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REGIONAL NETWORK ON PESTICIDES FOR ASIA AND THE PACIFIC

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Technical report: Consultation on pesticide residue analysis at Plant Protection Wing, Department of Agriculture Extension Dhaka, Bangladesh, 15 March - 9 April 1987*

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

Based on the work of Naresh Chandera Atreya, consultant on residue analysis

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Vienna

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1 Abstract

The report covers one month consultancy at the Pesticide Residue Laboratory of Plant Protection Wing of Department of Agriculture Extension in Dhaka, Bangladesh. Details of the instruments, equipment available for Residue Laboratory are given together with recommendations for further purchases. Details on the need to increase laboratory space, staff and their training are also given.

2 Summary of Recommendations

The consultant recommends that the following recommendations 2.1 to 2.5 to be implemented as soon as possible in order to carry out proper and meaningful residue analysis at Plant Protection Wing of Department of Agriculture Extension.

2.1 Accommodation

2.1.1 Extra rooms for pesticide residue analysis.

2.1.2 Purchase and fitting of 2 additional air conditioning units.

2.1.3 Purchase and fitting of one fume cupboard in the residue laboratory.

2.1.4 Separate supply of hot and cold water for residue laboratory.

2.1.5 To remove the existing toilet and convert this small room for washing up of residue laboratory glassware.

2.1.6 Purchase and fitting of large deep sink in glassware washing up room.

2.1.7 Purchase of one deep freeze ($-20^{o}C$) and a large fridge and place in the designated sample preparation room.

2.1.8 Purchase of dishwasher for glassware cleaning for residue analysis.

2.2 Staff

2.2.1 Present trained staff should continue to work permanently on residue analysis

2.2.2 One additional graduate in chemistry and one technical assistant to be recruited immediately and be trained in residue analysis.

2.2.3 Trained staff must be encouraged to remain in the laboratory to do analysis and must never be transferred to non-analytical jobs in the department.

2.2.4 Chemists should be given regular training at Plant Protection Wing.

2.3 Instrumentation

2.3.1 Purchase of Gas-Liquid Chromatograph fitted with Nitrogen Phosphorus Detector, recorder and all necessary spare parts.

2.3.2 Purchase of High Performance Liquid Chromatograph with UV detector, recorder, columns and other necessary spare parts.

2.3.3 Purchase of UV lamp.

2.3.4 Purchase of UV-visible spectrophotometer.

2.4 Equipment

2.4.1 Purchase of extra glassware for residue analysis.

2.4.2 Purchase of Rotary Evaporator.

2.4.3 Purchase of further supplies of chemicals, eg pure solvent various reagents and GLC and HPLC accessory chemicals.

2.4.4 Purchase of specific equipment for residue analysis.

2.5 Services

2.5.1 Separate hot and cold water supply.

2.5.2 Improvement in the services of the electricity.

2.6 Safety in the Laboratory

2.6.1 Safety equipments, eg spectacles, laboratory coats, eye wash bottles, gloves and proper screens all essential and must be used at all times in the residue laboratory.

3 Introduction

Plant Protection Wing of the Department of Agricultural Extension is located at Khamarbari, Farmgate, Dhaka. Mr A R Khan is the director of Plant Protection Wing which has four sections. These are:-

1) Pesticide and Quality Control; Residue analysis.
2) Aerial and ground operation.
3) Plant quarantine.
4) Diagnostic surveillance and forecasting.

The organisation and personnel chart of Plant Protection wing is enclosed in as appendix 1. The Deputy Director of Plant Protection wing, Mr M A Malik is in charge of pesticide residue and Formulation laboratories as well as controlling the registration of pesticides. The laboratories are served by one senior chemist, three chemists and three Technical Assistants. One senior chemist (part-time) and two chemists were attached to the residue laboratory for training purposes.

This report discusses in detail the present set up of the residue laboratory, training programme to the laboratory staff as well as recommendations for further residue analysis.

3.1 Background

Bangladesh is primarily an agricultural country. Its economy is largely dominated by agriculture which accounts for about 46% of its gross domestic product, 75% of employment and over 80% of export earnings.

Rice is the major crop, Bangladesh ranks fourth among the rice producing countries of the world. Rice, the staple food of the people of Bangladesh dominates farming; approximately 80% of the total acreage under agriculture is devoted to rice. Approximately 80% of the world's jute is produced in the country and it is a major source of foreign exchange, providing 56% of the total exports. Bangladesh is the world's fifth largest producer - exporter of tea in the world. Other major crops are tobacco, vegetables, fruits, pulses, cereals, potatoes and sugarcane. These crops are mainly for domestic consumption. Spices are also a major export and there is a hope for a surplus of potatoes for export in the future.

Bangladesh is a food deficit country and to increase production of food grains, the government has been pursuing various agricultural policies. These include education, research, training and use and control of pesticides.

Pesticides play a vital role in controlling pests and diseases and thus ensuring both quality and quantity of food. the pesticide administration and quality control section under the Plant Protection Wing of the Department of Agriculture requires extensive information on pesticides before they are approved by the Ministry of Agriculture. The information includes toxicology of pesticides, formulation and environmental behaviour including pesticide residues in crops and soils. The pesticide ordinance of 1971, modified in 1984 regulates the import manufacture, formulation, sale, distribution and proper use

of pesticides. The pesticide rules of 1985 lay down detailed requirements of inter alia the government chemist and the pesticide laboratory.

The pesticide laboratory was started in 1966 and after two moves was located at the Plant Protection Wing in 1982. At the time the laboratory was set up, assistance both material and technical, was given by GTZ (the German Technical assistance programme). The present residue analytical instruments were provided by BGD 80/003 project under the direct supervision of Mr D U Khan. The laboratory suffered a severe blow in 1984 when Mr S A Khan (Deputy Director in charge of Pesticide Registration and Quality Control) and Mr R Amin (Senior Chemist) were killed in an aeroplane crash.

4 Proposed Plan for Training in Pesticide Residue Analysis

The consultant will undertake the following plan for residue analysis during the period from 15 March to 9 April 1987 at the Department of Agricultural Extension, Plant Protection Wing, Dhaka, Bangladesh.

1) General discussion and presentation on residue and environmental analysis.

2) Discussion about the significance of residues, tolerances, Maximum Residue Level, no observed effect levels and Acceptable Daily Intake.

3) To set up some supervised residue trials for crops and soils. To demonstrate safe and effective application of a pesticide.

4) Procedures for taking crop, soil and water samples from the supervised farmers trials.

5) Procedures for transportation, logging and storage of residue and environmental samples. Procedures for sample preparation/reduction. To establish written guidelines (in Bangla) on items 3, 4 and 5.

6) To review existing policies and practices involving pesticide residues and residue analysis.

7) To provide hands on training on analysis of important pesticides residues. To adopt analytical methods suitable with the existing available equipments/instruments.

8) To provide practical guidance on trouble-shotting and maintenance of equipment, eg GLC etc.

9) To give comprehensive background on chromatographic techniques, eg TLC, HPLC, GLC, spectrophotometres etc.

10) To discuss good laboratory practice (GLP).

11) To discuss safety procedures in residue laboratory.

12) To discuss present state of art - residue and environmental analysis.

13) To submit a report of findings and recommendations.

4.1 Residue Laboratory - Present Set Up

There are well over 50 pesticides registered in Bangladesh. There is a growing pressure of Plant Protection Wing from various sources, eg exporters, trade association etc to provide information on the residue levels on major crops especially on those crops which are exported to various countries.

The Residue laboratory is a small room (13 ft x 12 ft) on the third floor of Plant Protection Wing. The major items of instruments/ equipment available in this laboratory are listed in Appendix II. At present no regular residue analysis is carried out on crops, soils or

water. It is worth mentioning that if residue analysis has to be done seriously on a regular basis, these must be a commitment by the higher management of Plant Protection wing to upgrade the existing facilities, eg space, equipments and retain and motivate the trained chemists. Equipment and reagents required for residue laboratory are in Appendix III and IV respectively.

4.2 Programme Implementation

On arrival at Plant Protection Wing on 15 March 1987, the director Mr M A Malik gave a brief description about the set up of their departments. I was introduced to Senior Chemist Mr Jaffar and later on with two graduates Mr Mohiuddin and Mr Mahbub who were assigned to be trained for residue analysis.

The programme was split up into theoretical tutorials and practical approaches regarding residue analysis.

4.3 Theoretical Tutorials

A detailed introductory lecture was given about residue analysis to all members of the staff of pesticide and Quality Control Section. The lecture and discussion was supported by a number of slides and overheads. The following points were highlighted during the discussions:-

1) Pesticide residue analysis is an extremely complex and specialised area of chemistry and is a big challenge to the residue chemist.

2) Need to prevent contamination and focus was drawn to the sources of contamination. There is a need for separate work up areas as well as equipment and glassware etc.

3) Particular attention was drawn to requirements of special glassware samples preparation extraction and clean up equipment and need to store residue samples in the deep freeze.

4) There is a need to keep the formulation and residue laboratories separately.

5) Why there is a need for sensitive and selective instruments?

In addition to introductory lecture, an overhead presentation was also given on the significance of residues, Maximum Residue Limit (MRL), Theoretical and Actual Residues, No-Observed Effect Level (NOEL), Acceptable Daily Intake (ADI) and Risk Evaluation to Consumers. Various tutorials were given during one month on chromatographic techniques, spectrophotometres, sample preparation/reduction, accuracy and precision, limit of detection and determination. Several consultant's notes and various papers/ literature/books were given to the chemists for retention.

Full details of the topics covered during tutorials are given in Appendix V.

4.4 Practical Training

Both chemists, Mr Mohiuddin and Mr Mahbub had some experience in analysis particularly in formulation chemistry. However, both of them were extremely keen and enthusiastic to learn residue analysis for various organochlorine, organophorous and carbamate pesticides.

The residue laboratory had only one gas-liquid chromatograph (DANI 3865) fitted with dual detector flame inoisation and electron capture (^{63}NI). There is no Nitrogen Phosphorus detector (NPD) therefore, no analysis of organophorous compounds by GLC was undertaken. There is no high performance Liquid chromatography technique available at Plant Protection Wing. The UV/visible spectrophotometer (PYE unicam SP8-400) is an old model and has been out of action for a long time.

Therefore, initial training on analysis was mainly undertaken on various organochlorine compounds, eg Dieldrin, Endosulfan etc.

4.4.1 Gas-Liquid Chromatography (Electron Capture Detector)

The GLC-ECD was bought during 1986 but was not used for residue analysis. Full training on the use of ECD was given to chemists. Linearity calibration curves were drawn for dieldrin, endosulfan. Various notes were given regarding setting up GLC-ECD and particular attention wad drawn on how to avoid contamination of the ECD. Simple techniques for cleaning the detector were demonstrated.

Various column packings, eg OV101, SE30, OV17, DC200 were made in the laboratory and detailed procedures were given on column packaging/column conditioning.

4.4.2 Residue Analysis for Crops, Soils and Water

Full practical training on the external standard methodology was given for dieldrin and endosulfan in crops, soils and water. In summary the following steps were discussed in detail:-

1) Logging of residue samples and procedures of storing samples (deep freeze was not available in the section, samples were stored in the fridge).

2) Preparation and sample reduction for residue analysis.

3) Extraction procedures - choice of solvents to extract weathered residues.

4) Recovery experiments - spiking and levels of recovery criteria.

5) Selective partition of various compounds, including back partition etc.

6) Column chromatographic clean up - Florisil calibration of column material and various elution patterns were done. Other absorbents, eg Alumina, Silica etc were also discussed.

7) Measurement of residues.

8) Calculations of results and expressing results after corrections for external recoveries.

Similarly analysis of dieldrin and endosulfan was done on various other substrates to demonstrate the robustness of the method. Limit of detection and limit of determination of the methods were established.

Limited work was done by thin layer chromatography to measure some organophorous residues, eg Diaznion, Carbofuran and Fenitrothion. However the practical residue analysis was not done because the UV lamp has been unusable for some time. Full detailed of the practical work is given in Appendix VI.

4.4.3 Conduct of Supervised Trials

Supervised residue trials on Rice, soil and eggplants were carried out at the Agriculture Training Institute, Dhaka.

The design and layout of these trails were fully explained and discussed. Mr Brian Crozier Consultant of the formulation Chemistry and Plant Protection Wing (UNDP/FAO Project BGD/80/003) provided all the help on the formulation of dieldrin and endosulfan. Various plot sizes (3 x 10 m or 3 x 5 m) containing rice, eggplants were sprayed with either dieldrin or endosulfan at double the recommended rates for demonstration purposes.

Particular attention was drawn to the following aspects of these trials:-

a) Choice and selection of the plot size and marking of boundaries.

b) Calculations of volume of formulated materials required for the area to be sprayed.

c) Accurate calculations of the volume of spray solutions, eg calibration of spray boom, speed of walking and volume of spray per time.

d) Various steps to be taken to avoid contamination of the residue samples from the formulated materials/spray solutions.

e) Samples of diluted spray solutions were taken for analysis in the formulation laboratory.

f) Proper disposal of spray dilution in the field.

g) How to take residue samples of rice plant, eggplant (fruit and leaves) and soil at various pre determined sampling intervals. Full demonstration of random sampling and weight/number of plants to be taken from the appropriate plots for residue analysis.

Photographs are attached in Appendix VII.

h) Samples of rice plant, eggplant and soils taken at 0 and 7 days after the application were analysed for dieldrin and endosulfan. Further samples will be taken at harvest intervals and will be analysed by the residue analysts. The results will be sent to the consultant for discussions.

5 Conclusions

Residue analysis is extremely complex and highly specialised branch of chemistry. There is a growing pressure to undertake residue at Plant Protection Wing. The Pesticide Residue Laboratory is now equipped to carry out limited analysis or organochlorine pesticides. There is an urgent need to improve the present facilities of the residue chemistry laboratory and detailed recommendations of the consultant must be strictly followed.

In summary there is a need to increase the laboratory space, to buy various equipment and instruments, eg deep freeze, GLC fitted with NP detector, HPLC, UV lamp for TLC methods, spectrophotometer as well as general glassware and reagents. In addition there must be a commitment from the higher management to employ more (at least 2-3) well qualified chemistry graduate for residue analysis. Efforts must be made to retain the trained staff for carrying out residue analysis. Previously analysts trained abroad were transferred to non-analytical jobs in the department and this seems to be an utter wastage of resources. The resources from various aid agencies can only be usefully benefited if the appropriate trained chemists are retained, motivated and were given a good direction.

There is a limitation of funds in Bangladesh. Serious consideration should be given to the funds and resources required to buy the basic instruments to fulfil the consultants recommendations.

Upon purchase of various instruments and equipment based on the recommendations and equipment based on the recommendations of the consultant an extensive and proper training programme should be undertaken at regular intervals. TLC analytical methods should be developed for various organophorous compounds, until chemists are well trained on other instruments.

Based on the discussions of the report in UNIDO Headquarters, the Bangladesh Government should make a strong request to seek assistance for strengthening of the Plant Protection Wing of Agriculture Extension according to the Consultant's recommendations.

6 Detailed Recommendations

6.1 Accommodation

The residue analysis section must have suitable accommodation. The present one small room is inadequate, and therefore the room next to residue room (401) should be joined to the existing laboratory. This has already been discussed with the director Mr A R Khan and deputy director Mr M A Malik and they both are in full agreement with my findings. The design of the laboratory should be such that all instruments, eg GLC, HPLC, UV Lamp and spectrophotometer are located in a separate area well away from the working up benches.

The extended residue laboratory must have the following items:-

a) Separate from the sample receipt and storage of samples in the deep freeze. This room is now available and should be solely used for this purpose.

b) The existing toilet should be removed and this room is to be used for washing up of glassware. Residue glassware must always be washed by chemists to avoid any contamination and all glassware is to be kept separate from formulation laboratory glassware. This room must be equipped with a deep sink with independent hot and cold water supply. A distilled water unit should be fitted in this room to have a separate distilled water for residue analysis.

c) Room No 401 should be fitted with another air conditioning unit.

d) Adjacent to Room 401, is also to be fitted with air conditioning unit.

e) A separate fridge is necessary to keep all standard solutions as well as crop/soil extracts. This can be located in the sample preparation room.

6.2 Staff

Mr Mohiuddin and Mr Mahbub have started doing limited residue analysis and they have been trained to do some analysis of organochlorine compounds. Both of them should always be working on residue analysis, otherwise extensive training given to them will be a waste of the consultants time. The consultant has given them enough residue work for the next 9 months. In addition, if residue analysis is to be undertaken on a routine basis, an additional graduate (in chemistry) and one technical assistant should be employed as soon as possible. There should be an extensive regular training programme on residue analysis in the laboratory. When the present staff (including new recruits) have been working continuously for a period of well over two years, then one or two chemists should be sent to an established laboratory in a developed country where analyses are carried out on a routine basis.

The consultant strongly suggests that the residue staff at Plant Protection Wing should be trained next year provided all recommendations submitted are completed. Any trained staff must not be transferred to non-analytical jobs in the department.

6.3 Equipment

6.3.1 Instrumentation

A gas-liquid chromatograph fitted with Nitrogen phosphorus deter, eg Hewlett Packard or varian should be bought to do residue analysis for organophorous and Nitrogen Containing pesticides.

It is also possible to fit NPDector on to the existing DANl 3865 series providing the existing manufacture makes this NPD for this series. When the instrument is ordered, sufficient spares, eg additional beads, connectors, tubings, columns, ferrules must be ordered. This instrument must be installed properly and evaluated according to manufactures specifications before paying off the final amount.

High Pressure Liquid Chromatograph (HPLC), eg Pye Unican PU4000 series.

HPLC is a well established technique for pesticide residue analysis and an instrument should be bought to do many pesticide analysis. It is relatively easy to use and chemists can be trained within a short time for routine analysis. Consideration should be given to buying this instrument which may already be available in some manufacturers laboratory, eg ICI Bangladesh Quality Control Laboratory has Pye Unicam HPLC and therefore in case of any problems guidance can be obtained from experienced chemists. Prepacked HPLC columns must also be ordered. Please get in touch with the consultant if you require any more information on GLC or HPLC for residue analysis.

A suggested list of equipment, glassware and reagents is attached on Appendix III.

6.3.2 Analytical Standards

Pure analytical standards together with the purity certificate of analysis should be obtained from the manufacturing companies. All analytical standard should be stored in the fridge.

6.3.3 Sampling

A guideline for procedures to take residue samples from the field has been translated into Bangla by Mr Mehbub after discussion with the consultant. Procedures were also given for the preparation of residue samples, reduction and storage of residue samples. Residue field data forms are also given for translation with Bangla.

6.3.4 Transport Facilities

Residue chemists have to conduct supervised trials and from time to time collect samples for residue analysis. It is, in my opinion essential to have a car solely for this purpose under the direct control of residue department.

6.3.5 Storage of Residue Data

Serious consideration should be given to store all data properly. This includes all records of field trials, laboratory notebooks and instrument traces.

7 Acknowledgements

The consultant would like to thank all members of the Plant Protection Wing for there kind co-operation. Thanks are also due to Mr D U Khan, National Project Co-ordinator and Mr Jaffer, Senior Residue Chemist of Plant Protection Wing for making resources available to the consultant.

The consultant was very impressed from Mr Mehbub and Mr Mubiddin, residue chemists of Plant Protection Wing for their keenness to learn residue analysis.

Particular thanks for Mr D U Khan, National Project Co-ordinator for providing all transport facilities for the Consultant.

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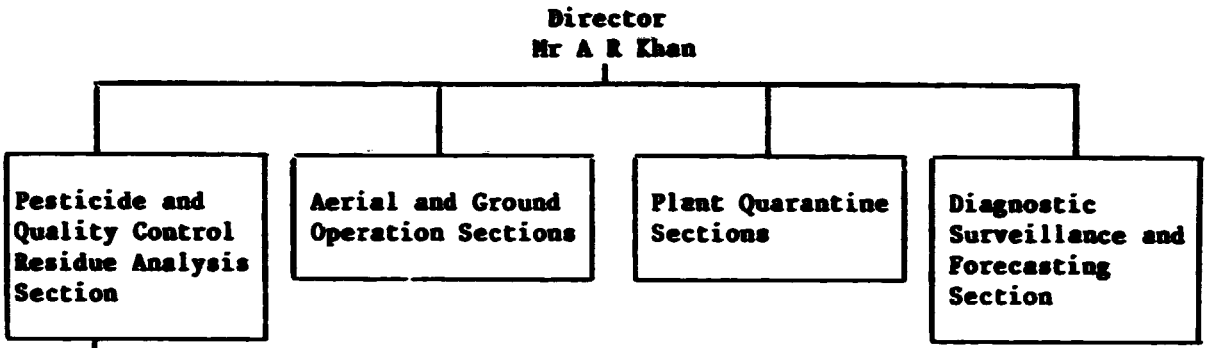
APPENDIX I

ORGANISATION AND PERSONNEL CHART

PLANT PROTECTION WING OF AGRICULTURAL EXTENSION



Deputy Director and Registration Manager

Mr M A Malik

Senior Residue Chemist

Mr Jaffar

Residues	Formulation
Mr Mohiuddin	Mr Afiqur
Mr Mehbub	1 Technical Assistant
	2 Laboratory Assistants

APPENDIX II

EQUIPMENT AVAILABLE AT THE RESIDUE LABORATORY, APRIL 1987

1 Gas-Liquid Chromatograph DANI 3865 FID/ECD.

2* PE550A UV/Visible Spectrophotometer.

3 Buchi Rotavapor R110 Rotary Evaporator.

4 6 Unit Soxhlet extraction apparatus.

5* Centrifuge Labofuge 600 Hereaus.

6 Ultrasonic Bath CL-UI Combilator.

7 Oven 0-300^{o}C Hereaus.

8 Water bath Haeke F3.

9 Mettler Analytical Balance.

10 Mettler 10 Kg Balance.

11 Sortorius 1103 2Kg Balance.

12 Sieve Shaker.

13 Vacuum/Compression Pump.

14* UV lamp-Comag.

15 Magnetic Stirrer/Hotplates x 4.

16 Griffin Flask Shaker.

17 KOTTERMANN Yolo Orbital Shaker.

18 Waring Blender

19 Ultra Turrax Grinder - Janke and Kunkel.

* Equipment in need of repair or replacement.

APPENDIX III

EQUIPMENT REQUIRED FOR RESIDUE ANALYSIS

1 Equipment for the initial preparation of samples, eg:-

a) Hobart Mincer - available from Glen Creston, Stanmorse, UK.
b) An ultra centrifugal mill, Retsch ZM1, fitted with 3 mm screen (Glen Creston).

2 Range of knives (large, medium and small).

3 Trays - 6-8 for sample preparation.

4 High speed macerator, eg laboratory mixer emulsifier available from Silverson machines Ltd, UK or Sorvall Omni - Mixture homogeniser available from Du Pont UK Ltd.

5 Deep freeze for storage of Residue Samples (down to -20°C).

6 Gas-Liquid chromatograph fitted with Nitrogen - phosphorous detector (NPD). Additional beads etc together with accessory should also be obtained.

7 UV-Visible spectrophotometer.

8 UV-lamp.

9 TLC tanks (at least 3 or 4) and TLC plate preparation equipment.

10 HPLC with ultra-violet detector.

11 Various HPLC columns 25 x 5 mm id with 5 mm ultrasphere ODS or similar packings suitable for valve injection.

12 Potentiometric pen recorder (10 mv).

13 Syringes for HPLC and TLC, eg Hamilton 25, 50 and 100 ul.

14 Glass columns for GLC 300 mm x 10 mm ID (to fit with Dani 2865 GLC-ECD and to fit GLC-NPD).

15 Graduated glass centrifuge tubes of 10 ml capacity calibrated down to 1.0 ml in 0.1 ml units.

16 Measuring cylinders stoppered - 250 ml, 100 ml and 50 ml (at least 12 of each size).

17 Separating funnels (500 ml, 250 ml and 100 ml) at least 12 of each size.

18 Rotary evaporator with good vacuum system.

19 Round bottomed flasks (250, 100 and 50 ml) (at least 12 of each size).

20 Pasteur pipettes - 10-12 boxes. there are essential in order to transfer small volumes of various solvents.

21 Hot and cold water supply - separate water tank supply.

22 Proper facility for washing up of glassware.

23 Chromatography columns - short.

24 Silylanising agent, eg Silyl 8.

25 Septa, 'O' rings etc.

26 Hair drier.

27 Safety glasses - lightweight.

28 Safety shields - for rotary Evaporator.

29 Fume cupboard.

30 Laboratory coats.

31 Eye-wash bottle.

32 Nitrogen or air supply for evaporating small volumes of organic solvents.

APPENDIX IV

REAGENTS REQUIRED FOR RESIDUE ANALYSIS

1 Solvents - Redistilled Acetone Methanol h-hexane, diethyl ether, dichloromethane chloroform, acetonitrile, ethylacetate.

Particular care must be taken to avoid contact with materials, eg plastics which may contaminate the solvents.

2 Granular anhydrous sodium sulphate - analae grade - BDH Chemicals Ltd, Poole, UK. Heat in an oven at 140^{o}C for 24 hours to remove volatile contaminants.

3 Glass wool.

4 Florisil (100-120 US mesh), Alumina (Neutral and basic) and silica.

5 Anti foam emulsion M30 - available from Hopkin and Williams - UK.

6 BOND ELUT^{TM} disposal columns (2.8 cm^{3}) containing 500 mg unbonded silica - Jones chromatography.

7 Celite 595 filter aid.

8 Acids - Con HCL, H_2SO_4 and glacial acetic acid.

APPENDIX V

THEORETICAL TUTORIALS

1 Introductory lecture on residue analysis - slide presentation.

2 Significance of residues in foods, maximum Residue Limit (MRL), Theoretical and Actual Residues No Observed Effect Level (NOEL) Acceptable Daily Intake (ADI), Examples of various Risk Evaluation to Consumers.

3 Discussion on guidelines for Residue sampling and sample preparation. These guidelines are written up in Bangla language.

4 Discussion about how to conduct a supervised residue trial.

5 Safety in the laboratory.

6 Chromatography:-

- a) General
- b) Paper
- c) TLC
- d) Column
- e) GLC
- f) HPLC

7 Spectroscopy:-

- a) UV/Visible
- b) Others

8 Internal Standards Use in Residue Analysis.

9 Good Laboratory Practice (GLP) and Good Agriculture Practice (GAP).

10 Various papers for residue analysis, eg FAO recommended methods, joint FAO/WHO Food Standard programme, Codex Alimentarious Commission Guidelines for Good Analytical Practice in Residues and recommendations for methods of Residue, technical monograph No 8, 1983, GIFAP Pesticide Residues in food, Pesticide Manual and various guidelines for sampling etc, were given for retention at Plant Protection Wing.

APPENDIX VI

PRACTICAL TRAINING

1 Topics covered in Practical gas-liquid chromatography.

a) Preparation of various column packing materials.
b) Injection techniques.
c) Measurement of Carrier gas flow rate and influence of glass flow rate.
d) Measurement of retention time and relative retention times.
e) Trouble shooting.
f) Decontamination of ECD.

2 Procedures of logging residue samples, storing samples etc, preparation of residue samples for various crops and soils.

3 Field Sampling Procedures.

4 Conduct of supervised trials - spraying (some photographs are attached in the Appendix VII).

5 Extraction, partition, column clean up and calculations of residue.

6 External standard methodology.

7 Receiving experiments procedures.

8 Criteria for acceptance of residue results.

9 Limit of detection and limit of determination.

10 Expressing residue results to two significant figures.

APPENDIX VII

CONDUCT OF SUPERVISED TRIALS

Photos showing preparation of formulation solutions in the field, spray the plot of crops, sampling rice plants, eggplants and soil.

Photograph 1. Preparation of formulation solutions of Dieldrin.

Photograph 2. Spraying eggplant crop.

Photograph 3. Sampling of Rice Plant

Photograph 4. Sampling of egg plant (Flint of leaves)

Photograph 5 and 6. Taking soil samples (0-5 cm) depth with the available tools.