desired the necessary budgetary, organisational, technical efforts and the high investment in time must be considered and included in the financial system and workplan.
- If a POPs laboratory can document (*via* its internal AC/AQ system) that its analytical results comply with external norms and fulfils the requirements of clients, the access to an accreditation scheme should be critically considered.
- Cooperation with other laboratories, working in similar fields, can be very useful (looking beyond one's own nose).
- Accreditation may be achieved as the existence of an accreditation certificates may increase the confidence in the working quality and results.
- The standardisation of analytical methods for POPs in environmental samples – often and controversely discussed – may be acquired, however, every laboratory should better stick to its own elaborated and successfully tested (*e.g.,* round-robin studies) standard operating procedures (SOPs).
- To maintain the state of the art/technical standard in a POPs laboratory the responsible lab manager has to assert thorough survey about improvement of instrumentation and other lab equipment, *e.g.,* accelerated solvent extraction (ASE) for saving solvents and of man power.

5 Addresses of Potential Donors

In order to enable the interested reader to get into contact donor organizations which could be engaged in terms of technical cooperation for the establishment of POPs laboratories or the improvement of existing laboratories which are virtually selected for future POPs analysis, a list is added which contains addresses of potential governmental institutions, that have been/are engaged in providing technical, personal and/or informational support on a project basis.

In the context of this report the author tried to contact some national and international organisations from which he assumed that they could be considered as potential donors with respect to the Stockholm Convention and the establishment of POPs laboratories in developing and threshold countries.

The author's inevitably limited knowledge obtained through inquiries (internet search, telephone calls, E-mails) can be summarized as such, that none of the below mentioned organisations is actually engaged in the SC-POPs Project. This does not necessarily mean that there might be no chances of acquiring funds and/or technical and/or informational support. In general such inquiries must be launched by the respective governments or via their authorized organizations to be answered adequately (see under Conclusions and Recommendations).

Alphabetical address list of potential donor agencies (not comprehensive):

Danish International Development Agency (DANIDA)
Asiatisk Plads 2
DK-1448 Kobenhavn K
Denmark
Tel: 45 33 92 00 00
Fax: 45 32 54 05 33
Email: um@um.dk
Web site: http://www.um.dk

Deutsche Gesellschaft für Technische Zusammenarbeit (GTZ) GmbH
Dag-Hammarskjöld-Weg 1-5
D-65760 Eschborn
Germany
Telefon: 49-(0)6196-79-0
Telefax: 49-(0)6196-79-1115
Web site: http://www.gtz.de/de/index.htm

Japan International Cooperation Agency (JICA)
6th-13th floors, Shinjuku Maynds Tower
1-1, Yoyogi 2-chome, Shibuya-ku
Tokyo 151-8558
Japan
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7 ACKNOWLEDGEMENT

The author would like to thank Mr. W. A. Schimpf, GTZ, Bonn, for his supportive discussions, the provision of documents and the thorough reviewing of the manuscript.