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RESTRICTED

PESTICIDE DEVELOPMENT PROGRAMME INDIA

DP/IND/80/037

INDIA

<u>Technical report: Pilot scale development of pesticide formulations</u> (with particular reference to suspension concentrates) *

Prepared for the Government of India

by the United Nations Industrial Development Organization, acting as executing agency for the United Nations Development Programme

Based on the work of Gerald L. Baldit, consultant in chemical engineering

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United Nations Industrial Development Organization

Vienna

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approximately 13 Rupees to 1 US Dollar.

INTRODUCTION

The writer was recruited by UNIDO for three months to assist in the development of pesticide formulation capabilities in India. The duties were set out as follows in Job Description No. DP/IND/80/037/11-64:

"The consultant will be required to advise and assist the technical personnel of the Pesticide Development Programme India on utilisation of laboratory data on pesticide formulation processes and equipments for establishing commercially viable plants. He will be required to give guidelines on preparation of techno-economic feasibility reports, design, selection and layout of formulation process machinery etc. for fms. plants. He will also assess the training needs of local personnel and at the end of his mission is expected to submit a report on his work carried out, his findings and recommendations"

After briefing in Vienna, the writer proceeded to New Delhi, arriving on 23 October 1986. He left New Delhi on 17 January 1987 for debriefing in Vienna.

This report is meant as a guide to the type of work required in translating a laboratory pesticide formulation into a process which can be handed over as the basis of an industrial production unit. For this reason, some of the action taken has been described in detail.

ABSTRACT

Instruction was given to the PDPI staff in the formulation Pilot Plant section in the use and maintenance of a 15 litre Dyno Mill for the preparation of suspension concentrate formulations of pesticides, in particular of carboxin.

A multipurpose suspension concentrate plant was designed with an output of approximately 400 litre / day. As a guide to the type of data required before a process can be handed over to a manufacturing works, a Model Manufacturing Manual for a 40% Carboxin concentrate was written and left with PDPI.

Recommendations are made regarding the setting up of a truly independent development centre, the equipping and staffing of the formulation pilot plant section, training of existing staff, general safety and hygiene and a possible ways in which the pilot plant and its staff can be utilised in the future.

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CONCLUSIONS AND RECOMMENDATIONS

A. Conclusions

Although the formulation pilot plant contains much equipment which has been imported though UNIDO, the shortage of raw materials and general lack of back-up facilities and simple equipment which could be purchased locally restricted the amount of useful plant work which could be carried out.

Nevertheless, it is hoped that it is now realised by all concerned that a lot of development work has to be done before the more complicated formulations such as suspension concentrates, microemulsions etc. can be handed over for full scale production. This is costly in time and materials and one must, therefore, be very sure that there is an adequate market demand and that the financial return will be such as to justify the effort involved.

B. Recommendations

For the sake of clarity, detailed recommendations have been made at the end of each Chapter. The major recommendations are as follows:

1)

4)

The proposal that the PDPI Centre be split off administratively and financially from Hindustan Insecticides Limited and be controlled by a Governing Body drawn from Government and Industry should be implemented immediately (Chapter I)

- Greater emphasis must be placed on safety, cleanliness and pollution control. For this purpose a senior member of staff should be appointed and trained as Safety Officer (Chap¬ter II, C).
- 3) There should be some rearrangement of equipment in the pilot plant and certain items of equipment purchased from local suppliers (Chapter II, B and iV).
 - In particular, a model suspension concentrate plant should be erected, at a capital cost of approximately Rs. 98,000/- (excluding the already imported Dyno mill and pump) (Chapter II, D).

5)

The Pilot Plant staff require further practical on-works training (say 6 months) in an established overseas pesticide formulation plant - preferably in an English-speaking country. Messers S.N.Gupta and R. Gururajan would benefit from such training (Chapter II, E).

- 6) A mechanical/electrical engineer with practical experience in a chemical plant should be recruited (Chapter II,E)
- A well equipped maint_{enanc}workshop should be installed and staffed by a skilled fitter/electrician (Chapter II, B & E)
- Further Pilot Plant trials should be carried out with the carboxin
 40% SC formulation to determine the optimum criteria for operating
 the Dyno Mill (Chapter III).
- 9) Work should be initiated immediately to develop a process for decontaminating wash waters from the suspension concentrate plant (Chapter II, D).
- 10) Pilot Plant staff should be encouraged to visit formulation factories in India to gain the confidence of formulators and to offer assistance in training personnel and developing processes and equipment (Chapter V).
- 11) A member of the PDPI staff should be nominated Packages Adviser and be sent overseas for training, preferably in the UK (Chapter IV).

Recommendation Nos. 5 and 11 are for action with UNIDO. The other recommendations are for action by the management of the PDPI Centre.

THE PESTICIDE DEVELOPMENT PROGRAMME INDIA

- 1 -

The PDPI is a UNDP/UNIDO assisted project of the Government of India. The main function of the programme is to strengthen and improve pesticide formulation in India by developing and testing new types of formulations, exploiting indigenous raw materials and training personnel from the numerous formulating companies in formulation techniques (both on a laboratory and a pilot plant scale), quality control of raw materials and finished products, selection of packages and general safety procedures both in plant and for prevention of environmental pollution. In addition to working on its own initiative, the PDPI Centre will accept sponsorship for the development of specific formulations from any commercial company. Although much of the work will be directed towards the use of pesticides in Agriculture, there will be requirements for formulations for Public Health and household use.

The PDPI is located in Research and Development Centre of Hindustan Insecticides Limited (a wholly owned Government company) at Udyog Vihar, some 25 km from Delhi). It is regarded as an HIL project administered by HIL's General Manager (Projects) and is dependent on HIL for local resources. The staff have, for the most part, been recruited from within HIL and, although not directly on HIL's payroll, they still regard themselves as belonging to HIL.

In the writer's opinion, these close ties with HIL present serious disadvantages and must cast doubt on the objectivity of PDPI. HIL is only one (admittedly the largest) of many Indian manufacturers, formulators and sellers of pesticides and it is only to be expected that other companies will be reluctant to entrust their new developments to a Centre administered by their main competitor.

This problem has not gone unnoticed by the Government and UNIDO and the writer strongly supports the proposal to make PDPI completely independent financially and administratively from HIL and recommend that this be implemented as soon as possible. The present proposal of Associate Membership of the Centre by companies and individuals is a good one and could form the basis of complete independence. Industry's representation on the Governing Board should reflect its financial contribution towards operating costs. The present system of budgetory

I.

control and provision of finance appears to be ill-defined. It is suggested that section leaders should be encouraged to draw up their own annual budgets for incorporation into the Centre's main budget. Once the budget has been accepted by the Board, section leaders should be made responsible for their portion and given authority to spend up to their budget. Expenditure should be monitored monthly.

Another general area that requires some action by management is the encouragement of the awareness by all staff on how their work interacts with other disciplines in the Centre. Personnel must never lose sight of the fact that the ultimate aim of all pesticide formulation work is to provide the end user(farmer, public health authority etc.) with products which can be used safely and economically in order to improve the agriculture and environment of the country.

The needs of the formulation laboratory have already been discussed by Woodford (December 1986). The requirements of a formulation pilot plant are given in more detail in Chapter II of this report.

II. The Formulation Pilot Plant

A. The Function of a Pesticide Formulation Pilot Plant

In order to avoid any confusion in the minds of the staff of PDPI, the writer consider it necessary to state clearly the function, as generally accepted in all developed countries, of a formulation pilot plant. It has four main functions:

1. To develop a laboratory formulation into a process which can be operated on a works scale.

2. To produce sufficient quantities of new formulations for field scale development trials and demonstrations.

3. To train people in the safe handling of chemicals and machinery

4. To pin point effluent treatment problems. A pilot plant should NOT be used for producing material for commercial sale.

The Building and Equipment

в.

The formulation pilot plant room is adjacent to the synthetic chemistry pilot plant room (part of HIL's Research Department). A major nuisance is caused by birds which enter through numerous holes in walls, despite wire netting having been put up over vents. It is hoped that this problem will be solved as soon as the trees on the estate are large enough to form natural nesting sites.

- 3 -

The open drain system inside the building is considered dangerous. It is an open invitation for people to discard unwanted rubbish into it.

Most of the basic machinery items in the pilot plant have already been listed by Mosinski (August 1985, Table 9). Since that data delivery has been taken of a Dyno Mill Type KD15 and a Silverson Stirrer Model DX. In addition there is an old Avery platform weighing machine (100 kg x 100g) with no tare device and an old bench balance (10 kg x 50g). Neither is suitable for use in a pilot plant where accurate weighing is essential.

Some of the items of equipment appear to have little relevance to the proposals for formulation work outlined in the 'Revised Workplan' (January 1986) and there has been too great an emphasis placed on powder formulation. The equipment has been set up around the walls of a very large room in such a way as to make access for services and operators difficult. One serious fault is the installation of two noisy air compressors inside the room.

As has already been commented on by Mosinski (August 1985) inadequate provision has been made for dust extraction when handling and grinding powders.

The store room attached to the plant room has no shelving for housing spare parts, samples etc. There is no workshop foruse in maintaining machinery and making small rapid changes to the layout of plant, pipework etc.

It is recommended that :

1)

The two air compressors be housed outside the building.

- An efficient dust extractor and air scrubber be installed outside the building with a main trunk duct going round the inside of the building with suitable branches off to all powder charging and discharging points.
- 3. A small well-equipped maintenance workshop should be provided. This should be able to assist in machine maintenance, carry out electrical installations, prepare pipework and generally assist in the operation of the pilot plant.

4. New weighing machines should be purchased viz.

2)

Avery Semi-Self Indicating Counter Scale type 1215 BFH : 2 kg x 2 g(with weights)

Avery Industrial Bench Platform Scale 3303CLS with tare:10 kg x 20g Avery Dial Scale 3205 CLE with tare : 100 kg x 200g (Approximate prices are given in Appendix 3).

- 5. There must be an adequate supply of small (25 1 downwards) drums, plastic vessels, scoops, adhesive and tie on lables, and means of handling, storage and labelling samples made on the plant.
- 6. The store room should be equipped with shelving. All samples should be stored neatly and properly labelled.
- 7. A model suspension concentrate plant be erected (see Chapter III). Although designed for suspension concentrates, it must be sufficiently flexible to allow, for instance, the Dynomill to be substituted by the Colloid mill for the preparation of emulsion flowable formulations.
- 8. A foot operated filling machine (50 ml to 500 ml capacity) similar to that offered by Rank & Co., should be purchased for filling out suspension concentrates and other fluids.

- 9. Consideration should be given to constructing one large cubicle with tiled walls and glass front in which powders can be formulated. It should be possible to arrange for the necessary equipment to be wheeled in, secured to the floor if necessary, and connected up with all necessary services (water, electricity, compressed air, dust extraction etc.)
- 10. The purchase of any other major items of formulation machinery should only take place after the advice of a Consultant, very familiar with the use of such equipment, has been obtained.
- 11. The open drains should have removable covers and be kept clean.

C. Safety and Hygiene

4.

In a unit handling toxic chemicals, the importance of strict safety measures and general hygiene and cleanliness cannot be over-emphasised. Mr. V.N. Dutta is preparing guide lines for safety in formulating plants. These guidelines must also be adhered to strictly in the Formulation Pilot Plant.

It is, therefore, recommended that :

- 1. A senior member of staff be designated 'Safety Officer' and be made responsible for all aspects of safety, not only in the pilot plant but in the whole Centre.
- 2. The entire pilot plant building (formulation and synthetic chemistry) be designated a Hazardous Area in which smoking is prohibited. Processes requiring Naked lights (e.g. welding) should be carried out only after sanction by the Safety Officer.
- 3. After taking advice from the fire fighting Authorities, adequate fire fighting equipment be purchased to deal with chemical and electrical fires.
 - An emergency shower, eye washing facilities and first aid kit be installed and clearly labelled.

- 5. The ablutions area must be kept clean and provided with ample water, soap and paper towels.
- 6. Signs pointing to shower, eye bath and emergency exits should be put up.
- 7. An industrial vacuum cleaner (e.g. Sturtevant model with special filters for toxic dusts and flameproof 2 hp motor) be purchased.
- 8. All personnel be instructed to clean up spillages as they are made and to keep all equipment, stores etc. thoroughly clean and tidy.
- 9. In order to help in keeping the working areas clean and free from toxic materials, the concrete floor should be coated with a suitable polyurethane (or similar) paint.
- 10. All operators should be issued with full covering overalls, rubber gloves and protective boots.
- 11. A good supply of dust mask and safety spectacles should be made available.

D. Suspension Concentrate Plant

As a result of experience gained in using the Dyno Mill (See Chapter III) a 400 l/day (1 shift) suspension concentrate plant has been designed (Drawing 1). Production is based on 100 litre batches and the product can be both oil and water based. It is estimated that the installed cost of the locally produced items (vessels, pumps, stirrers, filling machine, drains, water cooling unit and pipework) but not including a dust collector and effluent collection tank will be Rs. 98,000/-. The Dyno Mill and feed pump imported from Switzerland early 1986 cost S. Fr. 55,000/- delivered.

If similar equipment were made available in the PDPI formulation pilot plant, this could be used for training purposes and for producing development quantities of new formulations. Although designed as a suspension concentrate plant, the vessels are, of course, multipurpose and can be used for formulating other liquid products. It is recommended that -

 Locally manufactured stainless steel vessels (2 x 100 litre jacketed, 1 x 60 l and 1 x 25 l) together with all necessary portable stirrers, pumps and pipe fittings be purchased in order to assemble a model suspension concntrate pilot plant which could be transformed into a general purpose liquids formulation plant.

The vessels should be mounted on wheels to permit ease of movement.

- A water chilling unit capable of supplying a minimum of 1500 l/hr. demineralised water at 10°C and 2 bar pressure should be purchased (It is understood that this is already in hand).
- 3. The model liquids pilot plant area should be surrounded by a shallow drain covered with removable gratings to facilitate washing down and prevent contamination of the rest of the building.
- 4. The drain should connect to a large external settling tank into which all wash waters can be collected for decontamination.
- 5.

A programme should be initiated by the Formulation Section to work out a system for flocculating the carboxin containing wash waters and render the effluent fit for discharge into the normal sewerage system.

6. The electrical control panel supplied with the Dyno Mill should be fitted with a wattmeter and an ammeter in order to measure (a) the actual current consumption for ascertaining the optimum criteria for operating the system and b) to check on abnormal surges in current.

7. More glass beads of varying size ranges shall be purchased. These should preferably come from Indian glassworks.

E. Staffing

The following senior staff were allocated to the writer for work on the pilot plant:

Mr. V.N. Dutta Mr. S. Kumar Mr. S.N. Gupta Mr. R. Gururajan

(All the above have been trained as chemists) There were also two plant operators.

In addition the services of an electrician/general handyman and a sweeper were made available as required.

It is understood that Dr. Kumar is, in future, to be responsible for Conferences and Training. It will be noted that the senior staff are all chemists. They have all had plant experience in an HIL factory. Dutta and Kumar have both had some overseas training. The section would be considerably strengthened with the appointment of a good mechanical engineer with sound practical experience, preferably on a pesticide plant and some knowledge of electrical engineering. Gupta and Gururajan would both benefit by a good practical training session in an established overseas pesticide formulation factory, preferably in an English speaking country.

It is therefore, recommended that:

1.

A mechanical engineer, with practical experience of plant work, be engaged.

- 2. Messers Gupta and Gururajan be sent for training for spells of, say, 6 months each, to a pesticide formulation factory, preferably in an English speaking country.
- 3. A skilled fitter/electrican be appointed to do maintenance and light construction work (although attached to the pilot plant section, this person would be available to other sections of the PDPI as required.

III. DEVELOPMENT OF SUSPENSION CONCENTRATE PROCESSES

An important commission received by PDPI was from HIL for the development of a carboxin suspension concentrate for use as a seed treatment. When the writer arrived at the Centre, the bench formulation work was well advanced, a laboratory Dyno Mill having been used for size reduction.

It was, therefore, agreed that this product should form the basis of the initial work in the pilot plant.

Before any practical work could be done, plastic vessels and containers, flexible piping and various pipe fittings had to be purchased in the market, and a temporary "lash up" made.

The minimum meaningful batch which can be processed in the 15 litre Dyno Mill is about 50 litres (and this only takes about 20 minutes to mill). In order to test all possible parameters to identify optimum working criteria (e.g. bead charge, mill speed, feed rate, milling temperature) some 10 to 15 batches would have to be made and much of this material would have had to be discarded. Unfortunately HIL could only make available sufficient carboxin for two batches. Since it appeared unlikely that more material could be obtained from the USA in time it was decided to carry out some practice runs using a china clay suspension. This gave the Pilot Plant staff valuable experience in handling powders and liquids and demonstrated the problems associated with stripping, cleaning and reassembling the equipment and disposing of large quantities of wash waters containing finely divided solids.

This emphasises the need to define the parameters for flocculating suspension concentrates and decontaminating wash water. This work should be carried on parallel to the actual development of formulations.

The formulation process as received from the laboratory involved three stages - preparing and milling a pre-mix, mixing in a gel solution and finally mixing in a thickener containing dye. The formula contains a small quantity of ethylene glycol which, in the laboratory process, was divided between the three stages making measurement, on a plant scale

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somewhat difficult. Furthermore, since carboxin is soluble in ethylene glycol (4.5% in ethylene glycol and 700 ppm in the glycol/water mixture of the carboxin formulation at 50° C) it was considered undesirable to have glycol present at the milling stage when temperature of 40° C could easily be attained in the mill. Elimination of the glycol from the pre-mix meant that the concentration of solids would go upto about 50% and the formulation chemists expressed doubts as to whether this could be successfully milled. It was therefore decided to mill a mixture of 50% DDT WP and water which proved quite successful provided that the temperature of the cooling water for the mill was kept low (Ca 10° C).

Two batches of the carboxin 40% suspension concentrate were made and this showed up a few problems viz :

- The need for a rapid (less than 10 min) test for particle size during milling (The purchase of a Malvern Particle size Analyse has been recommended by Woodford).
- 2. The need for a very efficient stirrer for mixing the gel and thickener solutions into the product.
- 3. The need for the milled product to pass over a sieve to catch any glass beads which might get broken during the milling process.

Nevertheless, sufficient experience has been gained to enable a multipurpose suspension concentrate plant to be designed (see drawing No. 1).

A model manufacturing manual for a carboxin 40% suspension concentrate has also been drawn up and left with the Centre. It must be emphasised that this manual (parts of which are not complete) is only a model to show the types of data which must be made available before a new formulation can be successfully handed over to the works for commercial manufacture. This manual covers details of formula, equipment required with flow diagram, preparation of equipment, manufacturing process and cleaning of equipments. The section on decontamination of wash waters has had to be left blank as no process has yet been developed. The manual also indicates tentative internal specifications for the finished product and specifications for raw materials for quality control purposes. The provision of raw material and finished product specification is normally the responsibility of the analytical section working in close collaboration with the formulation section. Raw material specifications must be agreed with the suppliers. (N.B. An internal specification should be tighter than the one given to outside parties such as registration authorities etc.).

It is recommended that :

Further pilot plant trials with the carboxin formulations should be carried out to determine the following criteria:

i) The optimum concentration of carboxin in the pre-mix.

ii) The optimum bead size (only beads of diameter 0.9 to 1.5 mm diameter were delivered with the Dyno Mill)

iii) The optimum bead charge in the Dyno Mill

iv) The optimum peripheral speed of the agitator blades (there are three possibilities).

v) The maximum permissible grinding temperature (this will determine the requirement of cooling water for the mill and pre-mix vessels).

Note: A series of experimental runs using a sulphur suspension has been designed in order to obtain some data on the effect of making the changes listed above. It must be stressed that the results will not automatically be translated to carboxin or any other products.)

IV. PACKAGING

A. General

The packaging of pesticides for safer storage, transport and ease of use is a very important aspect of formulation development. Woodford has already drawn attention to the need for storage tests to be carried out alongside formulation development. The current workplan of PDPI includes 4 man-months of consultancy on packaging. This might be considered rather long. In the writer's view this kind of money could better be spent by sending a member of PDPI staff to say PIRA (the British Research Association for the Paper and Board, Printing and Allied Industries) for a month followed by 1 month in a pesticide formulating company which carries out its own design and testing of packages and labels.

B. Packaging material for Suspension Concentrates

As already recommended by Woodford, long term storage tests in packages made of different materials must be carried out. Although most suspension concentrate are aqueous based, there is no reason why an oil based product should not be developed one day.

From the writer's experience, polyethylene terephthalate (PET) bottles, now available in India, would be suitable for both types of suspension concentrate formulations.

C. Filling Machine

The pressure operated filling machine already purchased was found to be unsuitable for filling suspension concentrate as it tended to entrain air during operation. A foot operated locally made volumetric measuring machine borrowed from HIL's Delhi factory, worked very well with the carboxin 40% SC. On a 250 ml fill, the variation was + 0.7% and - 0.5%.

It is recommended that

- 1) a foot operated volumetric filling machine (50 ml to $\frac{1}{2}$ l) similar to that offered by Rank & Co. be purchased(See Appendix 3)
- 2. a member of PDPI staff be sent to, say, England for some training on package development and testing.

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V. PILOT PLANT TRAINING AND DEVELOPMENT WORK

A. Training

As has already been stated, one important use to which a pilot plant can be put is for training people in handling chemicals and machinery. Training courses, based on group of not more than five students, should be organised to demonstrate all aspects of pesticide formulation powders and liquids. Students should be potential plant managers and come prepared to carry out all the manual operations involved, including stripping down, cleaning and assembly of machines under the supervision of senior pilot plant staff.

The course should pay great emphasis on

- safety and hygiene
- clean working
- keeping adequate records of work done

B. Development Work

The pilot plant can also usefully be used for providing data for inclusion in Product Manufacturing Manuals and for improving established processes. It is essential that this work be carried out in close collaboration with the formulation section and, where appropriate, personnel engaged on commercial manufacture. This could well necessitate pilot plant staff visiting formulators' works to give advice on improving procedures.

It is <u>recommended</u> that pilot plant personnel be encouraged to visit formulation factories in India to offer assistance in training personnel and problem solving.

APPENDIX I

ACKNOWLEDGEMENTS

The writer would like to thank all those people in UNDP and at the PDPI Centre at Udyog Vihar for their generous assistance during this assignment. In addition to those mentioned in Appendix II and Chapter II.E the writer would also thank Mr. R.R. Pillai for his patience in typing this report from a poorly written manuscript.

The writer is indebted to Mr. A.R. Woodford for his helpful suggestions regarding the Dyno Mill.

APPENDIX 2

Major Contacts

UNDP

Dr. M. Kamal Hussein, UNIDO, Senior Industrial Development Field Adviser Mr. Sat Pal, Assistant to SIDFA

Hindustan Insecticides Ltd

Dr. S.P. Dhua, Chairman & Managing Director* Mr. Munni Lal, General Manager Projects

PDPI

Dr. Sushil K. Khetan, Head Mr. V.N. Dutta, Senior counterpart Mr. S. Kumar, Senior counterpart Dr.P.K. Ramdas, Group Leader, Formulation Section

*Dr. Dhua is also Chairman of the PDPI Governing Body.

- 16 -APPENDIX 3

Prices of Typical Equipment of Indian Manufacturer for Pilot Plant Use

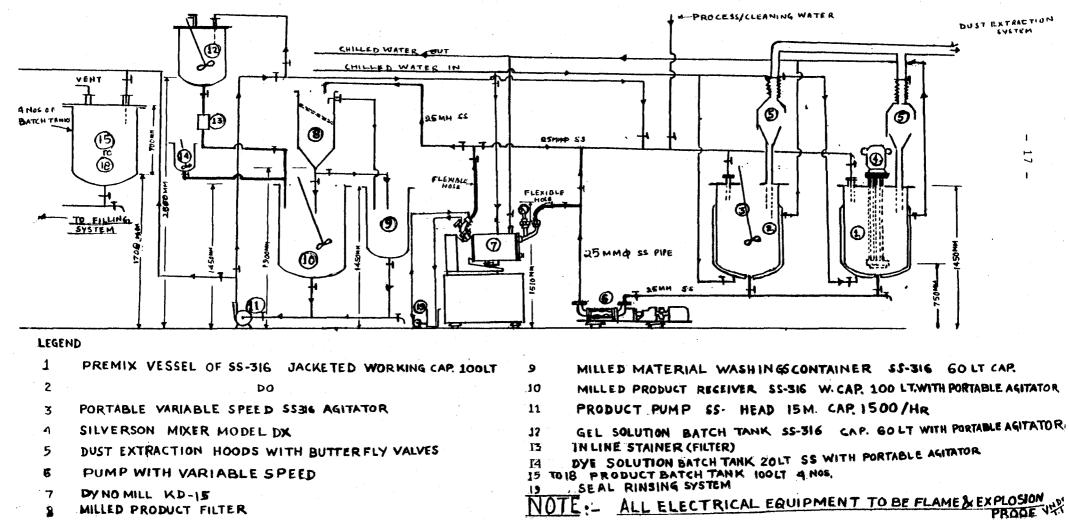
Avery India Ltd., Delhi

Semi-self-indicating Counter Scale type 1215 BFH, 2 kg x 2 g with set of weights	Rs. 5,700.00		
Industrial Bench Platform Scale 3303 CLS, $10 \text{ kg x } 20 \text{ kg}$ with tare device	Rs. 21,970.00		
Dial Scale 3205 CLE, 100 kg x 200 g with tare device	Rs. 29,570.00		
The above prices are delivered Gurgaon, excluding 23%taxes			
Rank & Co., Delhi			
25 1 S.S. vessel with bottom valve	Rs. 1,450.00		
50 l -do-	Rs. 1,850.00		
1001 -do-	Rs. 2,750.00		
100 l -do- and MS cooling jacket	3,250.00		
Foot operated S.S.filling machine for creams etc.	Rs.4,000.00		
Hand operated sealing machine for aluminium containers	Rs. 8,500.00		
Stirrer with 2 hp motor and reduction gear	Rs. 7,500.00		
High Speed stirrer with 2 hp motor and reduction gear	Rs. 9,850.00		
Extra for flame and explosion proof motors	Rs. 3,000.00		

The above prices are ex-works, all taxes included.

DRAWING NO. 1

FLOW SHEET OF MULTIPURPOSE SUSPENSION CONCENTRATE PLANT



MILLED PRODUCT FILTER

