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STRENGTHENING OF PESTICIDE DEVELOPMENT CENTRE
(PHASE II)

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
gas chromatography - mass spectrometry

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United Nations Industrial Development Organization
Vienna

* This document has not been edited.

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Explanatory notes:

Blue Star are local agents for Hewlett Packard Ltd in India.

GC	gas chromatography
HIL	Hindustan Insecticides Limited
MS	mass spectrometry
MSD	Mass Selective Detector (manufactured by Hewlett Packard)
PDC	Pesticide Development Centre

ABSTRACT

Title and number of project:

Development of pesticide analytical capability.
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Duty Station:

Pesticide Development Centre (PDC), Udyog Vihar,
Gurgaon, 122016, Haryana, INDIA.

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

Duration of project: Two months; April-May 1991.

Conclusions:

The staff of the Pesticide Development Centre were trained in the theoretical and practical aspects of pesticide analysis using gas chromatography - mass spectrometry, in order to exploit more fully the Hewlett-Packard 5970 Mass Selective Detector (MSD) which is installed in the Analytical Laboratory.

Recommendations:

To enable optimum use of the Mass Selective Detector (and all the other analytical and computerised equipment on site) it is absolutely essential that a reliable electrical power supply is obtained as rapidly as possible. Once this is provided, the present reluctance to use this valuable equipment will be removed. It is only through repeated practical application of the instrumentation to a variety of problems that experience and confidence will be obtained in the use this very powerful analytical technique.

I. INTRODUCTION

This report was prepared by Mr John P G Wilkins, UNIDO consultant in the analysis of pesticides using gas chromatography - mass spectrometry (GC-MS). It covers the period 4 April to 4 June 1991 (of which one week was personal leave). During this period the author was assigned to the Pesticide Development Centre (PDC) at Gurgaon (about 20km from New Delhi, near the Indira Gandhi International Airport).

PDC is a UNDP/UNIDO assisted project of the Government of India, being implemented by Hindustan Insecticides Limited (HIL) a Government of India Enterprise. Its purpose 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 Centre also assists and co-ordinates the activities of the UNIDO-sponsored Regional Project, RENPAP (Regional Network on Pesticides for Asia and 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 7 staff, and a wide range of modern analytical equipment, including a Hewlett Packard Mass Selective Detector (MSD) which was installed in 1989.

Unfortunately, for a variety of reasons, the potential value of the MSD has not been fully exploited. The objective of this consultancy was to help remedy this situation, through theoretical and practical training of the operators. The theoretical aspects were successfully completed, particularly mass spectrometric interpretation and computer-assisted data reduction. Less time was spent on practical training than was desired because the analytical part of the MSD was not operational for most of the consultancy period.

II. ACTIVITIES

When I arrived at PDC, it was found that the Hewlett Packard 5970 Series Mass Selective Detector was not operational. The engineers of the Hewlett Packard agents in India, Blue Star, traced the problem, after several days, to a faulty printed circuit board in the turbomolecular pump controller. The repair of this component was not possible, so a replacement had to be ordered from the USA. The original cause of this fault was attributed to an attempt to use the instrument with an inadequate electrical power supply.

Fortunately the computerised datasystem of the MSD, an HP 59970MS ChemStation, and the gas chromatograph, an HP 5890A GC, were functioning, so time was spent on software familiarisation and development, and GC maintenance and set-up.

Theoretical discussions took place throughout the period. One formal lecture, an introduction to the theory and application of GC-MS to pesticide analysis, was given.

A. SOFTWARE TRAINING AND DEVELOPMENT

Basic computer house-keeping practices were demonstrated, such as file transfers and archiving. Superfluous files which were occupying most of the computer memory were removed. Data reduction techniques were demonstrated, using test files and PDC data files, particularly the use of chromatograms of selected ions and limited mass ranges.

A powerful facility available on modern GC-MS systems is computer assisted identification of unidentified mass spectra. This is achieved by comparison with reference data. However, this facility must be used with caution as the results may be misleading. The quality of the results is dependent on the quality of the reference data, which is never totally reliable.

Examination of the Hewlett Packard supplied pesticide library (PESTICID.L computer file), which is a subset of 1,084 of the 38,000+ spectra in the main library (NBS_REVE.L), revealed several weaknesses; Names are given in Chemical Abstracts Service format, which are tedious, and sometimes impossible, to interpret (only the first forty characters are given). Molecular weight data, helpful when assessing library search results, are not included, so one has to calculate them from the molecular formulae. A large number of the pesticide library entries (over half) are not pesticides.

Another inconvenience is that the only way of retrieving a spectrum from a library database file is by its library entry number. So if one wishes to find out if a particular compound is represented in a database file, the only way to do it is by trial and error, making use of the fact that entries are compiled in ascending molecular weight order.

In order to avoid this cumbersome procedure, an alphabetic index of pesticides (and some related compounds) included in the supplied pesticide library was generated. Only compounds whose CAS registry number appears in the Pesticide Manual were included in this list, and of the 1084 entries in the pesticide library, only 480 met this criterion.

To facilitate spectral retrieval from the main database, for which generation of an alphabetic index was impractical, a plot of molecular weight vs library entry number was produced.

The library database which gives the most reliable results is one that contains relevant spectra, obtained using identical operating conditions to those used in acquiring the spectrum to be identified. In practice this means that whenever practicable, data generated on the MSD should be used to create a dedicated pesticide library of compounds of interest. A PDC library file (PDC.L) was created, and 230 entries made, using reference data provided by the author. Again, an alphabetic list of entries was produced.

B. GC-MS PRACTICAL TRAINING

Practical training was severely limited because of the difficulties with the instrumentation.

A guide to the identification of pesticide formulation contaminants using capillary GC-MS was prepared ("General approach for identification of pesticide formulation contaminants by GC-MS"). This includes practical information on GC set-up, silanisation of glassware (eg injection liners) and the selection and use of an appropriate GC-MS performance evaluation test mix

C. LITERATURE SEARCH

One day was spent at the Library of the Indian Agricultural Research Institute. Published information on the analysis of pesticide formulations of butachlor, DDT, endosulfan, malathion, phosphamidon and the synthetic pyrethroids was sought, and several useful papers and reports were found.

III. CONCLUSIONS

The main objective of the consultancy was severely impeded by the equipment difficulties described in the report. However, the staff of the Analytical Laboratory were given extensive theoretical training, and practical advice on the use of the Hewlett Packard Mass Selective Detector for pesticide analysis. A large proportion of the project was devoted to application and development of the Hewlett Packard software, with particular emphasis on the creation of a user library of pesticide mass spectra and making the data in the supplied pesticide library more easily accessible.

There is no doubt that the Hewlett Packard Mass Selective Detector is an extremely powerful analytical tool, and I am confident that this instrument can provide valuable information for PDC which it would find difficult or impossible to produce by other means. The staff who use the instrument are well qualified. They lack only practical experience.

A programme of practical work for the next few months has been produced.

IV. RECOMMENDATIONS

The management of PDC should make every effort to ensure the speedy provision of a stable, constant power supply to the Analytical Laboratory. Until this is provided it is difficult to envisage the successful deployment of the Hewlett Packard Mass Selective Detector.

Having discussed this problem with Blue Star staff, there appear to be three possible solutions; use of a generator (or "DG-set"), use of an uninterruptible power supply system, or connection of the facility to the more reliable Delhi supply. Considering these three options in turn:

i) Local diesel-powered generator:

The total power consumption of the MSD and associated equipment is estimated at 5kW. There are two generators on site at PDC, rated at 6 and 62.5kW. Because of the expense of running such generators, due to the high cost of diesel, use of the smaller capacity model solely for the MSD was thought to be the best solution. Unfortunately, this was not so, as the poor performance of this nominally 6kW model probably caused the breakdown of the MSD just before the start of the consultancy. A reliable 7.5kW generator is needed to run the MSD effectively. However if this option is pursued, it may be worthwhile considering a 20kW capacity system in order to supply all the analytical equipment on site, which also suffer from the erratic, local (Haryana State) supply. Use of the 62.5kW system, which is capable of supplying power to the whole site, will prove more costly in the long term.

ii) Uninterruptible power supply:

Such systems are expensive, occupy a lot of space and require careful maintenance. Their limited outputs usually limit their use to computers. This is probably not a practicable solution.

iii) Connection to the Delhi power supply:

The Delhi supply is much more reliable than the Haryana State supply. Connection requires a 500m feeder line. In the long term, this is the most attractive option. Implementation of this option has already been pursued for three years.

- iv) In order that the training given to the staff of PDC is reinforced and made effective, it is essential that the Mass Selective Detector is repaired and put into practical use as rapidly as possible. Uninterrupted practical application of the equipment to the analysis of a wide variety of pesticides is the best training, starting with relatively simple projects and tackling more complex ones as experience and confidence increase.

In a year or so, a further one month consultancy would be of great benefit in consolidating the theoretical training already given, with the practical experience which will have been acquired during execution of the proposed programme of experiments on the MSD.

The management should ensure that the development work with the MSD is allowed to progress with the minimum of distractions.

The possibility of a 3 month placement for the MSD operator at an appropriate research facility should be considered.

- v) The poor (largely non-existent) external telephone service available from PDC hindered progress throughout the consultancy, severely hampering communications with Blue Star and the UNDP office. This should be rectified as soon as possible to avoid the crippling inefficiency it causes.
- vi) Several reference texts and mass spectral database compilations (including a very powerful PC-based package) were recommended for purchase.

The latest release of Hewlett Packard ChemStation software should be evaluated with a view to purchase, as it offers several improvements. The possibility of purchasing the printed edition of the NBS/NIH Mass Spectral Compilation should also be explored. This compilation contains more complete spectral information than is contained in the HP database, in easily comprehended histogram format. As well as the complete IUPAC name, a drawing of the molecular structure for each entry is also included, which greatly facilitates its use.

- vii) The creation of an Indian MSD users' group should be encouraged, in conjunction with Hewlett Packard/Blue Star. This would provide a forum for the exchange of ideas and practical tips between MSD-users, who should realise the advantages of co-operation and mutual aid.

ACKNOWLEDGEMENTS

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UNIDO COMMENTS

(DP/IND/89/128)

The report clearly brings out the problems associated with having highly expensive analytical equipment with very little attention given to the quality of the power supply. The PDC has highly sensitive (both to power variation and dust) costly equipment and yet there is no sign of getting reliable power source. The author has given three alternatives and while all these are under consideration, there seems to be the constant problem of power failure damaging the expensive equipment.

The best solution is for the project to provide generators suitable for the analytical laboratory rather than waiting for the counterparts to get clearance from the Government to install another generator. The suggestion of connection to Delhi line should be seriously considered.

The expert should go again for additional consultancy when proper power supply is restored.