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PESTICIDE DEVELOPMENT PROGRAMME IN INDIA

DP/IND/89/128

INDIA

Technical report: Findings and recommendations*

Prepared for the Government of India
by the United Nations Industrial Development Organization,
acting as executing agency for the United Nations Development Programme

Based on the work of Dr. K. Pavel, consultant of quality
control of pesticides

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Vienna

* This document has not been edited.

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0. Explanatory Notes

- The numbering of divisions and subdivisions in this report was made according to International Standard ISO 2145.
- "Author" means the writer of this report.
- Abbreviations (in alphabetical order):

AAS	Atomic absorption spectroscopy
B.Sc.	Bachelor of Science
CIPAC	Collaborative International Pesticides Analytical Council
EPA	Environmental Protection Agency (US)
FAO	Food and Agricultural Organisation (UN)
FDA	Food and Drug Administration (US)
GLC	Gas Liquid Chromatography
GLP	Good Laboratory Practice
HIL	Hindustan Insecticides Limited
HPLC	High Performance Liquid Chromatography
ISO	International Organisation for Standardisation
M.Sc.	Master of Science
PDC	Pesticides Development Centre of the Government of India
PDPI	Pesticide Development Programme India
Ph.D.	Philosophiae Doctor
QA	Quality Assurance
QC	Quality Control
RENAPAP	Regional Network on Pesticides for Asia and the Pacific
TLC	Thin-layer chromatography
UNDP	United Nations Development Programme
UNIDO	United Nations Industrial Development Organisation
WHO	World Health Organisation (UN)

1. Abstract

Title of Project: Quality Control of Pesticides

Number of Project: DP/IND/89/128/11-60

Objective: To introduce the personnel of PDC to the principles of GLP and to inform them of the fundamentals of quality control.
To advise on general improvements in the analytical laboratory.

Duration of Activity: 6 weeks from 13 March to 24 April 1990 including a two weeks regional RENPAP workshop.

Location of activity: Pesticides Development Centre at Gurgaon (Haryana) near New Delhi.

Two lectures were given and one technical session was chaired by the author during the RENPAP workshop.

Two research institutes were visited in New Delhi

The following recommendations were made:

- to improve the electric power supply as soon as possible at PDC
- to ensure that instruments temporarily out of function be repaired as soon as possible
- to improve the reputation of the Pesticide Development Centre and thus achieve a higher utilization of the equipment
- to improve safety measures
- to give more consideration to protective measures regarding the environment

2. Introduction

This report was written by Dr. K. Pavel, Unido Consultant on Quality Control of Pesticides. It covers the period from 12 March 1990 to 24 April 1990. During this time the author was assigned to the Pesticides Development Centre (PDC) of the Government of India at Gurgaon, Haryana, near New Delhi. According to the Job Description he, in collaboration with his project counterparts, was expected to provide advice on improvements to the analytical laboratory and other activities in analytics and, in particular, on Good Laboratory Practice, Quality Control and Quality Assurance. Additionally it was expected that the author attend the "Regional Workshop on Pesticide Formulation Technology" from April 9 - 21 1990, organized by UNIDO and RENPAP (Regional Network on Pesticides for Asia and the Pacific).

Not all of these expectations were described precisely enough to start appropriate activities immediately. The promotion of newer formulations and advice on substitution of imported material by locally available raw materials did not comply with the special knowledge and experience of the author as an analyst.

A number of discussions with the general manager of PDC, Dr. Kawal Dhari, the head of the Analytical Dept. Dr. Pandey and Dr. Dhua of Hindustan Insecticides Limited (HIL) took place with the intention of clarifying the situation. On 16 March the author was informed that he was expected to give two lectures at the regional workshop on April 10th and April 19th and to take the chair of the technical session IV on April 19th. In a provisional schedule two themes for the lectures were suggested.

By visiting the different laboratories (analytical lab, labs for formulation development, for synthesis and for bio-assay) as well as the pilot plant, and talking to the staff the author endeavoured to get to know the personnel of the PDC and to get an impression of the work going on there. Part of the time was taken up with the preparation of lectures and later the attendance at the RENPAP workshop. Altogether the allotted time was too short to gain a satisfactory insight of the work of PDC to be able to make a profound judgement.

3. Activities

According to his understanding of his mission at the Pesticides Development Centre the author saw the objective of his work in discussing analytical problems which might possibly exist and to make the personnel of the analytical department familiar with the terms of quality control and quality assurance (Annex V) and with the principles of Good Laboratory Practice (Annex VI).

In a discussion with the members of the analytical department and some scientists of PDC about his programme of work the author was asked to give a brief survey of his field of activities in his company where he was head of an analytical research institute. Contrary to his expectations, no actual questions referring to analytical methods were put to the author. Actual instrumental problems seemed not to exist. However, a few other questions were asked, e.g. "What does Bayer do with the residues of the pesticide production, especially of phosphorus pesticides?" and "Does Bayer still produce phosphorus pesticides in spite of their relatively high toxicity?" or "What is the reaction of the people in Germany to big chemical accidents such as that in Bhopal?". The author gave relevant answers, which seemed to be accepted, but he expressed his opinion that the answering to such kind of questions was not the main purpose of his consultant activity as an expert on quality control and good laboratory practice.

To obtain further clarification of his task at PDC he asked for a list of problems or a programme of desired work in writing. Such a list was handed to him on 26 March. (Annex I)

3.1 Programme of work

The "Programme for Dr. Pavel" was suggested by Dr. Pandey, head of the analytical dept. at PDC, and is reproduced as Annex III. For carrying out this programme the author proposed a time table which assumed that the duration of his mission would be extended until approx. 5 May according to the UNIDO letter PRU/90/PRAS/APP/PRUS of 27 February 1990. (cf. Annex II). But on 29 March the author was informed that the end of his mission was fixed as 25 April. As a consequence the programme had to be reduced as shown in Annex IV Section a.

3.2 Findings

3.2.1 Purpose of PDC/Analyt.dept. and its equipment.

The Pesticide Development Centre (PDC), formerly Pesticide Development Programme India (PDPI), is a UNDP/UNIDO assisted project of the government of India. The research centre is located at Udyog Vihar, Gurgaon in Haryana, approximately 25 km from the centre of New Delhi. The complex of several buildings contains also analytical laboratories. These are equipped with sophisticated and expensive modern instruments including:

GLC-Instruments for the use of packed and capillary columns and different detectors.

GLC-Instrument with mass-spectrometric detector

HPLC-Instruments

Super critical fluid (SCF) chromatograph (still out of operation due to missing accessories)

Infrared spectrophotometer

UV/VIS spectrophotometer

Atomic absorption spectrophotometer (AAS)

Thermobalance (out of function due to missing accessories)

Preparative chromatography column with pump and fraction collector

Analytical balances, Thermostat, Ultrasonic bath,

Polarograph, Autoliner, Rotating evaporator,

Water purification set for the preparation of Water of HPLC-Quality.

Other instruments for the examination of chemico-physical properties of formulated pesticides are installed in the laboratories for formulation development. The analytical activities of PDC are not centralized in the analytical dept. but some are attached to other groups, e.g. the synthesis laboratory and the clay research laboratory. The actual "analytical laboratory" is mainly engaged in the determination of active ingredient contents in technical and formulated pesticides and in the development of analytical methods therefore. Samples are submitted by members of PDC, e.g. the formulation pilot plant or by external clients.

An important task of the PDC and the analytical dept. is to act as a centre for transfer of technology and information, which is realized during training courses, e.g. organized by RENPAP.

3.2.2 Personnel

The analytical group consists of 7 professionally trained persons. One Ph.D. as head of the analytical dept., four M.Sc.'s and two B.Sc.'s, all with professional experience of between 3 and 20 years. The state of training appears good in particular with regard to the handling of instruments and the methods for which each one is responsible. Some members of the staff have stayed in western countries for some time, e.g. in the USA, in Austria (Linz) and the UK. Most of them said that they were able to work with all instruments in the department. Some declared themselves as being experienced with only a few instruments. Due to the fact that only very few samples were submitted to the analytical laboratory for analysis during the time when the author stayed at the PDC, it was not possible for him to get a complete impression on the versatility of the personnel. During the RENPAP workshop the personnel of the analytical dept. was engaged in preparing and conducting experimental demonstrations in a training programme for chemical, chromatographic and spectrophotometric methods.

4. Recommendations

4.1 General

A most remarkable observation made by the author was the discrepancy between the copious and expensive equipment of the analytical dept. with instruments and the comparatively small number of analyses really carried out with the aid of this equipment. The efficiency of the instruments - perhaps with the exception of one GLC- and one HPLC-instrument - seems to be very low. Additionally, the very unsteady supply of electric power reduces the efficiency. Normally, electric power is supplied by the state of Haryana. But from experience this can be interrupted

without previous notice up to 5 and more times per day during working hours. In such cases a chromatographic or spectrometric analysis as well as certain computer work is interrupted and becomes worthless, and valuable time is lost. Under such circumstances it is possible, that not one single analysis is completed per day. In case of break-down of the power supply a diesel generator must be started.

It is strongly recommended that the second diesel generator, which is already in the power house, but not connected up be put into action as soon as possible, so that a steady power supply can be realized independent of the public supply from Haryana.

Other reasons for the insufficient utilization of the instrumentation are the low number of external orders for analyses and - partly - the fact that some instruments cannot be used because certain accessories are missing, e.g. a thermobalance, a supercritical fluid chromatograph, a Desaga autoliner etc. It is recommended that the reputation of PDC be improved and its facilities be more intensively promoted and that a part of the financial aid be put aside for repair, restoration and supplementation of instruments instead of buying new ones.

In the course of his lecture on GLP the author pointed out that cleanliness of facilities and instruments is a prerequisite for ensuring correct results. In this respect there is some room for improvement. It is not sufficient to put some chemical bottles in order, to wear laboratory coats, gloves and safety goggles only during a training programme in the presence of the participants of a training course. It should become selfunderstanding to observe a minimum of safety precautions at all times. An institution such as PDC, in particular the analytical dept. should be an example of cleanliness and a model in performance of GLP. These principles were discussed several times with the personnel and examples were given.

4.2 Safety precautions and environmental protection

The application of personal protective precautions was mentioned above. It should be added that bottles with eye rinsing liquid should be in each laboratory. Safety containers or cupboards should be available for the storage of low boiling and highly inflammable solvents.

Waste fluids e.g. from the laboratories and the pilot plant flow out of the buildings into an open drain around the buildings and finally onto a fallow field. Pesticides and formulation dyestuffs are to be found there. Solid wastes, e.g. tested samples are buried in the near neighbourhood. Within a radius of a few hundred meters around PDC there are several wells. In the neighbourhood of the institute the state of Haryana and the Community of Gurgaon have started laying out residential and agricultural areas. It should be remembered that based on the experience of other countries, e.g. in central Europe, pesticides can migrate over greater distances in the ground water. Thus a dangerous threat to the environment arises. The sooner this problem is given consideration the better, i.e. it is worth providing appropriate means to improve the situation.

Routine checks on safety and environmental precautions should be layed down in writing in the form of standard operating procedures.

4.3 Instrumental equipment

So far as can be judged after a relatively short stay at the PDC the sophisticated "High tech" analytical equipment can be considered as being almost complete. The progress in instrumental analysis forced ahead by the instrument-building industry is so fast that it is not always possible to work with the most modern instruments. Many developments and improvements are basicly not so important that it is absolutely vital to have the most recent model of an instrument. It is more important that the available instruments be utilized and that the personnel be trained in applying the different methods, e.g. for the determination of active ingredient contents and other parameters, than buying further expensive instruments. For the near future some

financial aid should be made available for the procurement of small laboratory equipment e.g. room thermometer, automatic burettes, piston burettes, motor-burettes, portable pH-meter, cork rings, piston pipettes and other glassware. A small vacuum cleaner would be useful for many purposes. A special equipment for thin-layer chromatography (TLC) i.e. a sufficient supply of precoated plates with fluorescent layer, developing chambers and UV-lamp should be available.

Means should be provided for the procurement of analytical reference materials, as the preparation of primary reference substances is very expensive, requires a considerable amount of experience and should be undertaken only in exceptional cases.

Finally a number of books e.g. CIPAC proceedings 1 - 3, FAO - specifications, Pesticide Manual, some ISO Guides and certain ISO Standards should be available in the library.

Comment on the Regional Workshop on Pesticide Formulation Technology.

In the course of the workshop 25 lectures were given in 9 days. All lectures showed a high scientific level and the amount of knowledge and information which was presented to the participants reached sometimes the limits of perceptive ability, particularly as differences in the pronuntiation of the English language and the noise of the fans and the air conditioning system in the conference room did not make understanding any easier. The distribution of copies of the manuscripts made it possible for the participants to read the lectures later. In any case at such conferences the speakers should be continually urged to speak louder, slower and more distinctly. (e.g. by a signboard)

As for the laboratory, pilot plant and experimental analytical demonstrations it must be noted that the number of approx. 20 participants per group is too large. For future workshops or training courses one should try to limit the number of participants per training or demonstration group - if possible - to less than 10 persons.

Nevertheless the workshop as a whole proved very useful, informative and of high quality, not least thanks to the engagement of the scientists of PDC.

Programme for Dr. Pavel

(proposed by Dr. Pandey on March 26th 1990)

1. Visits to local organisations:
 - 1.1 Central insecticide laboratory, Faridabad
 - 1.2 Shriram Institute for Industrial Research
 - 1.3 Bureau of Indian Standards, Shahibabad
 - 1.4 Artee Minerals, Faridabad
2. Activities during training programme:
 - 2.1 Lecture on Good Laboratory Practice
 - 2.2 Lecture on Quality Assurance/Quality Control of pesticide formulations
 - 2.3 Practical exercises, chemical analyses.
Practical exercises and problems and their solution
3. Laboratory Problems at PDC:
 - 3.1 Instruments maintainance
 - 3.2 Practical laboratory problems and their solutions
 - 3.3 Instrumental problems and their solutions
 - 3.4 Laboratory exercises, improvements and guidance
 - 3.5 Sample storage, disposal and sampling technique
4. Lectures for PDC:
 - 4.1 Good Laboratory Practice
 - 4.2 Maintainance of laboratory quality control,
sample handling
 - 4.3 Sampling techniques, storage and disposal
 - 4.4 Method development for pesticide estimation by
 - a) colorimetry / titrimetry
 - b) GLC / HPLC

Comments to "Programme for Dr.Pavel" (Annex I)

As for 1.: From the intended visits to local organisations only the Shriram Institute for Industrial Research (SRI) in Delhi was visited on 12 April together with Dr. Pandey, Dr. Shasha and Mr. Agarwal. A lively discussion with Dr. Dhingra, Chief of the Toxicological Services of SRI developed, particularly as Dr. Dhingra knows several colleagues of the author at Bayer AG in Germany, where Dr. Dhingra stayed for some time. The well equipped and well maintained analytical laboratories of SRI were visited. SRI is engaged in the analysis of various materials including pesticides and in developing new analytical techniques. Among others SRI elaborates data (analytical, physical-chemical, environmental and toxicological) which are also needed for registration purposes.

On 16 April the Indian Agricultural Research Institute in Delhi (Dr. Parmar) was visited together with Dr. Pandey and Dr. Shasha. After a lecture given by Dr. Shasha on slow release formulations of pesticides only a limited space of time was left for the author to give a short lecture on the (historical) development of analytical methods for pesticides and quality control during the last 30 years.

As for 2.: Lectures on Good Laboratory Practice, Quality control and Quality assurance were held at the RENPAP workshop (cf. Annex II). During the practical exercises some advice was given although only very few questions on analytical problems were raised by the participants.

As for 3.: On items 3.1, 3.4, and 3.5 several discussions took place. On item 3.3 detailed discussions arose on the problem of how to carry out preparative column chromatography to obtain highly purified fractions of pesticide active ingredients as reference materials. The author referred to his rich experience in this field. The item 3.2 is so vague that nothing can be said to this point.

As for 4.: 4.1: A lecture was held during the RENPAP workshop.
4.2 to 4.4 : Discussions on these items took place when inspecting the different facilities of PDC.

Time Table to "Programme for Dr. Pavel".

Scheduled by Dr. Pavel on 27 March.

		Remarks / actual course
27.3.	Tu } Discussions with members of PDC	
28.3.	We } analytical dept.	
29.3.	Th } Writing of lectures for the	
30.3.	Fr } RENPAP workshop	
2.4.	Mo	
3.4.	Tu	Holiday in India
4.4.	We }	} failed to take place } reasons unknown } no permission?
5.4.	Th }	
6.4.	Fr }	
9.4.	Mo	Regional Workshop, Inaugural session
10.4.	Tu	Key Note Session
11.4.	We	Technic. Session I, Demonstrations
12.4.	Th	" " II " Visit to Shriram Institute
13.4.	Fr	" " III "
14.4.	Sa	Sight seeing "Delhi and its Monuments"
15.4.	Su	Visit to Agra and Sikandra
16.4.	Mo	Special session on safety
		Visit to Indian Agricult. Research Institute
17.4.	Tu	Technic. Session IV Demonstr.
18.4.	We	" " V "
19.4.	Th	" " VI
20.4.	Fr	" " VII
21.4.	Sa	Valedictory Session
		Chairman Dr.Pavel + lecture Evaluation of course Debriefing with Dr.Sugavanam
23.4.	Mo	Lectures for PDC on GLP and sampling
24.4.	Tu	" " " on Titration in anhydrous solvents
25.4.	We	Lectures on PDC on storage and disposal of samples
26.4.	Th	Visit to local organisations
27.4.	Fr	" " " "
		} failed to take place } because the consultant } had to finish his mission } on 24 April
30.4.	Mo }	}
1.5.	Tu } concluding discussion with	
2.5.	We } analytical department	
3.5.	Th } preparation of final report	
4.5.	Fr }	
5.5.	Sa	Preparation of return
6.5.	Su	Return Flight

Section a: Reduced time table :

23.4.	Mo	Discussion on laboratory problems with Dr. Pandey, Mr.Taliwal and Mr. Goel. Afternoon: Summarizing discussion of the Workshop at UNDP-Office and at HIL-Bureau
24.4.	Tu	Preparation of return at PDC, Farewell party at Gurgaon.
25.4.	We	02.20 a.m. Flight to Frankfurt - Düsseldorf

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