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PRELIMINARY STUDY FOR THE CONSTRUCTION

OF

A POLY-FUNCTIONAL PILOT-SCALE LABORATORY FOR PLANT EXTRACTION

by

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Medicinal plants constitute a natural resource that is still not fully exploited by developing countries.

The aim of this study is to respond to some of the numerous problems associated with this field and to determine which of the available solutions would be more economical and specifically suited to developing countries.

For purposes of clarification, it is necessary to define the term "pilot-scale laboratory".

A pilot-scale laboratory is equipped to carry out experiments on a scale intermediate between that of a research laboratory and an industrial plant. The data obtained from such laboratories can then be applied to scale up a process and thus allow the undertaking of preliminary studies for the full industrialisation of such a process. Furthermore, it is possible to adapt a pilot-scale laboratory for the production of small quantities of materials required for sampling or to study the pharmacokinetics and chemistry of active compounds isolated in order to evaluate their properties.

Operating units can usually handle between 1 and 20 kg of raw materials, and consequently the size of the equipment has to be modified accordingly. A pilot-scale laboratory is not required to produce industrial quantities of products. Units rarely operate on a scale ranging from 80 to 100 kilograms, and are even less commonly used for scales of 150 to 200 kilograms.

The term "poly-functional" also needs to be defined. A poly-functional laboratory is equipped with the facilities required to carry out a variety of operations. To operate a production unit requires knowledge of flow technology (for the raw material and/or the finished product), methodology and yield.

The size of the apparatus installed will be calculated according to the extent of use by the team of operators (1 x 8, 2 x 8 or 3 x 8) in order that the costs of the initial investments are recuperated within the briefest of delays.

As opposed to a production unit, the direct aim of a poly-functional pilot-scale laboratory is not profitability, but the optimization of tests which will allow the selectior of the best methodology and the most appropriate operating mode.

The cost of building a laboratory adapted to the guidelines outlined above will greatly exceed that of a small production unit.

The sequence of operations to be carried out according to UNIDO's aim is listed in Appendix 1.

PREMISES

The area required for the installation of a poly-functional pilot-scale laboratory is of the order of 700 m^2. The location should ideally be chosen in the neighbourhood of both an industrial park and a research establishment or institute of higher education. The pilot plant can thus profit from the analytical and research facilities of these institutions. The building should ideally be built of modular units ; the building site should not only allow for an eventual expansion of the facilities, but should also be sufficiently large for the construction of one or more industrial units where the methods optimized at the pilot plant can be developed industrialiy.

The actual premises should consist of two separate buildings : the main construction (area 600 m^2) and an annex of approximately 100 m^2, located at an appropriate distance from the main building in a secure area. The annex is destined for the storage of solvents and flammable substances. It is used as a stockroom for both new and used solvents and is equipped with a light roof, with electrical and water installations ; it also contains shelving for storing 50 to 200 litre solvent drums, which should be manoeuvred with a fork-lift truck (or similar equipment).

The main building contains an area built with a high ceiling (8 m) which allows for the installation of tall apparatus (such as distilling columns etc.). It should also include an area destined for explosion-proof work and a series of premises and rooms furnished according to the guidelines described below.

1) Technical premises for the following :
   a) steam generator
   b) cooling system
   c) compressed air generator
   d) vacuum installation
   e) electrical installation
2) Plant storage and crushing
3) Laboratory
4) Offices
5) Cold room
6) Mechanical workshop
7) Drying installation (for both plants and finished products)
8) Poly-functional premises
9) Washing facilities
10) Bathroom installations

1) Technical premises

The area required for the technical premises will generally depend on the size of the equipment to be installed. Sufficient room should remain around the equipment to allow for easy movement ; furthermore, the possible future installation of more equipment should be planned. The technical premises should be both thermally and accoustically isolated from the rest of the building so as not to disturb work taking place in other areas of the building. The area should have direct outside access to avoid having to go through the rest of the building. The electrical control panel (power and lighting) of the main extraction site, equipped with explosion-proof equipment, can be installed in the electrical installation.

2) Plant storage and crushing (area required : 100 m^2 approx.)

The site provided for plant storage and crushing needs to be dry and have heating facilities ; it needs to be equipped with shelving and cupboards for the storage of sacks containing dried plants. The crushers, a scale and an efficient ventilation system for the aspiration and removal of dust particles should be installed in a separate area. Facilities for the storage of botanical samples should also be included in this area.

3) Laboratory (area required approx. 25 m^2)

Before undertaking a pilot-scale extraction on 1-20 kg of raw materials, it is necessary to carry out a small scale extraction on 10 to 100 g in the laboratory. It is often also in the laboratory that the final purification of products obtained from the pilot plant takes place.

4) Offices (area required approx. 25 m^2)

An office (15 m^2) is required to enable the director of the pilot plant to receive visitors, file documents, etc. An adjacent office (approx. 10 m^2) will serve as a secretary's office.

5) Cold Room (area required approx. 10 m^2)

This consists of a room, equipped with shelving, destined for the storage of products and work at + 4°C.

6) Mechanical workshop (area required approx. 25 m^2)

The workshop is equipped with a workbench and a set of electromechanical tools used for the maintenance of the pilot-scale laboratory.

7) Drying installation

This consists of a room equipped with apparatus for drying both plants and finished products.

8) Poly-functional premises

This is for storage and use of equipment not protected against explosions and is located some distance from the main work area.

9) Washing facilities

A room used for the maintenance, washing and drying of laboratory glassware.

10) Bathroom installations

Toilets, showers and washbasins.

EXPLOSION-PROOF PREMISES

Extraction site

The lay-out of this area is important. It needs to be equipped with the following items :

- Hot and cold water
- deionized water
- waste outlet
- glycol/water circuit operating at -15°C
- steam line at 3,5 bars
- low pressure steam line
- compressed air at 6 bars
- vacuum installation
- inert gas line
- electrical power - 380 v three-phase
  - 220 v monophase

The above installations must skirt around the room. Outlets every 3 m will allow for easy connection of equipment. Mounting the equipment on wheels or on carts in order to move it will add to the flexibility of the pilot plant.

The installation should be built out of reinforced concrete. In case of explosion, the building's light roof should blow out first. Several emergency exits are required, and their metallic doors should open towards the outside. The electrical installation must be protected against explosions.

Natural lighting is provided by windows in the upper part of the building and by transparent roof sections. The electrical lighting installation needs to be explosion-proof. Neon lights will be installed at two heights : directly below the roof and at a height of 3 m from the floor. A central lighting system and mercury vapor lamps should also be installed. Emergency lighting, powered by rechargeable batteries, must be installed near the exits. It should be able to function automatically in case of electrical power failures.

Suitable ventilation must be provided in the work area : ventilated heaters will be installed in openings in the roof and at ground level. In addition to ensuring air renewal, these will be used to maintain the room temperature

at + 18°C even if the outside temperature falls to -10°C. Air conditioning must be adapted to the climatic conditions of the country where the pilot plant will be situated. Removal of toxic fumes will be performed with mobile aspirators within the building as well as with ventilators installed in the roof of the building.

The extraction area will have a central gutter and a waste outlet connected via settling basins to a waste water processing plant. Used solvents will be kept in a storage building while awaiting removal and transport to a waste treatment plant.

Essential emergency equipment such as CO_2 and dry powder fire extinguishers, water hoses, gas masks, blankets, gloves, sand buckets etc. will be located inside the building in carefully selected areas. An alarm system, set off by breaking glass or by an electrical power failure must also be included.

PERSONNEL

The operation of the pilot-scale laboratory will be supervised by an 8- or 10-man team consisting of engineers, senior technicians, laboratory assistants, an electromechanical engineer and a cleaner. Particular care must be given to training the employees, who must on a permanent basis be able to ensure both the operation and maintenance of equipment. This will necessitate storage of a number of spare parts as well as having detailed instruction manuals available for each piece of equipment.

LIST OF NECESSARY EQUIPMENT

(see APPENDIX 2)

Potential uses

The various types of industry with which such a laboratory might collaborate include the following :

- pharmaceutical industry
- cosmetics industry
- farm-produce industry
- dyestuffs industry
- aroma and perfume industry, etc.

The laboratory can play a secondary role as both a teaching facility and research tool ; in collaboration with institutes of higher education it can offer introductory courses for students, vocational training for engineers, experimental training etc. ; there is also the possibility to prepare small quantities of pure substances necessary for basic research.

The pilot plant could also be used to undertake contract work. Billing for this work will in part or fully cover expenses incurred during the operation of the pilot plant.

Cost Evaluation

The cost of undertaking such a project is quite considerable and can vary enormously depending on the local conditions. We evaluate the cost of construction and initial equipment at approx. 15 M FF. In addition to credits necessary for initially equipping the pilot plant, it is also necessary to take into account funding for its operation, maintenance and periodic renewal of equipment. The annual budget, excepting salaries, is of the order of 2 M FF for a normal operation. It is difficult to impose such large costs on a developing country. It would therefore be desirable to adopt one of the following solutions:

- Construct a laboratory with international calling that would be financed by a group of interested countries and will work for them.

- Solicit help from a pilot plant already implanted in a developed country (such as the one at the CNRS, Gif-sur-Yvette, France, for example). Such an agreement could probably be discussed between the Management of the CNRS and UNIDO. The procedures optimized in such a laboratory can directly be applied to the implantation of small industrial installations at a much reduced cost and applicable to the realization of specific, non-poly-functional needs.

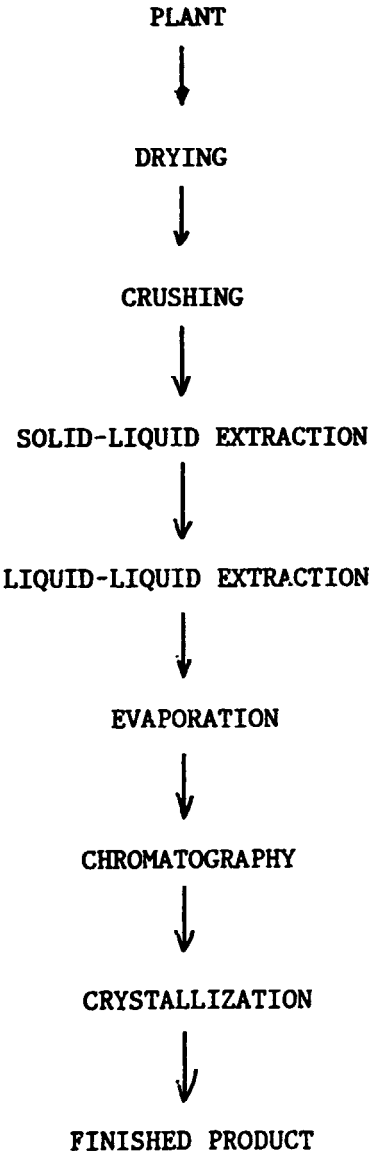
The French company Tournaire SA, BP N° 4, 06638 Le Plan de Grasse is specialized in the realization of such installations (see Appendix 3). It will be necessary to add additional equipment in order to adequately operate a plant extraction installation. The list below is a guideline :

- grinders/crushers for dried plants
- thin-film evaporators
- vacuum ovens for drying
- liquid-liquid extractors
- chromatography equipment
- fractional distillation equipment
- fluid production

These items will be distributed and located according to the specific needs of the operating unit in question.

As a general guideline, a list of the equipment required for the construction of a poly-functional pilot-scale laboratory is given in Appendix 2.

APPENDIX 1


PLANT
DRYING
CRUSHING
SOLID-LIQUID EXTRACTION
LIQUID-LIQUID EXTRACTION
EVAPORATION
CHROMATOGRAPHY
CRYSTALLIZATION
FINISHED PRODUCT

APPENDIX 2

Apparatus required to equip a pilot-scale laboratory

N.-B. The prices quoted are indicative and based on current market values. The actual cost may vary enormously depending on the desired quality of the equipment.

TECHNICAL PREMISES

Steam Generation : 250 kg/h of steam required at 4 bars. The generator should be supplied with soft water. Price 130.000 FF

Refrigeration : Refrigeration unit operating with glycol/water, equipped with air condenser. For glycol/water at - 15°C, 45000 Fg/h at 24 kW are required. External 100.000 kCal/h air condenser. Price 300.000 FF

Vacuum Installation : Vacuum provided by liquid ring vacuum pumps :

. 1 at 100 m^3/h Price 50.000 FF
. 2 at 25 m^3/h. Price 30.000 FF

Electrical Installation : 1 315 Oil transformer. Primary voltage 15000/20000, adjustable to 2.5%, equipped with normal accessories.
Price 70.000 FF

PLANT STORAGE AND CRUSHING

- Stainless steel crusher with sieve, equipped with 2.2 kW three-phase 220/380 v 50 Hz motor. Auxiliary motor to operate the oscillating shovel of the feeding apparatus. The whole mounted on a metal frame.
Price 115.000 FF

- All steel box balance with simple pendulum mechanism on a steel axis. Range 5 kg to 60 kg, graduated in 100 g increments.
Price 9.000 FF

- All steel balance, placed on the floor, with simple pendulum mechanism on a steel axis. Max. load 200 kg, graduated in 200 g increments. Price 4.500 FF

- Industrial type vacuum cleaner for dust removal. Price 3.000 FF

- Shelving and cupboards. Price 20.000 FF

OFFICES

- Office furniture and accessories. Price 15.000/20.000 FF

COLD ROOM

- Refrigeration Unit
- Shelving. Price 15.000 FF

DRYING INSTALLATION (for plants and finished products)

- Lyophilizer with trap capacity of 15 kg ice/24 h. Cooling power 4500 Fg/h. Two stage vacuum pump for 35 m^3/h capacity. Price 425.000 FF

- Stainless steel mobile atomizer, type "MINOR". Evaporation capacity: 1 to 6 kg water/h. Maximum inlet temperature 350°C ; outlet temperature 120°C. Price 165.000 FF

- Stainless steel-cased ventilated oven. Power 2000 w/220 v. Price 22.000 FF

- Stainless steel vacuum oven, heated by automatic circulation (thermosiphon). Price 70.000 FF

WASHING FACILITIES

- Washing machine for laboratory glassware.

Price 6.000 FF

- Double sink units equipped with hot, cold and deionized water.

LABORATORY

- Laboratory glassware, reagents, magnetic stirrers, rotary evaporators, chromatography columns, fraction collectors, thin-layer chromatography, melting point apparatus, stirrers powered by electric motors and compressed air, U.V. lamp, efficient hood, laboratory furniture, water baths, precision balance, pH meter, small laboratory equipment.

Price 150.000/200.000 FF

EXTRACTION PREMISES

Solid-Liquid extraction

- Stainless steel powder mixer with lid. Nominal capacity of 20 l, equipped with explosion-proof motor, chain and sprocket wheel drive, standard joints. Price 90.000 FF

- Stainless steel conical percolator, 80 l capacity, equipped with lid and drain tap. Price 20.000 FF

- 2 stainless steel Soxhlet type extractors, 20 l capacity, steam heated, with accessories for solvent evaporation.

Price 2 x 150.000 FF 300.000 FF

- Stainless steel floating-filter extractors, 150 l capacity, equipped with explosion-proof motor and accessories for use in steam distillation, extraction by hot or cold maceration, extraction under reflux and distillation.

Price 30.000 FF

- Stainless steel EF 2 extractors, 300 l capacity, which can be used for maceration, percolation, vacuum drying, filtration under pressure, reflux and solvent recovery. Price 500.000 FF

- All glass solid-liquid extraction apparatus consisting of the following :

. 150 l evaporating flask
. 1 DN 150 heat exchanger
. 1 DN 200 column, height 1000 m
. 1 DN 200 condenser
. 1 30 l extraction vessel

Price 65.000 FF

. 2 glass Soxhlet extractors, 20 litre capacity, each equipped with 1 20 l flask, 1 heat exchanger immersed in the 20 l flask, 1 condenser and 1 DN 150 Soshlet. Price 2 x 37.500 FF
75.000 FF

Liquid-Liquid Extraction

- Liquid-Liquid extraction apparatus consisting of the following :

. 2 centrifugal separators, type TA 1.04.525
. 1 separator, type TA 1.01.525
. 1 mixer, type ZA 1.66.525

Closed-system operation and impermeable to gases. The whole installation is equipped with explosion-proof motors.

Price 350.000 FF

- Liquid-Liquid extraction in glass apparatus. The equipment includes :

  . 2 50 l feed-vessels
  . 2 dosage pumps
  . 2 50 l receiving flasks
  . 1 DN 80 extraction column, made up of 4 elements and equipped with 15 x 15 Raschig rings
  . mobile glass tanks
  . cylindrical reactor vessels equipped with stainless steel frames, with 30, 50, 100, 150 and 200 litre capacities.

Price 162.000 FF

- DYNAVAR O type motor/agitator with a three phase (220/380 v - 50 Hz) explosion-proof motor, 1.1 kW at 1500 rpm, equipped with speed regulator and support column. Price 35.000 FF

- Type 316 stainless steel vessels and tanks in the following sizes : 5, 10, 20, 40, 80, 150, 300 and 500 litres.

- Super-D-canter SDC P 600 made of type 316 stainless steel, equipped with vibration-proof chassis and supports ; 5.5 kW 220/380 v 50 Hz motor.

Price 181.000 FF

- Stainless steel AS 16 supercentrifuge equipped with dosage head or separator, 3 kW 220/380 v 50 Hz motor. Price 220.000 FF

Distillation and Evaporation

- Distillation column and 50 l glass vessel equipped as follows :

  . 1 50 l evaporating vessel
  . 2 DN 150 heat exchanger
  . 1 50 l reaction vessel equipped with juice-pump
  . 1 DN 200 column, height 2000 mm, filled with 15 x 15 Raschig rings
  . 1 pneumatic timer

. 1 DN 200 condenser

. 1 trap, 1 cooler, and 5 and 50 l receiving flasks

Price 143.000 FF

. Automatic circulation (thermosiphon) glass extractor for 20 l flask, equipped with a 20 l flask, DN 150 heat exchanger, a DN 100 column filled with 8 x 8 Raschig rings, 2 20 l receiving flasks and a trap.

Price 88.000 FF

. DN 150 thin film evaporator equipped with a dosage pump and 5 and 10 litre receiving flasks.

Price 145.000 FF

. 1 steam-heated 20 l rotary evaporator.

Price 200.000 FF

Chromatography

. Glass column chromatography apparatus equipped with a) 200 l reaction vessel and b) DN 100 chromatography columns.

Price 65.000 FF

. Modulprep HPLC stainless steel chromatography columns, with diameters of 20, 40 and 80 mm, equipped with a pump, a control unit, refractometer, UV spectrophotometer, recording device and fraction collector. Price 500.000 FF

Miscellaneous equipment

. Stainless steel Buchner filters, 400 and 800 mm diameters.

Price 35.000 FF + 55.000 FF = 90.000 FF

. Distilled water apparatus with 50 litre/hour capacity.

Price 120.000 FF

. Ultrafiltration and Microfiltration laboratory units, for example Microlab 130 S equipped with both PVC and ceramic modules.

Price 200.000 FF

. Ventilation system for aspiration of toxic fumes equipped with mobile aspirator mounted on the extraction apparatus. Fan should have a 3600 m^3/h capacity, 1.5 kW at 2800 rpm.

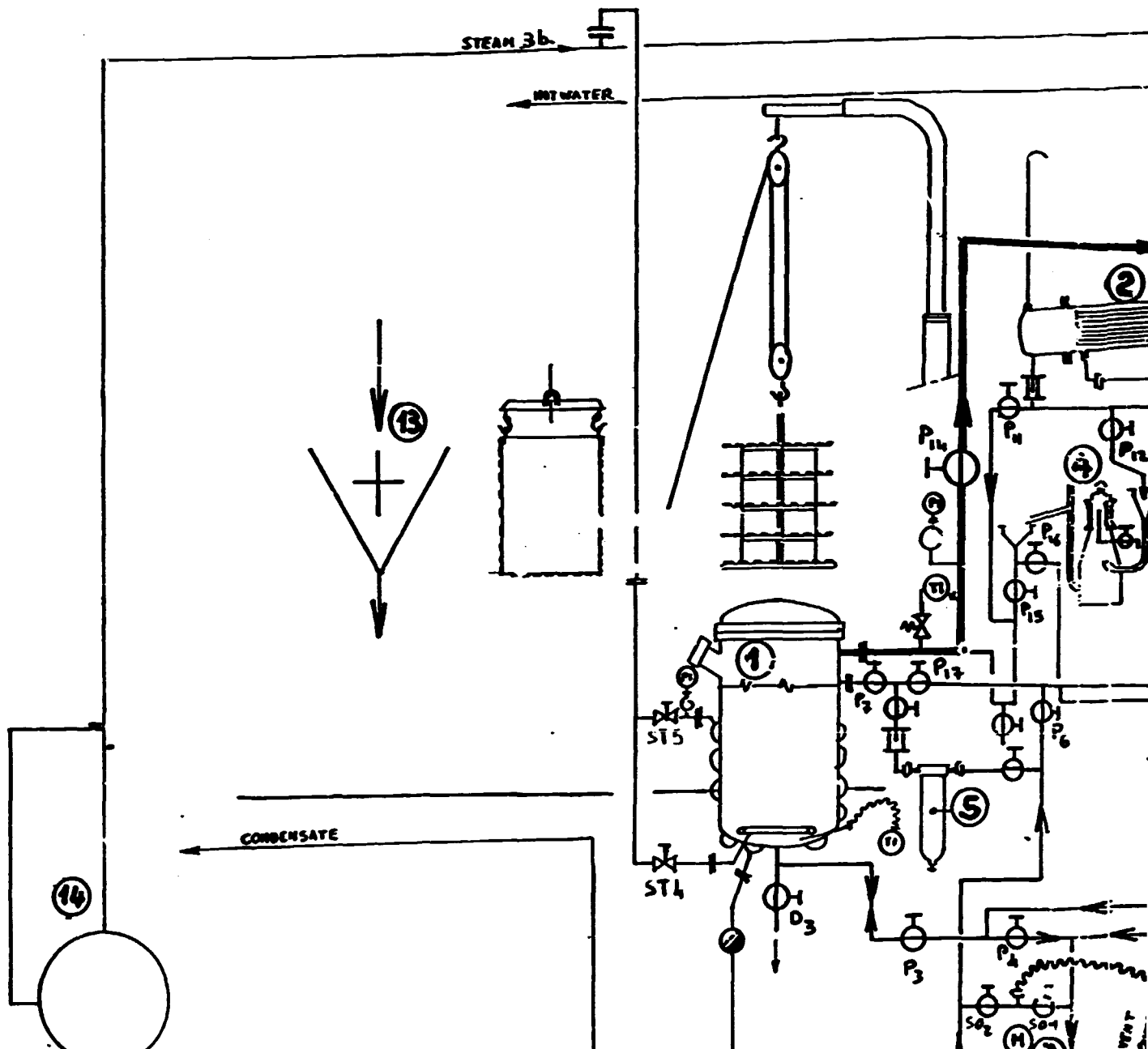
Price 77.000 FF

. Pneumatic temperature regulators.

APPENDIX 3

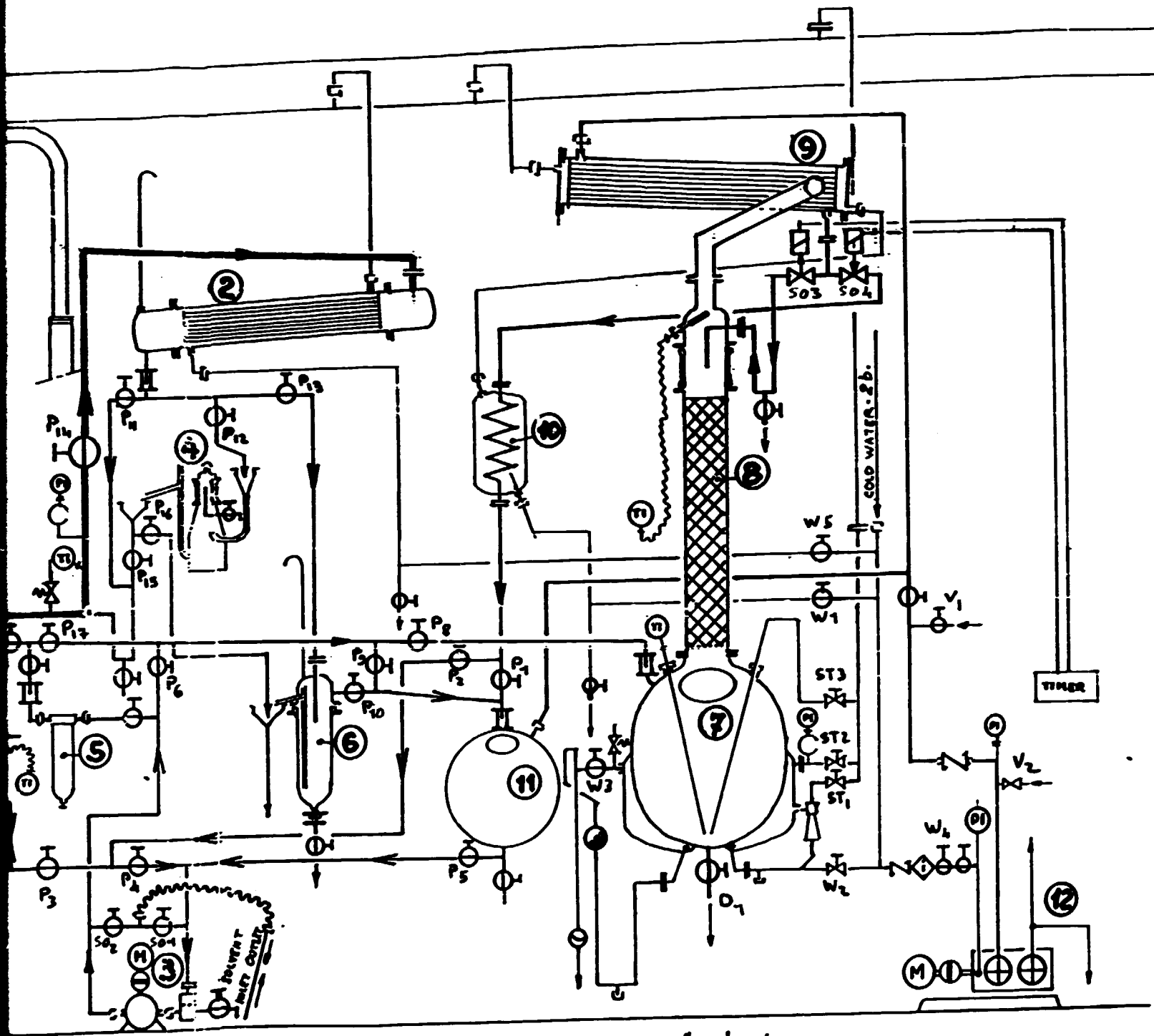
POLYFUNCTIONAL EXTRACTION UNIT TYPE CS 264

Stainless steel 250 l Extractor, condenser, Florentine vessel, decanter, circulation pump, vacuum evaporator, vacuum pump.



SECTION 1

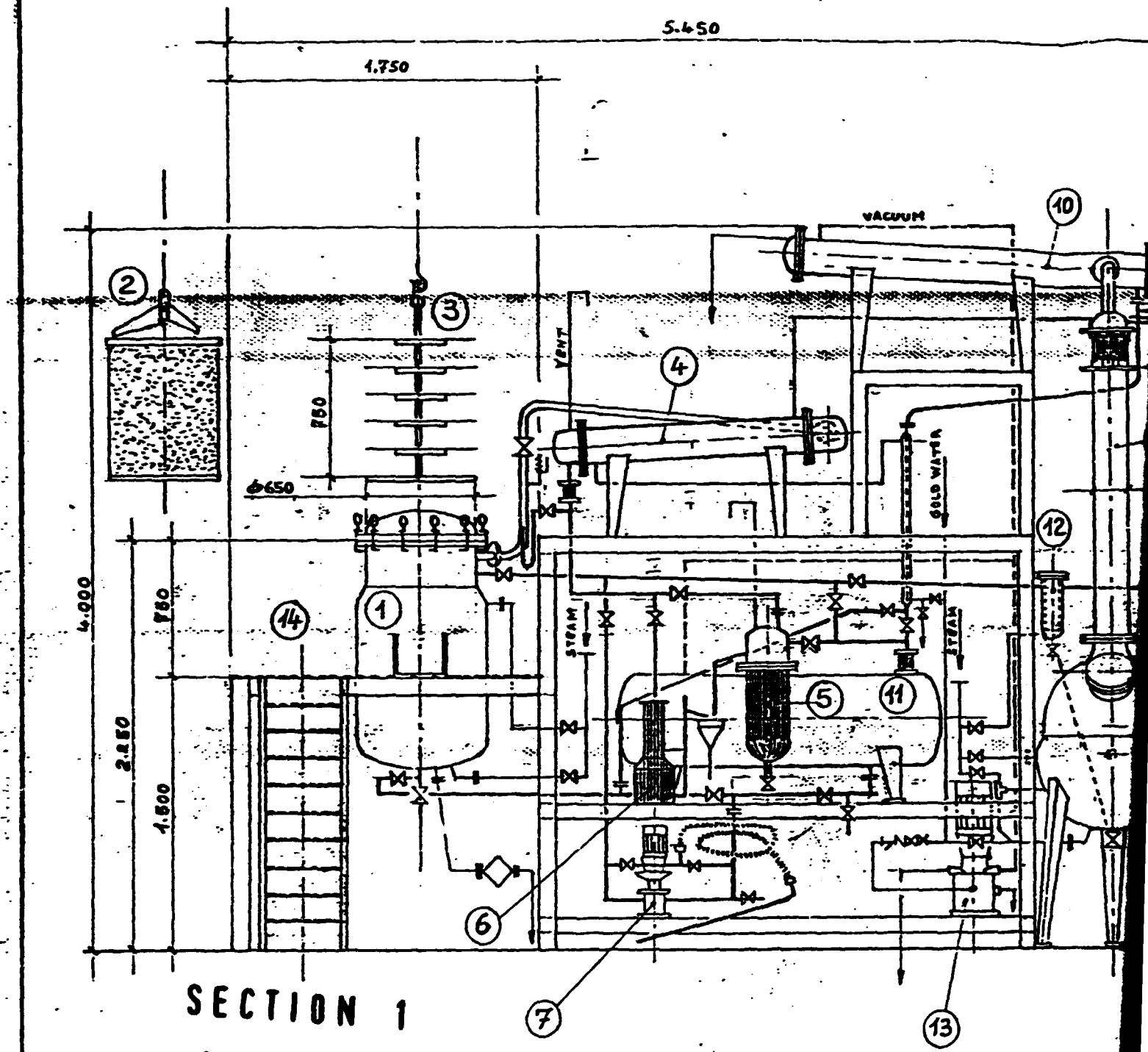
14	1
13	1
12	1
11	1



SECTION .2

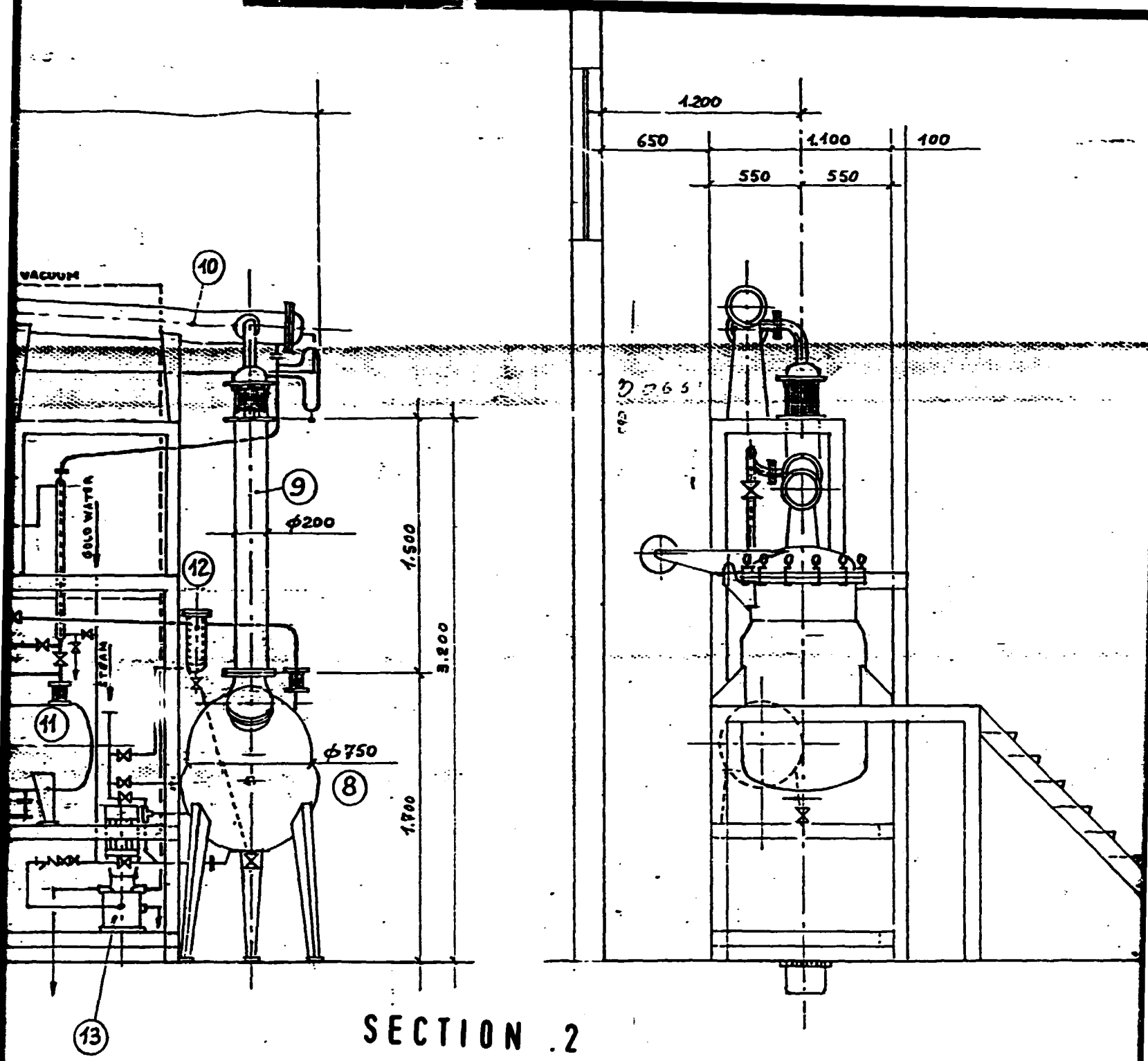
14	1	STEAM BOILER
13	1	CRUSHER
12	1	VACUUM PUMP
11	1	RECEIVING TANK

10	1	COIL TYPE COOLER
9	1	HORIZONTAL CONDENSER
8	1	COLUMN
7	1	BOILER
6	1	DECANTER
5	1	CARTRIDGE FILTER
4	1	FLORENTINE FLASK
3	1	SELF PRIMING PUMP
2	1	CONDENSER
1	1	EXTRACTOR



SECTION 1

REP	NO	DESIGNATION	CAP		REP	NO	DESIGNATION
					10	1	CONDENSER
					9	1	PACKED COLUMN
					8	1	BOILER
					7	1	CIRCULATING PUMP
					6	1	FLORENTIN FLASK
					5	1	DECANTER
14	1	FRAME			4	1	CONDENSER
13	1	WATER RING PUMP			3	1	PERFORATED GRIDS
12	1	SAFETY FILTER FOR MISCELLA			2	1	PERFORATED BASKET
11	1	RECEVER TANK	300L.		1	1	EXTRACTOR DOUBLE JACKET HEAT



SECTION .2

DESIGNATION	CAP.
ENSER	4 M2
ED COLUMN	
ER	250L.
LATING PUMP	
NTIN FLASK	6 L.
NTER	18 L.
ENSER	1,6 M2
ORATED GRIDS	
RATED BASKET	250L.
ACTOR DOUBLE JACKET HEATING	250L.

TOURNAIRE S A

NO	DATE	MODIFICATIONS

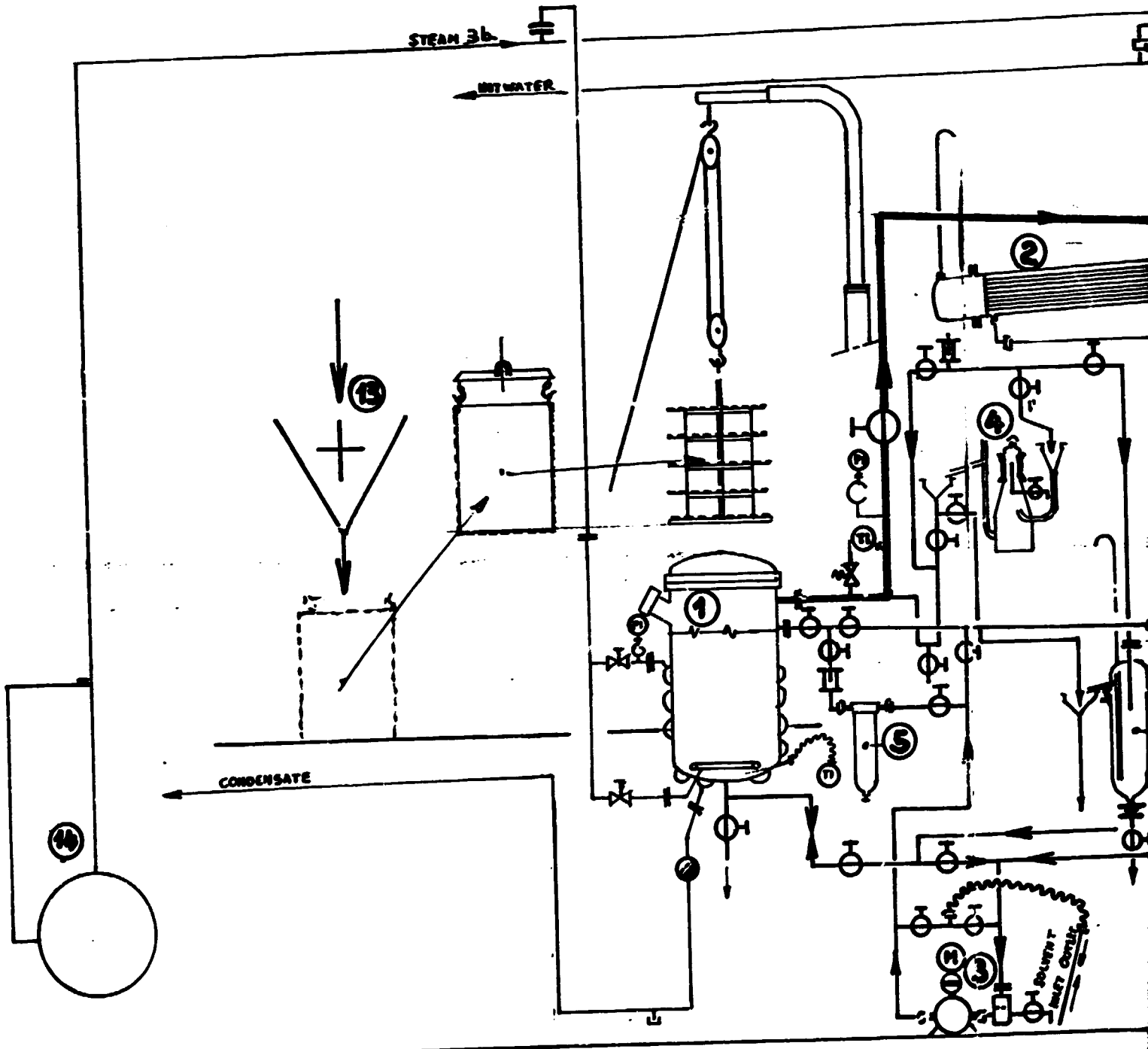
MULTI PURPOSE EXTRACTION UNIT

IMPLANTATION

DATE

PAR

CS



SECTION 1

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13	1	CRUCI
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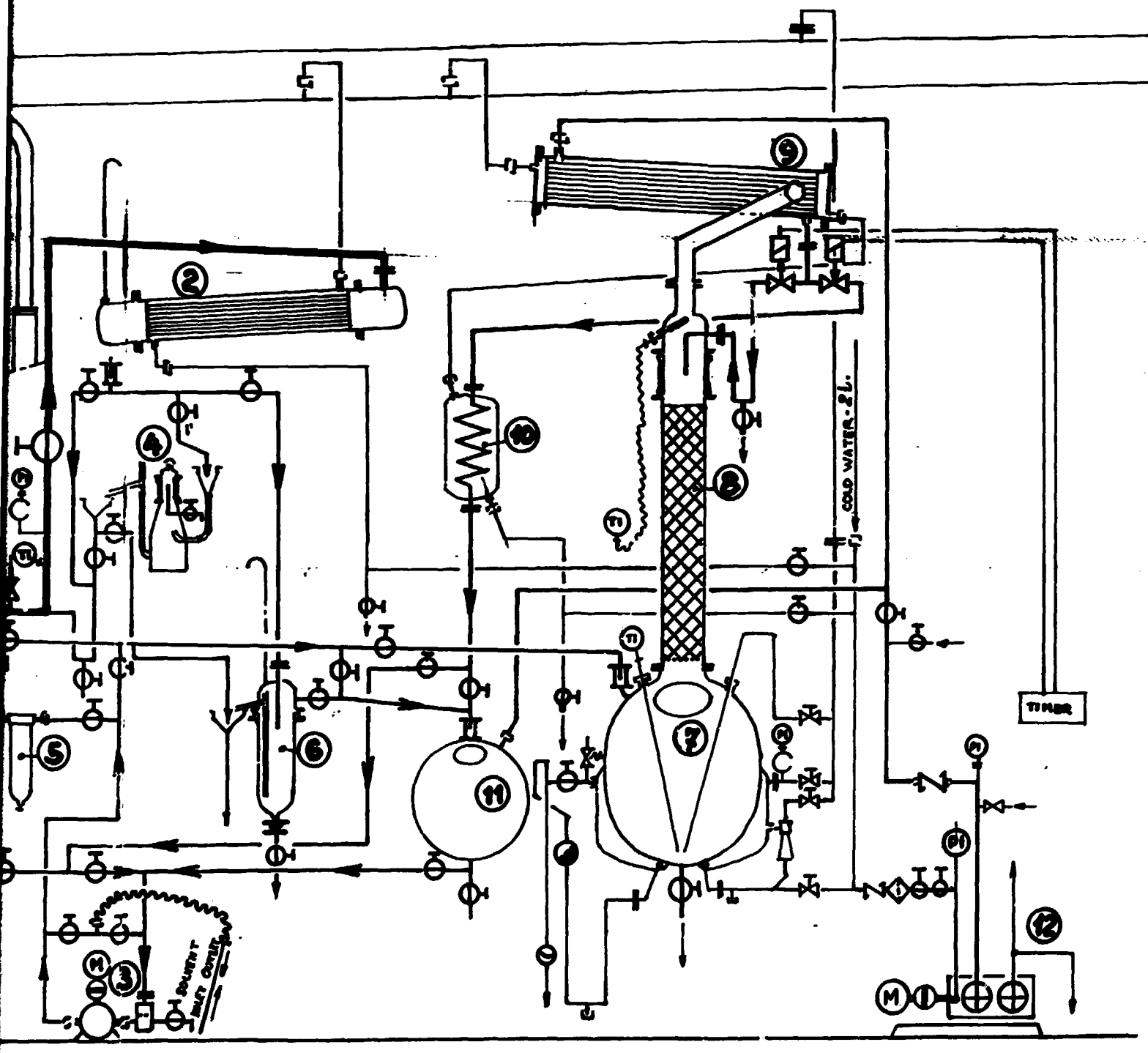
11	1	RECEI



SECTION .2

14	1	STEAM BOILER
13	1	CRUSHER
12	1	VACUUM PUMP
11	1	RECEIVING TANK
10	1	COIL TYPE COOLER
9	1	HORIZONTAL CONDENSER
8	1	COLUMN
7	1	BOILER
6	1	DECANTER
5	1	CARTRIDGE FILTER
4	1	FLORENTINE FLASK
3	1	SELF PRIMING PUMP
2	1	CONDENSER
1	1	EXTRACTOR

APPENDIX 3

POLYFUNCTIONAL EXTRACTION UNIT

This unit is composed of the following elements (cf. Schema) :

- Steam boilet
- Crusher
- Vacuum pump
- Receving tank
- Coil type cooler
- Horizontal condenser
- Column
- Boiler
- Decanter
- Cartridge filter
- Florentine flask
- Pump
- Condenser
- Extractor

and corresponds at a polyvalent pilot plant taking account the specifications prepared by the UNIDO.

O P E R A T I O N

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1. PRODUCTION OF ESSENTIAL OIL BY DISTILLATION

Unit preparation

Close the valves : -P 1 -P 2 - P 3 -P 4 -P 5 -P 6 -P 7 -P 8 -P 9
- P 10 -P 11 -P 13 -P 15
Open the valves : -P 12 -P 14 -W 1

Loading

Load the raw material in the extractor either with the perforated basket or with the perforated grids according to the structure of the raw material (avoid to use pulverulent raw material).

Close carefully the extractor which will be used as a still in this process.

Steam distillation

Open P 15 in case of cohobation or P 16 without cohobation.

Open the steam valve S T 4 and adjust the steam flow by control of the water flow in the florentine vase.

If the product to be treated need a higher pressure than the atmospheric, set the valve P 14 in order to increase the pressure in the still (extractor).

.../...

Hydro-distillation

Before loading fill in the still the right amount of water (the water circulating pump can be used for that).

Open and adjust the valve S T 5 in order to heat the still through the heat-exchanger. (the essential oil will be collected in the florentine flask).

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2. SOLVENT EXTRACTION

Loading

Load the extractor in the same way as § 1.

Solvent filling

Put the flexible pipe in a refrigerant drum open the valves : SO 1 - P 6 - P 9 - V 1 - W 2 -
Turn on the circulating pump and fill the receiver tank.
Turn off the circulating pump close the valves - SO 1 - P 9 -

Extraction

Open the valves - P 5 - P 6 - P 7 - P 14 - P 17

Turn on the circulating pump check the solvent level in the extractor through the sight-glass, turn off the pump at the suitable level.

Close the valves - P 5 - P 7 -

.../...

After the time required for the extraction, draw-off the miscella from the extractor : open the valves - P 3 - P 4 - P 6 - P 8 - turn on the pump and transfer the miscella in the boiler observe the liquid flow through the boiler sight glass. When the flow is off, turn-off the pump and close the valves - P3 - P 4 - P 6 - P 8 -.

Primary concentration and solvent recycling

Open the valves - P 1 - P 5 - P 6 - P 7 - P 11 - S O 3 - V 1
Heat the boiler either by water jacket or heat-exchanger direct steam according to the evaporating temperature of the solvent.
Refill the extractor with the circulating pump.
Another possibility is to by-pass the receiver tank and to fill the extractor by gravity with the distillate, for that :
close - P1 - P 5 - P 7 - open - P 3 - P 2
After the time required, draw-off the miscella as above.
Carry on the operation if necessary.

Stripping (solvent saving)

Open the valves - P 10 - P 13 -
Blow the extractor with the steam valve ST 4
Carry on the operation as long the solvent is flowing in the receiver tank.

Concentration under vacuum

Close all the "P" valves except P 1 (wide open) : open S O 3
Heat the boiler at the suitable temperature (water jacket operation).
Operate the vacuum pump for that :
- Open W 4 close V 1 open V 2 switch on the motor
- Adjust the vacuum value with the by-pass V 2
Carry on the concentration until the end of the solvent flow.
Collect the concentrate through the drain valve D 1 at atmospheric pressure (stop the vacuum pump open the vent V 1).

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3. LIQUID DISTILLATION AND RECTIFICATION

Loading

Load the boiler with the liquid to be treated with the circulating pump.
Open - SO 1 - P 6 - P 8 - V 1 -
Operate the circulating pump and load from a drum through the flexible pipe "solvent inlet/outlet".

Distillation

Close the valves - SO 1 - P 6 - P 8 -
Open W 1.
Heat the boiler either with direct steam heat-exchanger operation or with water jacket operation according to boiling temperature of the solution to be treated.
Adjust to the suitable boiling temperature.
Adjust the timer in order to set the reflux and drawing ratio with valves SO 3 and SO 4.

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4. BOILER HEATER OPERATION

Direct steam in the heat-exchanger

For evaporating temperature $> 95°C$ close the valve W 3 use the valve ST 2 for heating, the condensate steam will be eliminated through the steam trap.

Water jacket operation

Open the valve W 3 then fill the heat-exchanger with water through the valve W 2 use the valve ST 1 for heating, the condensate water will be eliminated by overflow through W 3.

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VALVES IDENTIFICATIONS & FUNCTIONS

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WATER VALVES

W 1 : Condenser water supply
W 2 : Water jacket supply
W 3 : Water jacket overflow
W 4 : Vacuum pump supply
W 5 : Condenser water supply.

STEAM VALVES

ST 1 : Boiler heating (water jacket operation)
ST 2 : Boiler heating (direct steam operation)
ST 3 : Boiler steam blower (cleaning)
ST 4 : Extractor steam grid supply
ST 5 : Extractor heat exchanger supply.

PROCESS VALVES

P 1 : Condensate and drawing-off ratio setting
P 2 : From boiler condenser + cooler to :
- Extractor
- Circulating pump

P 3 : From extractor to circulating pump
From boiler condenser to extractor (through P 2)

P 4 : From extractor throw P 3 to :
From boiler condenser + cooler through P 2 to :
- Circulating pump

P 5 : From receiver tank to circulating pump
P 6 : From circulating pump to :
- Extractor
- Boiler
- Receiver

.../...

P 7 : From circulating pump through P 6 to extractor
P 8 : From circulating pump through P 6 to boiler
P 9 : From circulating pump through P 6 to receiver tank
P 10 : From decanter to receiver tank
P 11 : From extractor condenser to extractor (solvent saving)
P 12 : From extractor condenser to florentin flask
P 13 : From extractor condenser to separator (stripping operation)
P 14 : From extractor to extractor condenser
P 15 : Cohobation
P 16 : Sewer

SOLVENT VALVES

P 17 : From circulating pump to extractor
SO 1 : From the solvent inlet/outlet flexible pipe to :
- the circulating pump
SO 2 : From the circulating pump to :
- the solvent inlet/outlet flexible pipe.
SO 3 : Reflux
SO 4 : Distillate.

DRAIN VALVES

D 1 : From concentrator
D 2 : From solvent tank
D 3 : From extractor.

+++

APPENDIX 3

POLYFUNCTIONAL EXTRACTION UNIT

This unit is composed of the following elements (cf. Schema) :

- Steam boilet
- Crusher
- Vacuum pump
- Receiving tank
- Coil type cooler
- Horizontal condenser
- Column
- Boiler
- Decanter
- Cartridge filter
- Florentine flask
- Pump
- Condenser
- Extractor

and corresponds at a polyvalent pilot plant taking account the specifications prepared by the UNIDO.

O P E R A T I O N

+++

1. PRODUCTION OF ESSENTIAL OIL BY DISTILLATION

Unit preparation

Close the valves : -P 1 -P 2 - P 3 -P 4 -P 5 -P 6 -P 7 -P 8 -P 9
- P 10 -P 11 -P 13 -P 15
Open the valves : -P 12 -P 14 -W 1

Loading

Load the raw material in the extractor either with the perforated basket or with the perforated grids according to the structure of the raw material (avoid to use pulverulent raw material).

Close carefully the extractor which will be used as a still in this process.

Steam distillation

Open P 15 in case of cohobation or P 16 without cohobation.

Open the steam valve S T 4 and adjust the steam flow by control of the water flow in the florentine vase.

If the product to be treated need a higher pressure than the atmospheric, set the valve P 14 in order to increase the pressure in the still (extractor).

.../...

Hydro-distillation

Before loading fill in the still the right amount of water (the water circulating pump can be used for that).

Open and adjust the valve S T 5 in order to heat the still through the heat-exchanger. (the essential oil will be collected in the florentine flask).

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2. SOLVENT EXTRACTION

Loading

Load the extractor in the same way as S 1.

Solvent filling

Put the flexible pipe in a refrigerant drum open the valves : SO 1 - P 6 - P 9 - V 1 - W 2 -
Turn on the circulating pump and fill the receiver tank.
Turn off the circulating pump close the valves - SO 1 - P 9 -

Extraction

Open the valves - P 5 - P 6 - P 7 - P 14 - P 17

Turn on the circulating pump check the solvent level in the extractor through the sight-glass, turn off the pump at the suitable level.

Close the valves - P 5 - P 7 -

.../...

After the time required for the extraction, draw-off the miscella from the extractor : open the valves - P 3 - P 4 - P 6 - P 8 - turn on the pump and transfer the miscella in the boiler observe the liquid flow through the boiler sight glass. When the flow is off, turn-off the pump and close the valves - P3 - P 4 - P 6 - P 8 -.

Primary concentration and solvent recycling

Open the valves - P 1 - P 5 - P 6 - P 7 - P 11 - S O 3 - V 1
Heat the boiler either by water jacket or heat-exchanger direct steam according to the evaporating temperature of the solvent.
Refill the extractor with the circulating pump.
Another possibility is to by-pass the receiver tank and to fill the extractor by gravity with the distillate, for that :
close - P1 - P 5 - P 7 - open - P 3 - P 2
After the time required, draw-off the miscella as above.
Carry on the operation if necessary.

Stripping (solvent saving)

Open the valves - P 10 - P 13 -
Blow the extractor with the steam valve ST 4
Carry on the operation as long the solvent is flowing in the receiver tank.

Concentration under vacuum

Close all the "P" valves except P 1 (wide open) : open S O 3
Heat the boiler at the suitable temperature (water jacket operation).
Operate the vacuum pump for that :
- Open W 4 close V 1 open V 2 switch on the motor
- Adjust the vacuum value with the by-pass V 2
Carry on the concentration until the end of the solvent flow.
Collect the concentrate through the drain valve D 1 at atmospheric pressure (stop the vacuum pump open the vent V 1).

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.../...

3. LIQUID DISTILLATION AND RECTIFICATION

Loading

Load the boiler with the liquid to be treated with the circulating pump.
Open - SO 1 - P 6 - P 8 - V 1 -
Operate the circulating pump and load from a drum through the flexible pipe "solvent inlet/outlet".

Distillation

Close the valves - SO 1 - P 6 - P 8 -
Open W 1.
Heat the boiler either with direct steam heat-exchanger operation or with water jacket operation according to boiling temperature of the solution to be treated.
Adjust to the suitable boiling temperature.
Adjust the timer in order to set the reflux and drawing ratio with valves SO 3 and SO 4.

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4. BOILER HEATER OPERATION

Direct steam in the heat-exchanger

For evaporating temperature > 95°C close the valve W 3 use the valve ST 2 for heating, the condensate steam will be eliminated through the steam trap.

Water jacket operation

Open the valve W 3 then fill the heat-exchanger with water through the valve W 2 use the valve ST 1 for heating, the condensate water will be eliminated by overflow through W 3.

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VALVES IDENTIFICATIONS & FUNCTIONS

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WATER VALVES

W 1 : Condenser water supply
W 2 : Water jacket supply
W 3 : Water jacket overflow
W 4 : Vacuum pump supply
W 5 : Condenser water supply.

STEAM VALVES

ST 1 : Boiler heating (water jacket operation)
ST 2 : Boiler heating (direct steam operation)
ST 3 : Boiler steam blower (cleaning)
ST 4 : Extractor steam grid supply
ST 5 : Extractor heat exchanger supply.

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DRAIN VALVES

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D 2 : From solvent tank
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A. ESCAUT

Gif, le 12 Décembre 1988

Mrs. Eva M. CAIRO
Recruitment Officer
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Vienna International Centre
P.O. Box 300
A-1400 VIENNA
(AUTRICHE)

Chère Madame,

Je vous prie de bien vouloir trouver ci-joint le complément du rapport qui m'a été demandé lors de mon voyage à Vienne, le 31 Octobre 1988, et de le transmettre à MM. MAJID et WIJESEKERA.

La page 17 (Appendix 13) devra être modifiée conformément au document ci-joint.

Je pense que de cette manière le rapport sera satisfaisant et je vous prie de faire le nécessaire pour obtenir le visa de conformité afin que les honoraires puissent être payés.

Je vous prie de croire, Madame, à l'expression de mes sentiments les meilleurs.

A. ESCAUT