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FEASIBILITY STUDY ON PESTICIDE MANUFACTURING IN IRAN

DP/IRA/88/002

ISLAMIC REPUBLIC OF IRAN

Technical report: Pesticide manufacture in Iran
Findings and recommendations*

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

Based on the work of R. Sales Barquets,
chemical technology expert

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United Nations Industrial Development Organization
Vienna

* This document has not been edited.

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C O N T E N T S

ANNEXES

I.- INTRODUCTION

The technical report of June 15th 1989 corresponding to the expert last visit was revised during the first days, stressing on the matters to be included in the working programme. Referring to the pilot plant, it was accorded after discussing about the different possibilities of design and execution that:

- The multipurpose pesticides and intermediates pilot plant of the characteristics recommended by the expert in the above mentioned report should be the best for covering the proposed objectives.
- To group all the equipment not locally available in one selected supplier and a single shipment, appears to be the most suitable.

A. Progress done from the expert last visit.

In general the development of the agrochemical sector in the Islamic Republic of Iran is following the guidelines stated from the beginning as recorded in the former technical report, and in accordance with the quinquennial plan recently elaborated by the Government, it will be increased.

In the synthesis laboratory of NIOC, the preparation of monochloroacetic acid (MCA) has been faced and an apparently good product has been obtained in the first experiments.

The management of the agrochemical R & D programme is interested in the following technologies:

- Atrazine
- Benomyl
- Glyphosate
- Pyrethroids, in a more advanced stage.

B. Working programme

It was elaborated according the recommendations of the last report with the following additions:

- Visit at the glass workshop in NIOC Research Center, and
- Seminar on the title: How to proceed R & D from lab to the pilot plant, at the amphitheater of NIOC Research Center.

The seminar was prepared by the expert and edited by the technical service of publications of NIOC Research Center.

II.- GLASS WORKSHOP AT NIOC RESEARCH CENTER

This workshop is endowed with the suitable facilities and skilled personnel for manufacturing the common laboratory glassware and vessels up to 20 l. about, including small spetial apparatus upon demand so as to repair the broken glass equipment from the lab.
In the opinion of the expert after examination of some samples, the work performed here is excelent and they are needing of designs and models for producing more sophisticated lab equipment.

III.- ASSESSMENT ON FORMULATING PESTICIDES

The technical grade pesticides must be formulated for their further application, unless a very rare exceptions.
The type of formulation to be selected depends of the physical and chemical properties of the active ingredient and the purpose of its application. So when both the conditions are known, we can have a first idea about the work to be done.
The principal types of formulation used in the agrochemical sector are:

- DP = dustable powder
- DS = powder for seed treatment
- EC = emulsifiable concentrate
- FLOW = flowable = suspension concentrate
- GR = granules
- LS = solution for seed treatment
- SG = water soluble granules
- SL = soluble concentrate
- SP = water soluble powder
- WP = wettable powder

There are specialized books and manuals on the matter, but perhaps the most suitable source of information is the technical literature supplied by the manufacturers of emulsifiers and related products. The most important companies producing emulsifiers and related products have a good technical service, in order to promote sales and at this purpose, sometimes they resolve the problem of any special formulation; after that, training and practical experience is the way to get a good knowledge of the activity.
For the moment, NIOC is interested in formulating the following products:

1 - copper oxychloride WP 59% and 84%

2 - sulphur WP 90%
3 - carboxin EC 20% and WP 50%
4 - carbendazime WP 75%
5 - tetradifon EC 8% and WP 18%
6 - benomyl WP 50%
7 - metaldehyde GR (slug baits) 5%
8 - dalapon-sodium SP 85%
9 - chlorthal-dimethyl WP 50% and 15% and GR 5%
10 - bendiocarb WP 76% and 80%
11 - methiocarb WP 75% and 50% and GR 4%
12 - benzene hexachloride WP 95% and 25% and EC 7% and 1%
13 - phosalone WP 25% and EC 3%
14 - diuron WP 80%
15 - TCA SL 47%

When formulating is faced in the lab the specifications of each formulation must be given and it is suposed that samples of the original formulations so as the required technical products are availables. Furthermore, the principal requirements for preparing the types of formulation involved in the above mentioned list are summarized as follows:

Emulsifiable concentrate (EC)

The laboratory must be endowed with the common glassware and equipment, specially 100 ml volumetric flasks, 10 ml measuring cylinders, dropper pipettes with teat, and all the necessary facilities for controling the final specifications.

Mostly of the formulations can be elaborated with two or three emulsifiers, selected from four or five basic compounds. When selecting solvents, solubility, physical and chemical properties, price and phytotoxicity are taken in acount.

A good emulsion depends of its hydrophilic/lipophilic balance (HLB) and the recommended method for doing it is that based in the preparation of titrated solutions, in 100 ml volumetric flasks e.g., with the technical product, selected solvent and emulsifier one by one, and then to balance by mixing at different proportions in 10 ml measuring cylinders. A blank solution prepared with technical product plus solvent, is used finally for optimizing the minimum allowable quantity of emulsifiers.

A practical example of formulation was developed in the laboratory by following this method.

Granules (GR)

In general the carrier is a mineral granule and the formulation is done by spraying it with the technical product mixed with aditives and solvent if necessary, into a blender.
Organic carriers are also used: in the case of methaldehyde slug bait, the carrier is bran and after blending, the mixture is extruded.

Soluble concentrate (SL)

The liquid and solid water soluble pesticides can be formulated as SL. Wetting agents are necessary and they must be water solubles and compatibles with the ionic character of the active ingredient.
Common laboratory equipment is needed so as the facilities for testing their final specifications.

Soluble powder (SP)

The water soluble pesticides in solid state, can be formulated as SP by adding soluble inert ingredients e.g. sodium sulfate, sucrose or any soluble powder not phytotoxic and easily available, and wetting agents; anticaking and flowing agents are sometimes needed.
The basic laboratory equipment is a variable speed mixer, acting at hight speed as a mill and all the necessary facilities for controling the final specifications.

Wettable powder (WP)

Solid and liquid pesticides can be formulated as WP. In the case of those in liquid state, a previous absorption in a suitable carrier or support is required.
Kaolin, kieselgur and other hydrophilic mineral powders so as sucrose are the common inerts; dispersing and wetting compounds are normally used for preparing the formulation and sometimes anticaking, flowing and stabilizing agents are used.
The basic laboratory equipment includes a mixer, a mill and a micronizer when necessary. In fact, in the case of micronized formulations if all the ingredients have been previously micronized by the suppliers, a mixer or blender is enough for formulating purposes.
The facilities for controlling the final specifications are required and in the case that micronized formulations are faced, a microscope with a calibrated micrometric ocular is convenient.

* When milling certain powders, safety measures must be undertaken in order to avoid deflagrations.

IV.- PRACTICAL WORK IN R & D LABORATORY

The chosen product for this work was monochloroacetic acid (MCA) and the technical report about its synthesis in the lab, elaborated by the Project Leader of Pesticides was firstly evaluated.

The process followed is a classic one described in the free literature, starting from acetic acid, chlorine and acetic anhydride as catalyst. The work performed was scientifically good, but its scope was not as required for scaling up the pilot plant stage.

Three new experiments were carried out under the supervision of the expert following the philosophy stablished in the Seminar (see annex 3) and the guidelines for the next trials were given. In these runnings the principal objectives are the purification step and to recover the catalyst.

In the first experiments according to the literature, after chlorination, MCA was distilled off; as the obtained product was not pure enough, it must be purified by crystallization.

Obviously to crystallize the product after chlorination, without distilling the MCA and then to recover the catalyst from the solvent, should be a simple and economical way and this is the goal. The results obtained following this procedure are promising, but definitive figures canot be given because unfortunately the analytical method for controlling this step of the process is not available.

When the analytical methods are ready, the chlorination step must be also optimized. Now the only way to study the changes affecting a step of the process. is to finalize it and wait for the final analytical result and that takes a lot of time.

To carry out a chlorination step in total darkness is essential in order to evaluate if the light affects the reaction.

The installation and equipment of the existing laboratory require to be adapted to the R & D task; by this reason some troubles have been recorded along the execution of the present experiments and the general recommendations have been given by the expert.

V.- BASIS OF DESIGN OF THE MULTIPURPOSE PESTICIDES AND INTERMEDIATES PILOT PLANT

The pilot plant will be housed into the existing building of 30 x 12 x 8 (high) m. The utilities production units will be located outside the building. The supporting metalic structure of modular design will be erected at three levels for permiting to carry out processes in cascade (by gravity).

The pilot plant will be provided with the utilities generally existing in an industrial plant and its design will permit to perform the following chemical

and physical operations:

- Reactions with addition of gases, liquids and solids at temperatures from -20 up to 200°C about and with different types of agitation and at variable speed.
- Distillation, Dean Stark separation and refluxing, at atmospheric pressure and under vacuum.
- Fractionating distillation in packed column of equivalent height of 15/20 plates.
- Separation of heterogeneous liquids by gravity.
- Filtration (clarification).
- Centrifugal separation of solids and liquids.
- Fluidized bed drying.
- Vacuum drying.

The design of the pilot plant will be endowed with a big agility for interlocking the existing equipment in order to simulate the flow diagram of the selected industrial process.

The major equipment list includes:

2 glass lined jacketed reactors of 100 l full glass equipped (condenser, separator, cooler, collector)

2 full equipped glass reactors 50 and 100 l over bath

1 full equipped glass fractionating column equivalent 20 plates with a 20 l reactor as boiler and automatic reflux ratio control

2 SS jacketed reactors of 100 l full equipped (condenser, cooler, collector)

1 SS centrifuge solid liquid separator

1 acid resistent centrifuge solid liquid separator

1 SS jacketed filter

1 SS bed fluid dryer

1 oven vacuum dryer

1 compact icemaker

3 diaphragm pumps

1 polivalent scrubber

2 tanks for collecting and neutralizing liquid effluents

3 balances 6-60-500 kg

1 elevator

Full safety devices

Spare parts

VI.- FINAL RECOMMENDATIONS

R & D laboratory. The installation of the existing laboratory must be adapted to the new task and endowed with the necessary equipment and chemicals for developing the required processes. A list including the principal lab equipment indicating prices will be sent by the expert.
Consequently the number of persons in charge of the task must be enlarged in accordance with the working programme; in general one qualified chemist requires two technicians.
When all the above exposed has been achieved, a training at home supervised by the expert should be essential.

Agrochemical formulation laboratory. When the equipment and materials recorded in chapter III have been provided, a training at home developing real examples of formulation should be convenient.

A visit to a foreign pilot plant and R & D centre of similar characteristics of those mentioned in the present report, should be very illustrative to the responsibles for the project while it is under study. At this purpose the facilities of E.I. Aragonesas in Huelva (Spain) and the I.Q.S. pilot plant and research centre can be visited by previous arrangement with the expert.

Annex No 1 (1 page)

LIST OF PERSONS MET

United Nations Development Programme, Teheran office:

Mr. Moghaddam, Programme Officer

National Petrochemical Company:

Mr. Nafissy, Agrochemical Manager Progect Evaluation

Mr. Hamzavi, Detail Engineer

National Iranian Oil Company Research Center:

Dr. Akbarnejad, Deputy Director of Technology Development Division

Dr. Hedayati, Manager Gas & Petrochemical Research Department

Mr. Jawad, Head of Process Eng. Department

Mr. Vafi, Head of Pesticides & Fertilizers Department

Dr. Soleimani, Project Leader of Pesticides

Mr. Afzali, Chemical Engineer Pilot Plant

Dr. Hashemolhosseine, Laboratory of Agrochemical Formulation

Mr. Sayyas, Laboratory of Agrochemical Formulation

Ms. Fatemy, Laboratory of Agrochemical Formulation

CHRONOLOGICAL ACTIVITY OF THE MISSION

18-10-89	Arrival in Vienna
19-10-89	Briefing at UNIDO Vienna office and arranging visa
20-10-89	Briefing at UNIDO Vienna office and collecting visa
21-10-89	Departure to Teheran
22-10-89	Arrival in Teheran. Briefing at UNDP Teheran office and visiting NPC office
23-10-89 to 25-10-89	Working meetings at NPC office
26-10-89 and 27-10-89	Holiday
28-10-89 to 01-11-89	Working meetings at NIOC facilities
02-11-89 and 03-11-89	Visiting UNDP office and Holiday
04-11-89 to 08-11-89	Working meetings at NIOC facilities
09-11-89 and 10-11-89	Visiting UNDP office and Holiday
11-11-89 to 13-11-89	Working meetings at NIOC facilities
14-11-89	Seminar at NIOC amphitheatre
15-11-89	Last working meeting at NIOC facilities
16-11-89 and 17-11-89	Debriefing at UNDP Teheran office and Holiday
18-11-89	Departure to Vienna and Barcelona