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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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This document has not been edited.

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### SUMMARY

This is the report of the third visit of the Chief Technical Adviser (CTA) to China (24 April - 21 May 1994). The purpose of the visit was to review progress against the Work Plan since the last visit in October/November 1993, follow up recommendations for the new R&D laboratories, the pilot plant and equipment, and to study in detail the flow sheet for the liquids pilot plant. Also to make further recommendations for recruitment of consultants, and plans for Fellowship Training and Study Tours, and to progress the requirements for the WG Sub-contract.

Discussions on the Project Programme and Budget took place at UNDP and CICETE in Beijing, and the technical progress work was carried out at NSCC, Nantong. A visit was also made to Shenyang Research Institute. The NPD, Mr Hong Chuanyi was present throughout all the discussions.

The Project Management Meeting was held at CICETE, Beijing on 12 May 1994 and was attended by the NPD, CTA and Dr S. P. Dhua (PMTA).

The project is moving according to plan. The recruitment of consultants, Training and Study Tours, and purchase of equipment are following closely the Budget for 1994. However, the requirements for WG development are about 3-6 months behind schedule, and plans have been made to focus on extrusion granulation technology to speed up the WG Sub-contract and training. Quotations from suitable contractors are being sought in USA, Japan and Europe.

The flow sheet for the liquids pilot plant has been simplified and made more flexible. Work on refurbishment of the pilot plant building in Nantong should start as soon as possible, so that it is operational in 1995.

Very good progress has been made with the new Research Centre building and it should be ready for occupation on schedule by July 1994.

RECOMMENDATIONS AND ACTIONS

1. The Work Plan and Timetable for 1994 for Recruitment of Consultants, Training and Study Tours, Equipment Purchase and WG Sub-contract should be followed as closely as possible to maintain the project on schedule.

ACTION: NPD/CTA/UNIDO.

2. Equipment purchases and installation should be progressed as quickly as possible in 1994.

ACTION: NSCC/CICETE.

3. Quotations should be sought for WG Sub-contract development and training in USA, Japan and UK/Europe, to be followed by a Study Tour in November 1994.

ACTION: NPD/CTA/UNIDO.

4. The Pilot Plant building should be refurbished as soon as possible, and the liquids pilot plant should be installed to become operational in 1995.

ACTION: NSCC.

5. Project Evaluation and Budget Revision should be carried out during the next visit in October/November 1994, in time for the TPR meeting.

ACTION: NPD/PMTA/CTA.

6. A second visit of a Packaging Expert should be made to NSCC, Nantong when the pilot plant equipment for liquids filling and packaging (Phase 1) has been installed during 1995.

ACTION: UNIDO.

## 1. PACKAGING WORKSHOP

The CTA attended a Packaging Workshop organised by NSCC and Mr.J.Hartmann, International Expert in Packaging Technology. This was held at the NSCC Conference Room, Nantong, on 28 - 29 April 1994. Delegates from NSCC, Nantong Packaging Standards Bureau, Plastic Bottle Suppliers and Bayer Agrochemicals China attended.

Mr.Hartmann has submitted a detailed report of his visit, and also held a de-briefing meeting with Dr.Sugavanam in Vienna. Recommendations have been given for purchase of package testing equipment, small bottle filling for liquids in Phase 1 of the Pilot Plant, and general International Standards which should be met for Packaging, Safety and Hygiene.

This was a good start to the introduction of new packaging materials and equipment for the Pilot Plant and later for small and large scale production filling lines for liquids and granules. A further visit to NSCC, Nantong should be made when the Pilot Plant (Phase 1) equipment has been installed during 1995.

## 2. EQUIPMENT REQUIREMENTS

All the recommendations and quotations provided by the CTA for laboratory and Pilot Plant formulation equipment were reviewed at NSCC, Nantong, and progressed (See Annex III of CTA's 2nd Visit Report, October/November 1993).

### 2.1 Laboratory Equipment

The Dyno KDL Bead Mill has arrived in Shanghai and will soon arrive at NSCC. The bead mill consists of interchangeable 600ml and 300ml chambers with a product pump. The suppliers will send one expert from Switzerland and two experts from Hong Kong to carry out installation and commissioning checks on 29 May 1994. (See also Section 2.2 Dyno KD25 Bead Mill for Pilot Plant).

Cost US\$ 17,300.

The Fritsch high speed rotor mill for powder grinding is also in transit from Shanghai.

Cost US\$ 7,500.

A contract has been signed with Hong Kong Agents for a high shear Silverson Laboratory Mixer Model L4R. Delivery to NSCC is awaited.

Cost US\$ 1,600.

A contract has been signed with Hong Kong Agents for a High Pressure Laboratory Homogeniser Type 8.30H for EW formulations. Delivery to NSCC is awaited.

Cost US\$ 11,000.

Contracts have been signed with Beijing Agents for an Electronic Analytical Balance, Electronic Formulation Balance and a Moisture Analyzer. Delivery to NSCC is awaited.

Total cost US\$ 13,000.

The equipment for producing pure water has been cancelled because de-ionised water can now be purchased easily in Nantong. This will save about US\$ 3,000.

A new quotation for a Haake Rotovisko RV20 has been received from the Beijing Agents. The new quotation is US\$ 55,000 compared to the original quotation of US\$ 37,000 obtained by the CTA. The increase is due partly to inflation and shipping and insurance costs, but mainly due to upgrading of the computer control system, printer and plotter. CICETE have negotiated with the Haake Agents for a better price on the computer package and a recent quotation has been received at US\$ 42,300, an increase of US\$ 5,300 over the original quotation. It is proposed to go ahead with this quotation which is valid until 30 June 1994.

The CTA has provided quotations for the following new items of equipment:-

#### Extrusion Granulation

Fuji-Paudal Kneader and Basket Granulator. Quotation from UK Agents, US\$ 25,220. However, a much lower quotation of US\$ 13,000 has been obtained from Fuji-Paudal, Japan, and it is proposed to place the order directly in Japan as urgently as possible. It will be possible to purchase a small laboratory fluid bed dryer in China to dry the granules.

#### Fluid Bed Granulation

Niro-Aeromatic STREA-1 Fluid Bed Granulator. Quotation from Niro UK, US\$ 37,120.

This quotation will not be progressed at present because the preferred process option for WG formulations is extrusion granulation.

The CTA visited Shenyang Research Institute to give a lecture and to tour their facilities. Shenyang have considerable experience of fluid bed technology and some of their equipment could be adapted easily to carry out laboratory work on WG formulations by fluid bed granulation techniques if NSCC have a need for this technology in the future.

## 2.2 Pilot Plant Equipment

The Dyno KD25 Bead Mill will soon be delivered from Shanghai to NSCC. The Pilot Plant has not yet been refurbished and, therefore, it will be necessary to set it up in a temporary position so that commissioning checks can be carried out by the Dyno experts on 29 May 1994 (see Section 2.1 for Dyno KDL Bead Mill).

Cost US\$ 70,000.

A contract has been signed with Hong Kong Agents for a Silverson AXR High Shear Mixer. A similar contract for two Silverson GX10 High Shear Mixers is awaiting a recommendation from the suppliers on the optimum R.P.M. for use with viscous liquids in 500 - 1,000 litre vessels.

Total cost US\$ 24,000.

A contract has been signed with Hong Kong Agents for a High Pressure Homogeniser for EW formulations, Type 10.50H. Delivery to NSCC is awaited.

Cost US\$ 23,160.

A contract has been signed with Beijing Agents for two Pilot Plant Electronic Balances. Delivery to NSCC is awaited.

Cost US\$ 9,400.

The quotation for a semi-automatic PPM Albro weight filler was discussed with the Packaging Expert, Mr. Hartmann. It was agreed that this quotation should be pursued along with others from Masterfil for volumetric piston-type filling in Phase 1 of the Pilot Plant.

PPM Albro cost US\$ 9,000 approximately,  
Masterfil cost US\$ 25,000 approximately  
(See Hartmann's report).

Small items of equipment for package testing should be purchased locally in China.

Cost US\$ 10,000 maximum.

(See Mr Hartmann's visit report, April/May 1994).

The CTA provided a quotation for a Micronette M500 powder micronising jet mill from Guseo, Italy. This mill has a throughput rate of 150 - 250 kg/hr. The quotation, without air compressor, was about US\$ 37,000.

## 2.3 Office Equipment and Transportation

The IBM PS/2 486 Computer and Hewlett-Packard Laser 4L Printer have been set up at NSCC and are working well.

Cost US\$ 6,750.

The Canon Fax machine is also set up and is in regular use.

Cost US\$ 2,050.



The Ricoh Photocopier will be set up with a voltage transformer in the New Research Centre.

Cost US\$ 4,500.

14 Air Conditioner units for the New Research Centre have been delivered to NSCC.

Cost US\$ 29,000.

A copying machine has been ordered from a local agent in China.

Cost US\$ 3,000.

The Dodge Maxiwagon was damaged in transit from USA to China. Repairs in Shanghai are now being negotiated by CICETE with the USA suppliers.

Cost US\$ 27,000.

A summarised list of all equipment items, status and cost is given in Annex I.

### 3. PILOT PLANT BUILDING AND FLOW SHEET

The Pilot Plant is an existing building in Nantong No.3 Pesticide Factory. The building has an upper floor but is in a very poor condition and needs to be completely refurbished before new equipment can be installed. The refurbishment work should start very soon so that new items of equipment can be installed and become operational as soon as possible in 1995. It will be necessary to provide a service lift to move materials from the ground to the upper floor.

A flow sheet drawing for a liquids pilot plant for SC, SE and EW formulations was prepared by NSCC. This was discussed in great detail, including comments made by Roger Weckersdorf, Kwizda, Austria. It was concluded that the original flow sheet was too complex and inflexible for a multi-purpose pilot scale development plant. A simpler and more versatile flow sheet was drawn, using flexible pipes and gravity feed where possible. The de-ionised water system was cancelled and the plant was scaled for batches of about 500 to 1,000 litres. Refrigeration will be required for the Dyno KD25 bead mill.

A flow sheet drawing of the simplified liquids pilot plant is shown in Annex II. This is regarded as Phase 1 of the Pilot Plant and it is proposed that this plant should be installed as soon as possible after refurbishment of the building. The new liquids filling line should be installed on the ground floor underneath the product storage vessels.

#### 4. SUB-CONTRACT FOR WG FORMULATIONS

It was agreed at the TPR meeting in October 1993 that the initial development of WG formulations should be carried out under a sub-contract arrangement outside China. US\$ 72,000 were allocated from the total Budget under BL 21-01 to facilitate speedy implementation.

After detailed discussion at NSCC, it was agreed that fluid bed granulation was not favoured because of the physico-chemical properties of NSCC's products and also the very high capital cost of plant scale-up. It was decided to seek quotations in USA, Japan and UK/Europe for the development of WG formulations by extrusion granulation, focussing particularly on a glyphosate WG formulation. The sub-contract arrangement should also allow for some training on extrusion granulation technology for at least two NSCC staff members.

Simultaneous WG development can take place at Nantong when the laboratory paste kneader, extrusion granulator and fluid bed dryer have been delivered. (See Section 2.1).

When suitable quotations have been obtained, a Study Tour will take place to decide on the preferred option, probably in November 1994 (see Section 5.2). The Study Tour may also include a visit to Fuji-Paudal in Japan, who are supplying the laboratory equipment.

#### 5. UNDP INPUTS

##### 5.1 International Experts

The CTA will visit the Project Site again in October/November 1994 and attend the TPR meeting.

Mr. J. Hartmann, Packaging Consultant, visited the project site in April 1994. Dr. M. Gimeno, Formulation Research Consultant, visited the project site in May 1994.

It is proposed that a further six consultants should visit the site during 1994. These are all listed in the Summary of Progress on Work Plan - May 1994 in Annex III.

A suitable Analytical Consultant and Safety Consultant will need to be identified.

It was suggested that Mr. K. Johnson, the Pollution Control Consultant, should spend one week of his mission at the Catalyst Project Site in Tianjin (CPR/91/122). This should apply also to Dr. Maramba, the Industrial Hygiene Consultant.

## 5.2 Training and Study Tours

This is going according to plan (see Annex III).

The Fellowship Training of four staff members at Imperial College, London, has been completed. Two people will visit CIPAC and the 8th IUPAC, USA in June/July 1994.

Three people will make a Study Tour of Japan, USA or Europe to decide on the preferred option for WG Sub-contract work and training in November 1994. Following the agreement of the WG Sub-contract work site, at least two chemists should undertake Fellowship Training in extrusion granulation technology at the end of 1994 or early 1995.

It is proposed to nominate two staff members for Fellowship Training at the Pesticide Regional Formulation Technology Workshop to be held at the IPFT, Delhi, India during December 1994.

The IBM computer and software programmes are now fully operational at Nantong. However, staff members at Nantong would benefit greatly from a short visit to NSCC by an IBM local representative to advise on the use of applications and tools in the software programmes.

## 5.3 Equipment

Nine items of laboratory equipment and five items of pilot plant equipment will soon be delivered to Nantong. Quotation enquiries are being negotiated for further items (see Section 2). It is expected that most of the laboratory items and some of the pilot plant equipment will be purchased in 1994 (US\$ 400 - 500,000). The remaining part of the equipment budget (US\$ 200,000 approx) will be spent in 1995 mainly on pilot plant items.

A summary of all equipment items is given in Annex I.

## 5.4 WG Sub-contract

This is now almost 3 - 6 months behind schedule. A decision has been made to focus on extrusion granulation technology, particularly for the development of a glyphosate WG formulation. Quotations for sub-contract development work and training will be sought in USA, Japan and UK/Europe. A decision on the preferred contractor will be made after the Study Tour in November 1994.

## 6. NEW RESEARCH CENTRE BUILDING

The building construction work on the new Research Centre has progressed rapidly since the CTA's last visit in October/November 1993. Most of the exterior work is complete and interior decoration is now being carried out. A service lift has been provided to all floors. Fourteen air conditioner units have been purchased for some rooms in the new building.

It is expected that the building will be ready for occupation in July 1994 and staff and equipment will be transferred during August and September. The Project TPR and Regional meetings are planned for October/November 1994 in the new Research Centre, Nantong.

## 7. FORMULATION DEVELOPMENTS

### 7.1 Suspension Concentrates (SC)

Further experience of SC formulation technology has been gained by making a 40% w/w carbendazim SC formulation. Locally available dispersing agents give higher viscosity than imported dispersing agents.

A mixed SC formulation has been made with 20% w/w carbendazim and 20% iprodione. This has good stability after 2 weeks at 54 C and more than 2 years at room temperature. Samples have been sent to Yunnan Province for field trials on tobacco. Polymeric 'comb' surfactants will be tested with a 25% w/w iprodione SC formulation to try to prevent crystal growth.

A 30% w/w clofentazine SC formulation (Apollo) has good stability for 2 weeks at 54 C and 2 years at room temperature. The SC has about 30% higher biological activity than the WP formulation. Field trials and toxicity testing will be carried out. This formulation, or the corresponding SE formulation (see below) may be the first new formulation output of the project.

Mixed SC's of mancozeb with systemic fungicides are being investigated to overcome the problem of pathogen resistance.

### 7.2 Suspoemulsions (SE)

A mixed suspoemulsion of 10% w/w clofentazine and 10% w/w fenpropathin has good storage after 2 weeks at 54 C and 1 year at room temperature. Samples are being made for field trials this year and in 1995, which could lead to registration in 1996 followed by initial test marketing.

### 7.3 Oil/Water Emulsions (EW)

Laboratory development of EW formulations has continued with phoxim, malathion and permethrin. A concentrated emulsion (CE) formulation has been made containing 80% malathion. Glycerine or urea give better physical stability than xanthan gum. The CTA will try to obtain a sample of PVA from ISP Europe to test as an emulsifier/stabiliser.

### 7.4 Seed Treatment Formulations

The CTA gave a lecture on the formulation and application of seed treatment formulations. There is a good market for seed treatments in China, and considerable interest in moving from powder to flowable seed treatments. The new equipment for SC's will also be suitable for flowable seed treatments (FS) formulations.

### 7.5 Controlled Release Formulations

This topic will be discussed in detail during Dr Gimeno's visit to Nantong. Recommendations for formulation development work will be made.

## 8. PROJECT MANAGEMENT MEETING, CPR/91/120

The CTA attended the Project Management Meeting at CICETE, Beijing on 12 May 1994. Mr Hong (NPD) and Dr Dhua (PMTA) were also present, along with members of CICETE, UNDP, FAO and the 122 and 123 projects.

Linkage between all these projects, 121, 122 and 123, was stressed for safety, hygiene and environmental aspects.

The meeting was in general agreement that the Pesticide Formulation Project, CPR/91/121, was making good progress and keeping more or less to schedule and to budget. The hardware and software inputs were now well in hand for the first two years of the project and the prospects of meeting the objectives were very good.

The Summary of Progress on Work Plan - May 1994 (see Annex III) was accepted, and it was proposed that the total Budget should be rescheduled to cover 1993 - 1997 (5 years). Revision of the budget would be carried out before the next TPR meeting to be held in Nantong in October 1994.

9. MISCELLANEOUS COMMENTS

The NPD and CTA visited Shenyang Research Institute to tour their facilities. The CTA gave a lecture on "Recent Trends in Pesticide Formulation Technology". Some parts of the Institute and equipment are quite old, but they have an experienced group working on fluid bed technology. Some of this equipment could be modified easily to carry out developmental work on fluid bed granulation of pesticide WG formulations.

The CTA gave NSCC a list of recent books and reports on pesticide formulations, surfactants etc, which they may purchase using the Miscellaneous Budget.

The level of spoken English at NSCC has improved noticeably and this should be encouraged further, along with increasing facility with computers.

During the next visit to Nantong in October/November 1994, the CTA will arrange for Mr L. Rogiers of ICI Surfactants, Belgium, to give a one-day seminar at NSCC on "Surfactants for Agrochemical Formulations." Mr Hong will arrange for members of Shenyang Research Institute and other interested organisations to be present.

10. ACKNOWLEDGMENTS

The CTA would like to thank once again Mr Hong Chuanyi (NPD) for his constant help and support as well as excellent arrangements and hospitality throughout the visit to China. Also the staff at NSCC, Nantong for their continuing enthusiasm and commitment to the project.

The CTA would also like to thank Professor Li Zong Cheng for his invitation to Shenyang Research Institute and for his hospitality at Shenyang.

ANNEX I

LIST OF EQUIPMENT ITEMS

<u>EQUIPMENT ITEM</u>	<u>STATUS</u>	<u>APPROX COST USS</u>
-----------------------	---------------	----------------------------

OFFICE AND TRANSPORTATION

IBM PS/2 486 Computer	Installed at NSCC	4,050
HP Laser Jet Printer	" "	2,700
Canon Fax Machine	" "	2,050
Ricoh Photocopier	Delivered to NSCC	4,500
14 Air Conditioner Units	" "	29,000
Dodge Maxiwagon	" " damaged.	27,000
Copying Machine	Now under repair Contract signed	3,000
		<u>TOTAL 72,300</u>

LABORATORY EQUIPMENT

Malvern Laser Sizer	Installed at NSCC	53,000
Waters Millipore HPLC	" "	48,000
Lab. Ultra-Turrax	" "	4,800
Lab. Powder Grinder	" "	2,500
Dyno KDL Bead Mill	In transit to NSCC	17,300
Fritsch Rotor Mill	" "	7,500
Silverson Mixer L4R	Contract signed	1,600
Lab. Homogeniser 8.30H	" "	11,000
Analytical Balance	" "}	13,000
Formulation Balance	" "}	
Moisture Analyser	" "}	
Haake Rotovisko RV20	Order now placed	42,000
Fuji-Paudal Kneader	" "}	13,000
Fuji-Paudal Granulator	" "}	
		<u>TOTAL 213,700</u>

<u>EQUIPMENT ITEM</u>	<u>STATUS</u>	<u>APPROX COST</u> <u>US\$</u>
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PILOT PLANT EQUIPMENT

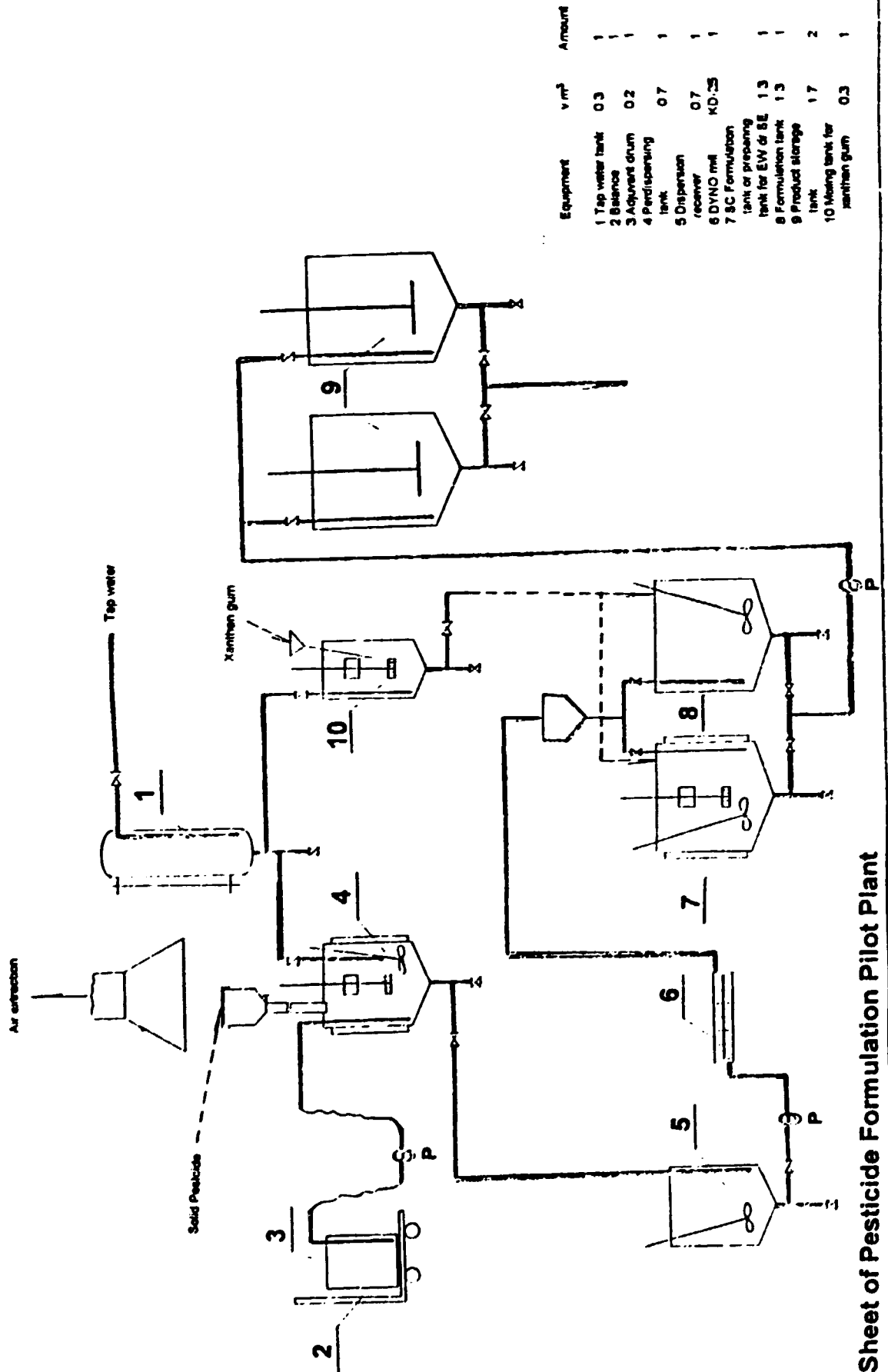
Dyno KD25 Bead Mill	In transit to NSCC	70,000
Silverson Mixer AXR	Contract Signed}	
Silverson Mixer GX10 (2 off)	" " }	24,000
Homogeniser 10.50H	" "	23,100
Electronic Balances (2 off)	" "	9,400
Micronette M500 Jet Mill	Under enquiry	37,000
Package Testing Equipment	" "	10,000
Liquid Filling Machines	" "	35,000
Extrusion Granulation Plant	Under research	100,000
Granules Filling Machines	" "	80,000

TOTAL 388.500

GRAND TOTAL FOR ALL EQUIPMENT 674.500



ANNEX II



Equipment	v m <sup>3</sup>	Amount
1 Tap water tank	0.3	1
2 Balance	0.2	1
3 Adjunct drum	0.7	1
4 Dispensing tank	0.7	1
5 Dispersion receiver	0.7	1
6 DYNO ml receiver	KD.25	1
7 SC Formulation tank or preparing tank for EW or BE	1.3	1
8 Formulation tank	1.3	1
9 Product storage tank	1.7	2
10 Mixing tank for xanthan gum	0.3	1

Flow Sheet of Pesticide Formulation Pilot Plant

PESTICIDE FORMULATION TECHNOLOGY      CPR/91/121

Summary of Progress on Work Plan    -    May 1994

1. Recruitment of Consultants.

BL11-01 . The CTA has visited China in April /May 1994 and will visit again in Oct./Nov. 1994 and attend TPR meeting .

BL11-02    Dr.M.Gimeno will visit China in May 1994 for 0.5 m/m. Another Formulation Research Consultant will be sought for a further visit later in 1994.

BL11-03    Mr.Zsifkovits, Kwizda, will visit China for 0.5 m/m in Summer 1994, and Dr.Damo, Hoechst, will visit China for 0.5 m/m in November 1994.

BL11-04    An Analyst will be required in September 1994 when all the new equipment has been transferred to the New Research Building.

BL11-05 . No suitable safety consultant has been identified yet.

BL11-06 . Mr. Johnson, UK, will visit China in October/ November 1994 to advise on pollution control and effluent treatment.

BL11-07 . Mr. Hartmann, Packaging Consultant, visited China in April 1994.

BL11-08 . It is proposed to invite Dr. Moramba, Phillipines, to advice on hygiene aspects near the end of 1994.

BL11-09 . An invitation to the IPM Consultant has been deferred because the Project Authorities are busy with other consultants.

2. Training and Study Tours

This is going according to plan.

Four people have been trained at Imperial Collage, London.

Two people will visit 8<sup>TH</sup> IUPAC,USA in July 1994.

Three people will visit organisations in Japan and USA or UK for subcontract of WG formulation in September 1994.

November

It is proposed to nominate two staff members for Fellowship Training at the Pesticide Formulation Technology Regional Workshop to be held at the IPFT, Delhi during December 1994.

Fellowship Training for at least two people will be necessary in Extrusion Granulation technology at the end of 1994 or early 1995.

### **3. Equipment Purchasing**

Some items of laboratory and pilot plant equipment are already in transit to Nantong. Other items have been ordered. A small number of items are still under price negotiation at CICETE. Enquires are being made for quotations for liquid filling equipment for the Pilot Plant.

### **4. Sub-contract for WG Formulations**

This is now about 3-6 months behind schedule. A decision has now been taken to focus on extrusion granulation technology and laboratory equipment will be purchased from Japan. Quotations will be obtained from USA and UK for sub-contract development of a glyphosate WG formulation and a decision will be made on the preferred contractor after the study tour in September 1994.

November

### **5. Outputs.**

The outputs for the PPER Document will be up-dated by July 1994.

All the Outputs for liquid formulations are on schedule. However, the Output for WG formulation is about 3-6 months behind schedule.

Controlled Release formulations will be discussed in detail during Dr. Gimeno's visit.

### **6. Budget for CPR/91/121**

This was originally set for 1992-1996 (5 years), However, funding was not available until 1993 and , therefore, it will be necessary to re-schedule the Budget to cover 1993-1997 (5 years).

UNIDO Comments

The report of the Chief Technical Adviser (CTA) gives a detailed account of the project activities carried out since the author's last visit. While most of the equipment has been either ordered or under consideration, the expert components will have to be phased-in according to requirements as agreed in the last tripartite review meeting. Decision to go for extrusion granulation for WDG will be taken up further by UNIDO. Recommendation to buy a semi automatic filling machine is important since trial runs and marketing of formulations under advanced development will have to be carried out for self sustainability of the research centre.

The lay out of the pilot plant should be modified to meet international standards and waste disposal facilities needed should be provided within the scope of the project.