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20181

Distr.
RESTRICTED

IO/R.267
15 April 1993

UNITED NATIONS
INDUSTRIAL DEVELOPMENT ORGANIZATION

ORIGINAL: ENGLISH

14 p.
diagrams

ASSISTANCE TO PESTICIDE FORMULATION TECHNOLOGY DEVELOPMENT

DG/CPR/91/121

PEOPLE'S REPUBLIC OF CHINA

Technical report: Findings and recommendations*

Prepared for the Government of the People's Republic of China
by the United Nations Industrial Development Organization

Based on the work of D. A. Knowles,
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* Mention of firm names and commercial products does not imply the endorsement of the United Nations Industrial Development Organization (UNIDO).

This document has not been edited.

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SUMMARY

This is a report of the first visit of the Chief Technical Adviser (CTA) to China (17 Feb to 3 March, 1993) to assess the technical requirements of a 5 year project aimed at the introduction of environmentally-friendly pesticide formulations to replace old formulations based on solvents, dusts and powders. Some project background discussions took place at the UNDP and CICETE offices in Beijing during 2 days, followed by 12 days of detailed discussions at the Project Authorities Site, Nanshen Chemical R & D Corporation (NSCC) at Nantong, Jiangsu Province. The National Project Director (NPD),Mr Hong Chuan Yi, was present throughout all the discussions, and acted as interpreter.

The state-of-the-art of conventional and recent pesticide formulations and process developments was reviewed in a 3 - day series of lectures by the CTA and discussions with technical staff at NSCC. This was followed by an assessment of future requirements for formulation areas of suspension concentrates (SC), oil-in-water emulsions (EW), suspoemulsions (SE) and water dispersible granules (WG).

The list of equipment in the Project Document was reviewed and modified where necessary. A few items have already been ordered and the CTA will send technical specifications of other essential items to NSCC. Some work with the new equipment will start in the old laboratories, but as soon as possible (mid - 1994) a move will be made to the new laboratories being built nearby. Pilot plant equipment will be installed in an existing empty building.

The whole Project Document (CPR/91/121) was discussed in detail and recommendations were made for Fellowships, Study Tours and visits by International Experts to Nantong. It was felt that a subcontract for WG development work is necessary to determine which of the various processes for WG's should be developed by NSCC.

Finally a plan for the first year of development work was proposed for approval.

Annexes are attached which show the job description of the CTA, the organisational links between the various parts of the Pesticide Project, and also an organisation chart of staff at NSCC, Nantong, along with names of people met during the visit.

1. FORMULATION TECHNOLOGY AND EQUIPMENT

During technical discussions with staff at NSCC, it became clear from their state of development and the type of active ingredients being handled by NSCC, that further formulation developments should have the following priority:-

1.1 Suspension Concentrates (SC)

NSCC have already done some promising work with 3 active ingredients using locally available dispersing agents and thickeners. A small laboratory open-topped bead mill is being used, which gives foaming problems and cannot be used continuously. A laboratory Colloid Mill is being used for premixing prior to bead milling.

It is proposed that NSCC should purchase some high shear mixing equipment for premixing and a laboratory scale horizontal bead mill such as a Dyno KDL or a Drais Mill. These homogenisers and bead mills will give much more efficient milling without the problem of aeration and can be used batch-wise or continuous. Both types of equipment are available in Europe from laboratory through to Pilot Plant and Production scales.

NSCC have already placed an order with IKA, Germany, for a small laboratory T25 Homogeniser, price US $ 7,734.40.

The CTA will send to NSCC details of technical specifications, price quotations and Agents in China for suitable homogenisers and bead mills as soon as possible.

The development of good suspension concentrate formulations with acceptable storage stability requires careful measurement of particle size distribution and structural elasticity and flow properties, ie. rheology. NSCC have already ordered a Malvern Laser Mastersizer E (with Service Contract), price £33,575. This instrument will be very suitable for measurements of particle size of suspensions, emulsions and wettable powders. NSCC are planning to send 2 staff from Nantong to Malaysia for 6 days in May 1993 for training with Malvern.

Controlled Shear Rheometers are essential for meaningful measurement of rheological structure and flow properties. Rheometers are available from a few suppliers in Europe. The CTA will send details to NSCC as soon as possible covering technical specification, price quotations and Chinese Agents, if any.

1.2 Oil-in-Water Emulsions (EW)

NSCC are very interested in converting some insecticide EC formulations to EW formulations to reduce the need for solvents. The CTA gave some advice on suitable emulsifiers and stabilisers for EW formulations. High shear mixer homogenisers, such as the equipment to be purchased for suspension concentrates, will probably reduce the emulsion droplet size to 2 - 5µm. However, in order to improve emulsion stability, it is necessary to reduce the droplet size down to 0.5 - 1µm. This can be done using a Valve Homogeniser. The CTA will provide a technical specification and price quotation for a suitable laboratory Valve Homogeniser. Pilot Plant and Production scale models are also available.

1.3 Suspoemulsions (SE)

NSCC have a requirement for mixed formulations where one active ingredient is solid and the other active ingredient is liquid. They have already started some laboratory development work on mixtures of SC and EW, known as Suspoemulsions SE formulations.

The CTA gave some advice on suitable dispersing agents, emulsifiers and stabilisers for SE formulations. All the equipment being purchased for SC and EW formulations will be suitable for SE formulations.

1.4 Water Dispersible Granules (WG)

Water dispersible granules are a recent development in pesticide formulation technology. They are much more environmentally friendly and eliminate the problems of solvents, dusts and powders. Packaging in paper cartons or water-soluble bags can be carried out very conveniently.

The CTA reviewed all the available technology for producing WG formulations. It is not possible to recommend a single preferred process until some laboratory work has been carried out with various pieces of equipment. It is proposed that this preliminary work should be sub-contracted to a Company in Europe which has all the suitable laboratory equipment for testing a few products. Once this work has been done, the CTA will be able to recommend the purchase of suitable laboratory and pilot plant equipment so that the development of specific formulations can take place at NSCC.

From our initial discussions with NSCC, it was suggested that extrusion granulation technology may be the most efficient and versatile process, but a final decision should await the results of the sub-contract work.

The active ingredients required by NSCC as WG formulations are, in order of priority:-

Metalaxyl, Bensulfuron, Decamethrin, Glyphosate and Chlorsulfuron.

1.5 Microencapsulation (CS)

Before any development work is started on microencapsulation formulations, some market research should be carried out to assess the market requirement for controlled release formulations.

Microencapsulation is complex technology and may involve the handling of toxic materials such as isocyanates.

It was proposed that microencapsulation should be given a low priority, and only introduced after the more important work on SC, EW, SE and WG formulations is well established.

2. PILOT PLANT

It is proposed that new equipment for pilot scale up development will be installed in an existing empty building at the NSCC production factory in Nantong.

The equipment for liquid and solid granular formulations will be scaled so that it will be capable of preparing a few litres or kilos for storage testing and small plot field trials, but at the same time large enough to carry out small production campaigns for test marketing, eg. continuous throughput rates of at least 50 litres or kilos per hour.

The purchase of pilot plant equipment will not be required until after the laboratory equipment has been purchased, ie. from 1994 onwards. The Pilot Plant should have a small laboratory for carrying out quality control tests.

The Pilot Plant will also have filling and packing lines for liquid and solid formulations.

Full safety, hygiene and environmental features will be included.

3. EQUIPMENT

3.1 Formulation Equipment

Some of the items of equipment mentioned in the Project Document for SC, SE, EW and WG formulations have already been discussed in Section 1.

Item 2. 3-6. A decision on these items for WG formulations must be delayed until the results of the sub-contract work are known.

Item 2. 7. A Rotovisko Rheometer complete with computer control and thermostat is essential for SC, SE and EW development. However, the allocated budget of $5,000 is totally inadequate. A budget price of about $50,000 would be more realistic.

Item 2. 8. A Zetameter is not necessary.

Item 3. 9-15. These items for WG development must be delayed until the results of the sub-contract work are known.

Item 4. 16-18. It was agreed that air or jet milling for powders and WG formulations is not necessary. Mechanical grinding is the preferred technique and NSCC have already ordered a small IKA M20 coffee grinder for making very small laboratory powder samples. The CTA will send details of larger laboratory powder grinders and suitable Pilot Plant mechanical grinding machines.

Item 4. 19. A micro-atomiser is not necessary.

Item 4. 20. Filling and packing machines for liquid and solid products will be required for the Pilot Plant. This information will be required at a later date. Filling and packing equipment is a specialised area, and it is proposed that an experienced engineering consultant should visit Nantong at a suitable time in the project when the equipment has been installed and is operational.

Item 5. 21. This is covered by comments on Rotovisko Rheometer (see Item 2. 7).

Item 5. 22. A Powder Tester is not necessary.

Item 5. 23. Particle Size Analyser has already been covered in Section 1.1.

Item 5. 24. NSCC already have a suitable Surface Tension meter.

Item 5. 25. NSCC will follow up locally to purchase a suitable instrument for measurement of contact angle for spray applications.

A new item of equipment for EW formulations is the Value Homogeniser previously discussed in Section 1.2. The CTA will send details of this item to NSCC.

The CTA will also send details of electronic balances suitable for formulation laboratory and Pilot Plant.

3.2 Analytical Equipment

Item 6. 26-27. NSCC have already ordered a Waters Millipore HPLC, price US $47,659.90 including installation costs and training 2 staff in Singapore for 2 days.

Item 6. 28. NSCC will discuss with local agencies the purchase of an electronic analytical balance covering the range 0.1mg to 200g.

Item 6. 29. The CTA will send details of a Moisture Content Analyser for WG development.

Item 6. 30. Fridges and ovens for storage stability testing, -30 to +50 C, can be purchased locally by NSCC.

Item 7. 31. An autoclave is not necessary.

Item 8. 32. These items will be required for the move into the new office and laboratory building.

4. PROJECT INPUTS

4.1 Government Inputs

These are all fixed and there are no problems, according to CICETE.

4.2 UNDP Inputs

4.2.1 International Experts

It is proposed to reduce the requirement for International Experts in Formulation Research/Technology (BL 11-02 and 11-03) because a substantial amount of advice and guidance in formulation research and technology would be provided by the CTA. The budget for the CTA BL (11-01) should, therefore, be increased to 12 m/m to allow for some Homebase work. A budget of 4 m/m each for BL 11-02 and BL 11-03 would be more realistic.

An analytical consultant (BL 11-04) will be required to help with advice and problem solving after the new analytical equipment has been commissioned, probably late 1993, or early 1994. The budget should be increased to 6 m/m.

It may be possible to combine Pollution Control (BL 11-06) and Hygiene (BL 11-08) into one International Expert with a total resource of 2 m/m.

The Packaging consultant (BL 11-07) should be an engineer with experience of filling and packing lines for liquids and solids.

4.2.2 Fellowships (BL 31-00)

Two fellowships are planned for 1993. The first fellowship is for 2 staff to study SC and WG technology at KWIZDA and at IPFT, India for a 3 - 6 month period. ($4,000 per month per person).

The second fellowship is for 2 staff to study SC, EW, SE, WG technology and rheological measurement at ICI and Rhone-Poulenc for 3 - 6 months during 1993.

A further 6 staff have been identified for further fellowships to be arranged in subsequent years. This makes a total of 10 staff and it was felt that at least 2 of these should be analysts who can study methods for chemical and physical analysis of pesticides.

It is unlikely that the total budget of US $300,000 for Fellowships will be required (BL 31-00)

4.2.3 Study Tours (BL 32-00)

It is proposed that a Study Tour should be set up for 6 staff from Nanshen and Shenyang to visit a number of companies in Europe, viz Hoechst, Oxon Italia, Ciba-Geigy, KWIZDA and others.

The study tour should be for about 4 weeks during November 1993 or early 1994.

4.2.4 Sub Contract (BL 21-00)

It is proposed that a sub-contract should be included in the Project Document with a budget of US $100 - 150,000. This funding can be found by transferring some of the funds from other budgets, such as International Experts and Fellowships.

The sub-contract work is required to investigate the optimum formulation and process technology for WG formulations, as described in Section 1.4. The sub-contract should be agreed and signed in 1993, so that laboratory work can start in 1994.

5. RECOMMENDATIONS FOR FIRST YEAR PROGRAMME

5.1 NSCC will continue the development of some SC, EW and SE formulations using ideas and suggestions arising from discussions with CTA.

5.2 CTA will send technical details of suitable equipment for liquid formulations and NSCC/CICETE will carry out purchasing procedures.

5.3 UNIDO/CTA will discuss the possibility of finding a suitable sub-contractor for WG development work in Europe. The contract should be signed in 1993 for work to start in 1994.

5.4 UNIDO will endeavour to set up two separate Fellowships for 4 staff in total to gain experience of liquid and granular formulations in Europe, and to include methods for physical measurement. Target starting dates mid to end of 1993.

5.5 UNIDO to set up a Study Tour for 6 staff to visit a number or European Agricultural Companies over a 4 week period. Target dates Nov 1993 or early 1994.

5.6 Analytical expert to visit NSCC, Nantong, near end of 1993 to give advice and to help with problem solving with new analytical equipment.

5.7 The date of the next visit by the CTA should be arranged to co-incide with the next TPR meeting in Beijing, probably October 1993. The CTA will also visit Shenyang to lecture on "Recent Trends in Pesticide Formulation Technology." Finally, the CTA will visit NSCC, Nantong, to review and monitor the programme and to agree plans for the forthcoming year.

6. MISCELLANEOUS COMMENTS

6.1 There is a need to improve the level of competence in spoken English amongst members of staff and plans are already in hand to send staff to a local College for training in English.

6.2 NSCC recognise the need to upgrade the level of safety and hygiene in the laboratory and production areas to bring them into line with International Standards.

General housekeeping levels need improvement and more emphasis placed on air extraction to control dusty operations.

6.3 Staff members would benefit from attending overseas formulation conferences and laboratory and pilot plant equipment exhibitions. It may be possible to link these visits with Fellowships and Study Tours in Europe.

6.4 The following Annexes are included at the end of this report:

ANNEX I CTA Job Description

ANNEX II Institutional Framework for the Subsector

ANNEX III Organisation Chart for Nanshen R&D Corporation

The staff met by the CTA during the visit are indicated on the charts.

7. ACKNOWLEDGEMENTS

The CTA would like to thank Mr Hong Chuan Yi (NPD) for his well planned arrangements and excellent hospitality throughout the visit to China. Also to the staff at NSCC, Nantong, for their friendly and helpful co-operation with this project.

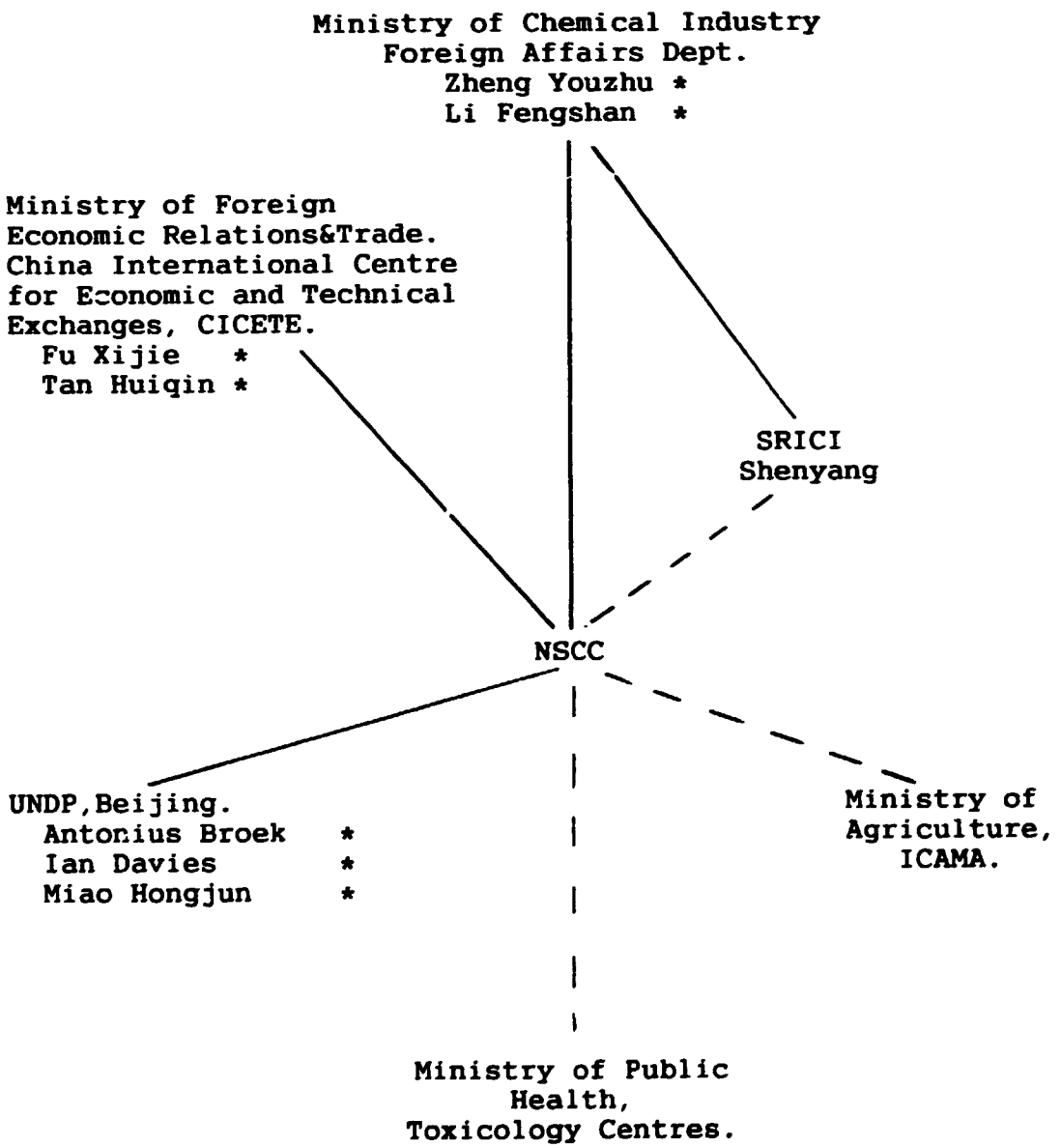
ANNEX I

CTA JOB DESCRIPTION

Title:	Chief Technical Adviser (CTA) BL 11-01.
Duration:	10 m/m (including home base).
Duty station:	Nantong, Jiangsu Province.
Purpose:	To assist China in developing pesticide formulations according to international standards making use of locally available raw materials,so that wastage of pesticide is minimised and user friendly formulations are introduced in the country.
Duties:	The consultant in collaboration with the NPD is expected to assist: --in modernising the existing laboratories at Nantong and provide advice in purchase of lab equipment, --in installation of lab equipment and assist in carrying out R&D work on formulations viz, SC, WG, WP,emulsions,controlled release and other formulations, --in carrying out physico-chemical property testing of pesticide formulations, --in advising the type of equipment that should be used for formulation purposes, --in identifying candidates for fellowship training, --in identifying experts, --in setting up pilot plants and scaling up new formulation products, --in setting up pollution control and occupational health safety standards, --giving lectures on recent developments in pesticide formulation, --submit technical report based on his work and with findings and recommendations, --prepare Terminal Report.
Qualifications:	Chemist,chemical technologist with extensive experience in pesticide development,production and useage. Must have experience of working in large pesticide formulation facilities at the top level with administrative responsibilities. Must be familiar with laboratory and pilot scale preparation of pesticide formulation, especially those which are aqueous-based, granules,mixtures etc. Experience in a developing country would be an advantage. He should have experience of international manufacturers of pesticides.

ANNEX II

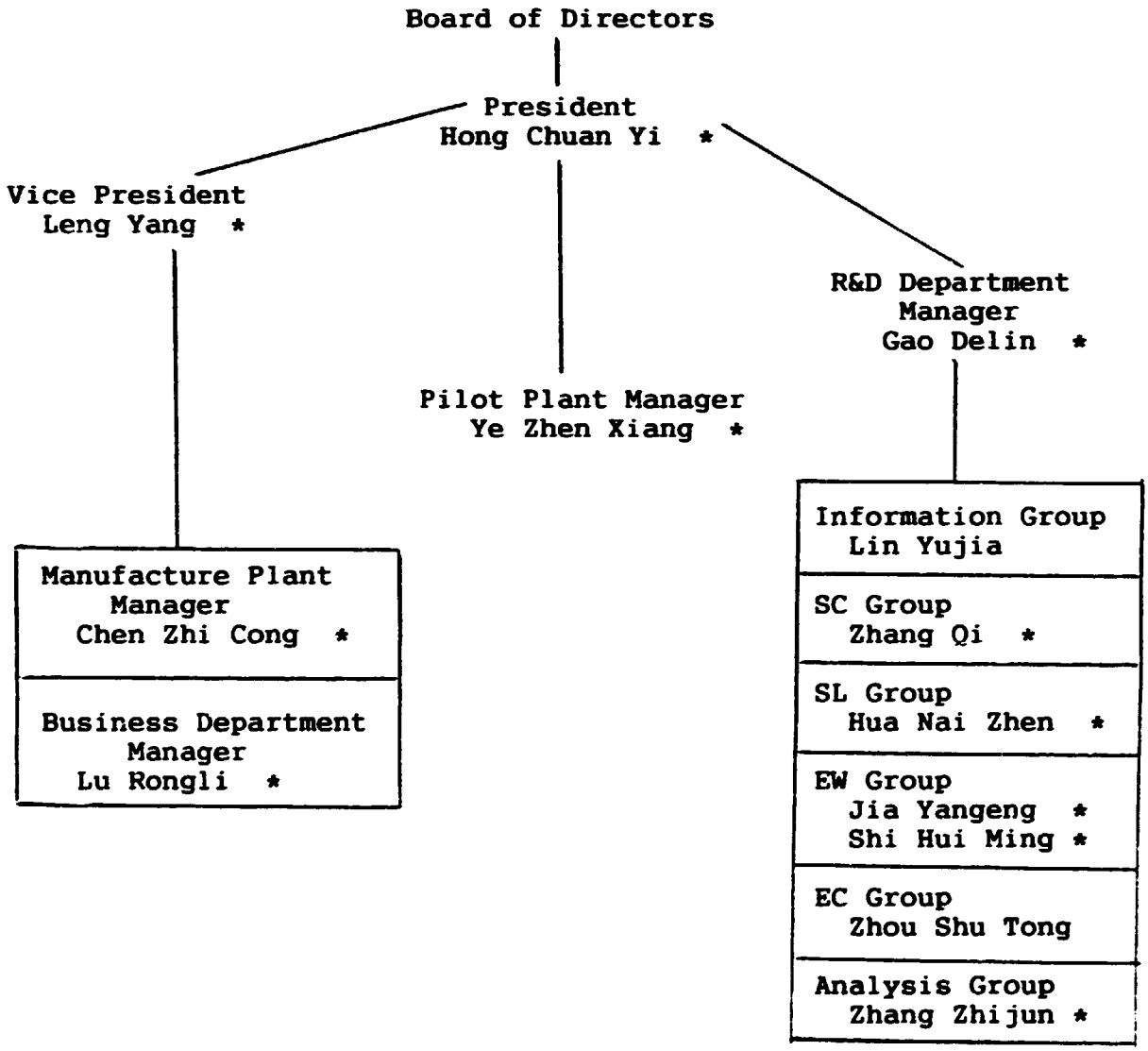
INSTITUTIONAL FRAMEWORK FOR THE SUB-SECTOR



* Staff met by CTA during visit.

ANNEX III

NANSHEN CHEMICAL R&D CORPORATION.



* Staff met by CTA during visit.

UNIDO COMMENTS

The visit of the expert is the first major activity in the project which will set the stage for future implementation. The report gives the inputs that are needed to start R&D work in high technology formulation. The most essential elements are the laboratory for laboratory scale operation and analysis. This can start in full swing once the new laboratory buildings are well set up.

The author's recommendation to start fellowships and also to look into the possibility of carrying out some experimental formulations outside the country in a reputable company should be discussed in the forthcoming meeting to allot funds from other budget line to subcontract arrangements. UNIDO can find a suitable subcontractor to do water dispersible granules in Europe.

Certain modifications given to the equipment list could be done and any savings likely to be incurred could be used for subcontract.

As the market in China is opening up, it is essential that bringing out high tech formulations should be in time and in accordance with market requirements with regards to safety, quality and bio-efficacy.