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ESTABLISHMENT OF A MULTIPURPOSE PESTICIDE PILOT PLANT

DP/EGY/81/006

ARAB REPUBLIC OF EGYPT

Technical report: Findings and recommendations*

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

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United Nations Industrial Development Organization
Vienna

* This document has not been edited.

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29-07-89 Arrival in Cairo and traveling to Alexandria.

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to

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02-08-89 Meeting with Mr. Khattab. Working meetings and supervising process in plant.

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04-08-89 Holliday.

05-08-89 Holliday.

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to

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09-08-89 Meeting with Mr. Tawfik. Discussions with Mr Sayed and Mr. Fawzy about the new project METHYL CHLOROACETATE and enlargement of the pesticide plant for producing simultaneously DIMETHOATE and MALATHION. Studing results with Mr. Said of the last batches and giving recommendations for batch 4/8.

and

10-08-89

11-08-89 Working meetings. Giving directions to Mr. Said for the elaboration of the following batches.

12-08-89 Holliday.

13-08-89 General revision of the work done up to date with Mr. Said, Mr. Fathy and Mr. Hany and final recommendations. Debriefing meeting with Dr. Khattab.

14-08-89 Departure to Cairo and Barcelona.

II.- INTRODUCTION

In November 1987 during the start up of the MALATHION production 3 batches were elaborated; the obtained quantity and quality of the final product was in progress but the standards of the project were not reached. As steam and vacuum utilities were not according with the project requirements, the trial were cancelled.

Later, ISMADYE elaborated 4 batches but analytical results are not available because at that time the G. L. chromatograph was out of service. A new batch (ref. 1/6) was elaborated in June 1989 but during the process the reactor RI-105 was overflowed and about 400 liters of crude MALATHION solution were lost.

In all the cases, separation of phases is by gravity because the centrifugal decanter DR-102 is out of specifications.

From the beginning there was a certain disorder in the process due to the following reasons:

- Failures in steam and vacuum utilities and in some details of the installation.
- Troubles in the analytical control and abnormal results, increased the difficult in adjusting the process.
- Technological changes introduced at the last moment in washings to enable separation of phases by gravity affected certainly the process.
- The quantity of toluene (435.2 kg/500 l) stated in the project is not enough for reaching the level of the thermometer well in RE-101 and by this reason, 300 l more of toluene have been added and this may affect also the process.
- Workers are not trained enough for this process; some bad operation has been recorded.

III.- WORKING PROGRAMME

The working programme was elaborated stressing on the following points:

A) Checking installation

During the first runnings in the start up stage, some working conditions may be adjusted under the supervision of the expert, then the installation must be adapted according to the project and further adjustements when necessary.

B) Providing an analytical support.

Analytical control of the final product is essential for evaluating the results; analytical control of the intermediate steps is a valuable working tool for leading the process on the right way.

C) Reviewing the project and topics in production start up.

The responsible of the plant, foreman and workers must be trained for following properly the process manual including the recommendations of the expert; this refers of course to the 3 shifts.

D) Stablishing working conditions for the next batches.

After study of the former batches and existing circunstances, some parameters of the process were balanced one by one as a general practice.

IV.- FINDINGS AND RECOMMENDATIONS

In general the maintenance of the plant is insufficient not affecting directly the yield, but increasing greatly the possibilities of operating problems.

Furthermore the addition of small deflections along the process gives a more important deflection in the final result.

RE-101

In the case that the extra addition of toluene injures the

condensation step, it is recommended either to reduce the quantity of toluene to the figure of the project and to install a bottom valve provided with temperature sensor, or to produce 2 batches of ADMTF as in the DIMETHOATE process, for 3 batches of MALATHION; in this case measuring carefully the ADMTF solution is essential because the proportions of ADMTF and DEM is a critical point in the condensation step.

DR-102

As this equipment is out of service, it is recommended to remove it from the plant in order to avoid further corrosion; its use for other purposes is to keep in mind. Annex No. 1 contains technical information about centrifugal decanters suitable for the MALATHION process.

TK-115

It is essential to install a glass cylinder for controlling the separation of phases at the discharge of the washing tank; in the now existing conditions is quite difficult to work properly.

RI-105

It is necessary to install a filter on the line feeding steam and nitrogen and a spetial valve for working under vacuum conditions, at the entrance of this line to the reactor. The existing valve is adecuate for steam but is unsuitable for vacuum conditions. Impurities and iron coming from the general steam pipe and from the nitrogen cylinders may injure the MALATHION and damage the vacuum valve.

V.- GENERAL REVISION OF THE PROCESS

A) ADMTF production.

It is assumed that ADMTF production is according with the standard as proved by results achieved in DIMETHOATE production. Despite some abnormal analytical results giving an equivalent amount higher than 100%, the practical yield is ca. 93% that means

530 kg of ADMTF as 100% obtained from 400 kg of phosphorus pentasulfide.

B) Condensation step.

The quantity of diethyl maleate (DEM) stated in the original project is more than necessary; to 530 kg of ADMTF corresponds 520 kg DEM not 590.5 kg as in the project. This point will be corrected according the results. The excess of DEM don't affects the final yield but decreases the purity of the final product.

In the condensation reaction, the effect of the excess of toluene coming from the ADMTF formation, is something to investigate in the pending trials.

In the opinion of the expert the conditions of the condensation as stated in the project are according with the good practice, but as it is normal in the start up stage, little adjustements in temperature, time and catalyst are in consideration.

C) Washing step.

Two aspects must be taken in account in this operation, the chemical aspect and the physical one. Generally, crude solutions of MALATHION are washed with aqueous sodium carbonate; washing with caustic soda solution in the conditions described in the project gives a good result. The control of free acidity after condensation and pH control during washing in both organical and aqueous solutions, demostrates that destruction of MALATHION is negligible.

MALATHION is easily emulsified with water and separation of phases becomes very difficult; in the project to resolve this problem, a big quantity of toluene in the organical phase and sodium chloride in the aqueous one are added, but in despite of this, laboratory trials prove that more than 5% of pure MALATHION remains in the aqueous layer and in the interphase decreasing the final yield. The excess of toluene is also inconvenient because prolongs the time of distillation.

In fact, the producers of MALATHION utilize centrifugal decanters for this purpose in order to avoid the problems above mentioned.

D) Final purification step.

This step involves elimination of toluene by distillation including the residual water coming from washings is also eliminated; steam stripping for taking off the trimethyl ester and final stripping to take off the residual water. To reach the moisture specifications according to the project requires the maximum available vacuum as stated in the project and recommendations of the expert; if necessary an excursion of about 1 hour at 100ºC is permitted.

The philosophy along that step is, time as short as possible, minimum temperature and maximum vacuum.

VI.- COMPARATIVE STUDY OF BATCHES 1/7, 1/8, 2/8, 3/8 and 4/8

Bx 1/7

Working conditions:

- ADMTF: As the project plus 300 l toluene
- Condensation: As the project
- Distillation: As the project

* Results:

- Quantity	956.0 kg
- Purity %	76.5 (Central laboratory)
	80.3 (External laboratory)
- Moisture	0.8 (Central laboratory)
	0.3 (Plant laboratory)
- Acidity	0.07
- Iron	6.1 ppm
- Insol.	0.18

* Remarks:

After the condensation step the contents in MALATHION was determined, but the column and detector were contaminated and consequently the GL chromatograph was out of service during 3 days, seeking the collaboration of an external laboratory.

Bx 1/8

Working conditions:

- ADMTF: As the project plus 300 l toluene
- Condensation: As the project
- Washings: As the project
- Distillation: As the project

* Results:

- Quantity 750.0 kg
- Purity 86.04 (Central laboratory)
  84.83 (External laboratory)
- Moisture 0.5 (Central laboratory)
  0.16 (Plant laboratory)
- Acidity 0.09
- Iron 6.8 ppm
- Insol. 0.8

* Remarks:

During washings about 400 l of organical solution of crude MALATHION were lost. This batch cannot be taken in consideration.

Bx 2/8

Working conditions:

- ADMTF: As the project plus 300 l toluene
- Condensation: As the project less 50 kg DEM
- Washings: As the project
- Distillation: As the project

* Results:

- Quantity 900.0 kg
- Purity % 87.1 (Central laboratory)
  85.81 (External laboratory)

- Moisture 0.4 (Central laboratory)
  0.29 (Plant laboratory)
- Acidity 0.07
- Iron nil
- Insol. 0.04

* Remarks:

This batch elaborated in similar conditions as 1/7 but 50 kg less of DEM yields the same quantity of MALATHION as 100% (perhaps a few kg more) but purity is increased despite differences in analytical results.

Bx 3/8

Working conditions:

- ADMTF: As the project plus 300 l toluene
- Condensation: As the project, less 50 kg DEM, plus 5 l TEM
- Washings: As the project

Distillation: As the project

* Results:

- Quantity 850.0 kg
- Purity % 72 (Central laboratory)
  n.a. (External laboratory)
- Moisture 0.8 (Central laboratory)
- Acidity n.a.
- Iron n.a.
- Insol. n.a.

* Remarks:

After condensation, polymerization has been observed as a thick layer, discarted before washings. Addition of 5 l catalyst triethyl amine gave a bad result in the present working conditions.

Bx 4/8

Working conditions:

ADMTF: As the project plus 300 l toluene

Condensation: As the project, less 50 kg DEM, plus 5 l TEM and

29 hours at 65°C

- Washings: As the project
- Distillation: As the project

* Results:

- Quantity	960.0 Kg	
- Purity %	66.2	(Central laboratory)
	n.a.	(External laboratory)
- Moisture	0.6	(Central laboratory)
- Acidity	n.a.	
- Iron	n.a.	
- Insol.	n.a.	

* Remarks:

In condensation step no polymerization has been observed; other steps are normal then, quantity and purity are abnormal according the available analytical result.

Generally, analytical results are erratic and some human errors in operating the plant have been recorded; in such a condition it is difficult to undertake conclusions, the only reliable conclusion is that the batch 2/8 is the best one.

VII.- DIRECTIONS FOR REACHING SPECIFICATIONS ACCORDING TO THE PROJECT

These trials are intended to obtain the best yield in the process, separating by gravity, maximum yield will be achieved only by utilizing centrifugal decanter.

The order of executing the trials depends of the analytical results and the operating conditions existing in the plant. It is essential don't start a trial without knowing the analytical results of the last one. During the adjusting stage one of the most important figure to take in consideration is the total amount of obtained MALATHION as 100%.

Recommendations for the batches following the 1/5

A)

- ADMTF: As habitual
- CONDENSATION:

  540.5 kg DEM; 10 kg hydroquinone; 25 h at 65ºC

- WASHINGS/DISTILLATION: As habitual

B)

- ADMTF: As habitual
- CONDENSATION:

  540.5 kg DEM; 10 kg hydroquinone; 30 h at 65ºC

- WASHINGS/DISTILLATION: As habitual

* Depending results and observations, batches A) and B) may be repeated with addition of 2 to 5 l of TEA catalyst.

C)

- ADMTF: As habitual
- CONDENSATION:

  520.5 kg DEM; other conditions: As the best batch

- WASHINGS/DISTILLATION: As habitual

D)

- ADMTF: As habitual
- CONDENSATION: As the best batch
- WASHING: With sodium carbonate
- DISTILLATION: As habitual

E)

- ADMTF: Reduce toluene quantity by producing 2 bx of acid with 600 kg of phosphorus pentasulfide each, for 3 bx of MALATHION. In this case, be very careful when measuring the ADMTF solution.
- CONDENSATION: As the best batch
- WASHING: As the best batch
- DISTILLATION: As habitual

F)

- ADMTF: Reduce toluene as in E)

- CONDENSATION: Exactly as the project process manual, but reducing the quantity of DEM to 520.5 kg
- WASHINGS/DISTILLATION: As the project and recommendations of the expert.

VIII.- FINAL RECOMMENDATIONS

It is essential to improve the maintenance of the plant and to correct the existing mechanical faults, in order to decrease the possibility of operating problems.

To train the personnel in operating the plant and reporting properly the data and remarks along the process.

The analytical methods must be checked in order to conciliate the results and minimize delays. A chemical analytical method for MALATHION determination, should be a standard for, e.g. J.Am. Anal. chem. 55, No.5, 1972 or any useful alternative method for controlling the production. The aim should be to reach FAO's specifications (annex 2).

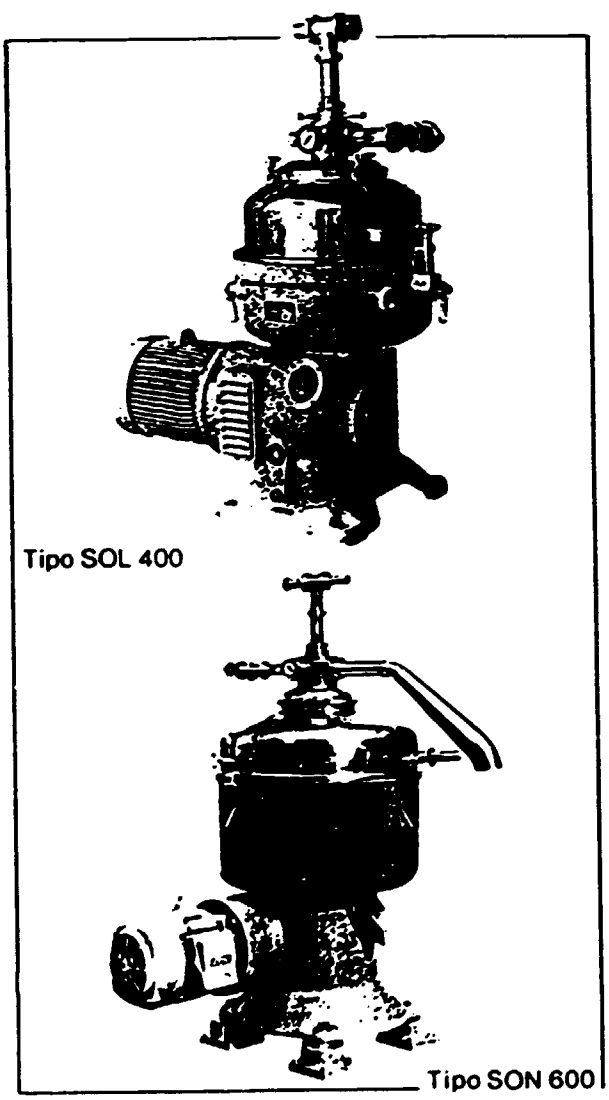
The Production General Manager of ISMADYE is kindly requested to comunicate to the UNIDO expert the results of the trials.

SEPARATORI CENTRIFUGHI A PULIZIA MANUALE TIPI SON-SOL

NEUTRALIZZAZIONE - LAVAGGIO - DEMUCILLAGINAZIONE DI OLII E GRASSI - SCREMATURA

CARATTERISTICHE TECNICHE

TIPO	SON SOL 400	SON SOL 600
Potenza Motore (HP)	10	20
Volume totale Tamburo (lt.)	20	50
Volume camera sedimentazione (lt.)	9	23
Velocità tamburo (lt.)	6000	4550
Peso netto (Kg.)	525	1335
Peso lordo (Kg.)	615	1530



Tipo SOL 400

Tipo SON 600

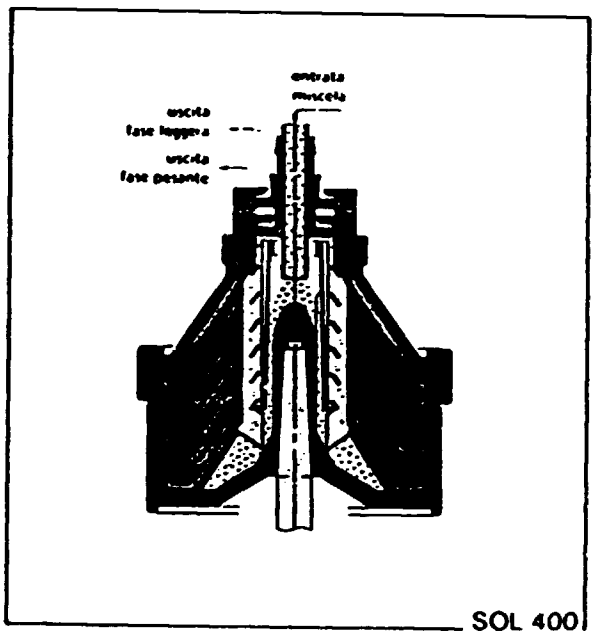
Materiali usati nella costruzione

- Baty in fusione di ghisa verniciata con resine epossidiche.
- Raccoglitori completamente in acciaio inossidabile AISI 304.
- Tamburo e tutte le parti a contatto con il prodotto in acciaio inossidabile AISI 304.

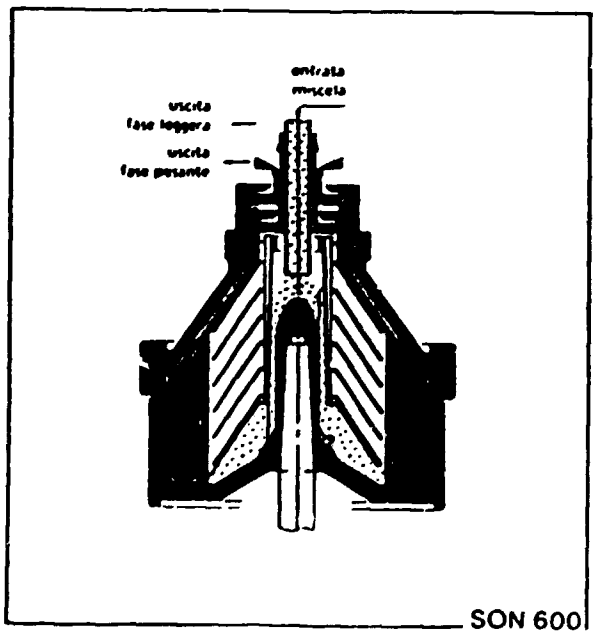
A richiesta del cliente si possono eseguire tutte le parti a contatto del prodotto in acciai speciali come AISI 316 - AISI 316L - INCOLOY 825 etc.

PRODOTTI E PORTATE (lt/h)

TIPO	SON 400	SON 600	SOL 400	SOL 600
Neutralizzazione	2500-3000	5500 6500		
Lavaggio	5000 6000	9000 11000		
Demucilaginazione	2000-2500	4500-5500	-	-
Scrematura (bassa percentuale di liquido leggero)	-	-	14000-16000	25000-30000



SOL 400



SON 600

PRESENTAZIONE

La Veronesi Separatori è lieta di presentare la propria serie di centrifughe utilizzate nei processi continui di neutralizzazione degli olii vegetali.
La versione normalmente utilizzata SON, permette di lavorare sia nella fase di neutralizzazione, per la separazione delle paste saponose, che in fase di lavaggio dell'olio stesso.
La notevole flessibilità della macchina, permette di lavorare sia nei processi continui che discontinui.
Inoltre per la loro semplicità costruttiva e robustezza consentono una estrema facilità di manutenzione non richiedendo personale altamente qualificato.
Queste macchine frutto di anni di studio e di esperienza su un notevole numero di impianti in Italia e all'Estero hanno permesso di raggiungere notevolissimi livelli di affidabilità ed una elevatissima efficienza nella separazione.

ESECUZIONE

La separazione avviene all'interno del tamburo che è del tipo a ritenzione di solido e fissato all'estremità conica dell'albero verticale.
La miscela da separare viene alimentata dall'alto attraverso il distributore.
Dal distributore il prodotto passa attraverso uno speciale interposto ad alette oppure si lamina attraverso una serie di dischi (in funzione del tipo di centrifuga) ove avviene la separazione delle 3 fasi.
La fase solida si deposita all'interno del tamburo mentre le due fasi liquide si scaricano a mezzo di turbine centripete che permettono la regolazione continua della separazione.
Le nostre centrifughe inoltre sono dotate di un sistema di ermetismo che evita il contatto del prodotto con l'aria e la possibile formazione di schiuma.

MALATHION TECHNICAL
FAO Specification 12/1/S/8

•1 DESCRIPTION

The material shall consist of malathion, together with related manufacturing impurities, and shall be a clear, colourless to light amber liquid, free from extraneous materials or added modifying agents.

•2 ACTIVE INGREDIENT

•2•1 *Malathion* [CIPAC *1B*; 12/1/M2/1•2]

The malathion content (g/kg) shall be declared (Minimum declared: 940 g/kg) and when determined, the content obtained shall not differ from that declared by more than ± 20 g/kg.

•3 IMPURITIES

•3•1 *Acetone insolubles* [CIPAC *1*; 12/1/M/1•7]
Maximum: 5 g/kg.

•3•2 *Acidity* [-/M/1•6]
Maximum: 5 g/kg calculated as H_2SO_4.

•3•3 *Iron* [-/m/1•4]
Maximum: 10 mg/kg.

•3•4 *Water* [CIPAC *1*; -/M/1•8]
Maximum: 1 g/kg.

•4 CONTAINERS

Containers should not be made of iron or steel unless specially protected to prevent contamination of the material with iron.

Note 1 For special purposes, where odour is important, e.g, products for use on flowers, a deodorized product should be specified.