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**STRENGTHENING OF PESTICIDE DEVELOPMENT CENTRE**

**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 John P. G. Wilkins,  
consultant in pesticide analysis**

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\* This document has not been edited.

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Explanatory Notes:

GC gas chromatography  
MS mass spectrometry  
MSD Mass Selective Detector (manufactured by Hewlett Packard)  
IPFT Institute of Pesticide Formulation Technology (previously known as PDC, Pesticide Development Centre)

ABSTRACT

Title and number of project: Development of pesticide analytical capability,  
DP/IND/89/128/A/11.57/J13426

Duty Station: New Delhi, India.

Objective of project: Training of IPFT staff in the application of gas  
chromatography - mass spectrometry to pesticide analysis.

Duration of project: One month, January - February 1993, return mission.

Conclusions:

The staff of IPFT received further training in the theoretical aspects of pesticide analysis, and practical training in the use of the Hewlett Packard MSD. This will enable them to use this facility more effectively in the future.

Recommendations:

The power supply problem is much less severe than previously, so there is probably no need (at the moment) to find an alternative to the Haryana State supply.

In order to consolidate the training given, and to maintain the performance of the MSD, it is essential that the instrument is used on a more routine basis (i.e. at least weekly).

Further training of the member of staff primarily responsible for the maintenance and use of the MSD, Mr Aggarwal, for an extended period (say three months) at a laboratory using GC-MS on a routine basis would be most useful.

## I. INTRODUCTION

This report was prepared by Mr John P G Wilkins, UNIDO consultant in the analysis of pesticides and related materials using mass spectrometry. It covers the period 18 January to 12 February 1993. During this period the author was assigned to the Institute of Pesticide Formulation Technology, Udyog Vihar, Gurgaon, 122016, Haryana. The Institute is situated about 20km from New Delhi, near the Indira Gandhi International Airport. This was the author's second visit to the laboratory.

IPFT is a UNDP/UNIDO assisted project of the Government of India, originally implemented by Hindustan Insecticides Limited (HIL). It is now in the process of becoming an autonomous body, under supervision of the Ministry of Chemicals and Fertilisers, Government of India.

Its objective is the strengthening and improvement of the pesticide formulation industry in India. To this end it provides technical, analytical, consultancy and training services to the industry. The IPFT also assists and co-ordinates the activities of the UNIDO-sponsored Regional Project, RENPAP (the Regional Network on Pesticides for Asia and the Pacific). The research and development facilities comprise the Clay Mineralogy Laboratory, the Pesticide Formulation Laboratory, the Pilot Plant, the Packaging and Safety Section, the Bio-Science Section and the Analytical Laboratory.

The Analytical Laboratory has seven staff and a wide range of modern analytical equipment, including a Hewlett Packard HP5970 series Mass Selective Detector (a "bench-top" GC-MS system), which was installed in 1989.

Theoretical and practical training was given in the use of the Mass Selective Detector during this consultancy.

## II. ACTIVITIES

The first part of the consultancy period was spent bringing the MSD into full operational service. Several relatively minor but troublesome hardware faults were overcome with the assistance of Blue Star Ltd engineers, the local Hewlett Packard agents. After about ten days the MSD was functioning satisfactorily, so a programme of practical training was undertaken and several analytical projects were tackled.

### A. SETTING-UP AND TESTING OF THE MSD.

Having demonstrated that the vacuum and tuning performance of the MSD was satisfactory when no GC carrier gas was being admitted, a new SGE capillary column (25m x 0.22mm id BP-1) was installed and connected to the MSD (direct coupling into the MSD). Hydrogen carrier gas was found to give better results than helium. Head pressure, septum and split vent flows were optimised. The performance of the GC and MS was tested by injections of methyl stearate (the recommended test compound), and shown to be acceptable.

### B. SOFTWARE DEVELOPMENT.

For security, a back-up copy of the operating software was made. File archiving procedures were demonstrated.

Mass spectrometric data for 20 additional pesticides were compiled into the computer library created by the author on his previous consultancy.

### C. PRACTICAL GC-MS APPLICATIONS.

In order to demonstrate the practical application of the MSD to real analytical problems, a range of technical and reference pesticide materials were analysed.

Compounds analysed included: parathion-methyl, cypermethrin, malathion, HCH, endosulfan, DDT, dicofol, quinalphos, phosphamidon, 2,4-D ethyl, monocrotophos, fenvalerate and dimethoate. Reference materials of several pesticide-related compounds were also analysed (e.g. monomethyl acetoacetamide, MMA, and cypermethrin acid chloride).

The set-up and application of "selected ion monitoring" (SIM) techniques were demonstrated, using technical BHC and malathion. The use of SIM mode data acquisition confers greatly enhanced sensitivity (50-100x better than that obtained using full scan mode). The presence of "chloro-HCH" isomers in technical BHC was demonstrated by detection of their very weak molecular ions at  $m/z$  322, 324 and 326. This required the use of SIM in order to obtain adequate sensitivity. A SIM method for the detection of low levels of isomalathion and two malathion homologues in technical malathion was also developed and successfully applied.

Many contaminants were detected and identified. Spectra not previously in the computer library were added.

Several "Standard Operating Procedures" (SOPs) were prepared in order to provide a source of reference for the practical work. These covered start-up and optimisation procedures and the selection of appropriate analytical parameters.

### III. CONCLUSIONS

"Hands-on" training in the GC-MS analysis of a wide range of technical pesticide materials was given. This presented an excellent opportunity for the staff of IPFT to explore and understand the factors important in optimising the experimental acquisition parameters, and to learn and apply interpretation techniques using "real" data.

There is no doubt that the Hewlett Packard MSD is an extremely powerful analytical instrument, and there is no reason why it should not contribute significantly to the future success of the Analytical Laboratory.

### IV. RECOMMENDATIONS

In order that the training given to the staff of IPFT is reinforced and consolidated, it is absolutely essential that the MSD is properly maintained and regularly used. Uninterrupted practical application of the equipment to the analysis of a wide variety of pesticides is without doubt the best training for the near future.

Further training of the principle operator, Mr Aggarwal, for an extended period (3 months) at a laboratory using GC-MS techniques on a routine basis for pesticide analysis would also be very beneficial.

Several reference texts and mass spectral database compilations were recommended for purchase.

The creation of an Indian MSD users' group should be encouraged, in conjunction with Hewlett Packard and Blue Star Ltd. Other forums for the exchange of ideas and practical advice should also be identified and the attendance of the operator encouraged.

Owing to a shortage of computer discs it was not possible to prepare a back-up copy of the 40,000 entry NBS Library. This should be performed as soon as the discs become available.

#### ACKNOWLEDGEMENTS

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UNIDO COMMENTS

The report is based on the return mission of the expert and clearly gives additional improvement effected by his counterparts to the operation of GC/MS equipment. Being an instrument of exceptional value to the institutes the consultant has given very constructive recommendation for continuous training of counterparts both in the institute and abroad and maintenance of a reference library for pesticide samples. The high level of training provided by the author would be very useful for the sustainability of the project.

A creation of MSD users group in India would be a good spin off for the project and would be even useful for comparative work and exchange of reference materials which will be cost effective.