OCCASION

This publication has been made available to the public on the occasion of the 50th anniversary of the United Nations Industrial Development Organisation.

DISCLAIMER

FAIR USE POLICY

CONTACT

Please contact publications@unido.org for further information concerning UNIDO publications.

For more information about UNIDO, please visit us at www.unido.org

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION
Vienna International Centre, P.O. Box 300, 1400 Vienna, Austria
Tel: (+43-1) 26026-0 · www.unido.org · unido@unido.org

17038-E

RESTRICTED

DP/ID/SER.A/1060
26 September 1988
ORIGINAL: ENGLISH

ESTABLISHMENT OF A MULTIPURPOSE
PESTICIDE PILOT PLANT

DP/EGY/81/006

ARAB REPUBLIC OF EGYPT

Technical report: Visit to Ismadye, Alexandria, Egypt

Prepared for the Government of the Arab Republic of Egypt
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, consultant
in pesticide production technology

Backstopping officer: B. Sugavanam, Chemical Industries Branch

United Nations Industrial Development Organization
Vienna

* This document has not been edited.

V.88-28962

CONTENTS

1. PLAN OF WORK

Meetings were held in the offices to study the problems with those in charge of each work area, after which the actual operations in the industrial plant and in the laboratory were observed, with special emphasis on the phases or points giving rise to the most conflict.

Information meetings were held periodically with the Chairman.

The following took part in the working meetings: Mr. Said M. Attyia, Mr. Fathy, Mr. Hany and engineers in charge of the mechanical and instruments workshop and Mrs. Eda, who is in charge of the research laboratory for methyl chloroacetate. Mr. Lotfi Khattab and Mr. Aly Taufik also took part in the information meetings.

2. STATUS OF THE PLANT AT THE BEGINNING OF THE VISIT

Dimethoate was being produced because demand for this product is strong at the present time. Moreover, during earlier attempts to produce malathion, the required quality and quantity standards had not been achieved.

Although the quality of the dimethoate produced met the specifications, the quantity fell short of the required capacity.

Study of the production sheets indicated that the quantity obtained fluctuated widely from batch to batch; dosing of intermediates was erratic; and the time required for crystallization was abnormally long. These things were happening for different reasons.

3. DIMETHOATE

3.1. Mechanical and instrumentation problems

The stirrer of the RI-103 was obviously inadequate. In order to improve its efficiency, I recommended that a second two-blade propeller should be installed on the same axis at one third the operating level. In taking the preparatory steps for calculating the estimated increase in power, I realized that the stirrer was turning the wrong way. When the direction of rotation was changed so that the stirrer turned the right way, stirring efficiency increased and crystallization time was shortened. I still recommend installation of the new stirrer for cases where a larger volume is being stirred owing to addition of more ice (because of a failure in cooling) or to recycling of product because of a failure in centrifuging or some other defect.

The MMA feed tube was also lengthened to the bottom in order to prevent losses through vaporization, especially with the new stirrer.

The pH meter probe caused frequent problems because no good maintenance service was available in Alexandria. In order to avoid the risk of an inaccurate reading, and the consequent error in neutralization and loss of yield, I recommended that samples should be taken manually and the pH determined externally using laboratory equipment, near the working area. In order to facilitate this work, I designed a simple device for the taking of samples by suction and gravity

emptying, which can be adjusted to a mouth of the reactor, in the place of the pH meter probe. Obviously, if the problem of maintenance of the probe is some day solved in a dependable way, using it would be more convenient.

3.2. Technological review

A detailed review was made of the process manual, with special emphasis on the points giving rise to most conflict. It was demonstrated that when the instructions in the process manual are carefully followed, production is good, of course provided that the equipment, instruments, power supply and raw materials are in line with project specifications.

As a supplement to the process manual, I provided some guidelines to correct minor deviations arising from poor practices and thereby to minimize possible losses.

3.3. Control of production

In this matter, there are notable defects that should be corrected, as indicated in point 10. I am referring to the plant's control laboratory, since the central analysis laboratory was not included in my visit.

I studied the analytical results for the acid and sodium salt in respect of production for the month of March, and the values do not appear to be accurate, since the weights of sodium salt obtained give values exceeding 100 per cent theoretical, and this is impossible; therefore either the analysis is inaccurate or the weights are incorrect.

I was assured that the final analyses of dimethoate are correct, having been checked externally. Therefore, in practice, I recommend carefully neutralizing the acid with the required amount of sal soda, using exactly the amount of MECA and MMA indicated in the manual, proportionally distributed for each batch of sodium salt obtained, and depending on the analytical result of the end product, adding small amounts of MECA and MMA alternately until the optimum result is achieved.

In the mean time, they should perfect their analytical techniques and check the results obtained.

3.4. Results obtained

The following data give a clear picture of the progress achieved:

BXS 13/14 May, of acid, corresponding to 3 BXS dimethoate: 1,620 kg.

BXS 15/16 May, of acid, corresponding to 3 BXS dimethoate: 1,660 kg.

* BXS 1/2 June, of acid, corresponding to 3 BXS dimethoate: 1,538 kg.

BXS 3/4 June, of acid, corresponding to 3 BXS dimethoate: 1,750 kg.

BXS 5/6 June, of acid, corresponding to 3 BXS dimethoate: 1,750 kg.

** BXS 7/8 June, of acid, corresponding to 3 BXS dimethoate: 1,700 kg.

---

* There was a loss owing to overflowing during centrifuging.

** Estimated minimum value, since at my departure a batch remained in the drier.

3.5. Increase in production

Ismadye is very interested in increasing its annual production of dimethoate and in fact is doing so by operating during the hot season of the year. I informed them of the precautions that they should take under their responsibility, especially during emptying of the centrifuges and filling of the drier, bearing in mind that the risk is less during the coolest hours of the day.

They also told me that they are studying a project for expanding the plant in order to increase daily production. In my opinion, this would be feasible when the production level and the training of personnel have been improved.

4. MALATHION

4.1. Mechanical and services problems

The DR-102 separating centrifuge does not meet the required flow specifications. This can be solved for the time being by separating the phases by gravity. The purpose of the separating centrifuge is to achieve efficient separation of phases speedily, leaving the organic phase with a minimum amount of water and thus minimizing the deterioration of the malathion.

After inspecting the RI-105 reactor, I recommended pointing the steam bubbling tube towards the stir vortex in order to improve the efficiency and homogenization of fluids.

In reviewing the worksheets for previous malathion production tests, I observed some anomalies that indicated defects in steam and vacuum during stripping. For this reason I arranged a dry run of the RI-105, and the outcome was that under thermodynamic conditions appropriate for condensation of the steam injected, the amount of water condensing was very small, and the vacuum pump frequently stopped. I left specific instructions for repeating the dry run tests and ascertaining whether the steam and vacuum met project specifications, since it would otherwise be impossible to achieve production standards.

4.2. Technological revi-

A detailed revision was carried out of the process manual, with special guidelines on gravity washing and phase separation and details on operational techniques in the distillation and steam stripping phase, which requires great skill on the part of the operator, but which, if adequate services are available, can give good results in three or four trials. Thus, it is very important to carry out dry run trials, reproducing process conditions, along the lines described in the previous section, before starting production.

It should be borne in mind that although in the case of dimethoate, operational faults on the whole cause a reduction in product, but not in quality, in the case of malathion, poor operation results not only in reduction in product but also in poorer quality of the end product obtained.

4.3. Production control

As long as accurate results cannot be obtained in determination of the acid produced in the first phase, it is recommended that exactly the amount of DEM indicated in the draft should be used and, if necessary, the proportions should be balanced taking into account the final analysis of the product obtained. However, this is impossible for the time being because the GL chromatograph needed for the final determination is out of order and cannot be used.

4.4. Conclusion

At the present time, it is not advisable to initiate production of malathion owing mainly to the defects observed in steam and vacuum and to the lack of a chromatograph.

Once these problems have been solved, production can be started and should take place normally, especially since the experience obtained with the production of dimethoate will be very useful because, on the basis of the results obtained, we can now be sure that the acid produced is of very good quality, something which is essential for malathion production.

5. METHYL CHLOROACETATE

5.1. Checking of the process in the laboratory

On my arrival, I was informed that, in the research laboratory, a number of synthesis trials had been carried out to prove the feasibility of the draft from Aragonesas. The yields were low, around 85 per cent, as compared with the figure of 95 per cent given in the draft, and this caused them to have doubts about its validity.

However, the reason for their low yields is simple: the process described in the draft is based on a classical Fischer esterification, but since the boiling point of methanol is lower than that of the ester/water azeotrope, part of the methanol always distills before reacting when standard laboratory equipment is used.

This disadvantage is overcome in our draft by feeding the reaction premixture through the bottom of a pre-heated RE-202 reactor.

In view of Ismadye's interest in verifying this, I provided them with precise instructions for reproducing these conditions in the laboratory.

In subsequent trials, the yields increased to 93 per cent.

5.2. Project feasibility study

The technicians at Ismadye have prepared a feasibility study of MECA at different yield levels, which states that what is set forth in the draft appears to be economic for them, where yields exceed 83 per cent.

There are other technologies for the production of MECA based on the use of super-heated methanol, but although they produce higher yields, the installations are expensive and can only be amortized by means of high production levels.

6. FUSED SODIUM SULPHIDE

This is the form required for the Egyptian market, and it appears that producing it from the aqueous solution will not pose a major problem. Satisfactory results have been obtained in the laboratory, but in industrial-scale trials, an unacceptable black product was obtained. In my opinion, this is due to contamination with iron. I provided technical information and recommended further laboratory trials under conditions that could be industrially reproduced.

7. RAW MATERIALS

The quality specifications required by the draft must be complied with. Special attention should be paid to P2S5, since a change in its granulometry could affect the feed flow, in which case the speed of the feed screw should be altered. The fumarate content in the DEM should also be monitored. In this respect, I shall be sending them more detailed information.

The volume of purchases and storage time are problems of logistics that they should solve themselves, in line with the delivery times of suppliers and their own manufacturing plans. Special attention should be paid to the storage of sensitive products such as P2S5, MMA, and in general all the raw materials should be stored under the conditions recommended by the supplier.

8. PLANT SAFETY AND MAINTENANCE

These matters appear to be somewhat neglected, and practices should be improved, following the guidelines given by the draft and my specific recommendations in this connection. It should be borne in mind that product spillage not only affects the workers' health, but also encourages corrosion and deterioration of installations and structures.

9. OTHER PRODUCTS

This plant is extremely versatile, and other products could be manufactured in it almost without additional investment. I recommended that Ismadye should carry out a market survey on insecticides in Egypt, and from among the resulting products, those should be selected which can best be adapted to the existing plant, beginning by the technologically simplest or most accessible ones. I offered my assistance for this work.

10. GENERAL RECOMMENDATIONS

In addition to the recommendations made in the foregoing sections of this report, which were outlined in detail to senior staff of Ismadye, the following deserve special mention:

- Vocational training courses should be organized for production personnel, covering subjects such as productivity, handling of chemical products, work safety and first aid in case of accident;

- The plant's control laboratory should be equipped with glassware and accessories, which are virtually absent at the present time;

- The technical production office should be provided with a few reference books such as the Handbook of Chemistry and Physics, the Merck Index and the Toxic and Hazardous Industrial Chemicals Safety Manual;

- Preventive maintenance of the plant should be provided, including periodic painting of metal structures and other parts exposed to corrosion;

- The production summary or statement should be communicated periodically to UNIDO or directly to me.