## 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

This document has been produced without formal United Nations editing. The designations employed and the presentation of the material in this document do not imply the expression of any opinion whatsoever on the part of the Secretariat of the United Nations Industrial Development Organization (UNIDO) concerning the legal status of any country, territory, city or area or of its authorities, or concerning the delimitation of its frontiers or boundaries, or its economic system or degree of development. Designations such as "developed", "industrialized" and "developing" are intended for statistical convenience and do not necessarily express a judgment about the stage reached by a particular country or area in the development process. Mention of firm names or commercial products does not constitute an endorsement by UNIDO.

## FAIR USE POLICY

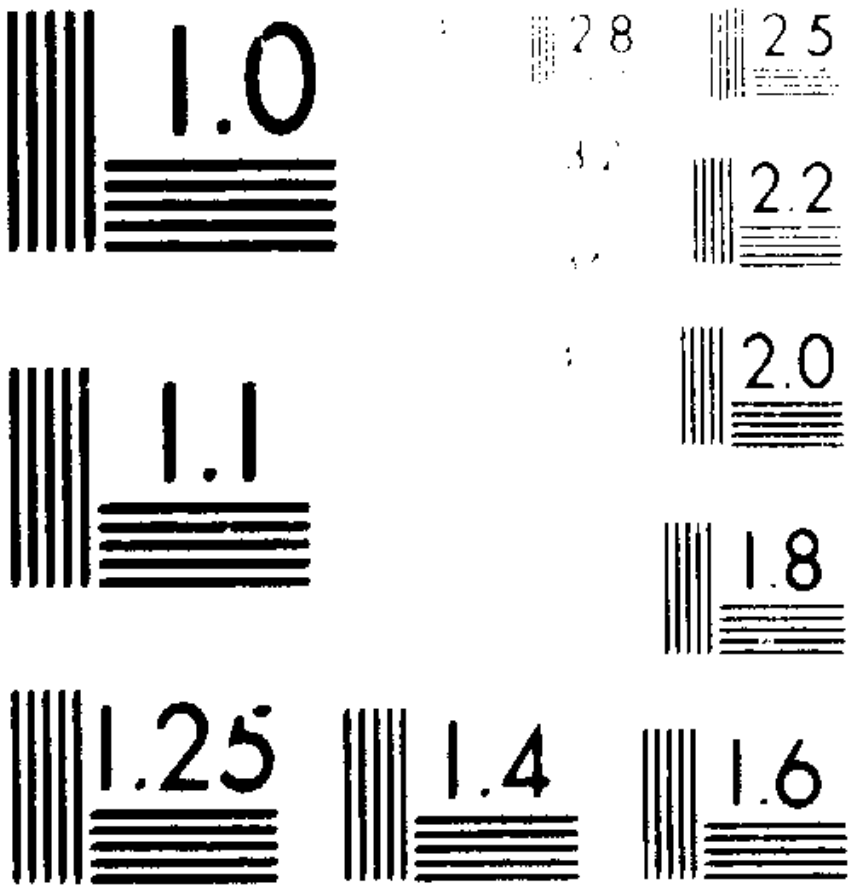
Any part of this publication may be quoted and referenced for educational and research purposes without additional permission from UNIDO. However, those who make use of quoting and referencing this publication are requested to follow the Fair Use Policy of giving due credit to UNIDO.

## CONTACT

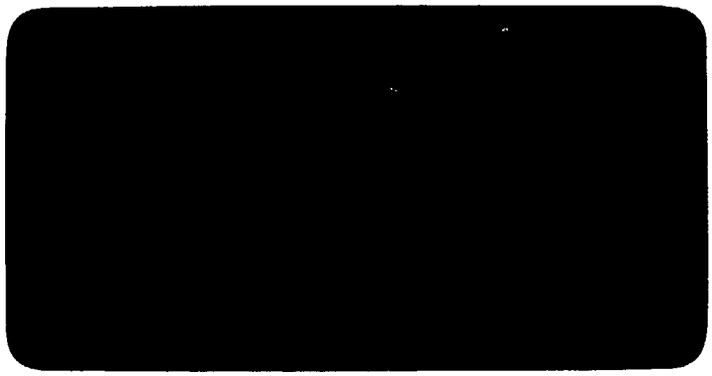
Please contact publications@unido.org for further information concerning UNIDO publications.

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


1.0
2.8
2.5
2.2
1.1
2.0
1.8
1.25
1.4
1.6

11382



Chimie Développement International

Project 60 03

11382

# Egypt STUDY OF A MULTIPURPOSE INSECTICIDE UNIT.

for

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

DP/EGY/81/006

November 1981

TABLE OF CONTENT

# 1 - INTRODUCTION

## 1-1. OBJECTIVES

At the Egyptian Government request, The United Nations Industrial Development Organisation is prepared to help in setting an unit for organo-phosphorous insecticides.

In former studies, it has been shown that such a project will have a potential effect on Egyptian Economy.

Furthermore, it appeared that the projected unit will be conveniently situated at the state's owned company : Dyestuffs & Chemical Co., Kafr el Dawar.

The present objectives of the mission are :

- to outline a scheme for a multipurpose organic synthesis plant with a preliminary production of 120 tonnes of dimethoate and 180 tonnes of malathion per year. The production of these two organo-phosphrous insecticides will fit with the very near future increase of Egyptian consumption . However this unit will contain almost all the necessary equipment for production of various organo-phosphorous insecticides.

- to examine the facilities of the DCC in order to identify equipment and other facilities which could be incorporated in the multipurpose pesticide plant.

- to give a list of equipment with their specifications
- to estimate the cost of the equipment to be provided on the UNIDO budget and Egypt Government budget.
- to estimate the costs of production in agreement with DDC's staff.

- to assist UNIDO in drafting a document to contact any producer of these compounds which may be interested in selling the necessary know-how for this project.

The assistance of the following individuals who contributed their time and information to this report is greatfully acknowledged.

- Dr Lotfi KHATTAB (General Works Director, ISMADYE)
- Dr Hassan Saïd MAHMOUD (Research Director, ISMADYE)
- Mr Mokhtar Kamel BARSOM (Technical Director, ISMADYE)
- Mr K. SZABO (Senior Industrial Development Officer, UNIDO).

## 1-2. REPORT CONTENTS

Process descriptions based on recent available technical data are given in Section 2.

The raw materials & Utilities consumptions and requirements are given in Section 3.

The equipment and their specifications are listed in Section 4.

The section 5 is an estimate of the equipment costs both in UNDP budget and Egyptian Government budget. This section include an estimate of inside and outside battery limit Investments.

In section 6 the costs of production of malathion and dimethoate are given.

After the conclusion (section 7) we include as annexes (1) safety, (2) economical figures, (3) preliminary lay-out, (4) reference terms for tender invitation.

# 2 - PROCESS DESCRIPTION

## 2-1. O-O DIMETHYLDITHIOPHOSPHORIC ACID

### 2-1-a. Background and Chemistry

O-O Dimethyldithiophosphoric acid is a phosphorous compound used in the production of organophosphorous insecticides such as malathion and dimethoate.

The chemical reaction is fairly straightforward, i.e. phosphorus pentasulfide is reacted with methanol in a solvent like toluene.

$$P_2S_5 + 4\ CH_3OH \longrightarrow 2\ \begin{matrix}(CH_3O)\\(CH_3O)\end{matrix}\!\!>\!\!P(=S)\!-\!SH + H_2S$$

However, phosphorus pentasulfide must be added carefully, because of the highly exothermic nature of the reaction. The safety aspects are described in Annex 1.

### 2-1-b. Process description

Figure 6003-PX-100 is a flow-sheet for the batch production of O-O-dimethyldithiophosphoric acid by reaction of methanol with phosphorus pentasulfide. The design is based on three shifts per day, 7 days a week and 7 200 hours per year schedule.

According to the following production schedule (table 2-1) two 12 hours-batches per day can be carried out.

TABLE 2-1

O-O-DIMETHYLDITHIOPHOSPHORIC ACID PRODUCTION SCHEDULE

| Operation | Number | Time | Item No |
|---|---|---|---|
| Methanol and toluene are charged into reactor | 1 | 0.5 hr | D102/R101 |
| Mixture is heated to 70-74°c | 2 | 0.5 hr | R101 |
| Phosphorus pentasulfide is slowly added under temperature control | 3 | 1.5hr | D101/R101/E101/C101 |
| Reaction mixture is maintained at 82°c | 4 | 3 hr | R101/E202/C101 |
| Excess Methanol and Toluene are distilled under slight vacuum (760-220 mmhg) | 5 | 2 hr | R101/T101/E101/ D102/E102 |
| Mixture is cooled down to 50°c | 6 | 1.5hr | R101 |
| Mixture is filtered and stored | 7 | 2 hrs | R101/H101/D103/ P101/D401 |
| Total | | 11 hrs | |

OPERATION N° 1

During the distillation of the previous batch (operation 5) the distillate (toluene and unreacted methanol) is sent by gravity to D102 (connected to the vacuum).
Fresh methanol and toluene are added to the measuring tank D102 before the next batch starts.
When the R101 has been emptied from the previous batch, the mixture in D102 is sent by gravity to R101.

OPERATION N°2

The mixture is heated by circulating steam through the jacket to 70-74°c boiling point. Then we switch to the cooling water.

OPERATION N°3

As soon as the water is circulated through the reactor jacket, the phosphorus pentasulfide solids are added from D101 to R101. The temperature in R101 is controlled by the $P_2S_5$ feeding valve and the flow of cooling water.

OPERATION N°4

The mixture is kept at this temperature for 2-3 additional hours.
During operations N° 4 and N°5, $H_2S$ produced is passed through E101 (to remove as much as possible the methanol and toluene) to the water vacuum jet C201 (C201 will act as an water absorber of $H_2S$ and the discharged water is send to the effluent treatment).

OPERATION N°5

When the reaction is finished, the steam is circulated through the jacket of R101 and R101 is now connected to E101 through the distillation column T101.

The pressure in E101 will decrease from 760 mm to 220 mm Hg in order to keep the product temperature at 80-85°c.
The distillate with a reflux divider is sent to D102.
All the methanol is distilled off whereas some toluene is left with the acid (acid concentration at the end of the distil lation step : 60 to 65 weight percent).

OPERATION N°6

The mixture is cooled to 50°c by circulating water through the reactor jacket.

OPERATION N°7

In order to eliminate the solids in the toluene acid mixture, the mixture is pumped through the filter H101 and collected in a surge tank D103. This mixture is then analysed prior to its transfer to storage (D401).

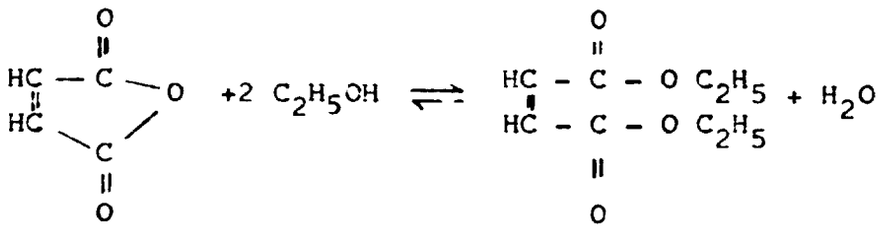
## 2-1-c. Equipment list

| | |
|---|---|
| C101 | Vacuum jet |
| D101 | Phosphorus Pentasulfide hopper |
| D102 | Methanol-Toluene tank |
| D103 | Acid surge tank |
| E101 | Condenser |
| E102 | Distillate cooler |
| H101 | Acid filter |
| P101 | Acid pump |
| R101 | $P_2S_5$ Methanol reactor |
| T101 | Methanol recovery column |

## 2-2 ETHYL MALEATE

### 2-2-a. Background and Chemistry

Ethyl maleate is an intermediate used in the production of malathion. The manufacturing process consists of reacting ethyl alcohol with maleïc anhydride in a suitable solvent such as dichloroethane



This reaction is a very standard esterification catalyzed by acids like sulfuric acid or p.toluene sulfonic acid (PTSA).In order to minimizecorrosion and to reduce product degradation, PTSA is usually selected.
The above reaction is an equilibrium and water has to be removed so as to get high conversion yields. This is achieved with solvent like dichloroethane which forms ternary azeotrope with water and ethanol.

### 2-2-b. Process description

Figure 6003-PX-200 is a flow-sheet for the batch production of ethyl maleate by the reaction of ethyl alcohol with maleïc anhydride. This section is flexible enough to produce the N-methylchoroacetamide.

The design is based on three shifts per day, 7 days a week and 7 200 hours per year schedule to be shared with the production of N-methylchloroacetamide.

According to the following production schedule (Table 2-2), one 24 hours batch per day can be carried out.

TABLE 2-2

ETHYL MALEATE PRODUCTION SCHEDULE

| Operation | Number | Time | Item N° |
|---|---|---|---|
| Dichloroethane and ethanol (fresh and recovered) are charged into reactor . | 1 | 1.0 hr | D206/D203/P202/R201 |
| Maleic anhydride flakes are charged into reactor and steam is open into jacket. | 2 | 3/4 hr | D204/R201/P201/E201 |
| Catalyst is charged into reactor . | 3 | 1/4 hr | R201 |
| Mixture is kept boiling by falling film evaporator and water is continuously removed from reaction mixture. System pressure is slowly decreased in order to keep the bubble point of the reaction mixture below 130°C. | 4 | 5 hr | R201/P201/E201/T201/ E202/D205/D209 |
| Excess ethanol and dichloro-ethane are distilled under vacuum. | 5 | 3 hr | R201/P201/E201/T201/ E202/D205/D206. |
| Crude product is cooled down to 50°c. | 6 | 2 hr | R201 |
| Sodium carbonate salt solution is sent into reactor and the two immiscible phases are thoroughly mixed. | 7 | 1/2 hr | D202/R201 |
| Agitation is stopped, heavy aqueous phase is decanted and drained. | 8 | 1 hr | R201 |
| Salt solution is sent into reactor and the two immiscible phases are thoroughly mixed. | 9 | 1/2hr | D202/R201 |
| Agitation is stopped heavy aqueous phase is decanted and drained | 10 | 1 hr | R201 |
| Washed product is distilled under vacuum to the surge tank. | 11 | 9 hr | R201/T101/P201/E201/ E202/D205/D207 |
| | | 24 hr | |

OPERATION 1

During the previous batch, recovered ethanol-dichloroethane have been stored in D206. The complement is added in D203. As soon as R201 is empty, D203 is emptied by gravity into R201 and D206 is emptied with P202 into R201.
The steam is opened into the jacket of R201.

OPERAITON 2

Weighted Maleïc anhydride flakes into the hopper are charged into the reactor R201, the recirculation pump P201 is started (R201-P201-E201-R201).
Steam is opened into the shell side of the falling film evaporator (E201).

OPERATION 3

PTSA is charged into reactor R201.

OPERATION 4

Mixture is kept boiling by falling film evaporator and water is continously removed from reaction mixture.
The initial boiling point of the raction mixture under atmosphere pressure is around 70°c however, as the raction proceeds, ethanol is consumed and the boiling point rises. System pressure is therefore slowly reduced to keep the solution boiling at a maximum temperature of 130°c. The ethanol dichloroethane water vapors are condensed overhead in E202 and the heterogeneous distillate is allowed to decant in the reflux drum (D205). The water-rich light phase is discarded and the dichloroethane-rich heavy phase is refluxed to the distillation column T201.
The discarded water phase is first stored in D206 connected to the vacuum through D205. When the operation 4 is finished, D206 is connected to the atmosphere and the water can be discarded . The D206 is connected again to the vacuum before operation 5.

OPERATION 5

Excess ethanol and dichloroethane are distilled under vacuum. The dichloroethane -rich light phase is refluxed to the distillation column T201 and the ethanol-rich heavy phase is sent to D206.
The recirculating pump P201 and steam into E201 and R201 are stopped.

OPERATION 6

The crude ethylmaleate into R201 is cooled down to 50°c by cooling water.

OPERATION 7

In order to remove the catalyst, a sodium carbonate solution is charged (from D203) into R201 where the mixture is agitated.

OPERATION 8

The agitation is stopped and the heavy aqueous phase is drained to waste.

OPERATION 9

The neutralized organic mixture is then washed by a sodium chloride solution (D302). Agitation.

OPERATION 10

The agitation is stopped and the heavy aqueous phase is drained to waste.

OPERATION 11

The washed organic phase is heated up and distilled under vacuum (with E101). The distilled ethylmaleate is recovered into D207.

## 2-2-c Equipment list

| | |
|---|---|
| D202 | Neutralization solution tank |
| D203 | Liquid raw materials measuring tank |
| D204 | Solid raw materials hopper |
| D205 | Reflux drum |
| D206 | Recovered solvent tank |
| D207 | Product tank |
| D209 | Water tank |
| E201 | Falling film evaporator |
| E202 | Condenser |
| E203 | Vent Condenser |
| R201 | Reactor |
| T201 | Distillation column |
| P201 | Recirculation pump |
| P202 | Product pump |

## 2-3 N-METHYLCHLOROACETAMIDE

### 2-3-a Background and Chemistry

N-methylchloroacetamide is one intermediate used in the production of dimethoate.

This compound is produced in a two-step process involving monochloroacetic acid, methanol and monomethylamine.

In first step, monochloroacetic acid is esterified by methanol, this intermediate is then reacted with monomethylamine to form N-methylchloroacetamide. The reactions are shown below :

$$ClCH_2 - COOH + CH_3OH \rightleftharpoons ClCH_2 - COOCH_3 + H_2O$$

$$Cl\ CH_2 - COOCH_3 + CH_3NH_2 \longrightarrow Cl\ CH_2 - \overset{O}{\overset{\|}{C}}N\overset{H}{\underset{CH_3}{\diagup}} + CH_3OH$$

It should be noticed that methanol is recycled and that the net consumption of methanol is only due to operation losses.

### 2-3-b Process description

Figure 6003 PX 200 is a flow-sheet for the batch production of N-methylchloroacetamide. This section is flexible enough to produce the ethylmaleate.

The design is based on three shifts per day, 7 days a week and 7 200 hours per year schedule to be shared with the production of ethylmaleate.

According to the following production schedule (Table 2-3), a complete batch can be carried out in 36 hours.

TABLE 2-3

N-METHYLCHLOROACETAMIDE PRODUCTION SCHEDULE

| Operation | Number | Time | Item N° |
|---|---|---|---|
| Cyclohexane and methanol are charged into reactor | 1 | 0.5 hr | D208/R201/P203 |
| Monochloroacetic acid flakes and PTSA are charged into reactor | 2 | 1.0 hr | D204/R201 |
| Mixture is heated to its boiling point (50°c) | 3 | 1.0 hr | R201 |
| Mixture is kept boiling | 4 | 2.0 hr | R201/T201/E202/D205 |
| Methanol-cyclohexane azeotrope is distilled off | 5 | 3.0 hr | R201/T201/E202/D205/D206 |
| Water-cyclohexane azeotrope is distilled off | 6 | 1.5 hr | R201/T201/E202/D205/D209 |
| Heavy phase of methanol cyclohexane azeotrope distilled previously is charged into reactor | 7 | 0.5 hr | D206/R201/D208 |
| Mixture is heated and kept boiling | 8 | 2.0 hr | R201/T201/E202/D205/ |
| Methanol-cyclohexane azeotrope is distilled off | 9 | 2.5 hr | R201/T201/E202/D205/D206 |
| Water cyclohexane azeotrope is distilled off | 10 | 0.5 hr | R201/T201/E202/D205/D209 |
| Crude ester is cooled to 60°c or below | 11 | 2.0 hr | R201 |
| Dilute caustic solution is charged into reactor. The two phases are allowed to decant and the aqueous phase is discarded | 12 | 2.0 hr | R201/P201/D203 |
| Process water and organic phase are charged under agitation into reactor.The two phases are allowed to decant and the aqueous phase is dicarded. | 13 | 2.0 hr | D203/R201 |
| Organic phase is charged into reactor | 14 | 0.5 hr | D203/R201 |

| Operation | Number | Time | Item N° |
|---|---|---|---|
| Excess cyclohexane is distilled off (80°c) | 15 | 2.0 hr | R201/T201/E202/D205/D207 |
| Ester is cooled from 80°c to -10°c | 16 | 4.0 hrs | R201 |
| Methanol monoethylamine mixture is charged into reactor, temperature is kept below 0°c. | 17 | 2.0 hrs | R201/D203 |
| Reaction mixture is kept at 0°c | 18 | 0.5 hrs | R201 |
| Excess methanol is distilled off | 19 | 3.0 hrs | R201/T201/E202/D205/ D206/D208/P202 |
| Unreacted ester is distilled under vacuum | 20 | 1.0 hr | R201/T201/E202/D205/D207 D203/P202 |
| Product is cooled | 21 | 1.0 hr | R201 |
| Dichloroethane is charged into reactor-cooling is maintained | 22 | 1.0 hr | R201 |
| N-methylchloroacetamide solution is pumped to storage | 23 | 0.5 hr | R201/D207 |
| Total | | 36 hrs | |

OPERATION 1

In D208, fresh methanol and cyclohexane are added to the recycled methanol and cyclohexane. Then the mixture is charged into the reactor R201.

OPERATION 2

Monochloroacetic acid flakes (D204) and p-toluene sulfonic acid (PTSA) are added to the solvents by gravity

OPERATION 3

Steam is opened into the jacket and reaction mixture is heated to its boiling point.

OEPRATION 4

Mixture is kept boiling with total reflux from E202-D205

OPERATION 5

In order to reach high ester yields, water produced by the reaction has to be removed. Unfortunately, the cyclohexane/water azeotrope cannot be distilled straightforward, it requires the previous distillation of the methanol-cyclohexane azeotrope (temp.maxi : 115°c).
In D205 the light phase is refluxed (cyclohexane rich phase) while the heavy methanol rich phase is charged into D206.

OPERATION 6

When the methanol is removed, the distillation of the water-cyclohexane is started up.
The cyclohexane rich phase only is refluxed while the water rich phase is charged into D209. At the end of the distillation the cyclohexane phase is charged into D209 too.

OPERATION 7

From D206, the heavy phase (methanol) is pumped into R201 the light phase (cyclohexane) is pumped to the solvent surge drum D208

OPERATION 8

Mixture is heated and kept boiling
(Identical to operation 3 and 4).

OPERATION 9

Distillation of cyclohexane-methanol azeotrope
Identical to operation 5 - From D206 the whole content is sent to D208

OPERATION 10

Distillation of cyclohexane water azeotrope.
(Identical to operation 6).

OPERATION 11

Crude ester is cooled down to 60°c or below .

OPERATION 12

From D202, dilute caustic solution is charged into reactor. After agitation the two phases are allowed to decant . The heavy organic phase is pumped to D203 while the light aqueous phase is discarded.

OPERATION 13

From D203 the organic is charged into R201 with process water. After agitation the two phases are allowed to decant. The heavy organic phase is pumped to D203 while the light aqueous phase is discarded.

OPERATION 14

The organic phase is charged again into R201

OPERATION 15

The excess cyclohexane is distilled off at 80°c. The distillate is charged into D207. (Note that every time D207 is emptied to D208 while the aqueous phase is discarded).

OPERATION 16

The temperature of methyl chloroacetate is then reduced to - 10°c by circulating the brine through the reactor jacket.

OPERATION 17

The Monomethylamine in solution in methanol is slowly added from D203 with stirring. During this addition the batch temperature is kept between -5°c and 0°c.

OEPRATION 18

The reaction mixture is kept below 0°c for 30 additional minutes.

OPERATION 19

Excess methanol is distilled off. The distillate through D206 is pumped to D208.

OPERATION 20

Unreacted ester is distilled off (Pressure 17 mm Hg) and collected into D207 from where it is pumped to D203 waiting for the operation n° 12 of the following batch.

OPERATION 21

The product is slightly cooled.

OPERATION 22

Dichloroethane is added to the reactor and the resulting solution is cooled to 50°c or below.

OPERATION 23

The solution is stored in D207.

## 2-3-e Equipment list

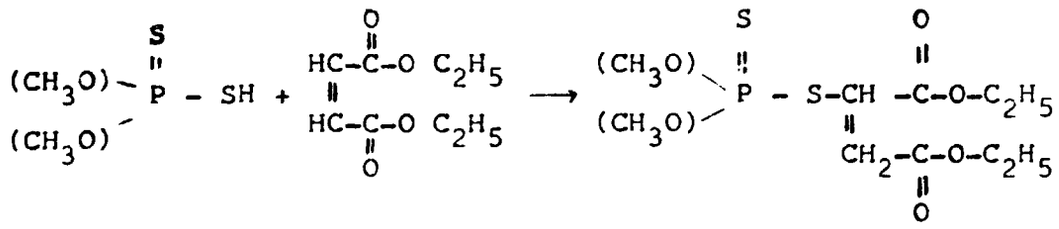
Identical to equipment list of 2-2-c, plus :

| | |
|---|---|
| D207 | Product tank |
| D208 | Solvent surge tank |
| P203 | Solvent pump |

## 2-4 MALATHION

### 2-4-a Background and chemistry

Malathion or O-O-Dimethylphosphorodithioate of diethyl mercapto succinate is a non systemic insecticide and acaricide having a fairly low mammalion toxicity. This very safe insecticide is a liquid of low volatily, which can cause skin irritation when its diethyl fumarate content is around 1 to 4 percent by weight. Malathion was developped by American Cyanamid which is one of the major producers. Malathion is produced by reacting O-O-dimethyldithiophosphoric acid with diethyl maleate :



In order to get a very low diethyl fumarate content, American Cyanamid improved the production process by stripping off the unconverted reactants in a wiped film evaporator This process is indeed very efficient since the conversion yield is around 94 %, based on ethyl maleate.

## 2-4-b Process description

Figure 6003-PX 300 is a flowsheet for the batch production of malathion by reaction of ethylmaleate with O-Odimethyl-dithiophosphoric acid in toluene. The design is based on three shifts perday, 7 days a week, 7 200 hours per year schedule to be shared with dimethoate production.

According to the following production schedule (table 2-4), two 12 hours batches per day can be carried out.

TABLE 2-4

MALATHION PRODUCTION SCHEDULE

| Operation | Number | Time | Item N° |
|---|---|---|---|
| Diethyl maleate is charged into reactor | 1 | 0.5 hr | D207/R302 |
| Recovered reactants from previous batch are charged into reactor | 2 | 0.5 hr | D306/R302 |
| Dithiophosphoric acid solution is charged into reactor | 3 | 0.5 hr | D301/R302 |
| Reaction mixture is heated to 90°c | 4 | 1.5 hr | R302 |
| Temperature is maintained and pressure is reduced to 20 mm Hg | 5 | 3.0 hr | R302/D304/E304/E305 |
| Mixture is cooled to about 70°c | 6 | 1.0 hr | R302 |
| Unconverted reactants are vaporized in agitated film evaporator and crude product is recovered in vacuum surge tank. | 7 | 4.0 hr to 5.0 hr | R302/D306/E303/D307/E304 |
| Crude product is cooled | 8 | 1.0 hr | D307 |
| Crude product and sodium carbonate solution are charged into second reactor | 9 | 0.5 hr | D303/D307/R303 |

| Operation | Number | Time | Item N° |
|---|---|---|---|
| Agitation - Decantation | 10 | 1.0 hr | R303 |
| Heavy organic phase is transferred to surge tank and aqueous phase is sent to waste | 11 | 0,75 hr | R303/D307 |
| Crude product and wash water are charged into reactor | 12 | 0,5 hr | D307/R303 |
| Agitation - Decantation | 13 | 1.0 hr | R303 |
| Heavy organic phase is transferred to surge tank and aqueous phase is sent to waste | 14 | 0,75 hr | R303/D307 |
| Organic phase is transferred into reaction | 15 | 0.5 hr | D307/R303 |
| Malathion is steam stripped at 50°c under vacuum (25 mm Hg) Malathion is cooled after steam stripping | 16 | 4.0 hr | R303/E306/D308 |
| Malathion is pumped to the drums through a filter | 17 | 2.0 hr | R303/H302-drums |

Total 12 hrs (R302)
12 hrs (R303)

OPERATION 1

Diethyl maleate is charged into reactor R302 from D207.

OPERATION 2

Recovered reactants in D306 are charged into reactor R302 by gravity.

OPERATION 3

Acid solution is charged into reactor from D301 by gravity.

OPERATION 4

The agitation and heating are started up.

OPERATION 5

In order to maintain the temperature at 90°c., the pressure will be decreased continuously to 20 mm Hg. During the operation most of the toluene is vaporized, condensed into E304, recovered into D304. At the end of this operation, the vacuum in D304 is stopped and the recovered toluene is sent to the toluene storage. On top of 301 a vent condenser E305 minimizes the toluene losses.

OPERATION 6

The mixture is slightly cooled down to 70°c.

OPERATION 7

The mixture is sent to the wiped film evaporator E303 in 4-5 hours, where most unconverted reactants are vaporized, condensed in E304, sent in D306. These unconverted reactants are sent back to R302 during the operation 2 of the following batch. The crude product is sent to the vacuum surge tank fitted with a cooling coil. (D307).

OPERATION 8

In D307 the mixture is cooled

OPERATION 9

From D307 the crude product is charged into R303 with sodium carbonate solution coming from D303.

OPERATION 10

The agitation is stopped and the phases are allowed to decant

OPERATION 11

The heavy organic phase is transferred to D307 and aqueous phase is discarded.

OPERATION 12,13,14

Identical to operation 9,10,11 with pure water.

OPERATION 15

The organic phase is transferred into reactor R303

OPERATION 16

In R303, the Malathion is steam stripped. The aqueous vapors are condensed in E306 to be sent into E308 before to be discarded

OPERATION 17

Malathion is pumped through the filter H302 to fill the product drums.

## 2-4-c Equipment list

Only some equipment of the reaction 300 are used :

| | |
|---|---|
| D301 | Liquid raw material measuring tank |
| D303 | Sodium carbonate and bicarbonate solution tank |
| D304 A/B | Solvent tanks |
| D307 | Surge drum |
| D308 | Ethyl maleate feed tank |
| E303 | Wiped film evaporator |
| E304 | Condenser |
| E305 | Vent condenser |
| E306 | Condenser |
| H302 | Cartridge filter |
| P302 | R302 Pump |
| P303 | R303 Pump |
| R302 | Reactor |
| R303 | Reactor |

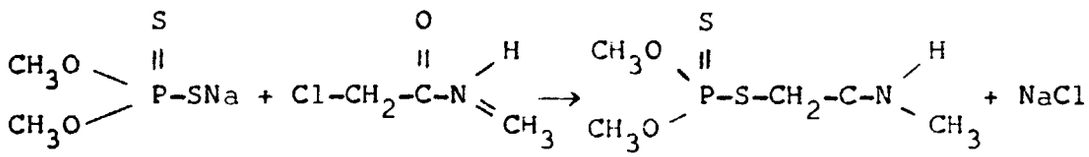
## 2-5 DIMETHOATE

### 2-5-a Background and Chemistry

Dimethoate or O,O Dimethyl S Carbomoylmethyl phosphorodithioate is a systemic and contact insecticide.

It is a white solid (m.p. 51-52°c) moderately soluble in water. Dimethoate was developed by American Cyanamid and Montecatini which are important producers.

Dimothoate is produced by reacting the sodium salt of O-O Dimethyl dithiophosphoric acid with N-methylchloraceta-mide



In theory, the reaction is very simple, but in practice dimethoate may react further with sodium dimetylphosphoro-dithioate to yield O,O S-Trimethylphosphorodithioate. The result is a low yield and a product difficult to purify.

In order to minimize this problem, various companies (American Cyanamid, Fisons) have developed two phases reaction system, whereby the dimethoate is extracted into the organic phase as it is formed.

Various water immiscible solvents and/or solvent mixtures have been used, however, a chorinated solvent/cyclohexanone mixture was selected because it seems to give satisfactory yields.

## 2-5-b Process description

Figure 6003-PX-300 is a flowsheet for the batch production of technical dimethoate (90 % purity) by reaction of N-methylchloroacetamide with O,O-Dimethyl-dithiophosphoric acid. The design is based on three shifts per day, 7 days a week, 7 200 hours per year schedule to be shared with malathion production. According to the following schedule (Table 2-5) one batch can be carried out in 12 hours.

TABLE 2-5

TECHNICAL DIMETHOATE PRODUCTION SCHEDULE

| Operation | Number | Time | Item N° |
|---|---|---|---|
| Dithiophosphoric acid solution is charged into extraction tank | 1 | 0.5 hr | R301 |
| First extraction with $NaHCO_3$ solution. The aqueous phase is transferred to surge tank | 2 | 2.0 hrs | R301/D301 /D303 |
| Second extraction identical to first one | 3 | 2.0 hrs | D303/R301/D301 |
| Third extraction with water | 4 | 2.0 hrs | R301/D301 |
| Rich aqueous phase is charged into reactors | 5 | 1.0 hr | D301/R302 R303 |
| Dichloroethane, cyclohexane and chloroacetamide solution are added to reactors | 6 | 0.5 hr | Storage + D207/ R302 + R303 |
| Reaction mixture is heated to 55°c | 7 | 1.5 hrs | R302 + R303 |
| Temperature is maintained at 55°c | 8 | 3.0 hrs | R302+R303/E301-E302 |
| Reaction mixture is cooled to 20°c | 9 | 1.5 hrs | R302 + R303 |
| Agitation is stopped-the two phases are allowed to decant | 10 | 1.0 hr | R302+R303 |
| Aqueous phase is pumped to section 400. Organic phase is added to extraction tank containing a sodium bicarbonate solution | 11 | 1.0 hr | R302+R303/R301 (D403) |
| Organic phase is washed by bicar bonate solution organic phase is pumped back to reactor containing Process water | 12 | 2.5 hrs | R301/R302+R303 |
| Organic phase is washed by water. Aqueous phase is pumped to surge tank, organic phase is transferred to solvent evaporator | 13 | 2.0 hr | R302+R303/R304 |

| Operation | Number | Time | Item N° |
|---|---|---|---|
| Crude product is heated up and vacuum pump is started | 14 | 1.0 hr | R304/E303-E304 |
| Solvents are distilled off under vacuum | 15 | 4.0 hr | R304/E303-E304-D304 |
| Final traces of solvents are distilled off and molten product is pumped to flaking unit | 16 | 6.0 hrs | R304/E303-E304-D304 D305/H301 |

Note : The extraction, reaction and concentration steps are carried out in parallel.

OPERATION 1

Dithiophoric acid solution is charged into R301

OPERATION 2,3,5

Two successive extractions by sodium-bicarbonate solution (D303) are done . A third extraction with pure water follows . The salt acid rich aqueous phases are transferred to the surge drum D301 .

After this operation the lean toluene left is sent to D402 to be stored before being vaporized in R101 when the acid production is stopped.

OPERATION 5

From D301 the rich aqueous phase is charged into the reactors R302 and R 303 together with some more process water.

OPERATION 6

Dichloroethane, cyclohexanone are added to R302 and R303 from storage - Chloroacetamide solution is added from D207

OPERATION 7

The mixture is heated 55°c

OPERATION 8

The temperature is maintained. The brine is circulated through the vent condensers E301-E302

OPERATION 9

The reaction mixture is cooled to 20°c with brine in the jacket

OPERATION 10

Agitation is stopped. The two phases are allowed to decant

OPERATION 11

The heavy organic phase is sent to R301 with bicarbonate solution from D303 while the lean aqueous phase is sent in section 400 to cyclohexanone recovery unit.
The organic layer is washed with a saturated aqueous sodium bicarbonate solution in R301.

OPERATION 12

Washed organic phase is pumped back to the reactors containing water.

OPERATION 13

After decantation the organic phase is transferred to solvent evaporator R304 while the water phase is sent again in section 400.

OPERATION 14

Crude product is heated up and vacuum pump is started. The crude product is recirculating (R304-P304-E303-R304) on a wiped film evaporator.

OPERATION 15

The solvents are distilled off by vaporizing in E303, condensing in E304. They are collected into the recovered solvent tank D304.

OPERATION 16

The concentrate product is sent from E303 to the product surge drum D305 while the traces of solvent are still removed in E303.
In D305 the crude product is maintained at a temperature not below than 70°c. The molten product is sent to the flaking unit H301.

## 2-5-e Equipment list

All the equipment listed in 2-4-c are used, except :

D306-D307-D308-E305- E306-H302

plus the following equipment :

| | |
|---|---|
| D305 | Product tank |
| E301 | Vent condenser |
| E302 | Vent condenser |
| H301 | Metal belt flaker |
| P301 | R301 pump |
| P304 | R304 pump |
| R301 | Extraction tank |
| R304 | Solvent evaporator |

## 2-6. INTERMEDIATES STORAGE RECOVERY SECTION AND VACUUM SYSTEM

### 2-6-a Introduction

Only storage of intermediates are considered.

The acid production in section 100 is not flexible due to the fact that for safety reasons it is necessary to use full tote bin of phosphorous pentasulfide. This section is slightly oversized and this oversized design requires the need of a storage (D401 + P401).

If we want to avoid any unnecessary storage of ethylmaleate or N-methylcholoracetamide, it is possible to ajust the production of these intermediates to the production of the final compound.

As the design, of the sections 200 and 300 has been done on the following basis.

- 120 tonnes/year of dimethoate and 180 tonnes/year of malathion
- 180 tonnes/year of dimethoate
- 65 % full vessels minimum

It is necessary to have :

. For dimethoate production :

76 % full vessels in section 300 and
65 % full vessels in section 200

. For malathion production :

65 % full vessels in section 300 and
73 % full vessels in section 200

These conditions will reduce the storage of the intermediates to the surge tank D207.

During the dimethoate production, some crude toluene is recovered. This requires a recovery unit. But during the production of dimethoate, the acid section 100 will be stopped 5 days after one week of acid production. These five days will allow the recovery of crude toluene by a simple evaporation. A storage of this crude toluene is therefore necessary (D402).

During the dimethoate production, a rather large volume (3m3/batch) of aqueous effluent is obtained. This effluent contains some cyclohexanone which can be recovered by distillation. The above volume is such that it is more advisable to have a continuous treatment (D403-T403-E403/D404). The section 200 and 300 require a vacuum system (C 401 - D105).

2-6- b Equipment list

| | |
|---|---|
| C401 | Vacuum jet |
| D401 | Acid storage tank |
| D402 | Crude toluene storage tank |
| D403 | Waste water storage tank with heating coil |
| D404 | Distillate receiver (cyclohexanone) |
| D405 | Hotwell |
| E403 | Condenser |
| P401 | Acid storage pump |
| T403 | Distillation column |

## 2-7 STORAGE

All the raw materials are in drums or bags in a central store. They will be brought in the process area when it is required. The liquid raw material will be pumped from the drums to the vessels by one of the five hand pumps.

The solid will be brought to the hopper level with the lift of the building.

The malathion (liquid) will be charged from its surge tank into the drums. After control analysis,the drums are sent to the central store.

The dimethoate (solid) will be charged from the flaking unit into bags (The bags may be into drum). After control analysis the drums are sent to the central store.

# 3 - RAW MATERIALS AND UTILITIES REQUIREMENTS

## 3-1. PAW MATERIALS CONTAINERS

All the liquid raw materials are in drums
All the solid raw materials are in bag except the $P_2S_5$ which is in tote bin.

## 3-2. UTILITIES FACILITIES

| | | |
|---|---|---|
| Water : | Temperature | 10°c to 35°c |
| | Alkalanity | 150 ppm |
| | Pressure | 6 atm |
| Steam | 3atm gauge | |
| Compressed air | 4 atm gauge | |
| Central vacuum | 76 mmHg | |
| Nitrogen gas | 3 atm g | |
| Brine (NaCl) | 3 atm g | - 15°c |

## 3-3. UTILITIES REQUIREMENTS

The required vacuum is 10 mmHg and a new vacuum system is required (C401-D405).

The steam pressure of 3 atm gauge will be sufficient excepted for a small amount which requires a pressure of about 10 atm. This pressure is available near the building before the pressure valves.

The quantitative requirements are :

| | | |
|---|---|---|
| steam | 3 atmg | 600 Kg/m |
| steam | 10 atm | 300 Kg/m |
| steam total | | 470 Kg/m |
| Water | | 30 $M^3$/hr |
| brine | | 225000 Kcal/hr |
| nitrogen | | few $NM^3$/hr |
| Power | | 11 Kw |

- see table 3-1 for detailed requirements.

## 3-4. RAW MATERAILS AND UTILITIES CONSUMPTIONS

The consumptions are given in the following tables :

Table 3-2 O-O Dimethyldithiophosphoric acid
Table 3-3 Ethyl Maleate
Table 3-4 Malathion from intermediate materials
Table 3-5 Malathion from raw materials
Table 3-6 N-methylchloroacetamide
Table 3-7 Dimethoate from intermediate materials
Table 3-8 Dimethoate from raw materials.

TABLE 3-1

UTILITIES REQUIREMENTS

| | Steam 3 atm g | Steam 10 atm g | Water | brine | Power |
|---|---|---|---|---|---|
| Section 100 acid | 50 Kg/hr | - | 5M3/hr | 22 000 Kcal/hr | 2 KW |
| Section 200 maleate | - | 157Kg/hr | 2m3/hr (or 10m3/hr* | 76 000 Kcal/hr | 3 KW |
| acetamide | 140 Kg/hr | 10 Kg/hr | 7m3/hr | 60 000 Kcal/hr | 2 KW |
| Section 300 malathion | 60 Kg/hr | 80 Kg/hr | 4m3/hr | 15 000 Kcal/hr | 3 KW |
| dimethoate | 253 | 17 | 11m3/hr | 90 000 Kcal/hr | 4.5 KW |
| Malathion Sections 100 + 200 + 300 | 110Kg/hr | 237 Kg/hr | 11m3/hr (or 19m3)* | 100 000 Kcal/hr * (or 37 000 Kcal/hr) | 8 KW |
| Dimethoate Sections 100 + 200 + 300 | 443Kg/hr | 27 Kg/hr | 23m3/hr | 172 000 Kcal/hr | 8.5 KW |
| Final requirements ** | 600 Kg/hr | 300 Kg/hr | 30 m3/hr | 225 000 Kcal/hr | 11 KW |

* If Twater 30°c.

** The maximum requirements including the 30 % over capacity

TABLE 3-2

RAW MATERIALS AND UTILITIES CONSUMPTIONS

O-O DIMETHYLDITHIOPHOSPHORIC ACID
per ton (100 %)

| | |
|---|---|
| **RAW MATERIALS** | |
| Phosphorous pentasulfide | 0.7960 t |
| Methanol | 0.4911 t |
| **SOLVENTS AND CHEMICALS** | |
| Toluene | 0.0390 t |
| **UTILITIES** | |
| Nitrogen | 12 $NM^3$ |
| Steam 3 atm | 0.25 t |
| Water | 22 $m^3$ |
| Brine | $0.1.10^6$ Kcal |
| Power | 24.2 KWhr |

TABLE 3-3

RAW MATERIALS AND UTILITIES CONSUMPTIONS

ETHYL MALEATE
per ton (100%)

RAW MATERIALS

| | |
|---|---|
| Ethanol | 0.6905 t |
| Maleïc anhydride | 0.6313 t |

SOLVENTS AND CHEMICALS

| | |
|---|---|
| Dichloroethane | 0.0913 t |
| p.toluene sulfonic acid | 0.0166 t |
| Sodium carbonate | 0.0206 t |
| Sodium chloride | 0.2189 t |

UTILITIES

| | |
|---|---|
| Steam 10 atm | 2.9 t |
| Water | 18 $M^3$ |
| Brine | 1.2 $10^6$ Kcal * |
| Power | 52.3 KWhr |

* If $T_{water} \leqslant 30°c$ Brine consumption = 0
Water consumption = 138 $M^3$

TABLE 3-4

RAW MATERIALS AND UTILITIES CONSUMPTIONS

MALATHION FROM INTERMEDIATE MATERIALS
per ton (97 %)

| | |
|---|---|
| **RAW MATERIALS** | |
| O-O dimethyldithiophosphoric acid | 0.5004 t |
| Ethyl Maleate | 0.5380 t |
| **SOLVENTS AND CHEMICALS** | |
| Toluene | 0.0092 t |
| Sodium carbonate | 0.0060 t |
| **UTILITIES** | |
| Nitrogen | 9.1 $NM^3$ |
| Steam 3 atm g | 0.4 t |
| Steam 10 atm | 0,8 t |
| Water | 4,4 |
| Brine | 0,1 . $10^6$ Kcal |
| Power | 34.6 KWhr |

TABLE 3-5

RAW MATERIALS AND UTILITIES CONSUMPTIONS

MALATHION FROM RAW MATERIALS

per ton (97%)

RAW MATERIALS

| | |
|---|---|
| Ethanol | 0.3715 t |
| Maleïc anhydride | 0.3396 t |
| $P_2S_5$ | 0.3983 t |
| Methanol | 0.2457 t |

SOLVENTS AND CHEMICALS

| | |
|---|---|
| Sodium carbonate | 0.0171 t |
| Sodium chloride | 0.1178 |
| Toluene | 0.0287 t |
| P.toluene sulfonic acid | 0.0089 t |
| Dichloroethane | 0.0491 t |

UTILITIES

| | |
|---|---|
| Nitrogen | 15.1 $M^3$ |
| Steam 3 atmg | 0.5 t |
| Steam 10 atm | 2.4 t |
| Water | 65 $M^3$ |
| Brine | 0.8 $10^6$ Kcal * |
| Power | 75 KWhr |

* If $T_{water} \leqslant 30°c$ Brine consumption = 0.15 $10^6$ Kcal
Water consumption = 130 $M^3$

## TABLE 3-6

## RAW MATERIALS AND UTILITIES CONSUMPTIONS

### N- METHYLCHLOROACETAMIDE
per ton (100%)

| | |
|---|---|
| **RAW MATERIALS** | |
| Monochloroacetic acid | 1.0124 t |
| Monomethylamine | 0.3640 t |
| **SOLVENTS AND CHEMICALS** | |
| Cyclohexane | 0.060 t |
| P toluene sulfonic acid | 0.028 t |
| Caustic soda | 0.054 t |
| Dichloroethane | Accounted in dimethoate production |
| **UTILITIES** | |
| Steam 3 atm g | 3.2 t |
| Steam 10 atm | 0.3 t |
| Water | 31 $M^3$ * |
| Brine | 1.9 $10^6$ Kcal * |
| Power | 62 KWhr |

* If $T_{water} \leqslant 30°c$ Brine consumption= 0.65 Kcal
Water consumption = 156 $M^3$

TABLE 3-7

RAW MATERIALS AND UTILITIES CONSUMPTIONS

DIMETHOATE FROM INTERMEDIATE MATERIALS

per ton (90 %)

| | |
|---|---|
| **RAW MATERIALS** | |
| O-O dimethyldithiophosphoric acid | 0.8219 t |
| N methylchloroacetamide | 0.671 t |
| **SOLVENTS AND CHEMICALS** | |
| Sodium bicarbonate | 0.8552 t |
| Dichloroethane | 0.1229 t |
| Cyclohexanone | 0.020 t |
| Toluene | 0.031 t |
| **UTILITIES** | |
| Steam 3 atm g | 6.5 t |
| Steam 10 atm | 0.7 t |
| Water | 130 $M^3$ |
| Brine | 0.65 $10^6$ Kcal |
| Power | 115 KWhr |

## TABLE 3-8

## RAW MATERIALS AND UTILITIES CONSUMPTIONS

DIMETHOATE FROM RAW MATERIALS
per ton (90 %)

| | |
|---|---|
| **RAW MATERIALS** | |
| $P_2S_5$ | 0.6542 t |
| Methanol | 0.4036 t |
| Monochloroacetic acid | 0.6793 t |
| Monomethylamine | 0.2442 t |
| **SOLVENTS AND CHEMICALS** | |
| Toluene | 0.0631 t |
| Cyclohexanone | 0.04 t |
| Cyclohexane | 0.02 t |
| p toluene sulfonic acid | 0.019 t |
| Dichloroethane | 0.1229 t |
| Sodium hydroxide | 0.036 t |
| Sodium bicarbonate | 0.8552 t |
| **UTILITIES** | |
| Nitrogen | 9.9 $NM^3$ |
| Steam 3 atm g | 8.85 t |
| Steam 10 atm | 0.9 t |
| Water | 170 $M^3$ |
| Brine | $2.10^6$ Kcal * |
| Power | 177 KWhr |

* If $T_{water} \leqslant 30°c$ Brine consumption = 1.15 Kcal
Water consumption = 255 $M^3$

# 4 - EQUIPMENT SPECIFICATIONS

## 4-1- SECTION 100

C101 Vacuum jet - 35 Kg/hr of $H_2S$ at 20°c.
Section pressure : 200 mm Hg. Steam and water as propelling system. SS316

D101 $P_2S_5$ hopper 0.5 $m^3$.CS.

D102 Measuring tank - 1$m^3$. Vacuum (200 mm Hg).80°c.SS316 (slight amount of $H_2S$).

D103 Acid surge tank - 1.1 $m^3$. SS316

E101 Condenser - 2$m^2$ - Shell and tube - SS316 or graphite. CS on utility side.

E102 Cooler - 0.5 $m^3$ - double pipe - SS316 - CS on utility side.

H101 Horizontal Plate Filter.
80 Kg/batch (12 hours) - Filtration time : 2 hours
SS316.

P101 Acid pump. Centrifugal - 1.5 $m^3$/hr - 45 m - SS -

R101 Reactor - 1.6 $m^3$ agitated - Jacketed - Enamelled -

T101 Distillation column - Ø 250 mm - 1.8 meters of Pall ring 1" .SS316

## 4-2 - SECTION 200

D202 Neutralization solution tank - 850 liters - CS

D203 Liquid measuring tank - 1 $m^3$ - P design : 6 bar g . SS 304

D204 Hopper - 1.3 $m^3$ - SS304

D205 Reflux drum - 150 liters - vacuum - SS304

D206 Recovered solvents tank - vacuum - coil-2$m^3$ - CS

D207 Product tank - vacuum - coil - 2$m^3$ - SS304

D208 Solvent surge tank - 2$m^3$ - CS

D209 Water tank - vacuum - 200 liters - CS

E201 Falling film evaporator - 5 $m^2$ - Vacuum - SS316

E202 Condenser - 25 $m^2$ - Vacuum - SS304

E203 Vent condenser - double pipe-1.5 m - Vacuum - SS304

P201 Recirculation pump - 6$m^3$/hr - head : 40m - Volumetric - SS316

P202 Product pump 3$m^3$/hr - Head : 20 m - centrifugal cast iron

R201 Reactor - 3.5 $m^3$ agitated - jacketed - coils : 250 m Vacuum - SS304

T201 Distillation column - Ø : 0,5 m - 3 meters of Pall ring 1" - Vacuum - SS304

## 4-3 - SECTION 300

D301 Liquid measuring tank - 1.5 $m^3$ - SS316
D303 Neutralization solution tank - 350 liters - CS
D304 A/B Solvents tanks - 2.3 $m^3$ - Coiling coil (0.4 $m^2$) - Vacuum - CS
D305 Product tank - 0.5 $m^3$ - Coil - Vacuum - SS
D306 Unconverted reactants tank - 0.3 $m^3$ - Vacuum - SS
D307 Crude product surge tank - 1.2 $m^3$ - Vacuum - Coil - SS
D308 Water tank - Vacuum - 100 liters - CS

E301 Vent condenser - double pipe - L : 1.5 m - SS
E302 Idem to E301
E303 Wiped film evaporator - 2 $m^2$ - Vacuum - SS
E304 Condenser - 10 m2 - Vacuum - SS
E305 Vent condenser - 1 $m^2$ - Vacuum - CS
E306 Condenser - 2 m2 - Vacuum - CS

H301 Metal belt flaker - 1m x 5m - cooling by water and brine - covered - sheet SS
H302 Centrifuge filter system - SS 304 shell

P301 Acid pump - centrifugal 6$m^3$/hr - Head : 20 m - SS316
P302 Centrifugal - 2$m^3$/hr - Head : 20 m - SS316
P303 Centrifugal - 3 $m^3$/hr - Head : 40 - SS316
P304 Volumetric - 2$m^3$/hr - Head : 25 - SS316
P305 Centrifugal - 4 $m^3$/hr - Head : 20 - Cast iron.

R301 Extraction tank - 3.4 $m^3$ - agitated SS
R302 Reactor - 2$m^3$ - agitated - jacketed - enamelled
R303 Reactor - 3$m^3$ - agitated - jacketed - steam Introduction pipe - SS316
R304 Solvent evaporator - 2.8$m^3$ - jacketed - vacuum SS316

## 4-4 - SECTION 400

C401 Vacuum pump - multistage steam jet with surface condenser - Flow of air : 10 Kg/hr - Suction pressure: 10 mm Hg - castIron and carbon steel

D401 Acid storage tank : 10 $m^3$ - SS
D402 Crude toluene storage tank - 5 $m^3$ - CS
D403 Waste water tank - heating coil 50 meters - 3.5$m^3$-CS
D404 Distillate receiver - 300 liters - CS
D405 Hot well - vertical tank - Flat bottom - 100 liters - removable cover - CS

E403 Column condenser - 4$m_2$ - Shell and tube -CS

P401 - Acid storage pump - Centrifugal - 1.5 $m^3$/hr - Head : 25 m - SS316

T403 Distillation column - Ø :0.3m - 3 meters of Pall ring 1" - CS

# 5- INVESTMENTS

## 5 - 1. INTRODUCTION

The investments have two sources :

1/ A total budget of 800.000 US $ is provided by the United Nations Development Program.

It has been shown that 100 000 US$ are devoted to the consultant personnel costs.

The 700 000 US$ left will be shared into :

- 100 000 US$ for the engineering book cost
- 150 000 US$ for know-how fees
- 450 000 US$ for equipments, piping instrumentation to be imported

2/ A total budget of 1448 000 Egypt £ (1 766 000 US$) is provided by the Egyptian'Governement.
This budget is provided for all the local expenses

## 5 - 2. EQUIPMENT COSTS

See Table 5-1 for the item costs. The total is :

350 000 US$ for UNOP budget
130 000 US$ for Egyptian Government

## 5 - 3. INSIDE BATTERY LIMIT INVESTMENTS

See Table 5-2 for the detailed calculations. The ISBL is :

700 000 US$ for UDNP budget
350 000 US$ for Egypt Government.

## TABLE 5-1

## EQUIPMENT COSTS in US$

| ITEM | UNDP budget | Egypt.Gov.budget |
|---|---|---|
| C101 | 1 000 | |
| C401 | 8 000 | |
| D101 | | 1 500 |
| D102 | 7 000 | |
| D103 | 7 000 | |
| D202 | | 2 000 |
| D203 | 7 000 | |
| D204 | 8 000 | |
| D205 | 2 500 | |
| D206 | | 7 000 |
| D207 | 10 000 | |
| D208 | | 7 000 |
| D209 | | 1 000 |
| D301 | 8 000 | |
| D303 | | 1 000 |
| D304 A/B | | 16 000 |
| D305 | 6 000 | |
| D306 | 4 000 | |
| D307 | 8 000 | |
| D308 | | 600 |
| D401 | 15 000 | |
| D402 | | 8 000 |
| D403 | | 12 000 |
| D404 | | 1 000 |
| D405 | | 600 |
| E101 | 2 000 | |
| E102 | 600 | |
| E201 | 10 000 | |
| E202 | 15 000 | |
| E203 | 500 | |
| E301 | 500 | |
| E302 | 500 | |
| E303 | 30 000 | |
| E304 | 7 000 | |
| E305 | | 1 500 |
| E306 | | 2 000 |
| E403 | | 3 000 |
| H101 | 10 000 | |
| H301 | 15 000 | |
| H302 | 20000 | |
| P101 | | 2 500 |
| P201 | 3 000 | |
| P202 | | 1 500 |
| P301 | | 3 000 |
| P302 | | 2 000 |
| P 303 | 3 000 | |
| P304 | 2 500 | |
| P305 | 3 000 | |
| P401 | 2 500 | |
| Storage Pumps | 5x1000 = 5000 | |
| R101 | | 21 000 |
| R201 | 30 000 | |
| R301 | 20 000 | |
| R302 | | 24 000 |
| R303 | 25 000 | |
| R304 | 20 000 | |
| T101 | 5 000 | |
| T201 | 15 000 | |
| T403 | 8 000 | |
| | 326 000 | 119 200 |
| Inspection and purchasing cost | 24 000 | 10 800 |
| TOTAL | 350 000 | 130 000 |

TABLE 5-2

INSIDE BATTERY LIMITS INVESTMENTS US$

| | UNDP | Egypt Govert |
|---|---|---|
| Purchased Equipment | 360 000 | 130 000 |
| Transport | | 50 000 |
| Equipment handling (6%) | - | 30 000 |
| Buildings (new insvestments) | - | 80 000 |
| Piping 10% | 40 000 | 10 000 |
| Instrumentation<br>Lab. and safety equipments | 30 000 | 10 000 |
| Insulation | - | 20 000 |
| Electrical | | 10 000 |
| Contingencies | 30 000 | |
| Direct costs | 450 000 | 350 000 |
| Know-how | 150 000 | |
| Engineering | 100 000 | |
| Total costs | 700 000 | 350 000 |

## 5 - 4. WORKING CAPITAL

The working capital is equal to 240 000 US$.

## 5 - 5. OUTSIDE BATTERY LIMIT INVESTMENTS

The project budget Government contributionis in total of 1 766 000 US$.

The inside battery limit investments as equal to 350 000 US$.
The working capital is equal to 240 000 US$

The difference (1 176 000 US$)is considered as equivalent to outside battery limit investments.

# 6 - COSTS OF PRODUCTION

## 6 - 1. INTRODUCTION

As the malathion and the dimethoate are produced in the same unit, all the costs due to the equipment use and the labor will be shared in accordance to the time required for the production of these two compounds.

The unit will be shared to produce 180 tonnes of malathion and 120 tonnes of dimethoate into :

- 40 % for malathion
- 60 % for dimethoate

## 6 - 2. MAINTENANCE COSTS

By taking the usual figures obtained in DCC , these maintenance costs are evaluated at 20 000 US$/year.

## 6 - 3. OVERHEAD COSTS

The general plant overhead is in DCC equal to 40 % of the operating costs.
The property taxes are negligible.
The Insurance on the whole building is equal to 4 000 US$/year.As only a quarter of the building will be used, 1 000 US $/year is the building insurance cost for the projected unit.
The Insurance on the equipment is equal to 8 000 US$/year.
The depreciation in DCC is equal to 5.5 % of the total investments.

## 6 - 3. COST OF PRODUCTION

The raw materials and utilities costs are based on the raw materials and utilities consumptions (Table 3-5 and 3-8) and on the raw materials and utilities costs (see Annex 2).

The labor costs are given in Annex 2

The production costs are summarized in :

- Table 6-1 for malathion
- Table 6-2 for dimethoate

## TABLE 6-1

## PRODUCTION COST SUMMARY

MALATHION (97 %)-180t/year

| Raw materials and utilities | US$/t Malathion 97% | US$/year |
|---|---|---|
| Raw materials costs | 1 151 | 207 180 |
| Chemicals and solvent costs | 47 | 8 460 |
| Utilities | 57.5 | 10 350 |
| Total | 1 255.5 | 225 990 |
| **Operating cost** | | |
| Labor costs | 124 | 22 400 |
| Maintenance | 45 | 8 000 |
| Total operating | 169 | 30 400 |
| **Overhead expenses** | | |
| General plant overhead (40% of operating costs) | 68 | 12 160 |
| Insurance building | 18 | 400 |
| Equipment | | 3200 → 3 600 |
| Depreciation (5.5% of total unit) | 165 | 29 700 |
| Total overhead | 251 | 45 460 |
| PRODUCTION COST ..... | 1 677 | 301 850 |
| Based on 100 % AI .. | 1 729 | |

## TABLE 6-2

## PRODUCTION COST SUMMARY

DIMETHOATE (90 %) - 120t/year

| Raw materials and utilities cost | US$/t Dimethoate 90 % | | US$/year |
|---|---|---|---|
| Raw materials costs | 1 758 | | 210 960 |
| Chemicals and solvent costs | 407 | | 48 840 |
| Utilities | 144 | | 17 280 |
| Total raw | 23 091 | | 277 080 |
| **Operating cost** | | | |
| Labor costs | 280 | | 33.600 |
| Maintenance | 100 | | 12 000 |
| Total operating | 380 | | 45 600 |
| **Overhead expenses** | | | |
| General Plant Overhead (40% of operating cost) | 152 | | 18 240 |
| Insurance building | 45 | 600 | 5 400 |
| Equipment | | 4800 | |
| Depreciation (5.5% of total investments) | 371 | | 44 550 |
| Total overhead | 568 | | 68 190 |
| PRODUCTION COST ..... | 3 257 | | 390 870 |
| Based on 100 % A.I. | 3 619 | | |

# 7 - CONCLUSION

## 7-1. SITE

Previous reports have shown that the DCC factory is the best site to settle the unit.

During the present mission this choice has been confirmed. All the facilities like effluent treatment, available utilities, buildings give a low production cost and the skilled personnel is an insurance for high capacity utilization of this unit. Furthermore, the operators are used to dangerous products manipulation and then little effort will be required for the present productions.

## 7-2. TONNAGE

The mentionned productions of 120t/year of dimethoate and 180t/year of malathion correspond to the expected consumptions increase in Egypt. Without any solid experience in the insecticide production, Egypt should not seek to abrupty eliminate imports at current consumption level.

A smaller unit (like 40t/year) would have brought the cost of production to the current prices of the imported compound.

In addition the calculations have shown that the budgets (both UNDP and Egypt Government budgets) are sufficient for such a tonnage.

## 7-3. TYPE OF UNIT

Malathion and dimethoate are two insecticides with low toxicity . Their consumptions in Egypt are and will stay at a high value. (American Cyanamid, Montecatini still invest in rather large unit).

The flexibility of the unit is such that almost all the other organophosphorous insecticides may be produced in this unit with little modifications.

Hence this unit will be profitable during a first period and will be a good basis to establish a large unit for malathion and dimethoate and to experiment productions of other organophosphorous insecticides.

## 7-4. FINANCIAL ASPECTS

The calculated production costs are :

| | | | |
|---|---|---|---|
| malathion | (97%) | 1 730 US$/ton | (100% A.I.) |
| dimethoate | (90%) | 3 620 US$/ton | (100% A.I.) |

The current prices are :

| | | | | |
|---|---|---|---|---|
| malathion | 3 000-3 400 | US$/ton | (10%) A.I. | non formulated |
| dimethoate | 5 000 | US$/ton | " | " |

These figures are so positive that we have to recall that the low costs of production are due mainly to the facilities of DCC. It should be noticed furthermore that the foreign currencies saving for Egypt will be equal to about 700 000 US$/year.

However the above mentionned production costs are calculated with recent technology . It is probable that the guaranties given by the know-how supplier will give higher production costs (higher raw material consumptions).

This increase will not exceed 500 to 600 US$/ton and will still leave a reasonnable margin for profits and a foreign currencies saving of about 500 000 - 600 000 US$/ year.

## 7-5. FORMULATION UNIT

The formulation unit is not included in the present project. But it has been noticed that :

- the Kafr El Zayat factory is only doing the packaging of already formulated dimethoate and malathion. Its formulation unit is used for other insecticides.

- the formulation unit may be reduced to a mixing tank, two or three hand pumps and a balance.

- the usual solvent for the formulated product is kerosene which is a local and inexpensive product (the surface active ingredients to be added are the only materials to be imported).

Hence it would be advisable to have a close look at this point and to see if such a formulating unit is necessary.

## 7-6. CONCLUSION

This project is the first step of a long term project . This first step will give the necessary experience for any future large capacity unit project. This experience will give to Egypt a very good position towards any foreign partner.

In addition, this first step will have a significant effect on Egypt economy.

Hence this project has to be considered as a priority.

# ANNEX I

## SAFETY

In such a plant, safety involves hazards generally associated with chemical operations, plus some specific problems.

### 1. SPECIFIC PROCESS HAZARDS

One process step requires specific precautions to be incorporated in the plant design and lay-out. It is the reaction between methanol and $P_2S_5$ to give dimethylthiophosphoric acid, which is the first step for various organophosphorous insecticides.

This reaction is exothermic and can easily get out of hand, and even becomes explosive.

Two features have been incorporated in plant design to prevent accidents and/or minimize their consequences.

#### a. Alarm systems

The mixture temperature is measured and indicated.
A temperature alarm is actuated when :

- the temperature reaches a preset limit
- the rate of temperature rise (obtained by differentiating the temperature versus time curve) tends to go above a preset limit.

In this manner, a dangerous acceleration of the reaction can be immediately detected and stopped, which is more effective than a simple high temperature alarm which can be already late when it starts.

#### b. Enclosures

Reactor R101 is installed within a reinforced concrete cubicle in order to contain the detrimental effects of an explosion

ANNEX I (cont.)

to other buildings. One side of the cubicle is open with a dyke in front of the opening , in order to stop any flying debris in case of explosion.

Instrumentation is such that no personnel is authorized within the cubicle while the reaction is in progress, and all useful indications are legible on a control panel located outside the cubicle. All operating parameters will be controlled by means of remotely operated hand control stations located on that panel.

## 2. GENERAL CHEMICAL PLANTS HAZARDS

Most of materials handled are flamable and/or toxic.

For this reason, each process area and storage areas are dyked. The dykes containing storage tanks are of such volume that they can contain the whole volume of the largest tank included there.

Fire-fighting facilities are provided.

Personnel working within the process buildings is proviced with the usual equipment plus gas masks in more critical area.

# ANNEX 2

## ECONOMICAL FIGURES

### 1. RAW MATERIALS, CHEMICALS AND SOLVENTS COSTS

| | | US$/ton (100% basis) |
|---|---|---|
| $P_2S_5$ | : imported | 1 300 $/ton |
| Methanol | : local | 250 $/ton (if imported 350) |
| Toluene | : imported | 400 $/ton |
| Caustic soda | : imported | 480 $/ton |
| Ethanol | : local | 260 $/ton |
| Maleïc anhydride | : imported | 1 400 $/ton |
| Dichloroethane | : imported | 350 $/ton |
| P toluene sulfonic acid | : imported | 1 500 $/ton |
| $H_2SO_4$ (96%) | : local | 70 $/ton |
| $Na_2CO_3$ | : local | 200 $/ton |
| Na $HCO_3$ | : local | 270 $/ton |
| NaCl | : local | 10 $/ton |
| Monochloroacteic acide | : imported | 900 $/ton |
| Monoethylamine | : imported | 800 $/ton |
| Cyclohexanone | : imported | 1 200 $/ton |
| Cyclohexane | : imported | 700 $/ton |

## 2. UTILITIES COSTS

(source = DCC)

| | |
|---|---|
| Compressed air | 20 US cts/$m^3$ |
| Power | 4 US cts/Kwhr |
| Water | 3 US cts/$m^3$ |
| Steam | 3 US $/ton |
| Nitrogen | 25 US cts/$Nm^3$ |
| Brine | 50 US$ /$10^6$ Kcal |

## 3. LABOR COSTS

Basis :

- 7 operators and 1 foreman per shift
(28 operators and 4 foremen in total)

- Supervisor 1 man.

This basis gives on DCC source a yearly cost of labor of 56 000 US $

# ANNEX 3

## PRELIMINARY LAY-OUT

### 1/ - Buildings sizes

Roof at 13.2 metres for the two rooms

| | | |
|---|---|---|
| 1st room | 6.5 m x 15 m | at level 0.0 |
| | 6.5 m x 18 m | at level 6.6 |
| 2nd room | 6 x 6 | at level 0.0 |
| | identical | at level 6.6 |

For the two rooms, steel structure may be added at level 3.3 and 9.9

### 2/ Principles of the lay-out

Ground level

R101 the methanol $P_2S_5$ reactor will be in an outside specific reinforced cubicle as described in Annex 1.
The acid storage tank (D401) and crude toluene tank (D402) will be outside.
The metal belt flaker (H301) for the dimethoate will be in an outside light building to be provided.
All the other equipment to be at the ground level will be inside one of the two rooms.

they are :

| | | Volume | Estimated diameter | |
|---|---|---|---|---|
| Section 100 | D103 | 1.1 $m^3$ | 0.9 m | room N°1 |
| | H101 | 1 m x 1 m | - | room N°1 |
| Section 200 | D206 | 1.8 $m^3$ | 1,1 m | room N°2 |
| | D207 | 2 $m^3$ | 1.2 m | room N°" |
| | D208 | 2 $m^3$ | 1.2 m | room N°" |
| | R201 | 3.5 $m^3$ | I.4 m | room N°" |
| Section 300 | D304 | 2.3 $m^3$ each | 1.3m | room N°1 |
| | R301 | 3.4 $m^3$ | 1.4m | " " |
| | R302 | 2 $m^3$ | 1.2 m | " " |
| | R303 | 3 $m^3$ | 1.4 m | " " |
| | R304 | 2.8 $m^3$ | 1.4 m | " " |
| Section 400 | D403 | 3.5 $m^3$ | 1.4 m | " " |

see figure 6003 SKE 001 and 6003 SKE 002

other levels

All the vessels from which reagents are charged by gravity are at the level 6.6

Room N° 2 - Section 200

Two items have to be embedded
-D204 to facilate the solids manipulations
-T201 to respect the gravity flow from

E202 (condenser)
D205 (reflux drum)
T201 (distillation column)
R201 (reactor)

All the other equipment are installed on the 6.6 level (D202_D203-E201-D209).

Room N° 1

SECTION 100

D101 - D102 and T201 will be installed on the 6.6 level while E101 will be slightly above the column T201. If the required height exceeds the possible one, T201 will be embedded.

SECTION 400

D403 will be installed on the 6.6 level.

Due to the flow gravity between E403 and T403 it may be necessary to embed the column T403.

SECTION 300

D301 - D303 - D306 - D307 are installed on the 6.6 level while for E303 it is required to have a gravity flow from it to D307 and D305.

E306 will be at any convenient place above D308 installed on the 6.6 level or 0.0 level.

E304 and E305 will be above D306.

See figure 6003 SKE003 and 6003 SKE004.

# ANNEX 4

The next step of the project consists into inviting companies capable of selling their know-how.

The invitation letter will include terms of reference with DCC facilities description, general conditions, know-how contractor duty , engineering contractor duty.

## 1 - DCC FACILITIES DESCRIPTION

### UTILITIES

| | | | | |
|---|---|---|---|---|
| Compressed air | 4atmg | 60N $^3$/hr | 20% | is available for the unit. |
| Nitrogen | 3atmg | 150NM$^3$/hr | 10% | " " " |
| Steam | 3atmg | 30t/hr | 50% | " " " |
| Brine (15% NaCl) | 3atmg-15°c | 2.10$^6$Kcal/day | 90% | " " |
| Central vacuum | 0.1atm | 150NM$^3$/hr | -exit gases are scrubbed by water in a bubbling tank. | |
| Water | - | 6atm - temperature from 10°c to 35°c | | |
| | - | 150mg/l $Na_2CO_3$ total alkalinity | | |
| | - | as required. | | |

Note : Steam at 16atmg maximum may be available

### EFFLUENTS TREATMENT

An aqueous wastes treatment unit (with lime ) is available and its capacity is very large.

### WORKSHOP

The workshop is able to do carbon steel equipments, excepted the huge and high pressure vessels. It has drilling, boring, lessing, rolling, shearing, bending machines, a 50t press , a barrel manufacture.

It is able to work with rubber, ceramic, head lining. SS and CS welding is done.

## 2 - GENERAL CONDITIONS

### BUILDING

Two rooms are available with 2 antiacid floors at level 0,0m and 6,6 m. The roof is at the level 13.2 meters. Their sizes are :

1/ 15 x 6.5 m at level 0,0 and 18 x 6,5 at level 6,6.

2/ 6 x 4,0 " " and level 6,6

Note : Any outside and inside structure may be added.

- Inside area 260 m2
- Outside area 600 m2

The lightings and other units in the building are antiexplosion proofs (Polish code).

### TYPE OF PRODUCTION

The two products will be done by campaigns in the same vessels. The design will be based on 3 shifts per day, 7 days a week, 7200 hours per year schedule with a 20-30 % over capacity.

The production of 120 t/yr of dimiethoate and 180 t/yr of malathion is required.

The FAO specifications to meet :

MALATHION :

Aspect : clear, colourless to light amber liquid
Purity : 94 % w/w minimum
Impurities : Acetone insolubles 0.5% w/w maximum
Acidity (as $H_2SO_4$) 0.5% w/w maximum
Iron 10 mg/kg maximum
Water 0.1% w/w maximum

DIMETHOATE :

Aspect : white crystalline powder or flakes
Purity : 87 % w/w minimum
Impurities : Omethoate 0.5% w/w maximum
Mineral acidity (as $H_2SO_4$) 1.0% w/w maximum
Acetone insolubles 0.5% w/w maximum
Water 0.5% w/w maximum

OTHER CONDITIONS

The instrumentation has to be minimized but not detrimental to safety.

The raw material storages are either in drums or bags except for the $P_2S_5$ tote bins.

Caustic soda solution (40%) is available (pipe).
Unnecessary storage of the intermediates like N-methylchloroacetamide acid ethylmaleate have to be avoided while an acid storage is admitted.

Liquid drums are discharged by hand pump fixed on the drum and solid bags are poured into the hoppers manually (except $P_2S_5$.

Equipment is available from the DDC'S store like two enamelled reactors (1600 and 2000 liters), several centrifugal SS pumps. ....

## 3 - KNOW-HOW CONTRACTOR DUTY

The company selling the know-how has to :

1/ - Give a process book containing :

- detailed process description
- equipment sketches and specifications
- Pipe and intrumentation lists
- safety procedures and safety equipment lists.
- control analysis procedures (raw materials, intermediates, products) and laboratory equipment list.

This process book will enable the Engineering contractor to give the detailed engineering book and the DCC to conduct safe production as required in General Conditions.

2/ - Garanty the raw materials and Utilities consumptions and requirements given in the contract.

3/ - Give a training to one or two Egyptian engineers in a dimethoate and malathion unit.

4/ - Offer technical experts for a 3 man-months period for performance tests and trial period on project costs while extra period will be at the know-how contractor's expense.

5/ - Offer experts on consultation contract if required.

6/ - Approve the Engineering book.

## 4 - ENGINEERING CONTRACTOR

The engineering contractor has to :

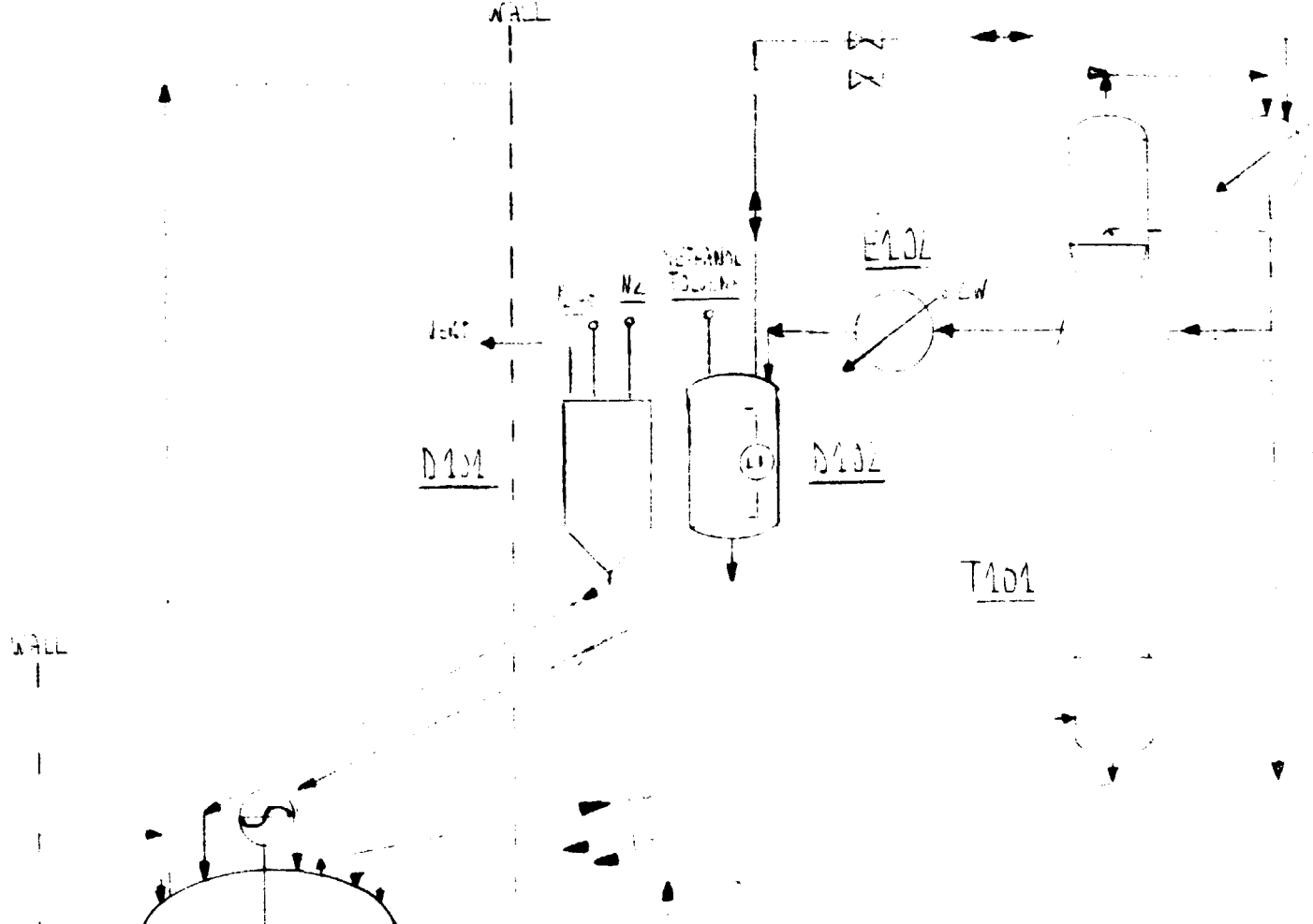
1/ - Give an engineering book containing all the necessary information required by the purchaser or the DCC workshop.

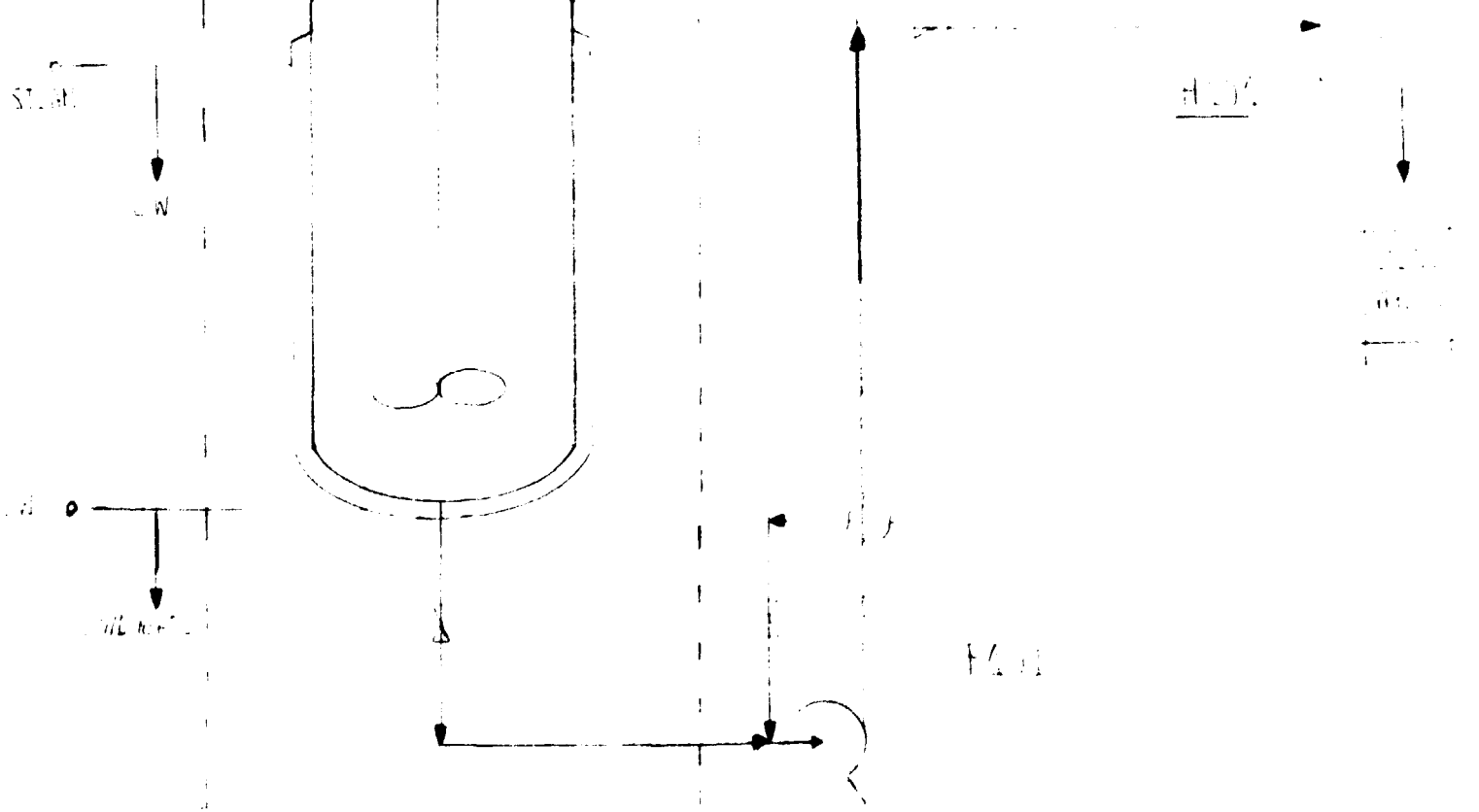
2/ - Give detailed lay-out and piping designs for erection of the unit by DCC with the necessary modifications to be done by DCC.

3/ Have its engineering book approved by know-how contractor

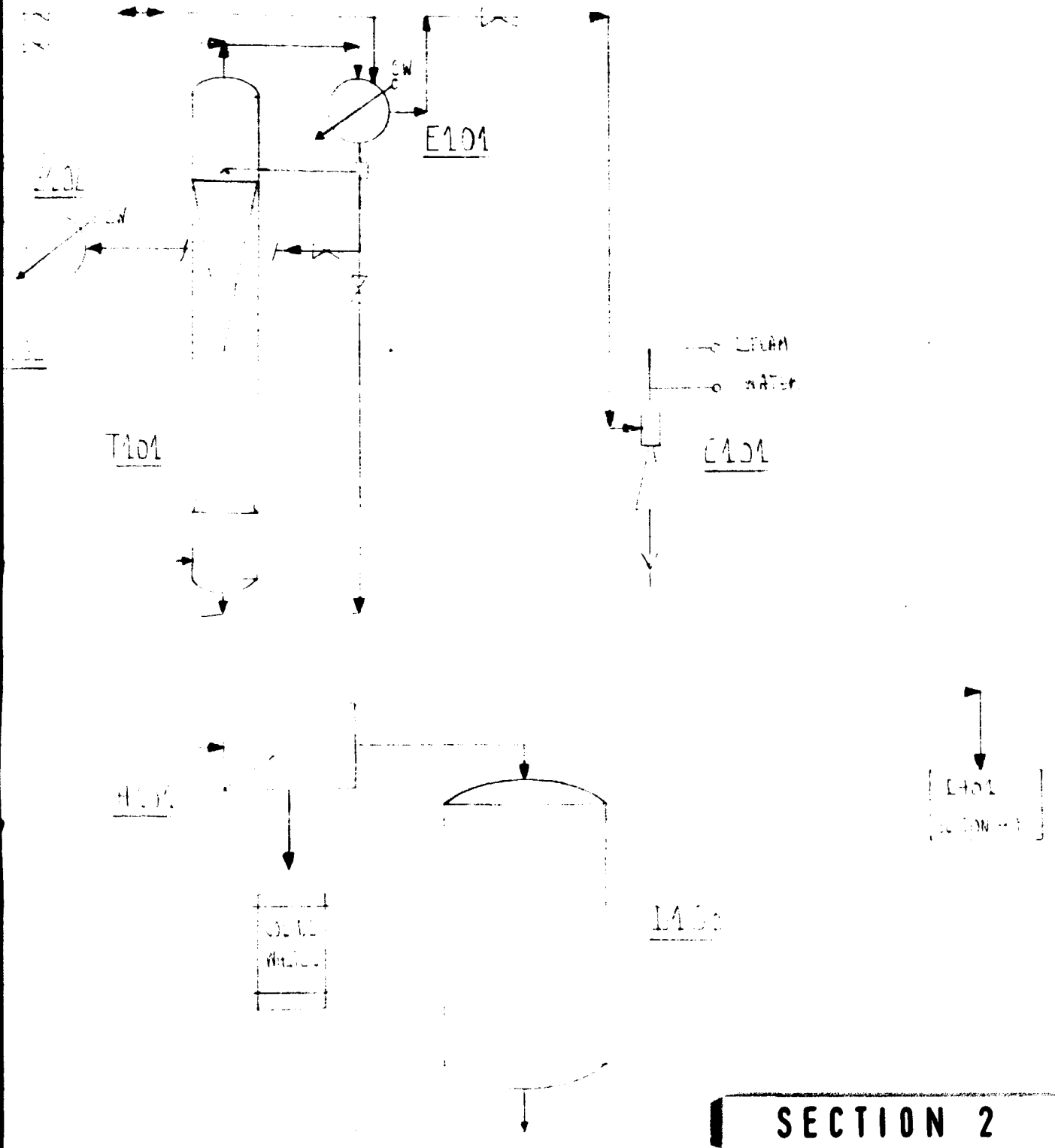
4/ - Offer assistance as required during the purchasing procedure (including inspection) at project costs.

NOTE : The DCC will collaborate in the preparation of the Engineering design under the supervision of the Engineering Contractor by providing 6 man-months of an engineer.


WALL
E102
CW
N2
D101
D102
T101
WALL



SECTION 1




# SECTION 2

CHIMIE DEVELOPPEMENT INTERNATIONAL

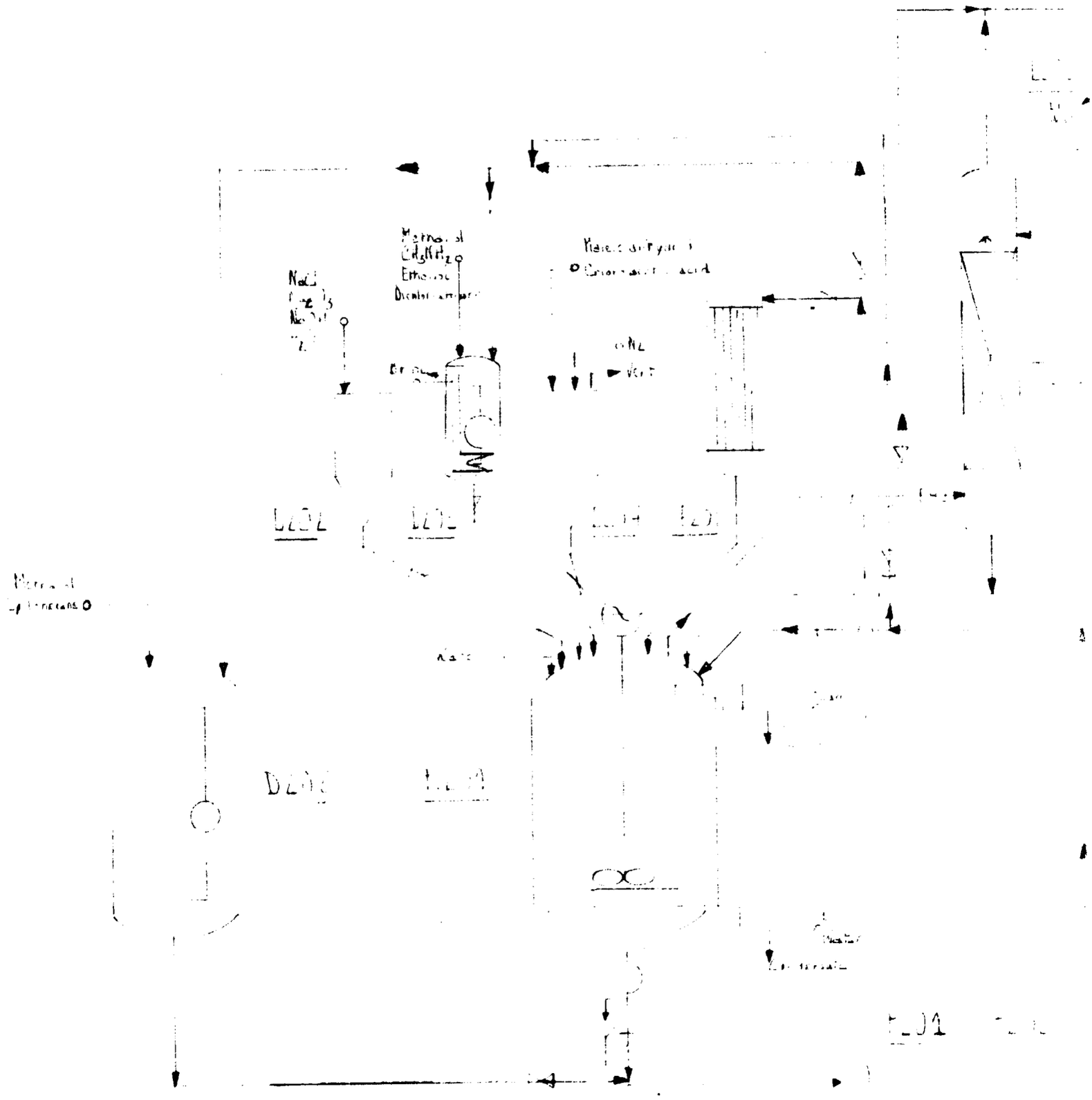
MALATHION-DIMETHOATE UNIT

SECTION 100

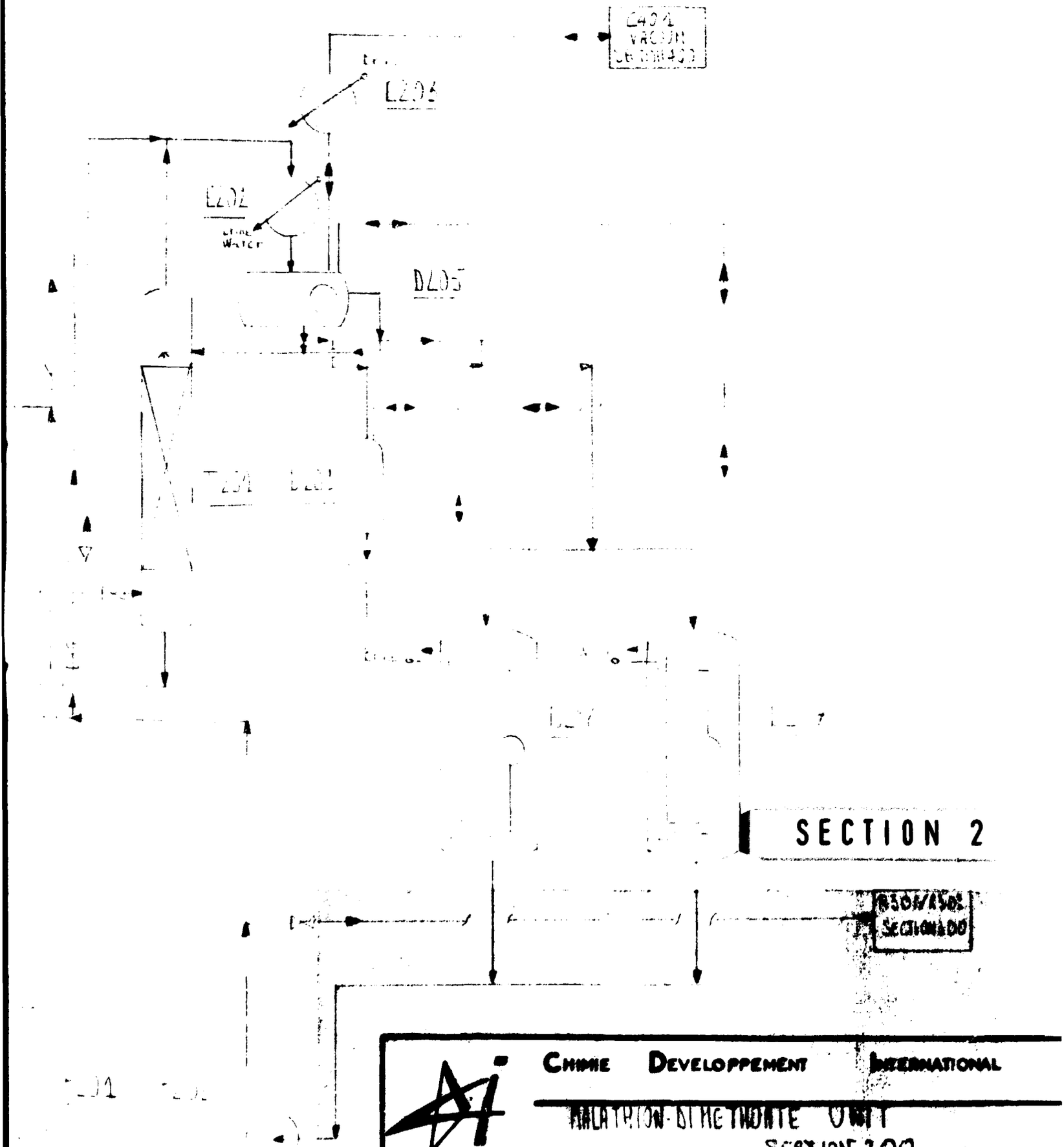
DESSINE PAR:

DATE:

CLIENT:



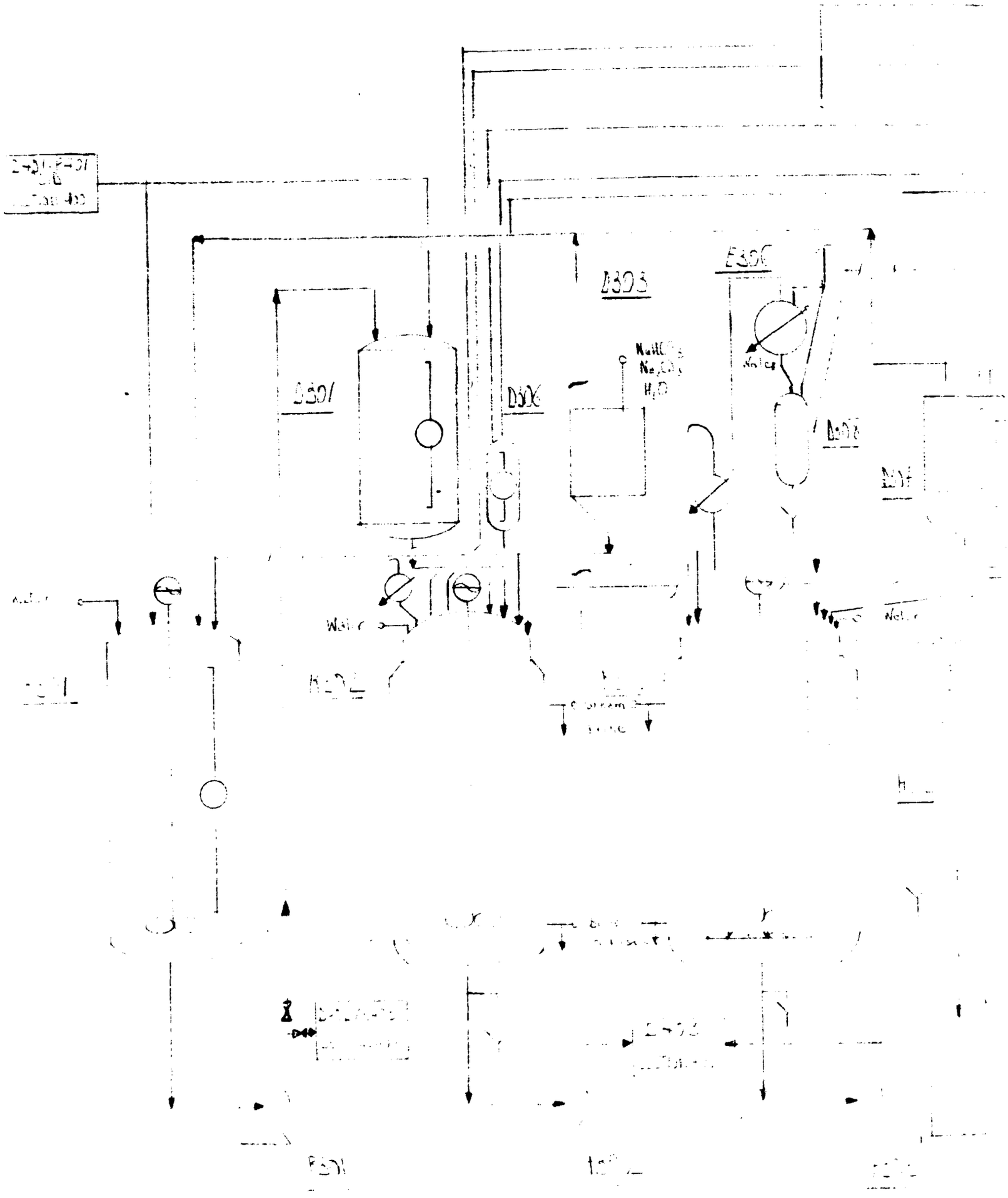
SECTION 1



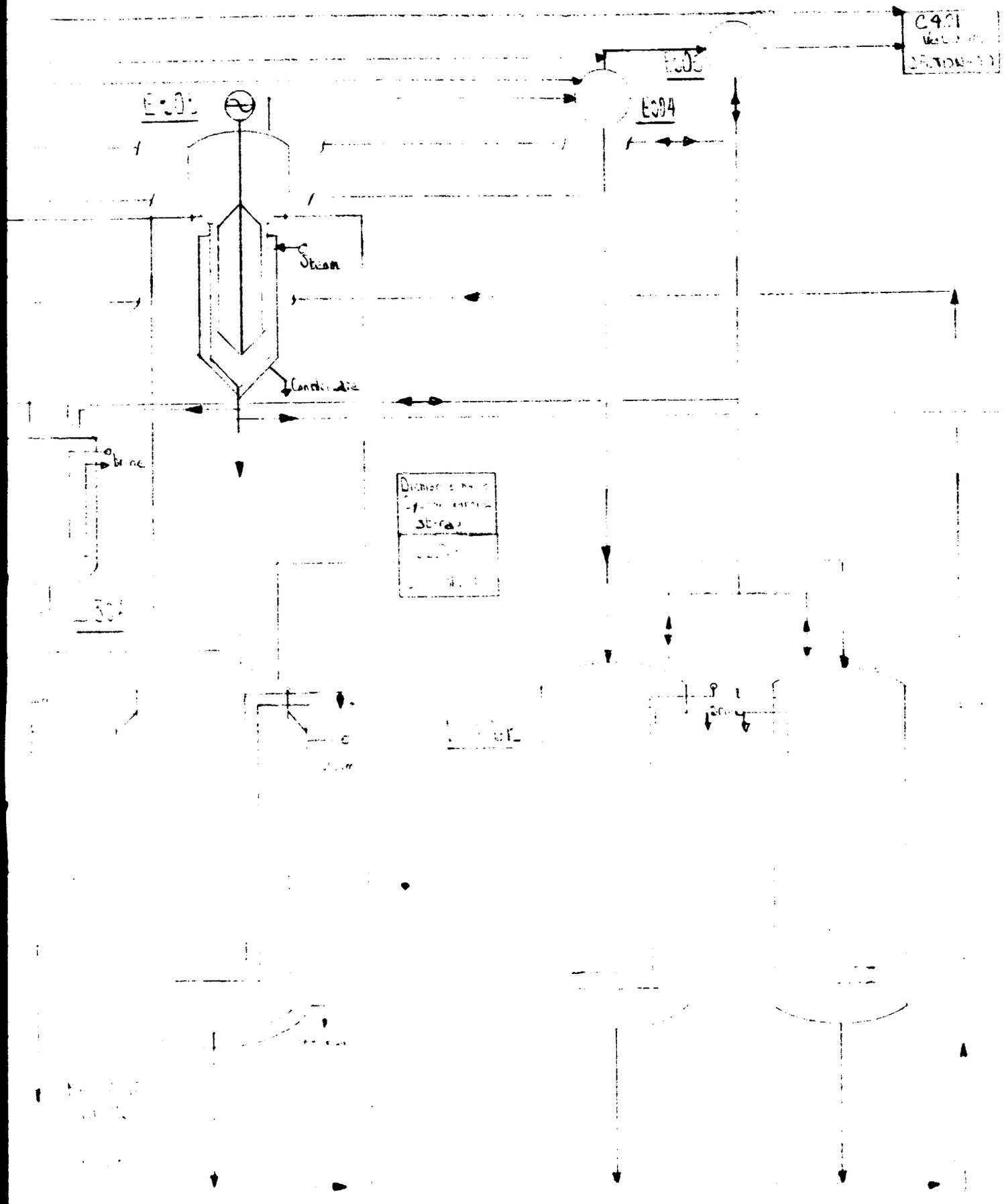

CHIMIE DEVELOPPEMENT INTERNATIONAL

MALATHION-DIMETHOATE UNIT
SECTION 200

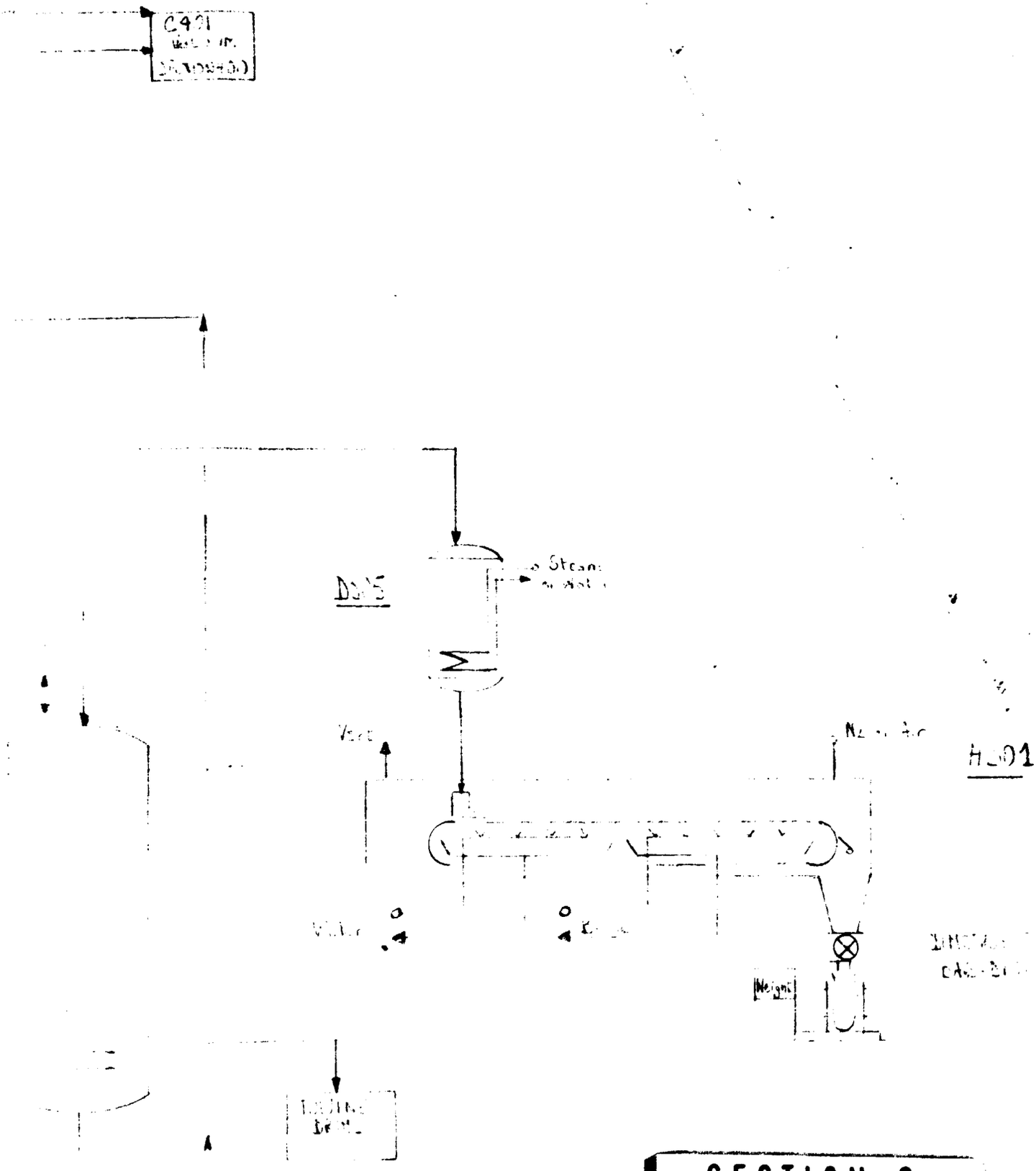
| | | |
|---|---|---|
| DESSINE PAR: EI<br>DATE: 9/11/11 | CLIENT:<br>UNIDO | 6003-PX-200 |



# SECTION 1




SECTION 2




# SECTION 3

CHIMIE DEVELOPPEMENT INTERNATIONAL

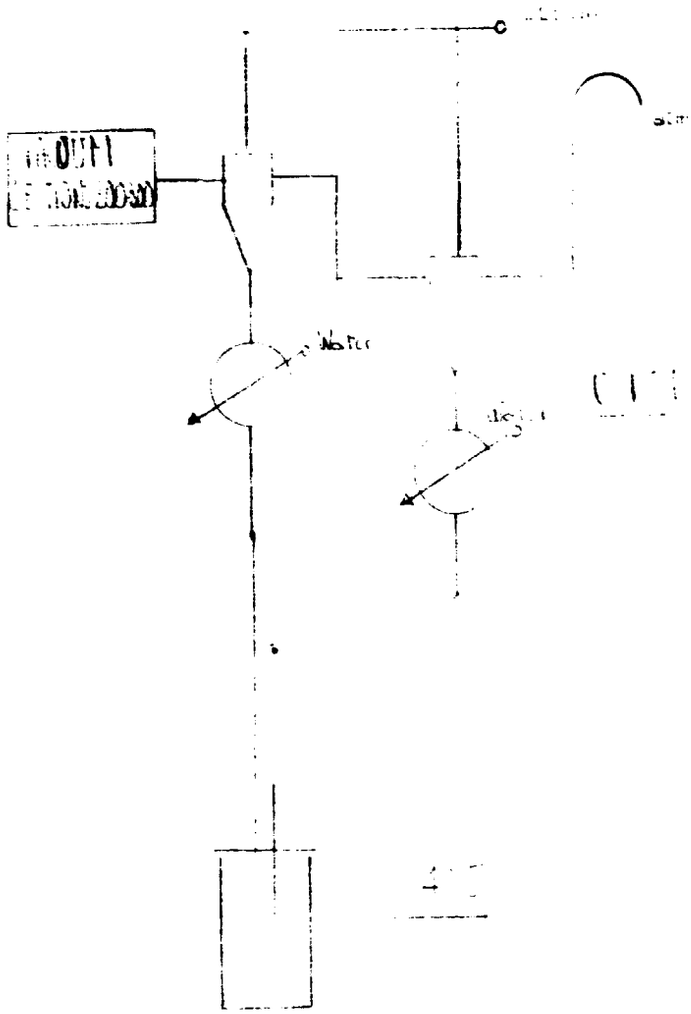
MALATHION DIMETHOATE UNIT

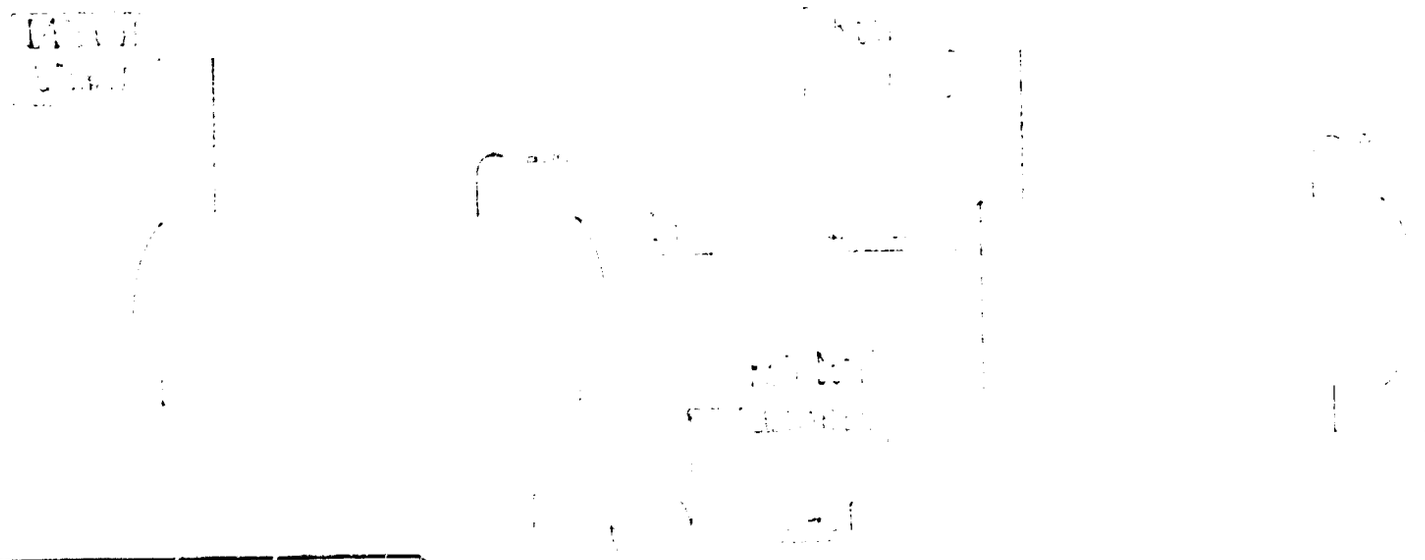
SECTION 200

DESSINE PAR: E.D

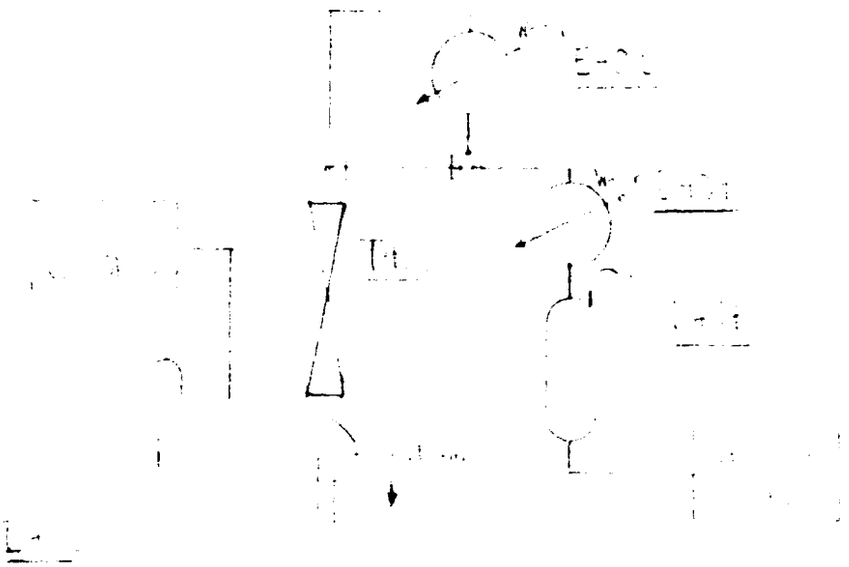
DATE:

CLIENT:





SECTION 1



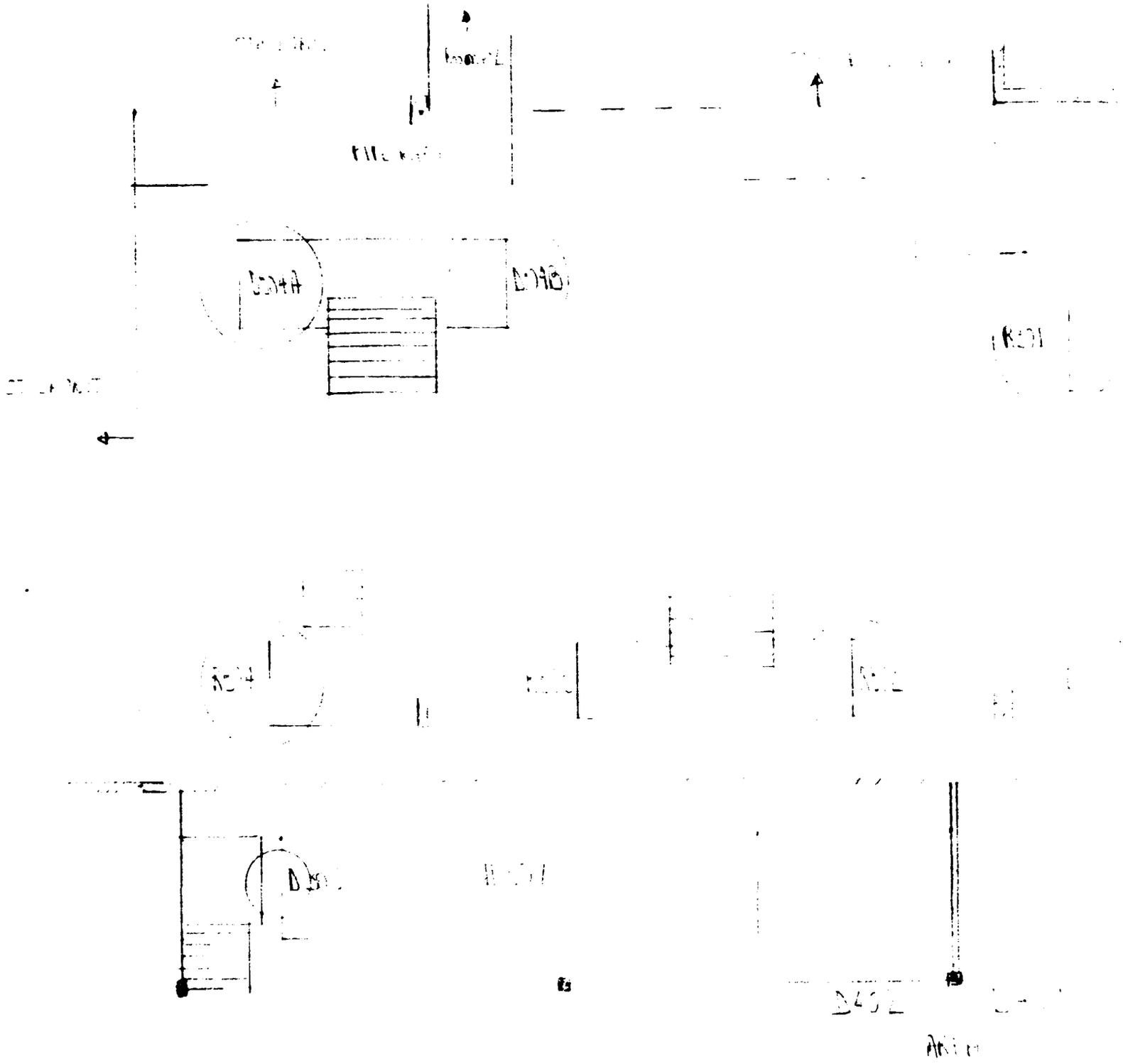
# SECTION 2

CHIMIE DEVELOPPEMENT INTERNATIONAL

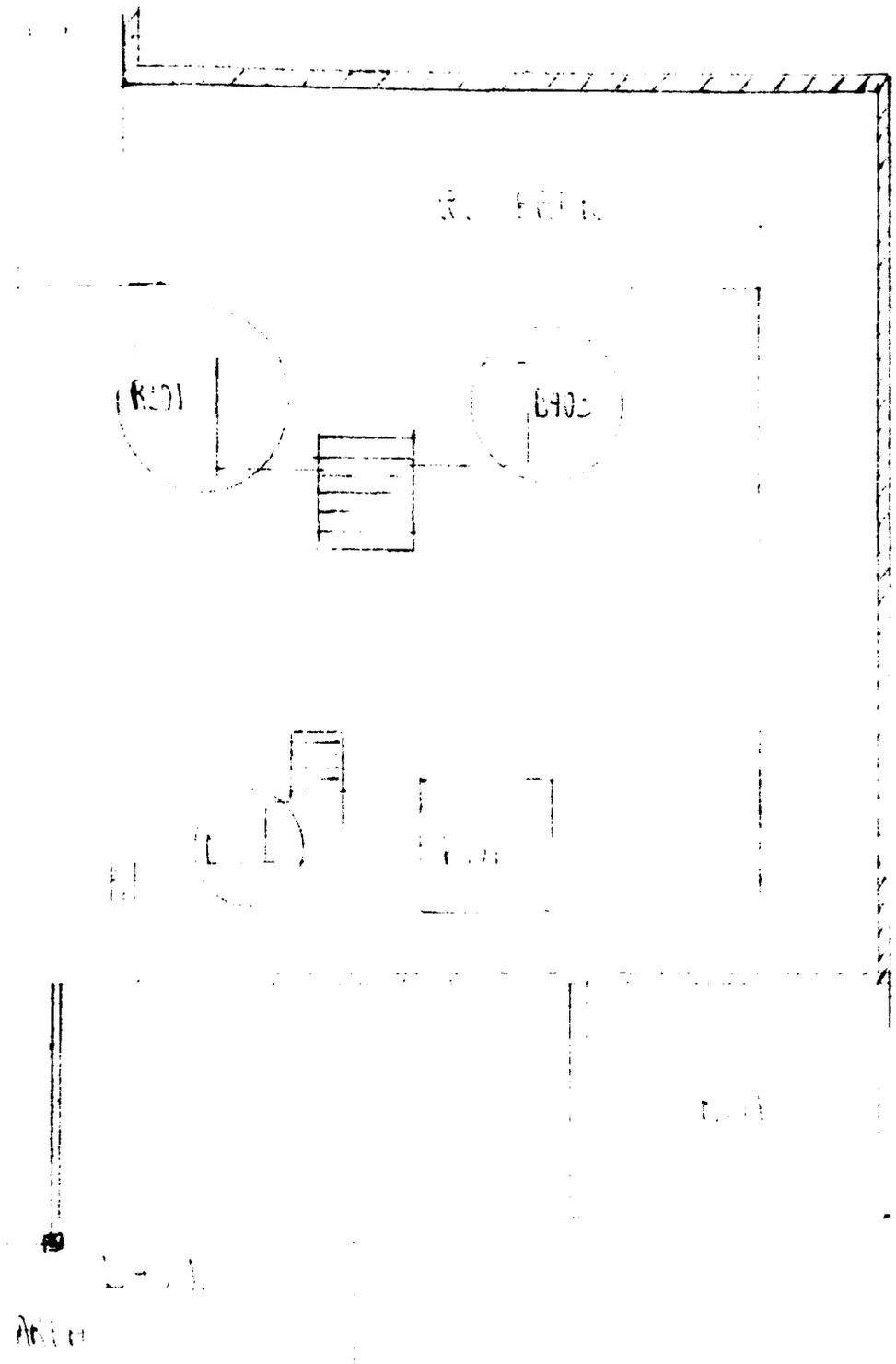
DESSINE PAR:

DATE:

CLIENT:



SECTION 1



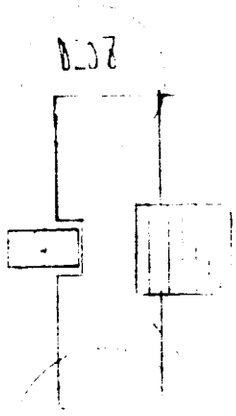
# SECTION 2

CHIMIE DEVELOPPEMENT INTERNATIONAL

DESSINE PAR:

DATE:

CLIENT:



SECTION 1

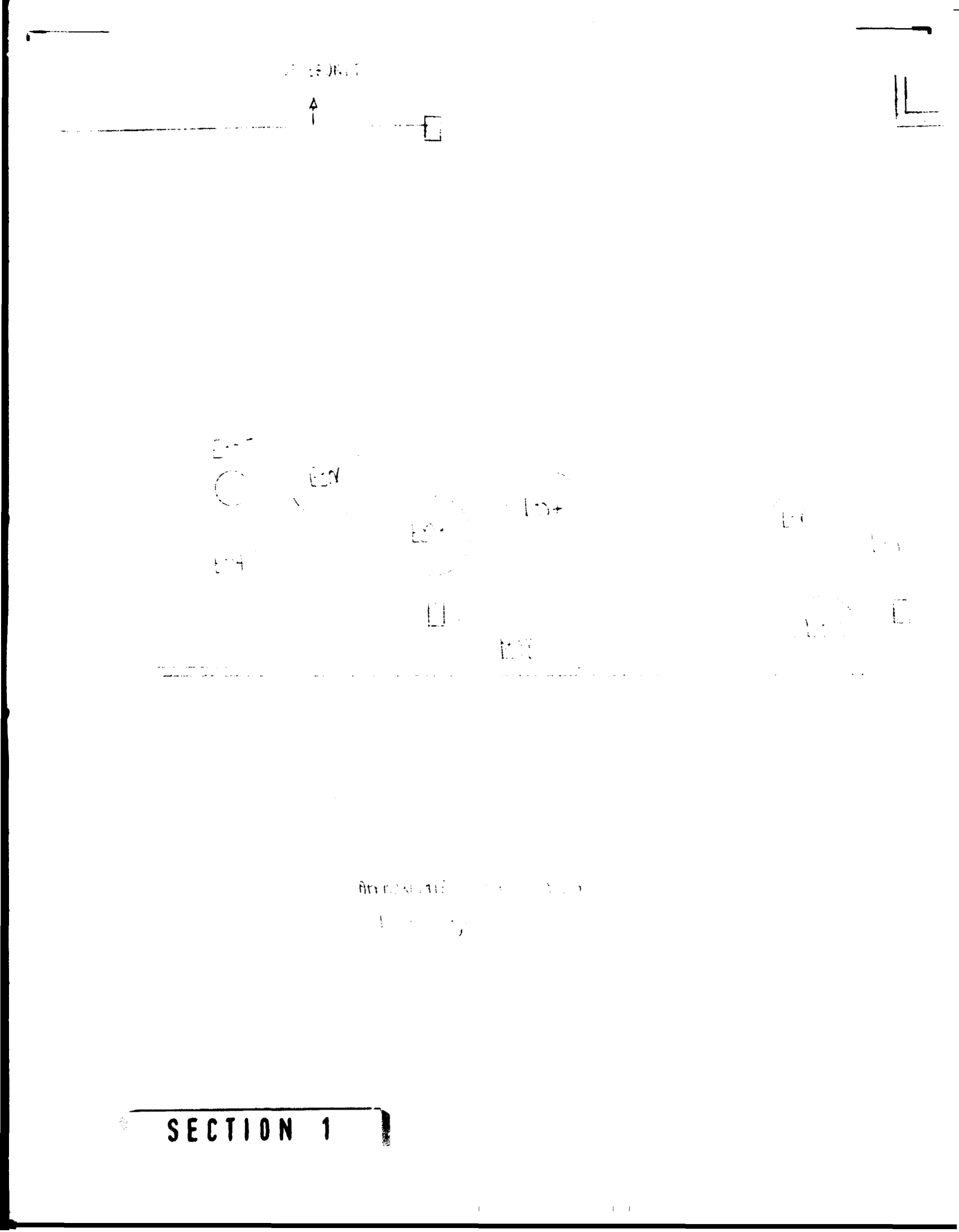
# SECTION 2

CHIMIE DEVELOPPEMENT INTERNATIONAL

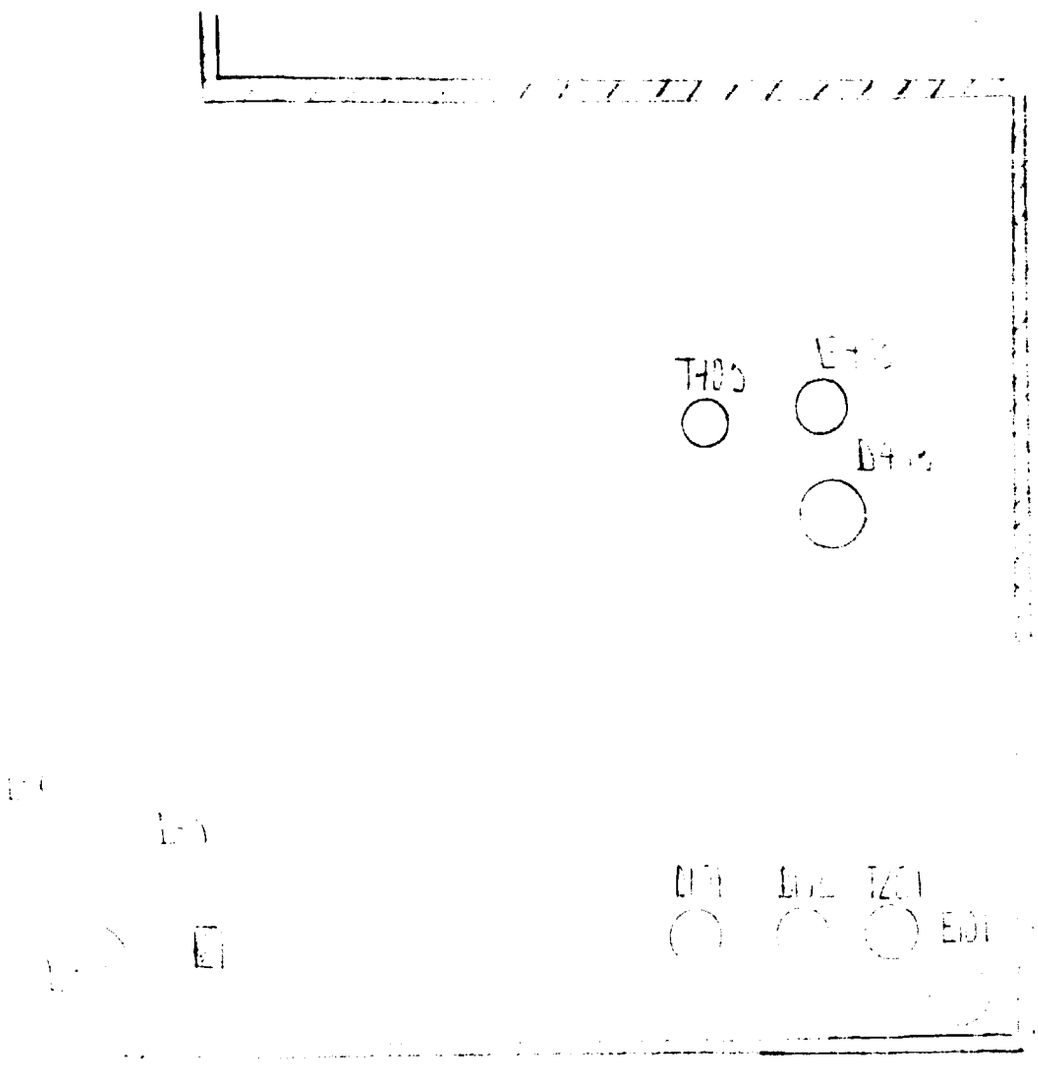
DESSINE PAR: E.D

DATE:

CLIENT:



# SECTION 1



# SECTION 2

CHIMIE DEVELOPPEMENT INTERNATIONAL

DESSINE PAR:

CLIENT: