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AN ECOTOXICOLOGY RESEARCH CENTRE IN PAKISTAN

US/PAK/90/294

PAKISTAN

Technical report: Pesticide residue analysis at
the Ecotoxicology Research Centre*

Prepared for the Government of Pakistan
by the United Nations Industrial Development Organization

Based on the work of Brian Crozier,
consultant in pesticide residue analysis

Backstopping Officer: B. Sugavanam
Chemical Industries Branch

United Nations Industrial Development Organization
Vienna

* This document has not been edited.

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ABSTRACT

Title: Consultant in Pesticide Residue Analysis

Number: US/PAK/90/294/11-02

Purpose: To instruct and train scientific staff at the NARC, Islamabad in the use of modern analytical techniques for the measurement of pesticide residues in biological tissues and environmental samples.

Duration: Twenty two days, 27 July to 17 August 1993
[Reduced from one month with the agreement of the backstopping officer.]

Conclusion: Only part of the essential supplies for the analysis of pesticide residues was available; practical instruction in extraction and clean-up techniques was impossible.

Lectures on the theoretical basis of analytical techniques were given.

Advice on the organisation of the laboratories and the siting of the equipment was given.

Recommendations: As soon as the generator for the gases for the GLCs and the appropriate supplies for the HPLCs are received, the manufacturer's agents should be asked to install the instruments and verify their correct performance. Whilst the agents are at the laboratory it is essential that all staff of the Ecotoxicology Centre are present to receive instruction in the use of the equipment.

Further instruction in the techniques to be used for the determination of pesticide residues should be given to all staff, either by training courses at suitable establishments or by further visits from experts.

ABBREVIATIONS AND ACRONYMS USED

ADI	Acceptable Daily Intake
CAC	Codex Alimentarius Commission
ESCAP	Economic and Social Commission for Asia and the Pacific
FAO	Food and Agriculture Organisation (of the UN)
FID	Flame Ionisation Detector (GLC)
FPD	Flame Photometric Detector (GLC)
GLC	Gas Liquid Chromatograph(y)
HPLC	High Performance Liquid Chromatograph(y)
MRL	Maximum Residue Level
NARC	National Agricultural Research Centre
NPD	Nitrogen Phosphorus Detector (GLC)
PARC	Pakistan Agricultural Research Council
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
UV/VIS	Ultra Violet/Visible Spectrophotometer
WB	World Bank
WHO	World Health Organisation (of the UN)

INTRODUCTION

UNIDO, in conjunction with other international agencies, eg., FAO, WHO, ESCAP, WB and UNDP, has established the RENPAP to promote the safe development and management of pesticides. There are 14 member countries in total, of which six have agreed to take the lead in specific topics. The details are given in the Job Description, see Annex I.

Pakistan has agreed to take the lead in Ecotoxicology and the project, of which this mission is part, is to establish an Ecotoxicology Centre at Islamabad. When fully established it is intended that the Ecotoxicology Centre, which will also have two smaller provincial laboratories under its control, will have a core of experts trained in the special techniques of ecotoxicology who will be able to help other countries in the RENPAP. It is expected that the Ecotoxicology Centre will provide a model for other countries and by holding workshops and training courses will greatly add to the safety in all aspects of the use of pesticides.

This report has been written by Brian Crozier, Campden Food and Drink Research Association, Chipping Campden GL55 6LD, England, Consultant in Pesticide Residue Analysis, based on the Job Description given in Annex I.

Reports of previous missions (see Annex VI) were very helpful in establishing the background to this mission, whose purpose was "to instruct and train scientific staff in the use of modern analytical techniques for the measurement of traces of pesticide residues in biological tissues and environmental samples."

I ORGANISATION

A. Accommodation

It had been arranged that the Ecotoxicology Centre would be housed in laboratories in the NARC, a large multi-disciplinary establishment on the outskirts of Islamabad. Three rooms were available on arrival, one an office and two laboratories. The laboratories were of basic layout each with two wall benches and one peninsular bench, the benches were covered with a laminate; there were ample cupboards and drawers under the benches and one set of wall cupboards. There were desks for four personnel in each laboratory with shelving above. Each laboratory had a sink, with double drainer and a supply of hot and cold water. The laboratories had obviously been recently refurbished; the walls repainted and the wood work (drawer fronts etc.) revarnished.

B. Staff

The laboratory staff is at present six qualified scientists, of varied disciplines, with two helpers. Only one of the scientists had any practical experience of analysing for pesticide residues, a number of years ago; the rest had qualifications in various aspects of ecotoxicology, eg., microbiology, ecology, entomology and toxicology. Two of this complement of staff were abroad on training programmes during the mission, a third member departed half-way through.

C. Equipment

The laboratory is starting from the absolute beginning; on arrival there was nothing moveable in either of the laboratories. The analytical equipment was still in the Central Stores in packing crates, glassware arrived five days after I did and the generator for the gas supplies for the GLCs was in transit somewhere.

D. Services

The laboratories are not air conditioned. There are only two power socket outlets to each bench. There does not appear to be a major problem with power failures but the voltage does fluctuate to a degree where it could damage delicate analytical instrumentation. Hot water is supplied to each of the laboratories by large individual water heaters.

E. Other Facilities

As NARC is a large multidisciplinary organisation, there will be very good facilities for carrying out trials to determine the fate of pesticides to crops, soil etc. in the future.

The library is quite large and has the facility to access worldwide information databases. The number of reference books on chemistry, analytical chemistry in general and pesticide analysis in particular is extremely small and I would suggest that a selection from the list in Annex IV be made for the laboratory.

II ACTIVITIES

Although the main purpose of the mission was practical training of the scientific staff in the analytical techniques and methods used in the determination of pesticide residues this was not possible because of the lack of any facilities. The service engineers representing the manufacturers of the instrumentation had provided a list of site requirements in June 1993 and were surprised to find that this had not been carried out by the time they arrived to install the instruments. The NPD had been requesting the manufacturer's representatives to come to a site meeting to discuss these points for specific changes, but had been unable to get a response from them. Whilst air conditioning for the laboratories could be installed later, voltage stabilisers were thought essential and without gas supplies for the GLCs and solvents for the HPLCs it would be impossible for them to verify the performance of the instruments.

Similarly without solvents or any other chemicals it was impossible for me to demonstrate any of the extraction or clean-up techniques to be used.

The main activities therefore took the form of lectures and talks on aspects of analytical chemistry and the determination of pesticide residues in various substrates. The list of lectures is given in Annex III. This is no substitute for actual hands-on practice and the instruction to be given by the engineers will be only the basics needed to operate the instruments. A period of some training for the staff, either in a modern laboratory or by further consultant's visits will be necessary.

III FINDINGS AND CONCLUSIONS

A. Accommodation

The laboratories are adequate at the moment to carry out the planned work on the determination of pesticide residues in environmental samples; however the long-term strategy is to have Sections dealing with 1) Environmental Chemistry, 2) Aquatic and Terrestrial Ecology and 3) Wildlife and Beneficial Species. Two laboratories will not be enough and the NPD should begin negotiations with the Director General at NARC for further accommodation to be available during the expansion of the Ecotoxicology Centre.

There should be a storage facility, preferably removed a short distance from the laboratory, for solvents, chemicals, pesticides and waste materials awaiting disposal. The building should be solidly built with adequate appropriate shelving and ventilation; and with restricted access.

For the short term, it will be practical to have the freezers for sample storage in the extraction and clean-up laboratory, but in the long-term, consideration should be given to a separate room.

If, as detailed in the Job Description, the Centre will be working to GLP, adequate secure space for archiving records and accommodating Quality Assurance staff must be found.

B. Staff

The organisation chart for the Ecotoxicology Centre expects a complement of 30 (not including QA and provincial laboratory staff). If the full complement is realised, seven of these are expected to be analytical chemists. This number will be sufficient for the foreseen workload and the total of seven should not be compromised. They should be qualified and trained in analytical chemistry, not seconded from other sections. Of the present staff, only Mr M Mumtaz has any practical experience of relevant analytical chemistry and he is in need of a refresher course at an appropriate establishment. All staff recruited to the Analytical Chemistry Section should have a period of training at a laboratory which is routinely carrying out analyses for pesticide residues.

C. Equipment

I expect the instrumentation to be satisfactory for the job for the foreseeable future. Because the laboratory is starting from scratch, there will be several items that will need to be obtained as the work in the laboratory gets under way. As a guide to the basic needs of a laboratory carrying out pesticide analyses the list given in Annex V may be used.

D. Services

Apart from the need to supply voltage stabilisers for the analytical instruments, the services supplied to the laboratory appear adequate.

E. Other Facilities

I assume equipment for supervised trials to be carried out on NARC sites will be available from the appropriate departments at NARC.

The Ecotoxicology Centre needs to be supplied with a selection of up-to-date reference texts on pesticide matters (see list in Annex IV).

When fully operational, staff of the Ecotoxicology Centre will require use of a suitable vehicle for sampling and attendance at trials sites for supervision of spraying, harvesting etc.

IV ACKNOWLEDGMENTS

The Author acknowledges the help and assistance received from staff at all levels with whom he has come into contact.

RECOMMENDATIONS**A. Notes on recommendations made in Dr Kirknel's report****1. Bench covering**

The parts of the benches where operations involving weighing samples and use of solvents and other chemicals take place should be covered with some disposable covering. Suitable materials, eg., "BenchKote", can be obtained from most laboratory suppliers.

2. Fume Cupboards/Hoods

There has been no physical progress as yet, plans and instructions are with the appropriate department at NARC.

3. Fire blankets and extinguishers

There are two extinguishers, dry chemical type, in the corridor outside the laboratories. I would suggest that at least one more be obtained, to be positioned in the Extraction/Clean-up Laboratory. Fire blankets need to be obtained.

4. Eye washes and Emergency Showers

At least two eye wash bottles are needed for each laboratory. It was proposed that there would be an emergency shower close to the exits from the laboratories.

5. Store room

There must be a store room for bulk solvents and chemicals; they must not be stored in the laboratory. As mentioned, there should be a refrigerator (spark proof) in the store.

6. Refrigerator; Freezer

A freezer will be needed for the storage of samples before analysis to prevent decomposition/metabolism of any pesticide residues present.

7. Voltage stabilisers

Authority to purchase suitable stabilisers locally has been given, the order has been placed.

8. Electrical outlets

The NARC electricians have prepared extension boards to be connected to the voltage stabilisers when obtained.

9. Waste chemicals

Some thought needs to be given to the problems of disposal of waste chemicals, especially solvents. Low temperature combustion was suggested.

B. Further Observations

1. Balances

I have not, in any of the documentation, seen evidence that analytical balances have been ordered. Naturally, in an establishment the size of NARC, there must be balances around that can be used, but this must be viewed as a short term solution.

2. Rotary evaporator

I have seen a copy of the order for a "rotary evaporator cooler", I am not convinced that this is actually a rotary evaporator without seeing the catalogue it was ordered from.

3. Sample preparation

For the analysis for pesticide residues in fruits, vegetables and other produce there is a need to process the substrate in order to allow the residues to be extracted. This entails the use of a food chopper and a food processor, macerator or tissue grinder.

4. Other items

There will be many items considered as basic laboratory equipment which will not be available because the Ecotoxicology Laboratory is starting from absolutely nothing. (See Annex V). Some of these items were on the list prepared by Dr Kirknel, but not on the order placed with the suppliers in Denmark.

5. Standard materials

Standard pesticide reference materials will be needed for the calibration of secondary standards, and for the preparation of appropriate solutions for recovery determinations.

C. Recommendations

1. Arrange for all necessary services and supplies for the analytical equipment to be obtained and installed in the laboratory.

2. When 1. above achieved, arrange for the local agents to commission the instrumentation and verify correct operation, using appropriate materials.

3. Organise appropriate training for senior analytical staff.

4. Organise on-the-job training for all analytical chemists.

ANNEX I

JOB DESCRIPTION

US/PAK/90/294/11-02

Post Title: Expert in Pesticide Residue Analysis

Duration: 1.0 m/m (split mission)

Date Required: As soon as possible

Duty station: Islamabad, Pakistan

Purpose of project:

To establish an ecotoxicology laboratory at the National Agricultural Research Centre (NARC), Islamabad belonging to the Ministry of Agriculture and study the fate and effects of pesticides in the environment and also make Pakistan effectively interact as Technical Coordinator giving inputs to eco-toxicology to the Regional Network on Pesticides for Asia and the Pacific (RENAPAP).

Duties:

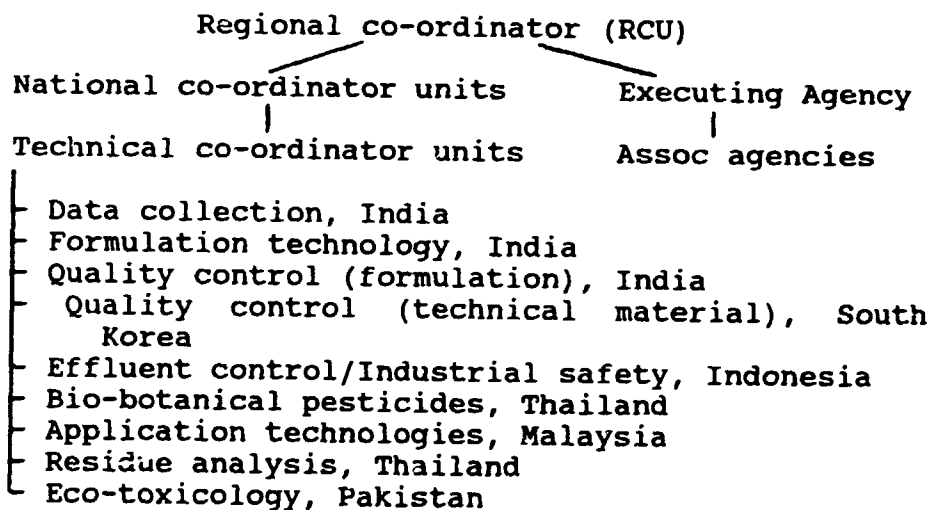
The expert is expected to instruct and train scientific staff in the use of modern analytical techniques for the measurement of traces of pesticide residues in biological tissues and environmental samples. This will involve demonstration of:

- sampling techniques for soil, water and other biological materials;
- clean-up procedures involving extraction, partitioning, chromatography, distillation and other techniques;
- use of GLC, HPLC, Spectrophotometry, Spectrofluorometry, TLC and other techniques;
- GLP procedures of working and recording data;
- new type residue methods.

Qualifications: The candidate must hold a degree in chemistry with long experience as a working analyst in the field of pesticide residues, preferably from a government establishment or have practical experience of multi-residue methods. He should not have been away from the bench for longer than 5 years.

Language: English

Background Information: United Nations Industrial Development Organisation (UNIDO) has been assisting 13 Asian countries through a network called Regional Network on Pesticides for Asia and the Pacific (RENAPAP) mainly to promote safe development of pesticides. Under this project specific areas have been assigned to different member countries to provide technical inputs as shown below.



Pakistan has taken up ecotoxicology based on the facilities available. These facilities need to be strengthened and from the contribution of Denmark to the UNIDO Industrial Development Fund (IDF) a project has been approved to strengthen the ecotoxicology laboratory of the NARC of the Ministry of Agriculture. The main aim is to link pesticide industries with the ecotoxicology laboratory and make use of the facilities for carrying out experiments on the fate of pesticides they produce for the Pakistan market. Self-sustainability (partly or fully) of the project is also considered to be an important factor by providing services to industries, government institutions dealing directly or indirectly with pesticides.

ANNEX II

PERSONS CONTACTED

United Nations Personnel

Dr O M Bovet Programme Officer

Pakistan Agricultural Research Council

Dr Umar Khan Baloch Director of Research (Crop Protection)
National Project Director

Mr Liaquat Ali Hashmi Scientific Officer

Ministry of Agriculture

Dr Habib-ur-Rehman Director of Soils and Plant Nutrition,
Peshawar

National Agricultural Research Centre

Mrs Shaheen Masud Director of Administration

Mr Mohammed Mumtaz Analytical Chemist

Dr Yousef Hayat Pesticide Microbiologist

Mr Ather Rafi Ecotoxicologist (Entomology)

Dr Sima Anwar (Mrs) Toxicologist

ANNEX III

LECTURES GIVEN TO STAFF AT NARC

1. Safety in the Laboratory
2. Basic First Aid
3. Good Laboratory Practice
4. Spectroscopy
 - 4.1 UV/VIS
5. Chromatography
 - 5.1 General Introduction and Overview
 - 5.2 Maintenance : Column Packing
 - 5.3 Trouble Shooting
6. High Performance Liquid Chromatography
 - 6.1 Equipment : Pumps, Columns, Detectors
 - 6.2 Methods
 - 6.3 Developments
7. Gas Liquid Chromatography
 - 7.1 Introduction : Components of the System
 - 7.2 Stationary Phases and Supports
 - 7.3 Special Techniques
8. Analysis for Pesticide Residues
 - 8.1 Considerations
 - 8.2 Extraction
 - 8.3 Clean-up
 - 8.4 Determination
 - 8.5 Recoveries
 - 8.6 Presentation of Results
9. Registration of Pesticides
 - 9.1 Need : Objectives : Responsibilities
 - 9.2 Data Requirements
 - 9.3 Trials
 - 9.4 Assessment
 - 9.5 Post-Registration Activities
 - 9.6 ADI and MRL
10. Mass Spectrometry of Pesticides

ANNEX IV

TEXTBOOKS FOR PESTICIDE ANALYSIS

1. Manuals and Reference Books

Many of these texts are updated at regular intervals, always specify the latest edition

- 1.1 Manual of Chemical Methods for Pesticides and Devices
US EPA. Published and distributed by the Association of
Official Analytical Chemists, Washington DC, USA
- 1.2 Official Methods of Analysis.
Editor W Horwitz
Association of Official Analytical Chemists, Washington
DC, USA
ISBN 0 935584 01 9
- 1.3 The Pesticide Manual
Editors Charles R Worthing and S Barrie Walker
British Crop Protection Council, England
ISBN 0 948404 01 9
- 1.4 Analytical Methods for Pesticides and Plant Growth
Regulators Volumes I to date
Editors G Zweig and J Sherma
Academic Press, New York, USA
ISBN 0 12 784314 0
- 1.5 The Agrochemicals Handbook
The Royal Society of Chemistry, Nottingham, England
ISBN 0 85186 416 3
- 1.6 Statistical Manual of the AOAC
[Statistical techniques for collaborative tests-W J
Youden and Planning and analysis of results of
collaborative tests-E H Steiner]
AOAC, Washington, USA
- 1.7 CRC Handbook of Chemistry and Physics
Editor in Chief R C Weast
CRC Press Inc, Boca Raton, FLA, USA
ISBN 0 8493 047 0
- 1.8 The Merck Index
Editor S Budavari
Merck and Co Inc, Rahway, NJ, USA

- 2. Pesticide Formulations: Analysis, Specification etc**
- 2.1 CIPAC Handbooks I, IA, IB, IC, D
Black Bear Press Ltd, Cambridge, England
- 2.2 CIPAC Proceedings Volumes I, II, III
Black Bear Press Ltd, Cambridge, England
- 2.3 Pesticide Formulation
Editor Wade van Valkenburg
Marcel Dekker Inc, New York, USA
ISBN 0 8247 1695 7
- 2.4 Formulation of Pesticides in Developing Countries
UNIDO, Vienna, Austria 1983
UN Publication sales number E.83.II.B.3
- 2.5 Pesticides, Preparation and Mode of Action
R Cremlyn
John Wiley & Sons 1978
ISBN 0 471 99631 9
- 2.6 Regulations of Agrochemicals: A Driving Force in their
Revolution
G J Marcos, R M Hollingworth, J R Plimmer
RSC, Nottingham, England
ISBN 0 8412 2089 1 (HB) 0 8412 2085 9 (PB)
- 2.7 Disposal and Decontamination of Pesticides
Maurice V Kennedy
ACS Symposium Series No 73 1978
ISBN 0 841 20443 0
- 2.8 The Pesticide Book
George W Ware
W H Freeman & Co, San Francisco, USA 1978
ISBN 0 716 70198 7

3. Pesticide Residues and the Environment

- 3.1 **Advances in Pesticide Science. Parts 1, 2 and 3**
IUPAC, Editor H Geissbuhler
Pergamon Press, Oxford, England
ISBN 0 08 022349 4
- 3.2 **FDA Pesticides Analytical Manual**
Food and Drug Administration, Washington DC, USA
- 3.3 **Manual on Analytical Methods for Pesticide Residues in Food**
Health Protection Branch, Health and Welfare Canada,
Ottawa, Canada
- 3.4 **Laboratory Manual for Pesticide Residues in Agricultural Products**
Compiled by R B Maybury, Pesticide Laboratory, Food
Production and Inspection Branch, Agriculture Canada,
Ottawa, Canada
- 3.5 **Principles of Environmental Sampling**
Editor L H Keith
ACS 1988
ISBN 0 8412 1173 6
- 3.6 **Environmental Chemistry**
S Monahan
Lewis 4th Edition 1984
ISBN 0 87371 238 2
- 3.7 **EPA's Sampling and Analysis Methods Database**
D Smith, W Mueller and L H Keith
Lewis 1990 3 volumes [on 6 diskettes]
Vol 1: Industrial Chemicals [4 diskettes]
Vol 2: Pesticides etc
Vol 3: Elements, water quality
- 3.8 **Food Contaminants: Sources and Surveillance**
Editors C S Creaser and R Purchase
RSC, Nottingham, England 1991
ISBN 0 85186 606 9
- 3.9 **Advanced Analytical Techniques
in Analytical Methods for Pesticides and Plant Growth
Regulators Volume XVII**
Editor J Scherma
Academic Press 1989
ISBN 012 784317 5

- 3.10 Pesticide Residues and Food Safety: A Harvest of Viewpoints
ACS Symposium Series NO. 446
Editors B G Tweedy, H J Dishburger, L G Ballantine, J McCarthy
ACS 1991
ISBN 0 8412 1889 7
- 3.11 Chemistry, Agriculture and the Environment
Editor M L Richardson
RSC, Nottingham, England 1991
ISBN 0 851 86228 4
- 3.12 Agricultural Chemicals and Pesticides
A handbook of the Toxic Effects
Editor Edward J Fairchild
Castle House Publications Ltd for the US Dept HEW 1978
ISBN 0 719 40002 3

4. General Analysis and Quality Control

- 4.1 Gas and Liquid Chromatography in Analytical Chemistry
Roger Smith
John Wiley & Sons Ltd 1988
ISBN 0 471 90980 7
- 4.2 Chromatography Today
C F Poole and S K Poole
Elsevier, Amsterdam 1991
ISBN 0 444 88492 0 (HB) 0 444 89161 7 (SB)
- 4.3 Chromatographic Enantioseparation
Methods and Applications
Stig Allenmark
The Ellis Horwood Series in Analytical Chemistry
Simon and Schuster 1991
ISBN 0 13 132978 2
- 4.4 Principles and Practice of Chromatography
B Ravindranath
The Ellis Horwood Series in Analytical Chemistry
Simon and Schuster 1991
ISBN 0 13 721523 1
- 4.5 Chromatography: Concepts and Contrasts
J M Miller
Wiley 1988
ISBN 0 471 84821 2

- 4.6 Chromatographic Integration Methods
N Dyson
RSC Monograph Series, Cambridge, England 1990
ISBN 0 85186 587 9
- 4.7 Modern Analytical Chemistry
Editor J N Miller
The Ellis Horwood Series in Analytical Chemistry
Simon and Schuster 1991
ISBN 0 13 005018 0 (HB) 0 13 005042 3 (PB)
- 4.8 Total Quality Management
J S Oakland
Heinemann 1989
ISBN 0 434 91479 7
- 4.9 Data Analysis in the Chemical Industry
Volume I: Basic Techniques
R Caulcutt
Ellis Horwood
ISBN 0 745 80727 5
- 4.10 Statistics for Analytical Chemistry
J C Miller and J N Miller
Simon and Schuster 2nd Edition 1987
ISBN 0 138 45421 3
- 4.11 Quantitative Analysis using Chromatographic Techniques
E Katz
Wiley 1987
ISBN 0 471 91406 1

5. Gas Chromatography

- 5.1 **Basic Chromatography**
H McNair and E J Bonelli
Varian Inc., 1974
- 5.2 **The Packed Column in Gas Chromatography**
W Supina
Supelco Inc., Bellefonte, PA, USA. 1974
- 5.3 **Gas Chromatographic Detectors**
D J David
Wiley Interscience, NY, USA. 1975
- 5.4 **Detectors in Gas Chromatography**
Jiri Sevcik
Elsevier Scientific Publishing Co. 1975
ISBN 0 444 99857 8
- 5.5 **Chromatographic Systems. Maintenance and Troubleshooting**
J O Walker, M T Jackson Jr and J B Maynard
Academic Press Inc., 2nd Edition, 1977
ISBN 0 12 732052 0
- 5.6 **Analytical Gas Chromatography**
Walter Jennings
Academic Press Inc. 1987
ISBN 0 12 384355 3
- 5.7 **Modern Practice of Gas Chromatography**
Editor Robert L Grob, 2nd Edition 1985
ISBN 0 471 87157 5
- 5.8 **Gas Chromatography. A Practical Course**
Gerhard Schomburg
VCH 1990
ISBN 3 527 27879 6
- 5.9 **Classical Split and Splitless Injection in Capillary GC**
K Grob Jr and A Huthig
Verlag 1988

6. High Performance Liquid Chromatography

- 6.1 High Pressure, High Resolution Liquid Chromatography and its Application to Pesticide Analysis and Biochemistry
D A Schooley and G B Quistad
in Progress in Drug Metabolism, Vol 3.
Eds J W Bridges and L F Chasseau
John Wiley and Sons Ltd 1979
ISBN 0 471 99711 0
- 6.2 Introduction to High Performance Liquid Chromatography
R J Hamilton and P A Sewell
Chapman and Hall 1982
ISBN 0 412 23430 0
- 6.3 Maintaining and Troubleshooting HPLC Systems. A User's Guide
Dennis J Runser
John Wiley & Sons Ltd 1981
ISBN 0 471 06479 3
- 6.4 High Performance Liquid Chromatography
M T Gilbert
Wright, Bristol, England 1987
ISBN 0 7236 0897 0
- 6.5 Introduction to Modern Liquid Chromatography
L R Snyder and J J Kirkland
John Wiley & Sons Ltd, NY, USA 2nd Edition 1979
- 6.6 Basic Liquid Chromatography
E L Johnson and R Stevenson
Varian, Palo Alta 1978
- 6.7 High Performance Liquid Chromatography
Editors P R Brown and R A Hardwick
John Wiley & Sons Ltd 1988
ISBN 0 471 84506 X
- 6.8 Practical HPLC Method Development
L R Snyder, J L Glajch and J J Kirkland
John Wiley & Sons Ltd 1988
ISBN 0 471 62782 8
- 6.9 Reversed-phase High Performance Liquid Chromatography
Ante M Krstulovic and Phyllis R Brown
John Wiley & Sons Ltd 1982
ISBN 0 471 05369 4

6.10 Troubleshooting LC Systems: A Comprehensive Approach to
Troubleshooting LC Equipment and Separations
John W Dolin and Lloyd R Snyder
Humana Press, Clifton NJ, USA

6.11 High Performance Liquid Chromatography
S Lindsay
Wiley 2nd Edition 1992
ISBN 0 471 93180 2 (HB) 0 471 93115 2 (PB)

6.12 High Performance Liquid Chromatography
P R Brown and R A Hartwick
Wiley 1989
ISBN 0 471 84506 X

6.13 Practical HPLC
V Meyer
Wiley 1988
ISBN 0 471 91140 2

6.14 Trace and Ultratrace Analysis by HPLC
S Ahuja
Wiley 1992
ISBN 0 471 51419 5

6.15 HPLC Tips and Tricks
C Gertz
LDC Analytical 1990

7. Thin Layer Chromatography

7.1 Practice of Thin Layer Chromatography
Joseph C Touchstone
John Wiley & Sons Ltd. 1992
ISBN 0 471 51419 5

7.2 Thin Layer Chromatography. Reagents and Detection
Methods Volume 1A
Jork, Funk, Fischer and Wimmer
VCH 1989
ISBN 3 527 27834 6

7.3 Chromatographic Science Series Volume 52.
Modern TLC
Editor N Grinberg
Marcel Dekker 1990
ISBN 0 8247 8138 4

8. Mass Spectrometry

- 8.1 Quadrupole Storage Mass Spectrometry
R E March and R L Hughes
John Wiley & Sons Ltd 1989
Vol 102 in Chemical Analysis: A Series of Monographs on
Analytical Chemistry and its Applications
ISBN 0 471 85794 7
- 8.2 Applications of New Mass Spectrometry Techniques in
Pesticide Chemistry
Editor J Rosen
John Wiley & Sons Ltd 1987
Vol 91 in Chemical Analysis: A Series of Monographs on
Analytical Chemistry and its Applications
ISBN 0 471 83280 4
- 8.3 Liquid Chromatography/Mass Spectrometry
Applications in Agricultural, Pharmaceutical and
Environmental Chemistry
ACS Symposium Series No. 420 Editor M A Brown
ACS 1990
ISBN 0 8412 1740 8
- 8.4 Mass Spectrometry
E Constantin and A Schnell
The Ellis Horwood Series in Analytical Chemistry
Simon and Schuster 1990/ [1991]
ISBN 0 13 555525 6 (HB) [0 13 553363 5 (PB)]

9. Other Techniques

- 9.1 Supercritical Fluid Chromatography
Editor R M Smith
RSC, Nottingham, England 1988
ISBN 0 851 86577 1
- 9.2 Analysis with Supercritical Fluids: Extraction and
Chromatography Editor B Wenchawiak
Springer 1992
ISBN 3 540 55420 3
- 9.3 UV-VIS Spectroscopy and its Applications
H-H Perkampus
Springer 1992
ISBN 3 540 55421 1

10. Pesticide Usage

- 10.1 Handbook on the Use of Pesticides in the Asia-Pacific
Region
Asian Development Bank, Manila, Philippines 1987

ANNEX V

MINIMUM BASIC INSTRUMENTATION AND EQUIPMENT FOR A PESTICIDE ANALYSIS LABORATORY

This list gives main items together with suggested spares and consumables

1. Major instrumentation with Accessories and Spares

Gas Liquid Chromatograph, with packed and capillary injectors, fitted with suitable detectors, eg., FID, ECD, NPD, FPD etc.

Gas supplies - nitrogen (or helium), hydrogen, air - from Nitrox or other suitable generator (with maintenance kit)
Gas filters for oxygen and moisture
Glass columns, ready packed or empty together with suitable liquid phases and support materials
Capillary columns, bonded phase
Injection port liners
Septa, silanised glass wool. O rings, syringes, ferrules, copper tubing, Swagelock fittings, chart paper(?)
Manufacturer's recommended service kit

High Performance Liquid Chromatograph, with variable wavelength UV detector

Selection of columns of various lengths and packings
Guard columns
Degasser/debubbler for solvents
Syringes, injection loops
Swagelock fittings and connectors, ferrules, stainless steel and PTFE tubing, chart paper(?)
Solvents
Manufacturer's recommended service kit

UV/Visible Spectrophotometer, scanning range 200 to 1100 nm

Spectrophotometer cells, various sizes
Spare deuterium and tungsten bulbs
Chart paper
Manufacturer's recommended service kit

Infrared recording spectrophotometer, double beam

Sodium chloride cells, various path lengths
Chart paper
Manufacturer's recommended service kit

2. Laboratory Apparatus and Small Instruments

Balance, analytical, 200g X 0.1mg, with tare
Balance, laboratory, 2kg X 0.1g, with tare
pH meter/potentiometric titrator
electrodes, buffer powders
Karl Fischer titration apparatus
solvents, reagents
Deioniser
spare cartridges or resins
Melting Point apparatus with tubes
Flash Point apparatus
Ultrasonic bath
Microwave oven
Centrifuge
assorted tubes and 100ml IP75 tubes
Sieve shaker and range of sieves
Laboratory oven X 2
Furnace with crucible and tongs
Steam bath, thermostatted water bath, cooled bath, water
bath for emulsion stability test
Refrigerator and deep freezer
Liquidiser / homogeniser / blender
Vegetable / fruit chopper
Tissue grinder
Hotplate/magnetic stirrer with fleas
Rotary shaker
Orbital shaker
Ball grinding mill with containers and balls
Rotary evaporator with spare clips and flaske
Heating mantles of various sizes
Vacuum pump / compressor
Water vacuum pump
Whirlimix
Desiccators
Stirrer motor and rods with varying lengths and paddles
Hair drier / heat gun
Viscometer, or range of viscometer tubes
TLC plate maker (or plates), tanks, sprayer etc
UV lamp
Stop clock, stop watch, interval timer
Thermometers, general and melting point

3. Glassware etc

No numbers are specified, this will depend on the type of work and number of expected samples

Volumetric flasks and stoppers to fit

1,5,10,20,25,50,100,200,250,500,1000 ml

Graduated cylinders, both lipped and stoppered

5,10,25,50,100,200,250,500,1000 ml

250 ml for suspensibility test

tap density cylinders

Pipettes, bulb and graduated

1,2,5,10,20,25,50 ml

pipette fillers

Pasteur pipettes and teats

Burettes

10,25,50 ml

Beakers, squat and tall form

10,25,50,100,250,500,1000 ml

Quickfit glassware

adaptors, condensers, distillation columns, flasks (round bottomed, one and two necked, 50,100,250,500 ml), still heads, splash heads, chromatography columns, separating funnels, thermometers, gas adsorption apparatus, Dean and Stark fitments, Soxhlet extractor with thimbles, conical flasks (25,50,100,250,500 ml), iodine flasks (250 ml), range of stoppers

Reagent bottles, labelled, 500,1000 ml

Buchner flasks and funnels with range of papers

Sintered glass funnels, range of porosities

Filter funnels, varying sizes, range of papers to fit

Test tubes and racks

Mortars and pestles, various sizes

Polythene squeeze bottles

Tubing: glass, polythene, rubber, vacuum, and tubing clips

Tubing connectors T and Y

Glass rods

Weighing bottles and funnels; disposable weighing boats

Glass vials, varying sizes with screw caps

Glass jars, varying sizes with screw caps

4. General Equipment

Spatulas, palette knives, scoops, spoons
Forceps
Scalpels and blades
Scissors
Bowls and buckets, polythene
Cleaning brushes (bottle, burette, stiff, bristle, soft)
Paint brushes, varying sizes
Aluminium foil
Cotton wool
Glass wool
Cork rings
Paper tissues
Butane burner with cartridges, tripods and gauzes
Vials, caps and liners
Crimper/decapper
Retort stands, clamps (various), bosses, rings
Scaffold rods and bases
Laboratory jacks
Multimeter
Assorted tools
Knives
Self-seal polybags or polybags and sealer

5. Safety and Disposable items

First Aid kits
Eye wash bottles
Safety glasses and goggles; safety shields
Dust masks and ear defenders
Laboratory coats
Protective gloves (nitrile, polythene, heat resistant)
Solvent store cupboard
Acid store cupboard

6. Special chemicals etc.

GC phases and supports
Silylating agents
Aluminium oxide, silica gel, Florisil (various grades)
CVS solutions
Indicator papers, pH papers

ANNEX VI

REFERENCES AND REPORTS CONSULTED

1. Calderbank, A. DP/ID/SER.A/1043
UNIDO report: Environmental toxicology related to pesticides in Pakistan
DP/RAS/85/023 1988
2. Calderbank, A. DP/ID/SER.A/1311
UNIDO report: An Ecotoxicology Research Centre in Pakistan
DP/RAS/85/023 1990
3. Fletcher, K. IO/R.254
UNIDO report: Establishment of and Ecotoxicology Centre
US/PAK/90/294 1992
4. Kirknel, E. DP/ID/SER.A/1640
UNIDO report: Findings and recommendations
DP/RAS/88/031 1993

UNIDO'S SUBSTANTIVE COMMENTS ON THE REPORT

The report of the expert gives a clear picture as to how the eco-toxicology laboratory should be developed so as to cover all aspects relevant to Pakistan and then in the Asia Region. The NARC Centre is ideally suited for setting up of such a centre and has highly qualified scientists who need further upgrading of their skills.

Delays in handing over of the laboratories to the project has affected the preparedness of the centre to receive and install equipment. This should be taken up as an urgent step to properly install equipment to carry further work in the laboratory.

While the proposed training arrangements are going according to plans so that experiments could start when the fellows return and they are well set up for the eco-toxicology workshop planned in March 1994.

The various recommendations including equipping the library will be taken up in the next tripartite review meeting and some of the requirements regarding g.l.p./quality assurance should be incorporated from the very beginning.