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PRODUCTION OF PHYTOCHEMICALS AND ESSENTIAL OILS
FROM MEDICINAL AND AROMATIC PLANTS

DP/TUR/83/003

TURKEY

Terminal report*

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

Based on the work of Mr. M.B. Narasimha, expert
in chemical technology

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Vienna

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TITLE : Production of crude phytochemicals and essential oils from Medicinal and Aromatic Plants - First Phase.

PROJECT : DP/TUR/83/003.

STATION : Eskisehir, Turkey.

DURATION : Mid-June to Mid-September, 1986.
Mid-April to Mid-August, 1987.

OBJECTIVES:

1.1 To establish R & D infrastructure, pilot plant and analytical facility and train technical manpower. To exploit untapped, abundant and varied natural flora of Turkey in the establishment of industry to produce:

- crude extracts from medicinal plants.
- isolation of active principles from the crude extracts for therapeutic use.
- crude essential oils from aromatic plants.
- terpeneless essnetial oils.
- isolation into groups of close boiling essential oils by fractional distillation.

1.2 To serve as a focal point:

- for training technical manpower.
- to generate process knowhow.
- to build capability in transfer of technology
- to build a strong base in design and engineering.

- Research and development centres, like Medicinal Plants Research Centre, with built-in infrastructure and trained technical manpower is expected:

  - to generate technically feasible and economically viable technologies in the utilization of natural flora of Turkey.

  - to provide R & D and analytical instrument facilities for use by the industry when established.

  - to provide advisory and consultancy services.

SUMMARY:

The Medicinal Plants Research Centre of the University of Anatolia, has been able to establish from scratch, during the first phase of the project, with the active assistance and co-operation of UNIDO/UNDP and its experts, basic infrastructure facilities to conduct R & D activities to produce crude extracts and essential oils based on some of the plants indigenous to Turkey and contacts with appropriate industrial units and other academic institutions in Turkey.

The project has made considerable progress, in the processing of essential oils and phytochemicals on bench-scale and at pilot-plant level. Mention may be made about the very significant achievement made in demonstrating an improved method, which resulted in increased processing capacity and enhanced quality of rose oil in the factory of the largest rose oil producers of Turkey viz., M/s. Gulbirlik, Isparta.

The project has also diversified itself, into extremely useful area of developing expertise, in the design and engineering with the design, fabrication and installation of 2 m^3 essential oil distillation unit, a multipurpose fractionation

facility with latest internal packings, vacuum filter and thus encouraging indigenous workshops in their fabrication.

Building basic infrastructure, to design simple, but appropriate plants and equipment based on, optimization studies and fabrication of such units indigenously, would save, not only time and money but also conserve valuable foreign exchange and hence is a compulsive need of developing countries.

1.0 EXPERIMENTAL WORK:

1.1 Bench Scale

- Origanum
- Origanum fine powder - currently not processed within Turkey - mainly exported.
- Salvia Scalaria L.
- Salvia Triloba
- Salvia Cryptantha
- Storax [Liquidambar Orientalis]
- Rose Domascena
- Capsicum
- Mentha-several species
- Laurus Nobilis
- Glycyrrhiza Glabra
- Chamomila
- Ruscus Aculeatus
- Gypsophila.

1.2 Pilot Plant Scale

- Origanum leaves
- Origanum powder
- Salvia Triloba
- Laurus nobilis [Bay leaf]
- Mentha - several species
- Capsicum - several varieties
- Gypsophila

2.0 INSTALLATION AND COMMISSIONING:

2.1
- Tournaire Pilot plant 500 litres capacity
- 2 M^3 essential oil distillation unit
- Vacuum filter - an accessory to Tournaire pilot plant.

2.2 All Glass Equipment imported from Europe

- Buchi All Glass "Chem Reactor CR 100" - capacity: 100 litres
- Buchi Rotavapor R-1501/E - capacity: 10 lit.
- Buchi Rotavapor R-151/Standard - capacity: 10 lit.
- Soxhlet 12 K. 55 316 - 12 kg/batch capacity

2.3 Modifications to Tournaire - Pilot Plant

- Crusher
- Rectification column - Tournaire's Supply
- Vacuum filter [addition to the pilot plant].

3.0 DESIGN AND ENGINEERING:

- 2 M^3 essential oil distillation unit
- Fractionation column
- Vacuum filter-accessory to Tournaire pilot plant
- Field distillation still 5 kg/batch
- Three stage counter-current percolators [Battery system] using discarded jet-plane exhaust pipes, capacity: 500 kg/batch.

4.0 TRAINING OF ENGINEERS:

4.1 Two graduate chemical engineers have been well trained both in bench-scale process development and pilot plant extractions and essential oil distillation. Both of them can independently operate pilot plants.

Three graduate chemical engineers and a Pharmacy graduate have been trained in all aspects of laboratory and bench-scale process development, all of them have become well conversant in this profession.

4.2 As a result of their association with the Project, two graduate chemical engineers and a pharmacist, have obtained Master's degrees. Three more chemical engineers are expected to obtain Master's degrees in the near future.

CONCLUSIONS:

The Medicinal Plants Research Centre is progressing in the right direction in fulfilling its objectives, in strengthening infrastructure, process development, know-how and design and

engineering. However, it needs further strengthening to enable it, to carry out:

1] Fractionation of essential oils;

2] Isolation of active principles from crude extracts;

3] To develope technologies in the utilization of abundantly available but untapped waste farm products viz., corn cobs, tea waste and tobacco waste, etc.

4] To generate appropriate process data necessary for design and engineering, very essential for the transfer of know-how to industry.

5] To develope capability, to transfer technology on turn-key basis, to user industry.

ADDITIONAL EQUIPMENT SUGGESTED:

1] Four-stage centrifugal type liquid-liquid extractor, with additional clarification and separation bowls, with explosion proof motor, suitable pumps with explosion-proof motors, starters, inter-connecting pipes and fittings; with necessary accessories and spares. All contact parts in SS.316.
Capacity: about 150 - 200 litres/hour.

2] Pressure Leaf Filter with suitable pump with explosion-proof motor and starter, pipe and fittings, leaves with spares. All contact parts in SS.316.

3] Spray Drier complete with all accessories
Evaporation: 50 - 100 litres/hour. All contact parts SS.316.

4] Open Pan evaporator with stirrer motor and starter flame-proof.
Capacity: 100 litres. All contact parts SS.316.

5] Stirred Reactor, Jacketed, suitable for steam heating 4 kg/cm/^{2} with suitable condenser, refluxing arrangements, receivers, water-ring vacuum pump, motors and starter, flame-proof type - all contact parts SS.316.
Capacity: 150 litres.

RECOMMENDATIONS:

To consolidate the gains made during the 1st phase of the project and extend its activities in the industrial utilization of abundantly available plant-based raw materials and to strengthen the expertise in the design and engineering of pilot, proving, & commercial plants, it is recommended to continue its activities into Phase-II.

INTRODUCTION:

Medicinal and Aromatic plants are widely grown all over the world. Due to large scale urbanisation in most of the countries, a large variety of these flora in most cases untapped - are abundantly available in the developing countries.

It has been reported that "about 25% of modern drugs are directly or indirectly derived from plant products".

Turkey is endowed with rich, abundant and varied natural flora, which is awaiting for scientific processing to yield, value added phyto-chemicals and essential oils.

Some of the major plants grown in Turkey are:

- Gypsophilla
- Glycyrrhiza Glabra

- Laurus Nobilis
- Salvia Sclarea
- Origanum
- Liquidambar Orientalis - organised cultivation
- Capscicum
- Rose Domascena - organised cultivation.

Presently no plant based industry, established on scientific lines, exists in Turkey, except perhaps Rose oil industry, which is in existence since mid 1950 - needing modernisation.

It is imperative to develop simple technologies, to help establish small scale industries for the extraction and refinement of intermediate products for export in the first phase. At a later stage, when necessary confidence, expertise and infrastructure is available, the intermediates can be further processed to semi-finished/finished products for internal consumption and/or export.

It is a well-known fact that every plant material is unique by itself, hence there is no universal process technique and processing plant, suitable to process all the plant materials. However, some plant materials could be processed in well-designed multi-purpose units.

Essential oils of plant origin are, complex mixtures of compounds of widely differing compositions and boiling points and form important constituents of cosmetics,perfumery, which contribute fragrance, flavour and preservation of food.

These oils composed mainly, terpene hydrocarbons, sesquiterpenes and oxygenated [perfumery] compounds.

The methods and equipment employed for the recovery of essential oils in some developing countries are often obsolete, resulting in poor and inconsistant quality of oils and consequent poor returns.

The crude oils thus produced are invariably exported, where they are subjected to refining, fractionation and blending to the extracting standards and specific needs, thereby earning enormous profits.

GENERAL METHODS OF RECOVERY OF ESSENTIAL OILS:

Depending upon the nature of plant materials, its end use and the state of development of the country of origin the following techniques are in practice in the recovery of essential oils:

- Extraction with super-critical carbon dioxide
- Extraction with volatile solvents
- Cold pressing
- Steam distillation.

The most commonly practiced and simplest of all the operations followed in the recovery of essential oils from the aromatic plant materials, is steam distillations.

Although steam distillation of aromatic plants is well-known and simple but data on optimum conditions, needs to be worked out on individual raw materials, since every plant material is unique by itself and cannot be generalised.

All plant materials are to be distilled immediately after harvesting, to prevent loss of volatile oils, due to evaporation, oxidation and resinification. However, if it must be stored before distillation, it should be dried under shade and as far as possible, free from air circulation.

STORAGE OF ESSENTIAL OILS:

Essential oils when stored improperly, deteriorate in quality, due to oxidation, resinification, hydrolysis, polymerization, etc.

These oils should therefore be free from moisture. Small amounts can be dehydrated by addition of anhydrous sodium sulphate, followed by filtration. Commercial quantities, however, needs to be processed in a super centrifuge.

Essential oils freed from moisture should be stored in well-fitted, tightly closed, conical top, containers of aluminium at low temperatures and protected from light.

Dark coloured hard glass bottles are suitable for storing small quantities of oils.

For added protection against oxidation, layer of air above the oil, should be replaced with inert gases viz., Co_2 or N_2.

Essential oils with higher content of phenols viz., Laurus Nobilis, Origanum, Thyme, when distilled by farmers in field distillation stills, are generally dark coloured due to the metallic impurities. Such oils can be decolorized by treating with either powdered tartaric acid or its concentrated aqueous solution.

ACTIVITIES CARRIED OUT:

The Medicinal Plants Research Centre of the University of Anatolia Eskisehir, Turkey has been able to establish during the first phase of the Project DP/TUR/83/003, with active assistance and co-operation of UNDP/UNIDO and its experts, basic infrastructure facilities to conduct R & D, scale-up operations, to produce, crude extracts and essential oils based on some of the plants indigenous to Turkey and contacts with appropriate industrial units and academic institutions of Turkey.

Through a series of discussions with the National Project Co-ordinator and his colleagues and visits to the Project site, which was being readied at the time of expert's first

tenure in June, 1986, process developmental programme on bench-scale was initiated in the Department of Pharmacy of the University of Anatolia, on the following plant materials:

- Gypsophila
- Glycyrrhiza Glabra
- Capscicum
- Ruscus Aculeatus
- Rose Domascena
- Liquidambar Orientalis
- Mentha - Several species
- Laurus Nobills
- Salvia Sclarea
- Salvia Triloba
- Chamomila
- Origanum
- Origanum fine powder.

To facilitate percolation of powdered Glycyrrhiza Glabra with hot water, a laboratory model, all glass four stage percolator, has been set-up, fabricated within the University.

BENCH-SCALE AND PILOT PLANT EXPERIMENTAL RESULTS:

- Origanum leaves
- Origanum powder [sponcered by Istanbul Exporter of Organization]
- Salvia Triloba
- Laurus Nobills [Bay leaf]
- Mentha - several species
- Capscicum - several varieties
- Gypsophila

Gypsophila

Gypsophila is one of the major plant resources of Turkey, about 46 species of it grow in the Anatolia region alone. About 250 - 500 TPA of it is being exported.

Gypsophila, contains sugars, resins in addition to triterpenoid saponins known as glycosides. The saponins are usually used in food and drug industry.

The saponins which are glycosides with a non-sugar aglycone portion termed sapogenins. These form molecular complexes with several compounds and are not easily separated in a pure form. They are classified according to its chemical composition into two groups: steroidal and triterpenoid. Saponins of Gypsophila comes under tritepenoid groups.

Saponins are soluble in water and alcohol and are excellent emulsifying agents, but are acrid in taste.

Experiments were conducted on bench scale, using polar and non-polar solvents.

It has been reported in the literature that crushed Gypsophila roots were de-resinified by extraction with petroleum ether and then suponins were extracted with ethanol.

This method, if followed in Pilot plant or commercial plants would involve the use of large amounts of petroleum ether and consequently more solvent losses, which may lead to serious fire accidents, since the plant materials should be totally freed of petroleum ether and then dried before extraction with ethanol.

The procedure worked out, involved refining of crude saponins [relatively small amounts] obtained by extraction of Gypsophila with ethanol.

The results of these extensive studies on bench scale when extended to the pilot plant and or proving plant scale, would be of immense value in the establishment of small or medium scale commercial units.

The experimental work, based on Gypsophila bicolor roots obtained from Van region, eastern part of Turkey. The study established conclusively optimum conditions, viz. particle size, time of extraction, solvents, and the rate of extraction.

Bench Scale Experiments

Soxhlet and Boiling Extraction of G.bicoler Roots with 96% of Ethanol

Time [hours]	0.25 mm Averate particle size		0.3 mm Average particle size		0.46 mm Averate particle size		1.45 mm Average particle size	
	Soxhlet Extraction Yield	Boiling Extr. Yield	Soxhlet Extr. Yield	Boiling Extr. Yield	Soxhlet Extr. Yield	Boiling Extr. Yield	Soxhlet Extr. Yield	Boiling Extr. Yield
	%	%	%	%	%	%	%	%
2	6.20	10.59	6.16	10.29	7.20	9.09	4.94	9.08
4	11.16	11.02	11.77	11.36	13.32	12.16	5.11	9.62
6	14.30	11.33	16.34	12.40	14.58	13.45	10.26	11.06
8	15.46	13.67	16.48	12.49	15.62	13.61	10.34	11.23

Soxhlet and Boiling Extraction of G.bicolor Roots with Water and 80% of Ethanol

Time [hours]	80 % Ethanol		Water	
	Soxhlet Extr. Yield	Boiling Extr. Yield	Soxhlet Extr. Yield	Boiling Extr. Yield
	%	%	%	%
4	20.44	21.55	20.48	24.59
6	25.38	23.66	21.19	26.93
8	25.97	24.10	21.61	27.43

Bench Scale Experiments

	Type	Time	% of the Yield
Petrolium Ether Extraction for Deresinification of 96% Ethanol, 80% Ethanol and water extracted crude Saponins	Soxhlet	3 hrs.	0.95
	Soxhlet	3 hrs.	9.37
	Soxhlet	3 hrs.	21.48
Direct ethanol extracted crude Saponins	Soxhlet	3 hrs.	3.46
Direct ethanol extracted crude Saponins	Boiling	2 hrs.	18.19
Direct water extraction	Boiling	1.5 hrs.	58.53
Direct water extraction	Soxhlet	9 hrs.	46.66

0.3 mm average particle size, 4 - 6 hrs. time of extraction and 96% ethanol gave the best results.

Extractions with water, resulted in the maximum yields, however, the extract contained some resins and sugars. Alcohol extract contained less of these.

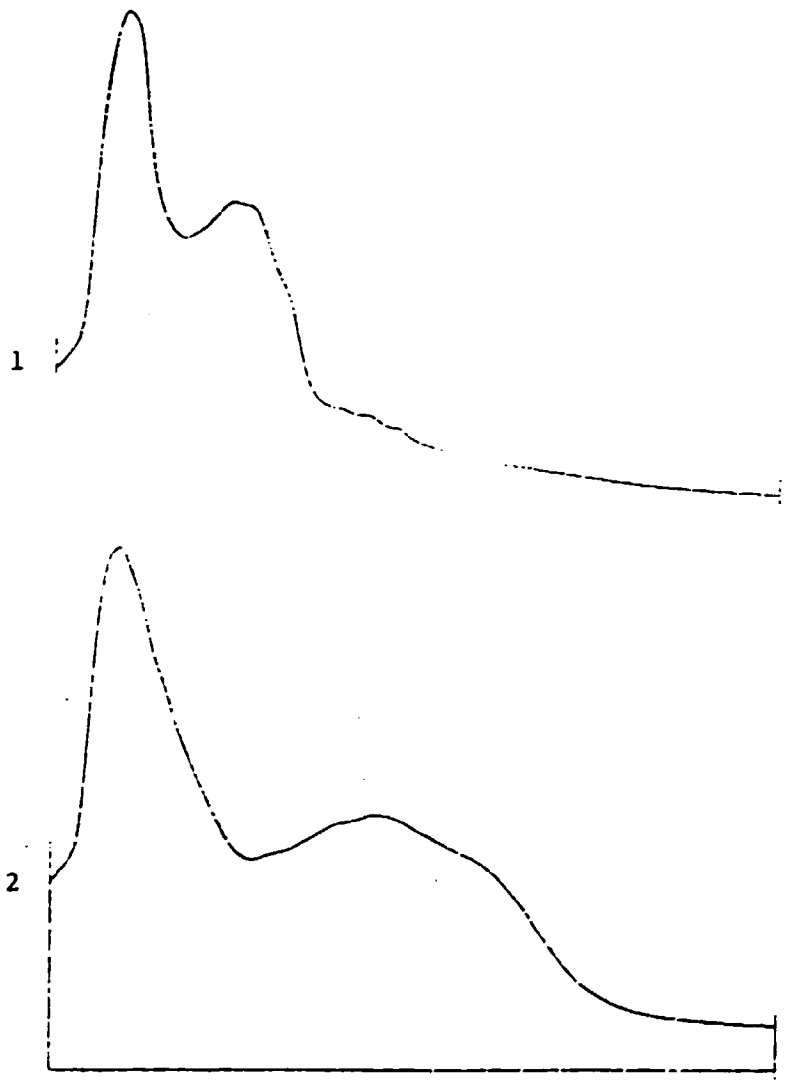
The extractions with water, 96% Ethanol and 80% Ethanol as solverts were worked out by using soxhlet and direct boiling. In the soxhlet extraction with water, foaming interfered with the extraction operation. However, extraction with alcohol in soxhlet, the yield was higher than the extraction directly in boiling alcohol. In the soxhlet extraction the concentration difference between Gypsophila and alcohol higher than the boiling extraction. For this reason the quality of saponins obtained in the soxhlet extraction was much better. However, when particle size increased to 1.45 mm the saponins obtained the boiling extraction were of good quality.

Saponins were precipitated in the cold medium. Precipitation was done either cooling the extract or dropping chilled acetone into it. But precipitation of saponins using water extract was done by addition of some alcohol after the extract was concentrated. By this method the saponins that were precipitated with acetone were white in color, however the yield was very low. But the saponins that were precipitated with alcohol after the extraction with water were not white in color but the yield was more than the other. Ether was also used for precipitation of saponin instead of acetone but no difference was noticed.

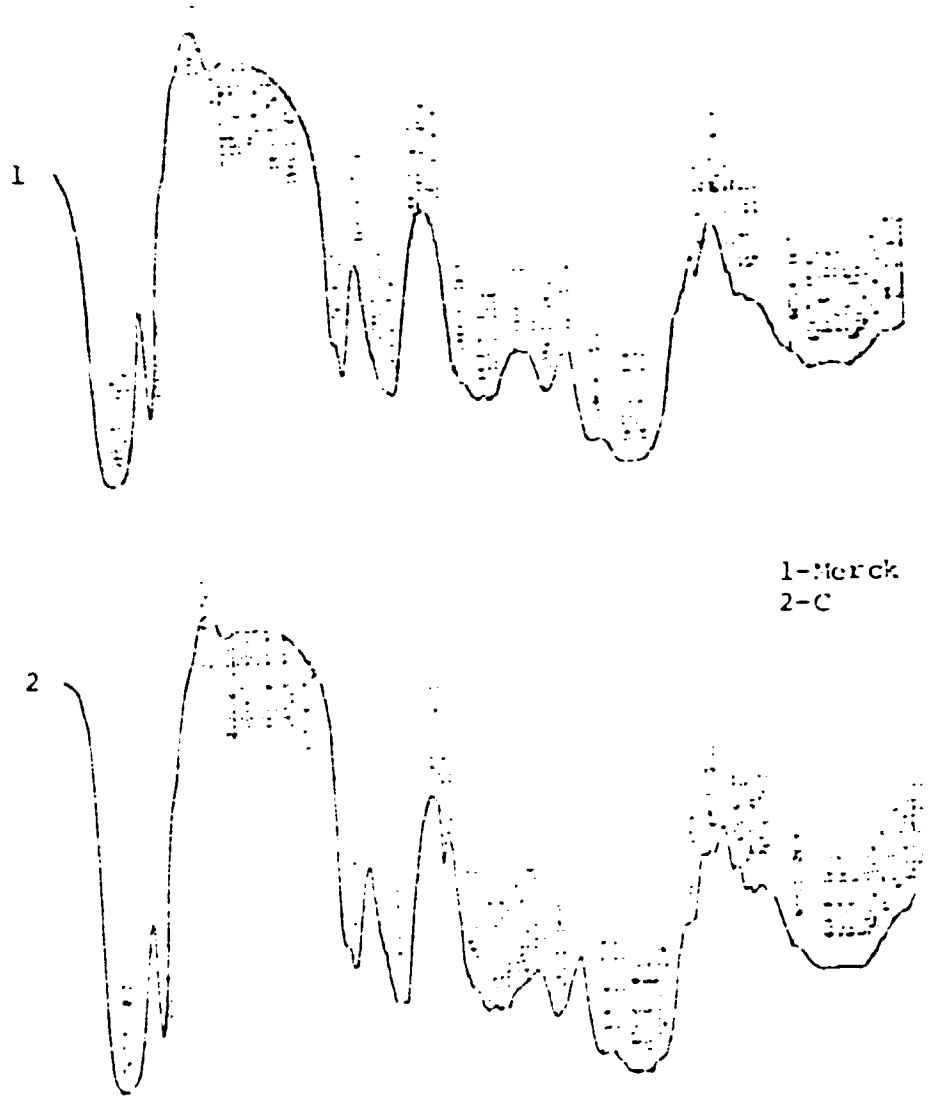
The spectroscopic and chromatographic evaluation of saponins obtained in this studies and that of Merck sample were done, using UV, IR and TLC instruments.

UV and IR spectrums of Merck saponin and the saponins coded [C] that was obtained by precipitation from the alcoholic extraction of the roots are shown in the following sheets.

1-Merck
2-C



These figures show UV spectrums of Merck Saponin and the experimental saponins, coded 'C'.



These figures show IR spectrums of Merck Saponin and the saponins coded 'C'.

It was noticed from the results thus obtained that there was not much difference between these samples. However, slight deviations showed in this evaluation was due to presence of some impurities in the experimental samples.

Capsicum:

The genus capsicum, which is commonly known as chilli, red chilli, hot red pepper. Kabasco is a member of the solanaceae, that originated in central and south America. These are of medicinal and culnary value.

Several varieties of capsicum are largely cultivated in Kahramanmarash, Kayseri, and Bursa regions of Turkey. Its production in Turkey during 1982-1986 was around 3800 TPA.

Capsicum species forms and accumulates several pungent materials in its fruits. Pungency is due to the presence of a group of compounds, known collectively as capsaicin.

Generally polar solvents viz., methyl, ethyl alcohols, acetone, are used for the extraction of crude extract known as oleoresin. The oleoresin contains impurities such as fats, lipids, pigments in addition to capsaicin.

In this work, it was aimed to establish extraction parameters, of hot red peppers from several parts of Turkey and the capsaicin content in them. For this purpose six hot red peppers, were collected from the above mentioned regions of Turkey.

Experimental Results [Bench Scale]:

A total of about 30 extractions have been made, using six varieties of capsicum cultivated in Turkey using: [i] Ethanol, [ii] Acetone, and [iii] n-Hexane. Ethanol gave maximum yields of oleoresin.

Extraction of Capsicum

Sample		1		2		3		4		5		6	
Solvent		OLEORESIN %	CAPSAICIN IN OLEORESIN	OLEORESIN %	CAPSAICIN IN OLEORESIN	OLEORESIN %	CAPSAICIN IN OLEORESIN	OLEORESIN %	CAPSAICIN IN OLEORESIN	OLEORESIN %	CAPSAICIN IN OLEORESIN	OLEORESIN %	CAPSAICIN IN OLEORESIN
Ethanol	a]	33		28		26		45.2		30.6		24.7	
	b]	40				27		54.0		27.6		31.4	
	c]							41.5				36.6	
	d]											34.5	0.40
	e]											20.0	0.32
Acetone	a]	13.5		14.6	0.286	11.2		21.4		9.6		14.2	
	b]	11.0		13.4		10.6		21.5		8.0		12.4	
n-Hexane	a]			14.5	1.1	8.5	1.68						

Note: 1] Percentages on MFB. [moisture free basis]

2] [b] - [e]: Cold percolation in 4-stage extractor.

3] [b] - [d]: A sample from bulk of 300 kg. lot.

Bench-Scale Ethanol Extraction Results:

Sample	Yield, % [MFB]	Capsaicin % [in Oleoresin]
1.	36.5	0.42
2.	28.0	0.80
3.	26.5	0.18
4.	47.0	0.28
5.	29.0	0.87
6.	36.0	0.13
7.	22.0	0.61
8.	30.0	0.58
9.	24.7	1.49
10.	5.6*	4.46
11.	12.0	2.50
12.	24.4	1.44
13.	31.2	1.88
14.	26.0	0.22

* Cold Ethanol extraction.
MFB: Moisture free basis.

The Effect of Particle size:

The following results were obtained by the extraction of sample 6 with Ethanol [96%] [different particle size].

Extraction time: 7.0 hours.

Particle Size [mm]	Yield, % [MFB]
Fine	22.1
0.42 - 0.5	26.4
0.50 - 0.595	23.1
0.595- 0.707	23.0
0.707-0.841	22.0
0.841- 1.19	18.3
1.19 -	13.0

Optimum particle size: 0.5 - 0.8mm.

The Effect of Extraction Time:

Using sample 6 having 0.59 mm average particle size the following data was obtained:

Time [hrs.]	Yield, % [MFB]
1/2	8
1	12
2	15
3	17
4	20
5	22
6	22
7	23

Optimum Extraction time: 4 - 5 hrs.

The Effect of Storage Time :

Extracting sample 6 kept at 10 - 20°C in a dry medium for 10 months storage [86, August - 87, April] the following results were obtained:

Time [month]	Yield, % [MFB]
1	36
2	34
3	32
4	19
5	21
8	22
9	22
10	21

Pilot-Plant Extraction [of Sample (6)]:

Sample 6 was extracted in 500 L Tournaire extractor with ethanol [96%]. The results as follows:

Batch	Quantity [kgs]	Ethanol [lit]	Time of Extraction [hrs.]	Oleoresin [kgs]	Yield % [MFB]
1	23	200	7	5.65	27
2	25	200	5	4.86	21

Note:-

1] ist batch, boiling alcohol extraction

2] 2nd batch, extraction at 40 - 45°C.

Licorice from Glycerrhiza Glabra

Licorice contains about 7% of the triterpenoid glycoside. glycrrhizin. The major use of licorice is as a flavouring agent in tobacco and confectionery, its derivatives are also used in pharmaceuticals.

Various methods have been worked out in the laboratory using water as solvent viz.,soxhlet, boiling in water and percolation. The parameters like.extraction time, solid/water ratio, extraction temperature, and the yield and quality of licorice have been worked out.

Experimental Results

Boiling in Water

Amount of solid	:	15 gm.
Amount of water	:	150 ml.
Total extraction time	:	9 hrs. [3 x 3]
Moisture content	:	7.9%
Total mother liquor	:	111 ml.
Total dry extract	:	3.18 gm.
Yield %	:	25.44%

Soxhlet Extraction

Amount of solid	:	15 gm.
Amount of water	:	150 ml.
Extraction time	:	14 hrs.
Moisture content	:	7.9%
Total mother liquor	:	106 ml.
Total dry extract	:	3.3 gm.
Yield %	:	23.9%

Percolation System

Number of Percolators	Raw Mat. [gm.]	Temp.of Water °C.	Temp. of Percolator °C.	Flow Rate [ml/min]	Time [hr]	Dry Ex. [gm.]	Yield [%]
1	60	90	80	3.5	5.0	15.1	27.1
1	80	90	80	4.6	2.5	16.56	22.2
2	40+40	90	80	13.0	2.7	17.16	23.0
4	4x50	90	1-60	9.4	3.25	41.88	22.54
			2-54				
			3-35				
			4-30				
4	4x50	90	1-60	9.3	5.25	40.2	21.6
			2-48				
			3-41				
			4-36				
4	4x50	90	1-53	12.0	4.0	50.42	26.77
			2-43				
			3-39				
			4-32				
4	4x25	90	1-64	7.4	6.0	23.16	25.52
			2-47				
			3-41				
			4-33				

Percolation System

NUMBER OF EXPERIMENT	MOISTURE CONTENTS [%]	NUMBER OF PERCOLATORS	RAW MATERIAL [Gm]	TEMP. OF WATER [°C]	FLOW RATE [ML/MIN.]	TEMP. OF PERCOLATOR [°C]	TOTAL EXTRACT [ML]	DRY EXTRACT [Gm]	TIME [Hr]	% YIELD
1	7.08	4	200 ::	90	9.4	1 - 69 2 - 54 3 - 35 4 - 30	1840	41.88	3.25	22.5
2	7.08	4	200 ::	85-90	9.3	1 - 60 2 - 48 3 - 41 4 - 36	2910	40.2	5.25	21.6
3	7.08	4	200 ::	85-90	12.0	1 - 53 2 - 43 3 - 39 4 - 32	2825	50.42	4.0	27.1
4	9.15	4	100	85-90	7.4	1 - 64 2 - 47 3 - 41 4 - 33	2650	23.16	6.0	25.5
5	9.15	1	25	85-90	-	80	2850	-	-	30.12
6	6.87	4	80 :::	85-90	4.0	-	1730	27.2	4.4	31.6
7	6.87	1	60	90	3.5	80	1030	15.1	5.0	27.1
8	6.87	1	60	70	7.3	45 - 50	700	9.9	1.6	18.0
9	6.87	1	80	90	4.6	80	700	16.56	2.5	22.2
10	6.87	2	80	90	13.0	80	2100	17.16	2.7	23.0

:: 50 grams in each Percolator; ::: 20 grams in each Percolator.

Soxhlet Extraction

1] 20 gr. raw material, 19.5 hr., 28.9% yield
2] 20 gr. raw material, 10 hr., 20.3%
3] 15 gr. raw material, 02.0 hr., 14.53%
4] 15 gr. raw material, 24 hr., 20.9% [alcohol extrac.]

RESULTS: Batch System

Number of Experiment	Moisture Content [%]	Raw Material [gr.]	Water [ml.]	Temperature [°C.]	Time [min]	Total Extract [ml.]	Total Dry Extract [gr.]	% Yield
1	7.08	10	80	Ambient	10	63	1.18	12.7
2	7.03	10	80	Ambient	20	60	1.19	12.8
3	7.08	10	80	Ambient	30	61	1.17	12.6
4	7.08	10	80	50	10	55	1.05	11.3
5	7.08	10	80	50	20	50	1.04	11.2
6	7.08	10	80	50	30	50	1.34	14.42
7	7.08	10	120	Ambient	10	105	1.01	10.9
8	7.08	10	120	Ambient	20	110	1.25	13.45
9	7.08	10	120	Ambient	30	110	1.05	11.3
10	7.08	10	120	50	10	100	1.52	16.4
11	7.08	10	120	50	20	100	1.38	14.8
12	7.08	10	120	50	30	95	1.61	17.3
13	6.87	10	200	Ambient	10	185	1.29	13.85
14	6.87	10	200	Ambient	20	185	1.26	13.5
15	6.87	10	200	Ambient	30	190	1.41	15.1
16	6.87	10	200	50	10	180	1.58	17.0
17	6.87	10	200	50	20	175	1.93	20.7
18	6.87	10	200	50	30	165	1.98	21.3

4-Stage Percolators

Plant Material: Glycyrrhira Glabros Root

Experiment No.	Weight of Material	No. of Stages used	Solvent used	Rate of Flow	Temperature °C.	Dry Extract gm.	% [MFB]
1. Moisture content	200 gr. [6.39%]	4	Water	-	1st.: 65 - 75 2nd.: 50 - 55 3rd.: 30 - 40 4th.: 27 - 33	41.9	22.38
2. Moisture content	200 gr. [5.35%]	4	Water	-	1st.: 57 - 65 2nd.: 45 - 55 3rd.: 38 - 43 4th.: 34 - 36	47.8	25.25
3. Moisture content	200 gr. [5.84%]	4	Water	-	1st.: 47 - 57 2nd.: 41 - 47 3rd.: 35 - 40 4th.: 31 - 33	49.5	26.28
4. Moisture content	100 gr [12.0%]	4	Water	-	1st.: 60 - 65 2nd.: 45 - 50 3rd.: 40 - 45 4th.: 30 - 35	23.16	26.32

Storax:

Storax has been known from ancient times. It is used as antiseptic, as fixative in perfume and in soap and tobacco industry.

Visited Storax growing and processing areas in Marmaris, to study the methods used in the processing of storax.

Presently storax balsom is being extracted from balsom smeared chips in a very crude manner, by boiling the chips in water, heated directly by burning wood and then pressing the mass by hand operated hydraulic press to separate the crude balsom from chips. The crude balsom thus obtained contains about 25% water, 7 - 10% bark dust and other impurities.

Series of experiments were conducted to extract balsom from balsom smeared chips by the use of various solvents on bench scale.

Extraction of Refined Balsom from crude Balsom obtained from Marmaris, Turkey:

Solvent	n Hexane	Ethyl Acetate	Ethyl Ether
Yield %	25.5	74.69	74.2
Acid Number [50 - 85]	32.8	62.88	67.8
Saponification Number [160 - 200]	-	-	194.3
Ester Number	-	-	126.45
Total Balsomic Acid [%] [28.5%]	-	30.13	30.93

Balsom Smeared Shavings

Solvent	Toluene	n-Hexane	Ethanol	Chloroform
Yield [%]	28.478	9.423	28.34	29.44
Acid Number [50 - 85]	54.87	-	73.08	69.49
Saponification Number [160 - 200]	172.00	134.3	164.50	164.00
Ester Number [107 - 122]	117.40	-	91.80	94.60
Total Balsomic Acid [%] [28.5%]	16.90	13.18	12.00	9.50

Extraction of Pure Balsom from Crude Balsom [From Marmaris]

	Fresh Crude Balsom [1987]						Old Crude Balsom [1986]					
	Yield of pure Balsom W/W %	Water Content V/W % *	Impurities W/W % **	Acid No.	Saponif. No.	Total Balsomic Acid	Yield of Pure Balsom W/W %	Water Content V/W %	Impurities W/W %	Acid No.	Saponif. No.	Total Balsomic Acid
				USP [50-85]	USP [160-200]	USP min.28.5%				USP [50-85]	USP [160-200]	USP min.28.5%
Ethyl Acetate	61.9	28.9	9.2	57.1	196	31.3%	74.69	7.65	17.65	62.81		30.11
n-Hexane	17.8	28.9	53.3	25.4	191	29.7%	25.48 [4 extrns.]	7.65	-	32.87		
Ethyl Alcohol	44.7	28.9	26.5	57.4	179	29.0%	75.67	7.65	16.68			
Fresh Chips [1987]							Fresh Chips [1987]					
Ethyl Acetate Acetate	18.1	14.4	67.5	54.3	230	29.8%						

* = Water: Estimation by direct distillation as per Turk Farmakopesi.

** = Impurities bark chips, powder, Tannins, Pigments, etc.

Good quality balsom according USP should contain 28.5% balsomic acid, a very important parameter. The balsom extracted by all the solvents contained balsomic acid above the limits specified in USP. However, the yield of pure balsom obtained by ethyl acetate was 61.90%.

Ethyl alcohol and n-hexane yielded 44.7% and 17.8% respectively.

USP and B.P. methods were used for analysis.

Camamile:

Camamile known as Papatya in Turkish, yields pale blue or greenish blue oil, the principal constituents of this oil are camomelene, angelic acid and its iso-amyl and isobutyl esters. The characteristic colour of oil is due to the presence of a pigment azulene.

It is used as a perfume in shampoo and as a tobacco flavour.

Several batches of fresh flowers were hydrodistilled in the laboratory and pilot plant.

In the laboratory, in addition to the use of the conventional clevenger apparatus, a laboratory model fractionating column was also used. In the later apparatus, the yield of oil was almost double. This was due to the fact that some of the water soluble constituents of the oil was separated in the column, thus increasing the overall yield of oil.

Due to non-availability of a good large size fractionation facility, this very important technique, remained untested on large scale.

Dry Flowers		Clevenger Apparatus	Laboratory Column
Dry Flowers : 100 grams Water : 1 litre Time : 3.5 hours		0.12%	0.3%
Dry Flowers : 100 grams Water : 1.5 litres Time : 3.5 hours		0.15%	0.4%
Dry Flowers : 80 grams Water : 1.0 litre Time : 3.5 hours		0.25%	0.625%
Dry Flowers : 80 grams Water : 1.0 litre Time : 3.5 hours		0.25%	0.625%
Dry Flowers : 80 gm [old] Water : 1.0 litre Time : 3.5 hours		0.25%	0.5%
<u>Pilot Plant</u> [Hydrodistillation]			
Dry Flowers : 5.0 kgs. Water : 5.0 litres Time : 2.5 hours		0.11%	-
Dry Flowers : 5.0 kgs. Water : 5.0 litres Time : 3.0 hours		0.113%	-
Dry Flowers : 5.0 kgs. Water : 5.0 litres Time : 3.0 hours		0.11%	-

Percent of oil obtained in the laboratory clevenger apparatus = 0.12%.

LAURUS NOBILIS L.

Laurus Nobilis L. grows wild in Mediterranean countries. The aroma of its oil is fragrant and taste, aromatic and bitter.

The dried leaves yield about 2 - 3% oil on steam distillation.

Cineol constitutes the chief component of its essential oil. The other components are α-pinene, α-phellandrene, linalool, α-terpeneol, geraneol, etc.

Several batches of dried leaves varying from 35 kgs to 50 kgs per batch have been distilled. It has been observed that:

- because of size and shape of dried leaves, these could not be packed well in the distillation still, a condition, very essential for effective recovery of essential oil. Sprinkling of small amounts of water at the time of packing, was a satisfactory answer.

- Some of the oil separated easily on the top of the water, however, some formed an emulsion,

  The emulsion from the separator has been introduced into the second separator, through a layer of petroleum ether, the oil recovered by distilling off the solvent.

Experimental Results

Steam Distillation - Laurus Nobilis

Batch No.	Qty. of Dry L.N. leaves [kg]	Time of Distil-lation [hr]	Rate of Steam Lit/kg.of charge/hr	Vol. of ml. di-rect Sap^{n}. ml.	Oil through pet.Ether ml.	Total [%]
1	45.0	4.0	1	757 [1.68%]	50 [0.11%]	1.79
2	47.0	3.5	1	985 [2.1 %]	80 [0.17%]	2.27
3	49.5	4.0	1	835 [1.69%]	125 [0.25%]	1.94
4	49.0	4.0	1	950 [1.94%]	[0.12%]	2.06

Total oil content in the charge material = 2.4%.

Rate of Distillation - Batch Charge

S.No.	Time [min]	Interval [min.]	Yield of Oil % of Total	Quantity/Batch [kg.]	Steaming Rate kg/kg of charge/hour
1.	60	60	51.37	45	1
2.	120	60	19.81		
3.	180	60	6.97		
4.	240	60	not collected		
5.	360	120	21.86		

Rate of Distillation - Laurus Nobilis

[Pilot Plant]

S.No.	Time [min.]	Interval [min.]	Weight of Batch			
			34 kg.	34 kg.	38 kg	38 kg
			Steam rate kg/kg/hr	Steam rate kg/kg/hr	Steam rate kg/kg/hr	Steam rate kg/kg/hr
			1.38-1.45	1.38-1.45	0.79-1.06	0.76-1.06
			Oil Obtained			
			Vol. ml..%	Vol. ml..%	Vol. ml..%	Vol.ml..%
1.	60	60	51.37	88.0	490	71.63
2.	120	60	19.81	72.5	112	16.37
3.	180	60	6.97	15.5	34	4.97
4.	240	60	not collected	not collected	15	2.19
5.	360	120	21.86	80.0	33	4.82

Steam Distillation of Laurus Nobilis

No. 1

Batch Size: 45 kgs.

Time of Distillation: 4 hrs.		Vol [cc]	%
Percentage of Oil recovered	- Direct separation:	757	1.68
	- Through Pet.Ether:	50	0.11
	Total	807	1.79

Rate of Steam : Approximately 1 litre per kg. of bay leave per hour.

No. 2

Batch Size: 47 kgs.

Time of Distillation: 3.5 hrs.		Vol [cc]	%
Percentage of Oil recovered	- Direct separation:	985	2.1
	- Through Pet.Ether:	80	0.17
	Total	1065	2.27

Rate of Steam : Approximately 1 litre per kg. of bay leave per hour.

No. 3

Batch Size: 49.5 kgs.

Time of Distillation: 4 hrs.		Vol [cc]	%
Percent of Oil recovered	- Direct separation:	835	1.69
	- Through Pet.Ether:	125	0.25
	Total	960	1.94

Rate of Steam : Approximately 1 litre per kg. of bay leave per hour.

No. 4

Batch Size: 49 kgs.

Time of Distillation: 4 hrs.		Vol [cc]	%
Percentage of Oil recovered	- Direct separation:	950	1.94
	- Through Pet.Ether:	60	0.12
	Total	1010	2.06

Rate of Steam : Approximately 1 litre per kg. of bay leave per hour.

Weight of Batch : 34 kgs.
Steam Rate : 1.38 - 1.45 kg/kg of material
Overall recovery of oil: 1.08%

S.No.	Time Cumulative	%	Interval
1.	60 min.	51.37	60 min.
2.	120 min.	19.81	60 min.
3.	180 min.	6.97	60 min.
4.	360 min.	21.86	180 min.

As determined in Clevenger Apparatus [3 hrs.]

Oil content in the feed 2.01 %

In the marc 0.039%

Weight of batch : 34 kgs.
Steam Rate : 1.38 - 1.45 kg/kg of material

Overall recovery of Oil: 1.08%

S.No.	Time Cumulative	Oil [ml]	% Cumulative
1.	60 min.	188.0	51.37
2.	120 min.	260.5	71.17
3 .	180 min.	286.0	78.14
4.	360 min.	366.0	100.00

Oil in feed 2.01 %
Oil in Marc 0.016%

Weight of batch : 38 kgs.
Steam Rate : 0.79 - 1.6 kg/kg of material

Overall recovery of Oil: 1.80%

S.No.	Time Cumulative	Oil [ml]	% Cumulative
1.	30 min.	315.0	46.05
2.	60 min.	490.0	71.64
3.	120 min.	602.0	88.01
4.	180 min.	636.0	92.98
5.	240 min.	651	95.18
6.	330 min.	684	100.00

Oil in feed 2.32 %
Oil in Marc 0.016%

Weight of Batch : 38 kgs.

Steam Rate : 0.79 - 1.06 kg/kg of material.

Overall recovery of Oil: 1.80%

S.No.	Time Cumulative	%	Interval
1.	30 min.	46.05	30 min.
2.	60 min.	25.58	30 min.
3.	120 min.	16.37	60 min.
4.	180 min.	4.97	60 min.
5.	240 min.	2.19	60 min.
6.	330 min.	4.82	90 min.

Oil in Feed 2.07 %

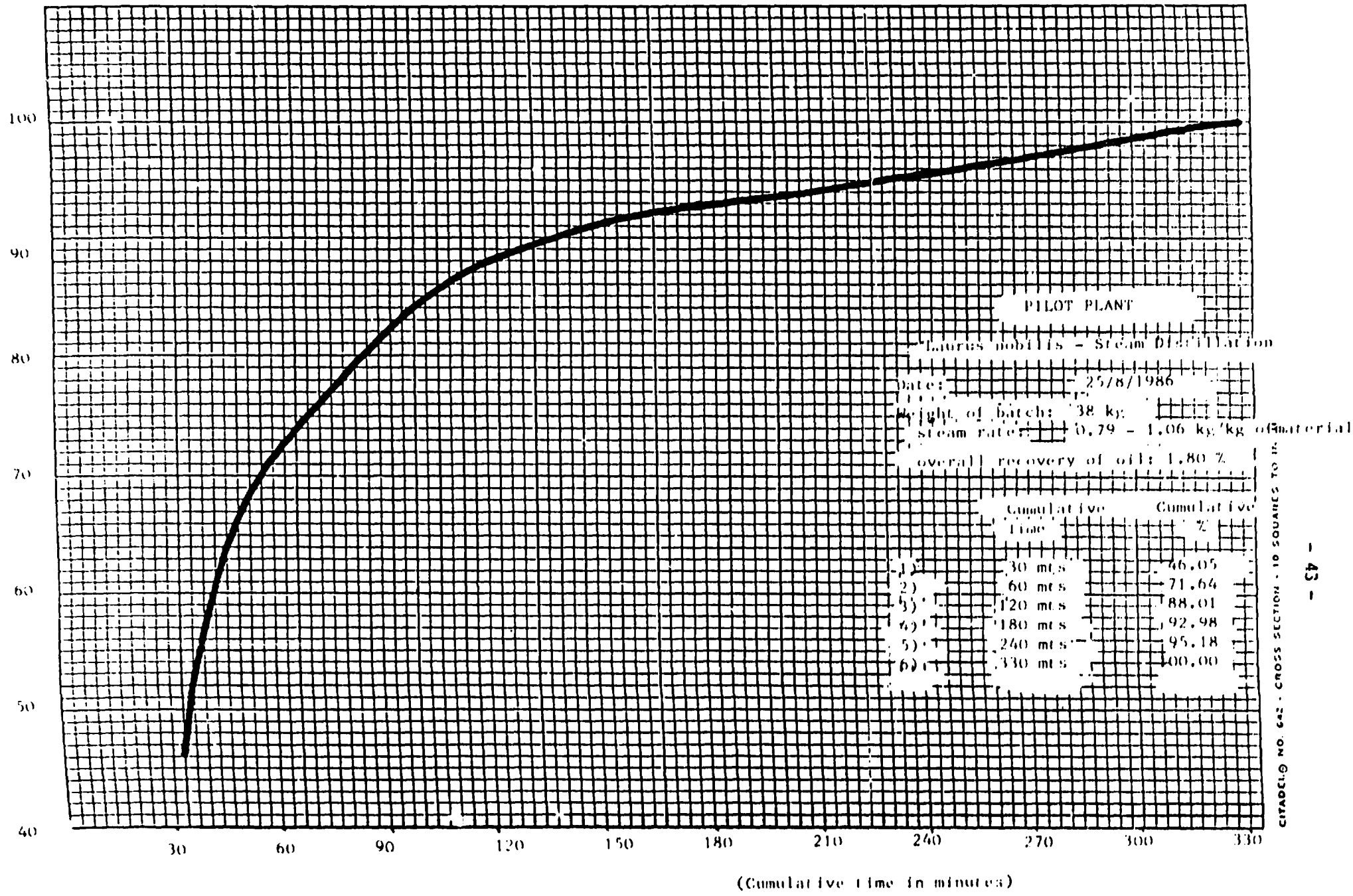
Oil in Marc 0.058%

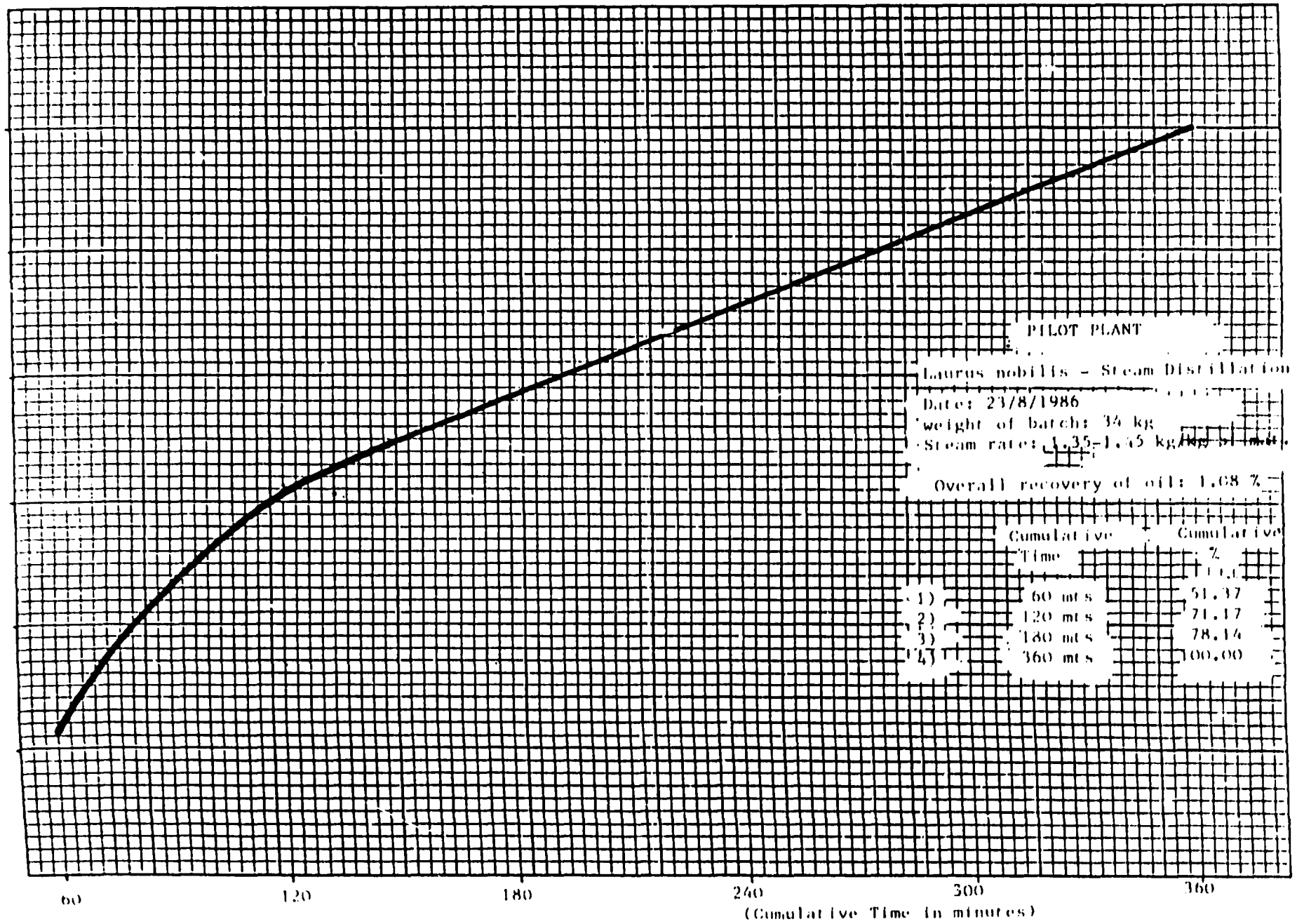
Analysis Oil of Laurus Nobilis L.

1. Gas Chromatogram: Total volatile oil
2. IR Spectrogram : Total volatile oil
3. Refractive Index: $n_D^{20} = 1.4625$
4. Optical Rotation: $\overset{20}{D} = -2.15$
5. Solubility : Very soluble in Ethanol [96 percent]
6. Weight per ml. : 0.95 gm/ml.

Laurus Nobilis L.

Components	Pilot Plant	Clevenger	Pilot Plant
	%	%	
α-pinene	7.5984	5.0866	5.1982
Camphere	0.0891	0.4268	0.5208
β-pinene	5.3584	3.9846	4.1007
Sabinen	9.6635	7.8307	8.3658
Myrcen	0,7906	0.4876	0.4528
α-terpinen	0.4803	0.3044	0.3242
D-Limonen	1.4305	1.2475	1.1753
1,8-Cineol	49.4749	53.8726	58.9781
γ-terpines	0.9913	0.6321	0.69
p-Cynol	1.0166	0.9804	1.0968
Terpinolin	0.1933	0.1601	0.1464
Linalool	0.3913	0.1047	0.1625
Linalylacetate	0.4326	0.2745	0.3256
Terpinen-4-ol	1.6541	2.433	1.7941
Terpenyl acetate	9.3156	10.5119	7.1499
α-Terpineol	1.2525	2.0003	1.3298
Geranyl acetat	0.2577	0.1046	0.0641


PILOT PLANT
Laurus nobilis - Steam Distillation
Date: 25/8/1986
Weight of batch: 38 kg
Steam rate: 0.79 - 1.06 kg/kg of material
overall recovery of oil: 1.80 %
Cumulative Time
Cumulative %
1) 30 mts 46.05
2) 60 mts 71.64
3) 120 mts 88.01
4) 180 mts 92.98
5) 240 mts 95.18
6) 330 mts 100.00
100
90
80
70
60
50
40
30
60
90
120
150
180
210
240
270
300
330
(Cumulative time in minutes)
CITADEL® NO. 642 - CROSS SECTION - 10 SQUARES TO IN.


PILOT PLANT
Laurus nobilis - Steam Distillation
Date: 23/8/1986
Weight of batch: 34 kg
Steam rate: 1.35-1.45 kg/kg of mat.
Overall recovery of oil: 1.68 %
Cumulative Time
Cumulative %
1) 60 mts 51.37
2) 120 mts 71.17
3) 180 mts 78.14
4) 360 mts 100.00
100
90
80
70
60
50
60
120
180
240
300
360
(Cumulative Time in minutes)
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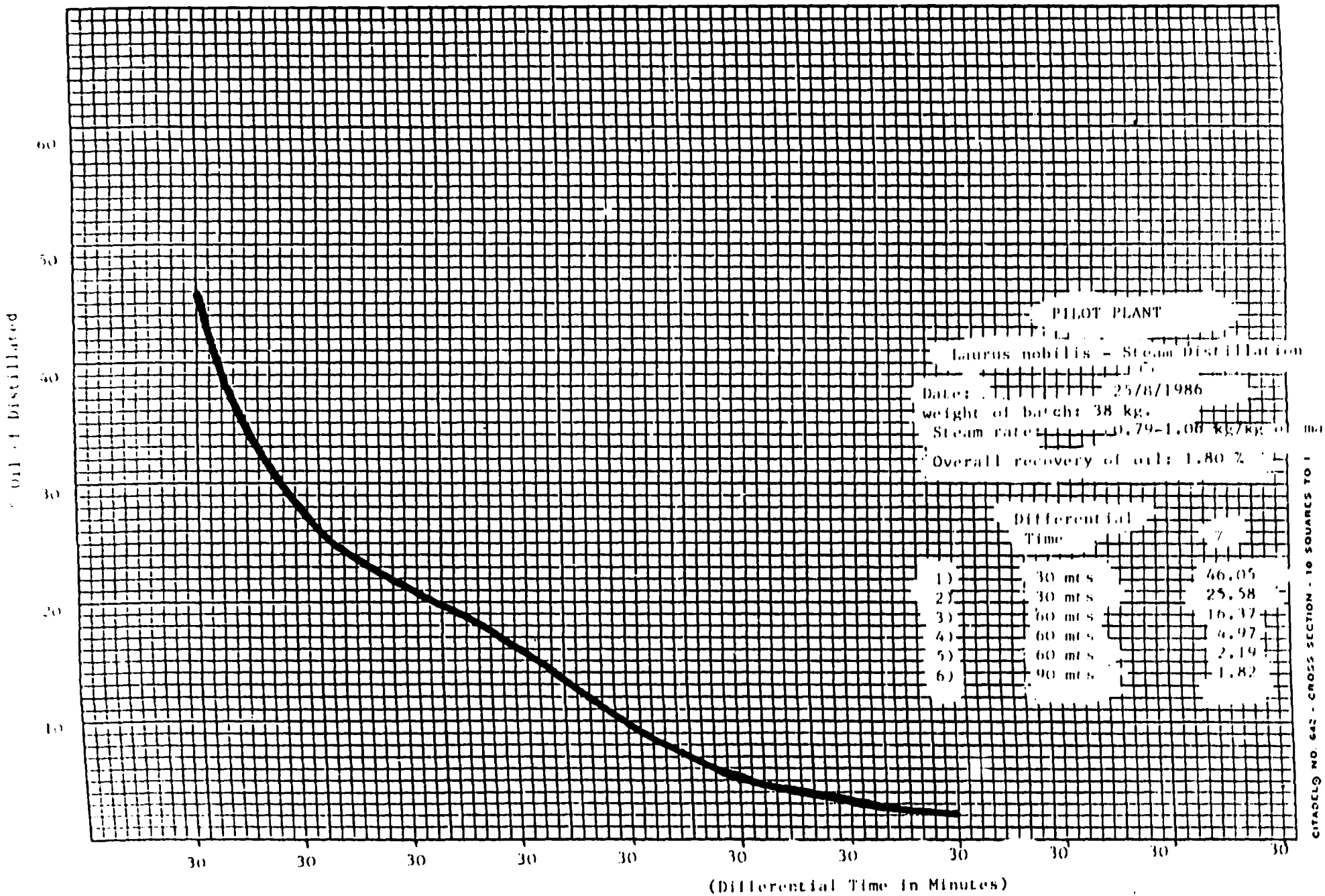
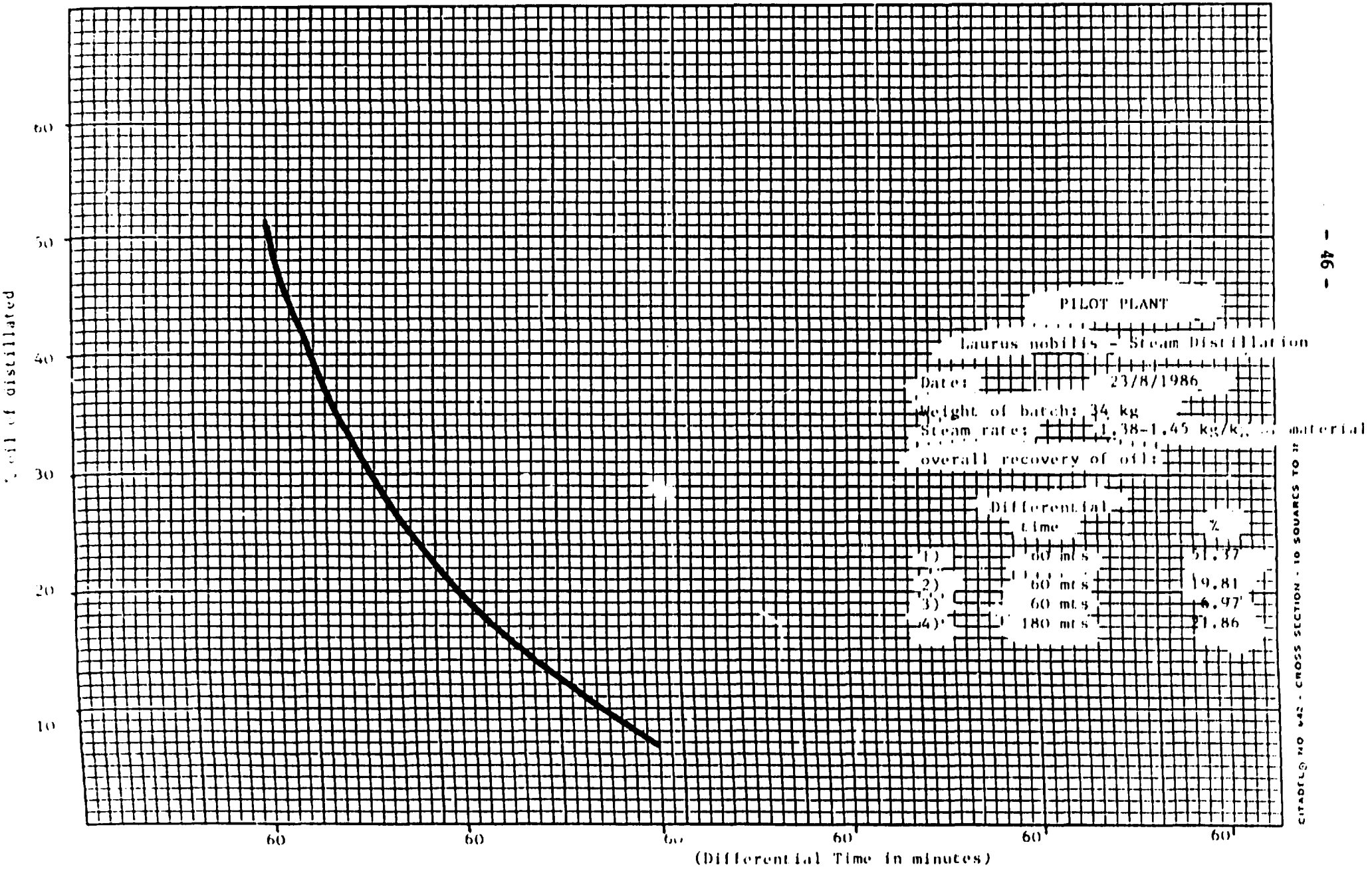

PILOT PLANT
Laurus nobilis - Steam Distillation
Date: 25/8/1986
weight of batch: 38 kg.
Steam rate: 0.79-1.00 kg/kg of mat.
Overall recovery of oil: 1.80 %
Differential Time
1) 30 mts 46.05
2) 30 mts 25.58
3) 60 mts 16.37
4) 60 mts 4.97
5) 60 mts 2.19
6) 90 mts 1.82
60
50
40
30
20
10
30 30 30 30 30 30 30 30 30 30
(Differential Time in Minutes)
CITADEL® NO. 642 - CROSS SECTION - 10 SQUARES TO 1


PILOT PLANT
Laurus nobilis - Steam Distillation
Date: 23/8/1986
Weight of batch: 34 kg
Steam rate: 1.38-1.45 kg/k... material
overall recovery of oil:
Differential time
1) 60 mts 51.37
2) 60 mts 19.81
3) 60 mts 6.97
4) 180 mts 21.86
60
50
40
30
20
10
60
60
60
60
60
60
(Differential Time in minutes)
CITADEL® NO 642 - CROSS SECTION - 10 SQUARES TO

SALVIA TRILOBA

Rate of Distillation - Salvia Triloba [Pilot Plant]

S.No.	Time [min.]	Interval [min.]	Weight of Batch [Kg.] 54		49.3		50		42.5	
			Steam Rate of Flow Kg/Kg/hr. 0.6 - 0.99		0.46 - 1.08		0.51 - 0.63		0.79 - 1.5	
			Oil Obtained							
			Vol.ml.	%	Vol.ml.	%	Vol.ml.	%	Vol.ml.	%
1.	10	10	447	40.14	460	32.43	427	40.61	335	43.34
2.	20	10	365	32.75	448	31.58	215	20.45	245	31.69
3.	40	20	225	20.21	370	26.08	300	28.53	96	12.42
4.	70	30	47	4.22	98	6.91	68	6.47	41	3.75
5.	130	60	15	1.35	28.5	2.01	17.5	1.66	12	1.55
6.	190	60	8	0.72	14	0.99	14.0	1.33	56	7.24
7.	250	60	6.5	0.52	-	-	10.0	0.95	-	-

SALVIA TRILOBA L.

Oil Content - Predistillation and Post Distillation

Time of Distillation = 3 hrs.

Batch No.	Oil in the Feed [%]	Oil in Marc [%]
1.	1.42	0.06
2.	1.80	0.10
3.	2.50	0.11
4.	2.08	0.05

Analysis Report of Oil of Salvia Triloba L.

1. Gas Chromatogram : Total volatile oil.
2. IR Spectrogram : Total volatile oil.
3. Refractive Index : $n_D^{20} = 1.4715$
4. Optical rotation : $\overset{20}{D} = 1.38$
5. Solubility : Very soluble in Ethanol [96 percent]
6. Weight per ml. : 0.91 gm/ml
7. Cineol Content : 41.6%

Composition of Steam Distilled Oils of Salvia Species

Components	Salvia Triloba L. [%]	S.Cryptantha [%]	S.Sclarea P/Plant [%]	S. Sclarea Clevenager [%]
	[P/plant]	[P/plant]		
α -pinene	5.7203	14.7793	0.0361	0.0249
Camphene	3.3044	9.4754	-	-
β -pinene	4.5465	7.2089	0.0622	0.0611
Sabinene	0.1638	0.5217	-	-
Myrcene	3.313	2.1801	1.5238	1.822
α -terpinen	0.3329	0.2691	-	-
D-linonine	1.3982	2.2141	0.3387	0.7307
Cineol	41.6461	18.0258	-	0.2657
γ -terpinen	0.4949	0.6588	0.6283	0.8415
p-cynol	1.1758	0.2778	-	-
Terpinolen	0.138	0.2599	-	-
Camphor	7.2708	8.8213	-	-
Linalool	0.508	0.6922	14.3782	20.4821
Linaly acetate	1.1596	2.961	63.3779	37.1179
Caryophyllene	0.6486	0.2626	-	0.1398
α-humulene	0.6341	1.7088	0.0924	0.0848
Borneol	2.0206	1.9074	5.1807	8.3021

Laboratory Distillation of Salvia Sclerea L.

S.No.	Weight of charges [gm.]	Yield of Oil		Time
		Vol.in ml	%	
1.	50	0.2	0.4	
2.	10	0.04	0.4	
3.	25	0.11	0.44	
4.	25	0.11	0.44	
5.	10	0.04	0.40	

Since oil content in S.Sclarea was too low no experimerts were conducted in Pilot plant.

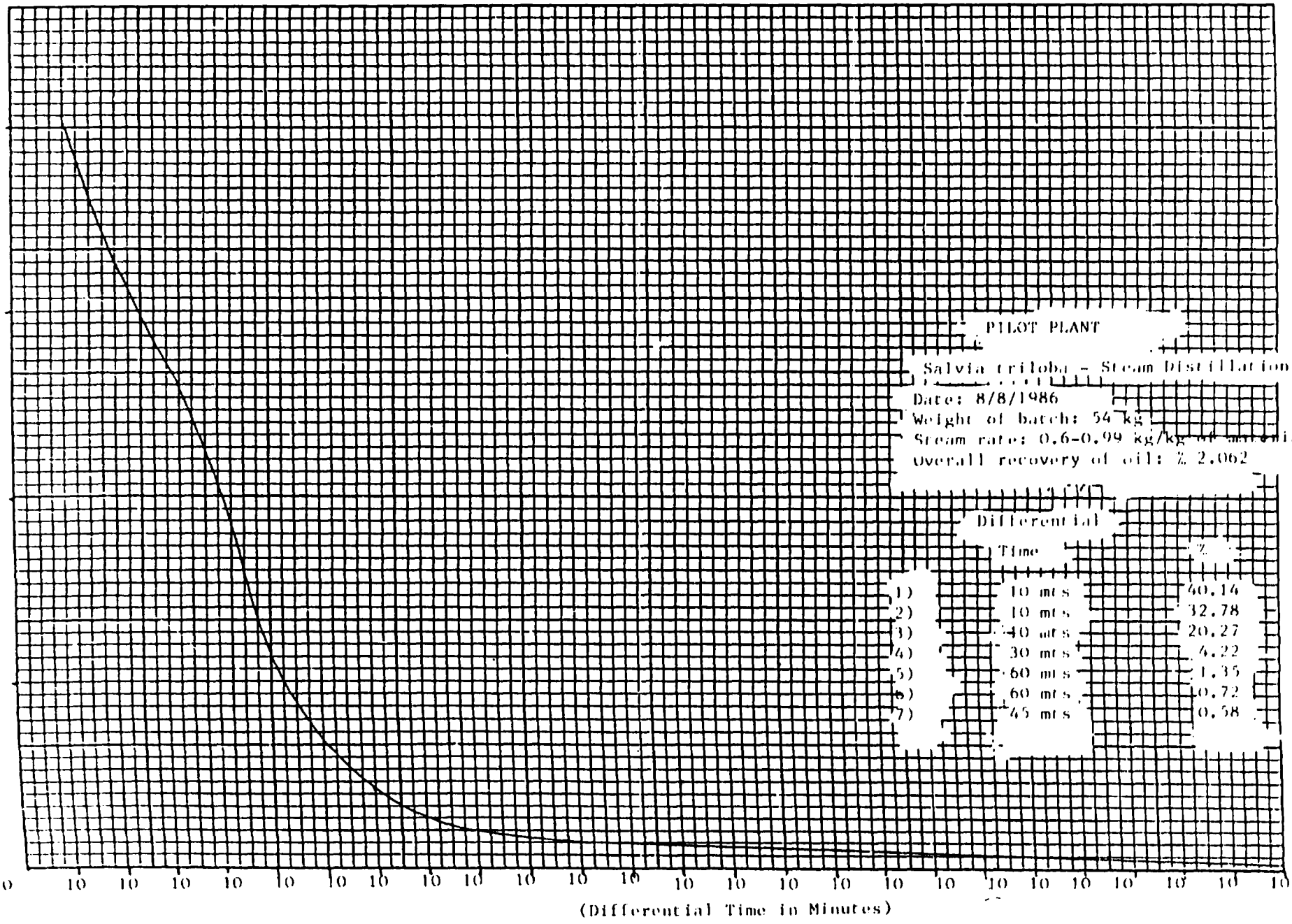
Salvia Cryptantha

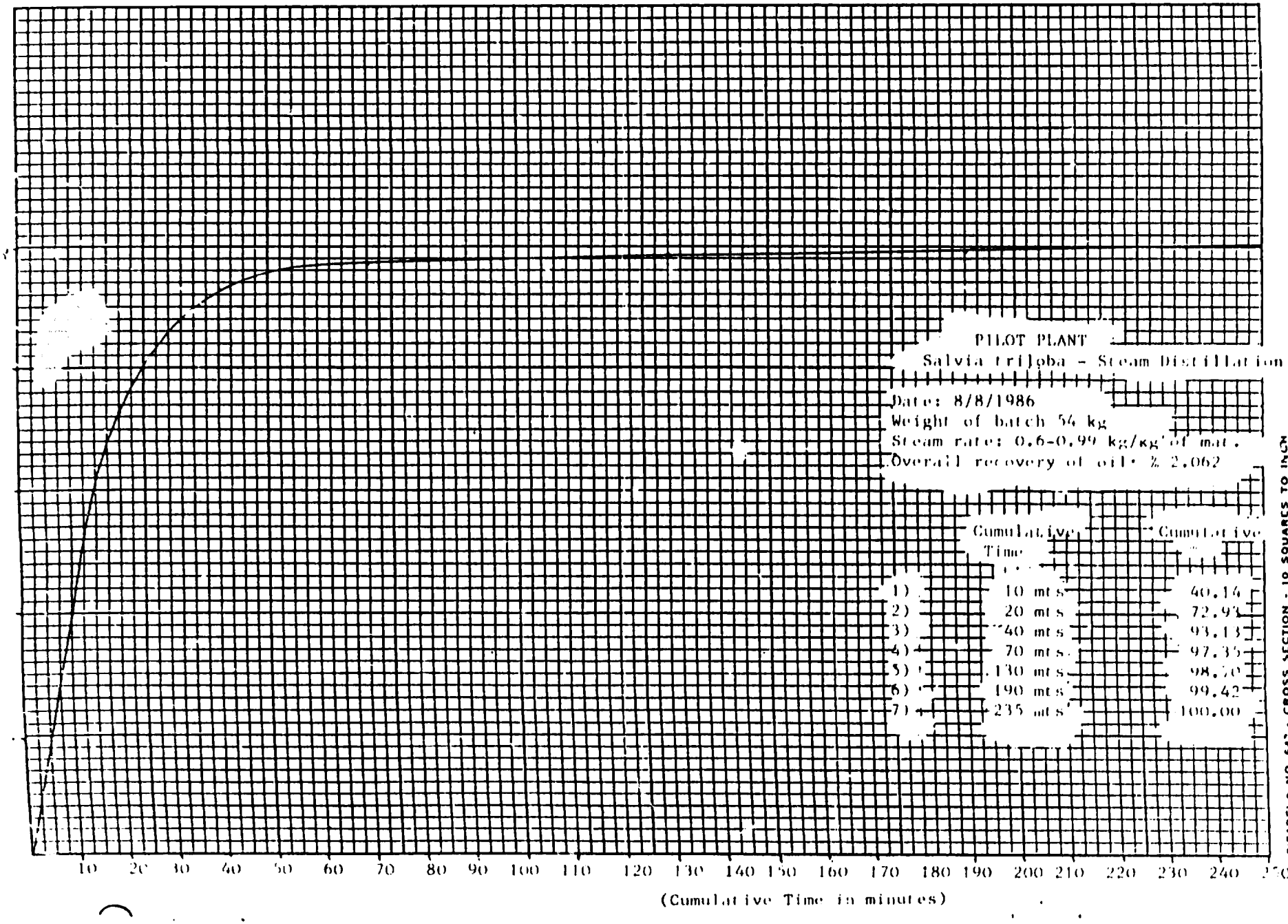
In the laboratory the yield of oil was 0.1%, and in Pilot plant [3.5 hrs.] yielded 0.015% oil. No further experiments were conducted.

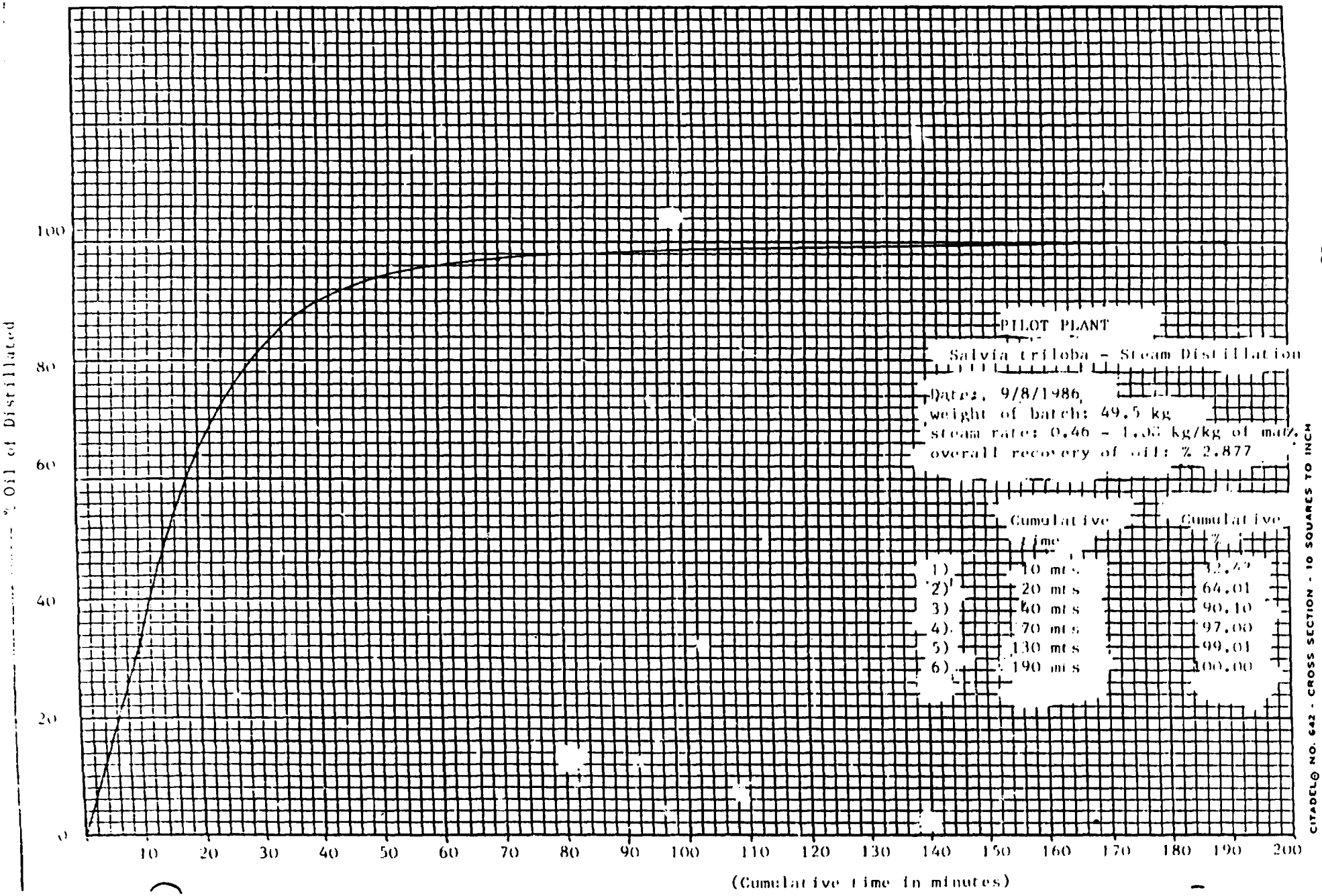
Steam Distillation of Salvia Cryptantha

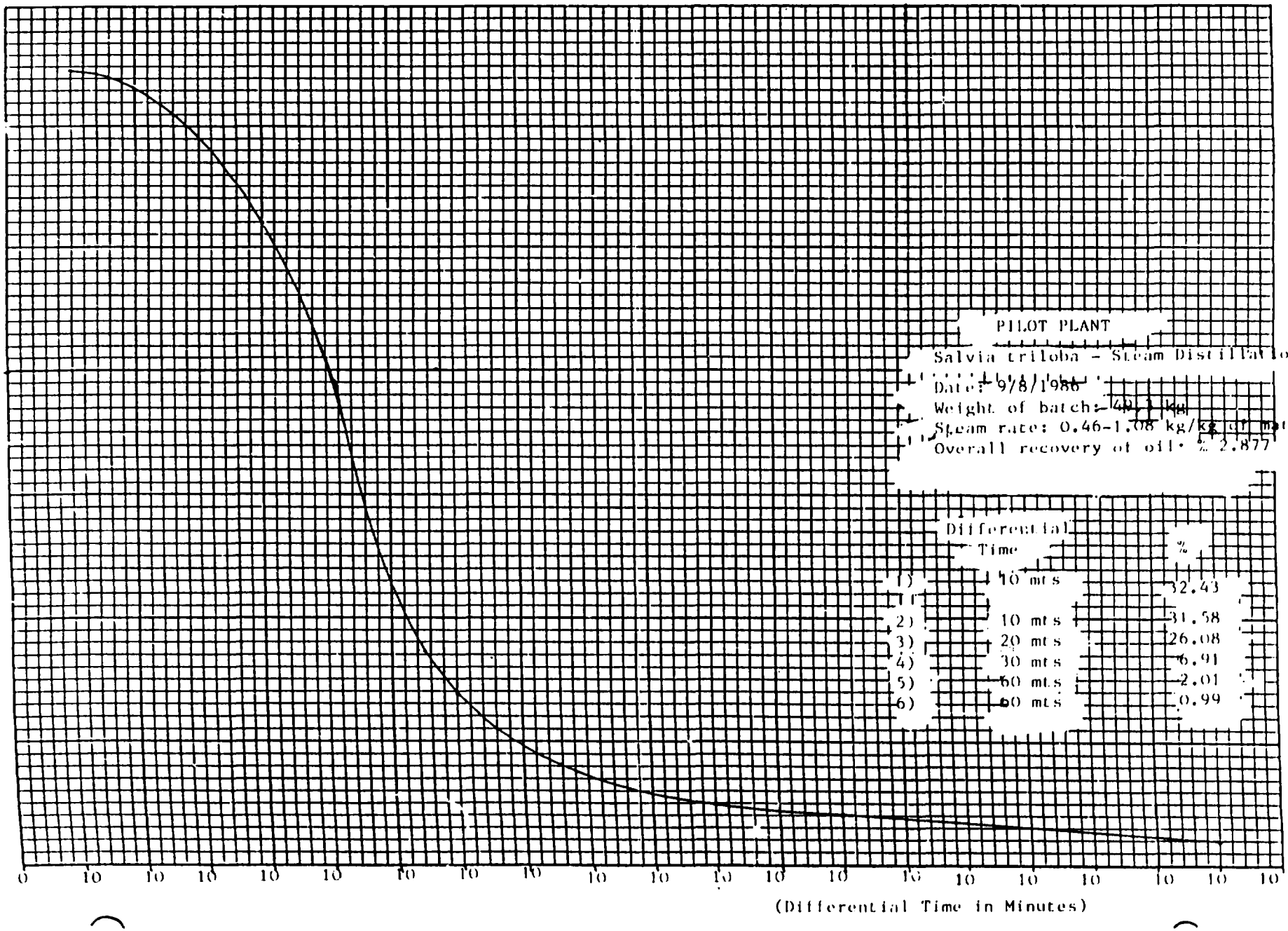
Salvia crytanta sample collected from nearby Kanlipinar was steam distillated.

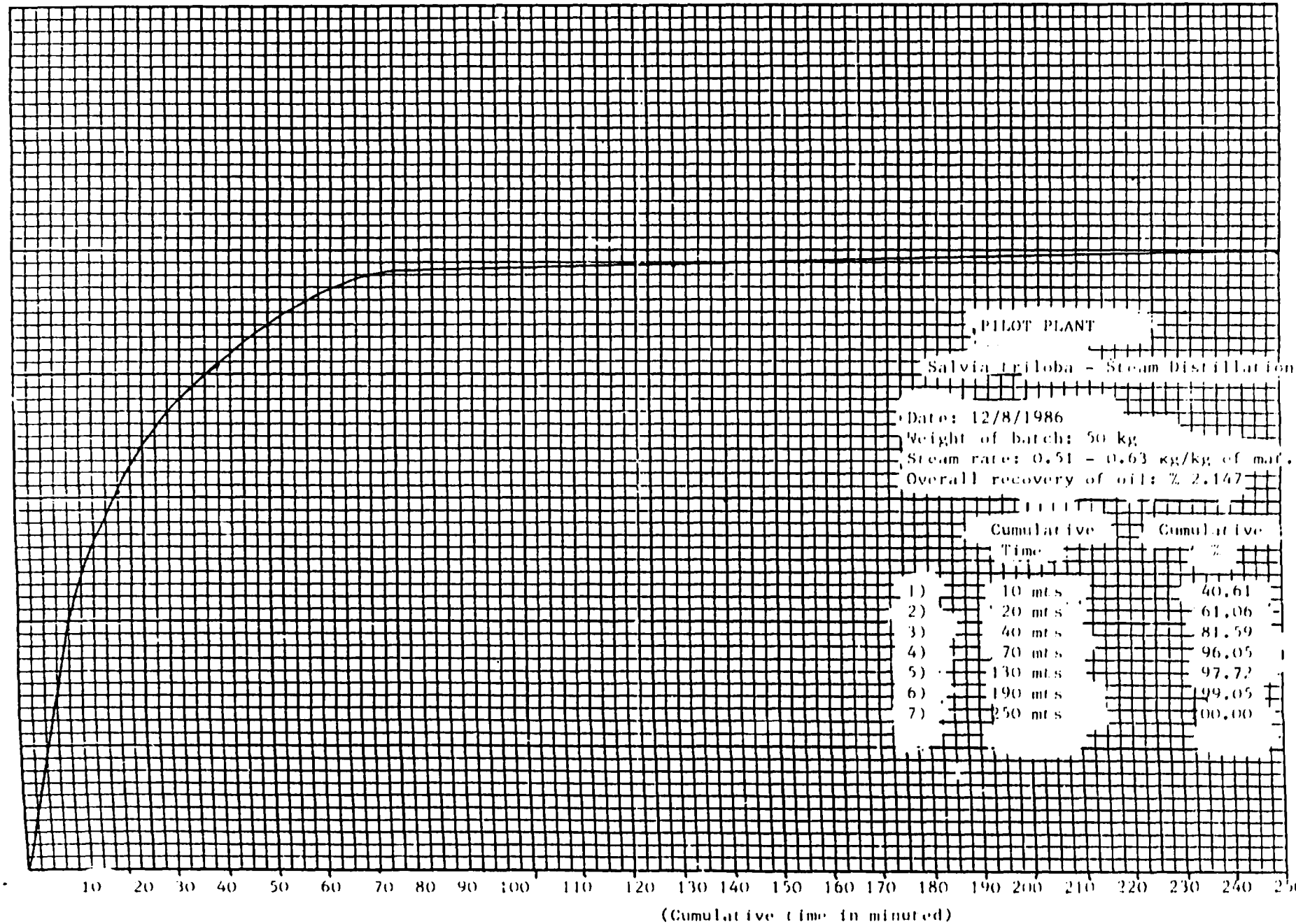
Quantity	:	40 kgs.
Time of Distn.	:	3.5 hrs.
Steam Rate	:	0.86 kg/kg/hr.
Volume of Oil	:	6 ml.
Oil - %	:	0.015% [0.15% in the lab.]

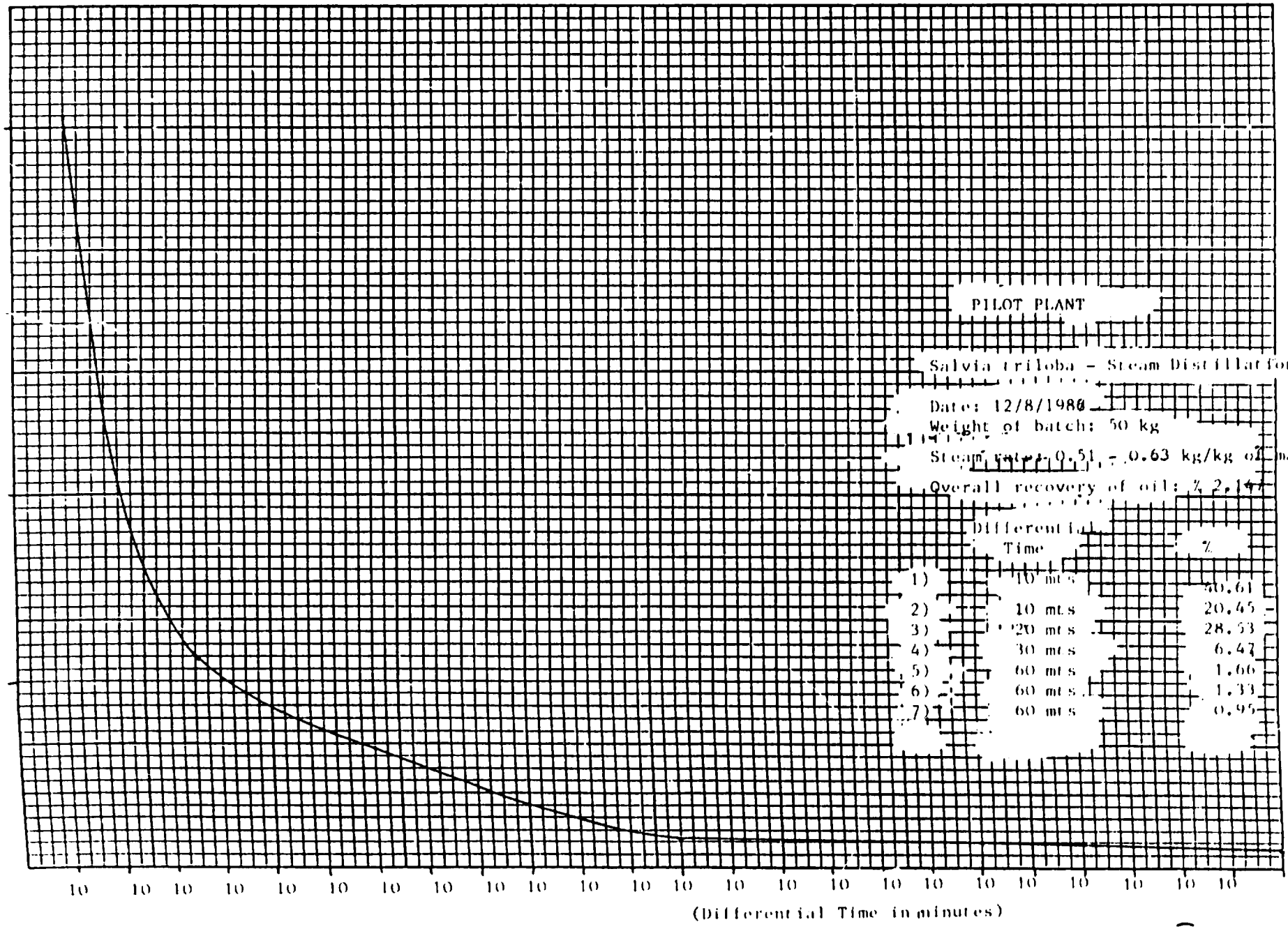

PILOT PLANT
Salvia triloba - Steam Distillation
Date: 8/8/1986
Weight of batch: 54 kg
Stream rate: 0.6-0.99 kg/kg of material
Overall recovery of oil: % 2.062
Differential
Time
%
1) 10 mts 40.14
2) 10 mts 32.78
3) 40 mts 20.27
4) 30 mts 4.22
5) 60 mts 1.35
6) 60 mts 0.72
7) 45 mts 0.58
% Oil of distillated
40
30
20
10
0
10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10
(Differential Time in Minutes)
CITADEL® NO. 642 - CROSS SECTION - 10 SQUARES TO I

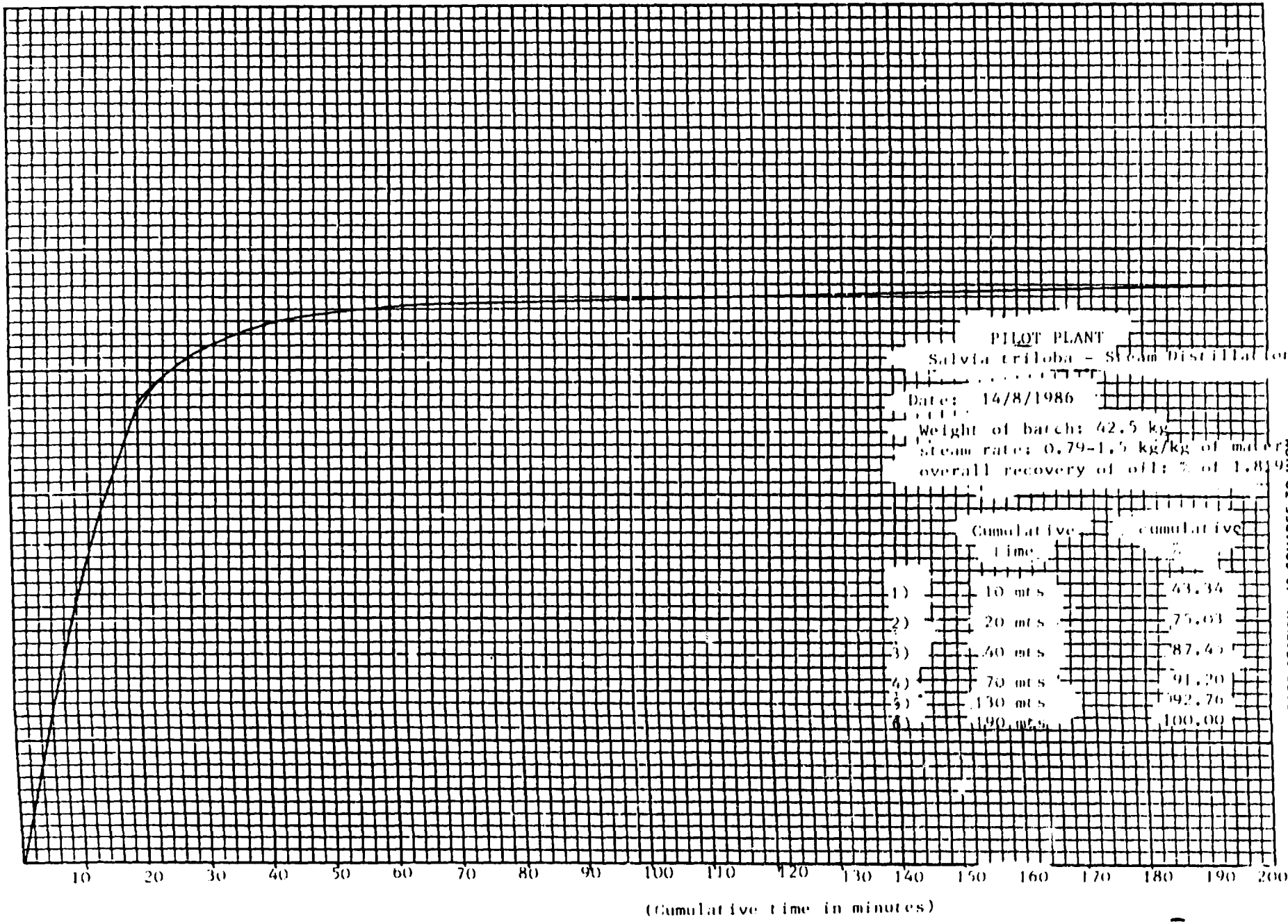

PILOT PLANT
Salvia triloba - Steam Distillation
Date: 8/8/1986
Weight of batch 54 kg
Steam rate: 0.6-0.99 kg/kg of mat.
Overall recovery of oil: % 2.062
Cumulative Time
Cumulative
1) 10 mts 40.14
2) 20 mts 72.93
3) 40 mts 93.13
4) 70 mts 97.35
5) 130 mts 98.50
6) 190 mts 99.42
7) 235 mts 100.00
Oil % Distillated
80
60
10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240
(Cumulative Time in minutes)
CITADEL© NO. 642 - CROSS SECTION - 10 SQUARES TO INCH

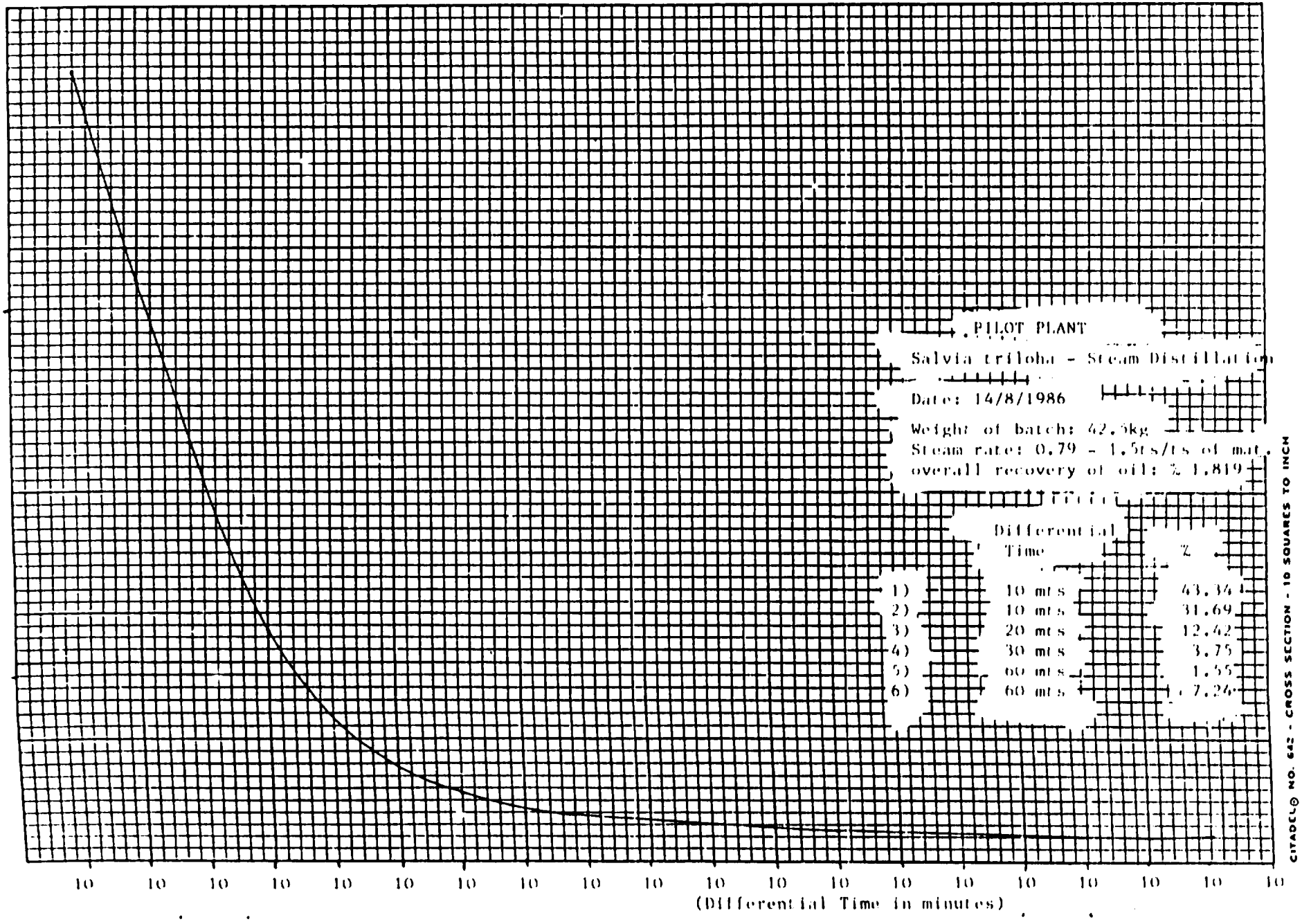

% Oil of Distillated
100
80
60
40
20
0
10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200
(Cumulative time in minutes)
PILOT PLANT
Salvia triloba - Steam Distillation
Date: 9/8/1986
weight of batch: 49.5 kg
steam rate: 0.46 - 1.33 kg/kg of matr.
overall recovery of oil: % 2.877
Cumulative time
Cumulative %
1) 10 mts 32.42
2) 20 mts 64.01
3) 40 mts 90.10
4) 70 mts 97.00
5) 130 mts 99.01
6) 190 mts 100.00
CITADEL® NO. 642 - CROSS SECTION - 10 SQUARES TO INCH


PILOT PLANT
Salvia triloba - Steam Distillation
Date: 9/8/1986
Weight of batch: 40.1 kg
Steam rate: 0.46-1.08 kg/kg dr mat.
Overall recovery of oil: = 2.877
Differential Time
1) 10 mts 32.43
2) 10 mts 31.58
3) 20 mts 26.08
4) 30 mts 6.91
5) 60 mts 2.01
6) 60 mts 0.99
30
20
10
0
10
(Differential Time in Minutes)
% Oil of Distillated
CITADEL® NO. 642 - CROSS SECTION - 10 SQUARES TO IN.


% Oil of Distillated
100
80
60
40
20
10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250
(Cumulative time in minutes)
PILOT PLANT
Salvia triloba - Steam Distillation
Date: 12/8/1986
Weight of batch: 50 kg
Steam rate: 0.51 - 0.63 kg/kg of mat.
Overall recovery of oil: % 2.147
Cumulative Time
Cumulative %
1) 10 mts 40.61
2) 20 mts 61.06
3) 40 mts 81.59
4) 70 mts 96.05
5) 130 mts 97.72
6) 190 mts 99.05
7) 250 mts 100.00
CITADEL® NO. 642 - CROSS SECTION - 10 SQUARES TO INCH


40
30
20
10
PILOT PLANT
Salvia triloba - Steam Distillation
Date: 12/8/1980
Weight of batch: 50 kg
Steam rate: 0.51 - 0.63 kg/kg of
Overall recovery of oil: % 2.14
Differential Time
%
1) 10 mts 40.61
2) 10 mts 20.45
3) 20 mts 28.53
4) 30 mts 6.47
5) 60 mts 1.66
6) 60 mts 1.33
7) 60 mts 0.95
10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10
(Differential Time in minutes)
CITADEL® NO. 642 - CROSS SECTION - 10 SQUARES TO INCH


PILOT PLANT
Salvia triloba - Steam Distillation
Date: 14/8/1986
Weight of batch: 42.5 kg
Steam rate: 0.79-1.5 kg/kg of material
overall recovery of oil: % of 1.81%
Cumulative Time
cumulative %
1) 10 mts 43.34
2) 20 mts 75.03
3) 40 mts 87.45
4) 70 mts 91.20
5) 130 mts 92.76
6) 190 mts 100.00
% Oil of Distillated
100
80
60
40
20
10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200
(Cumulative time in minutes)
CITADEL® NO. 642 - CROSS SECTION - 10 SQUARES TO INCH


% of oil distillated
40
30
20
10
10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10 10
(Differential Time in minutes)
PILOT PLANT
Salvia triloba - Steam Distillation
Date: 14/8/1986
Weight of batch: 42.5kg
Steam rate: 0.79 - 1.5ts/ts of mat.
overall recovery of oil: % 1.819
Differential Time
% 1) 10 mts 43.34
2) 10 mts 31.69
3) 20 mts 12.42
4) 30 mts 3.75
5) 60 mts 1.55
6) 60 mts 7.24
CITADEL® NO. 642 - CROSS SECTION - 10 SQUARES TO INCH

Origanum

Origanum used for steam distillation as a raw material was collected from Izmir.

This raw material contained 2-2.63% of oil according to laboratory experiments. Three batches steam distilled in 2 m^3 capacity still.

	I Batch	II Batch	III Batch
Weight of material [kg]	127	100	30
Steam Rate [kg/kg of material]	0.2 - 0.6	0.5 - 0.8	0.7 - 1.8
Overall Recovery of Oil	2.24%	2.54%	1.55%

The oil content in the feed before distillation 2.63% and after distillation [MARC] was from 0.068 to 0.450%.

Carvacrol constitutes major constituent of the essential, about 75%.

STEAM DISTILLATION

Net weight: 127 kgs.
Steam Rate: 0.265 - 0.581 kg/kg/hr.

	Interval [min.]	Volume of Oil [ml]
1. Sample	15	220
2. "	15	292
3. "	30	413
4. "	30	630
5. "	30	440
6. "	30	362
7. "	30	335
8. "	45	73
9. "		10

Overall yield of oil = 2.185%

ORIGANUM POWDER

Distillation studies were conducted for the maximum recovery of oil from non-saleable origanum plant powder sponsored by a private party in Izmir, Turkey.

This raw material contained 1.5 - 2.2% of essential oil. Seven batches were steam distilled in a still of 2 m^3 capacity.

Experimental Results [Origanum Powder]

Batch	I	II	III	IV	V	VI	VII
Weight of material[kg]	197	249	250	945	148	134	171
Steam Rate [kg/kg of material]	0.4-0.87	0.4-0.6	0.2-0.6	0.5-1.1	0.4-0.73	0.3-0.6	0.5
Overall recovery of oil [%]	0.25	0.28	0.35	0.67	0.71	0.55	0.20

Analysis of Oil - Origanum Onites L.

1] Gas Chromatogram : Total volatile oil

2] IR Spectrogram : Total volatile oil

3] Refractive Index : $[n]_D^{20} = 1.506$

4] Optical Rotation : $[\alpha]_D^{20} = -0.25$

5] Solubility : Very soluble in Ethanol [96 percent]

6] Weight per ml. : 0.95 gm/ml.

Oil of ORIGANUM ONITES

Components	%
Pinene	1.322
Camphene	0.3787
β-Pinene	0.0973
Myrcene	1.7141
α-Terpines	1.5215
D-Limones	0.2302
Cineol	0.2526
α-Terpinen	5.8186
p-Cymol	6.4196
Terpinoles	0.2358
Linalool	0.175
Borneol	0.9697
Thymol	1.0863
Carvacrol	74.1976

Experimental Results of Ruscus Aculeatus Roots-Dried and Crushed

FERMENTATION		HYDROLYSIS		EXTRACTION				HYDROLYSIS			RESULTS
Solvent	Time	Acid	Time	Solvent	Type	Time	Yield Total %	Acid	Time	Yield Total %	
Water	48 hrs	7% HCl	2 hrs	n-Hexane	Soxhlet	3 hrs.	2.52	-	-	-	Total Ruscogenine 2.52%
Water	48 hrs.	-	-	n-Hexane	Soxhlet	3 hrs.	0.85	-	-	-	0.85%
-	-	7% HCl	2 hrs.	n-Hexane	Soxhlet	3 hrs.	2.45	-	-	-	2.45%
-	-	-	-	n-Hexane	Soxhlet	3 hrs.	0.51	-	-	-	0.51%
-	-	-	-	Et-OH[96]	Soxhlet	3 hrs.	14.21	7% HCl	2 hrs.	18	2.57%
-	-	-	-	Et-OH[96]	Direct Boiling	3 hrs.	21.3	7% HCl	2 hrs.	33	7.0%

Installation and Commissioning

Tournaire Pilot Plant - 500 litre capacity, consisting of:

- Extractor cum essential oil distillation still
- Miscella distillation still with packed column
- Heat exchangers
- Circulation pump
- Water ring vacuum pump
- Receivers
- Inter-connecting pipes and fittings
- Supporting steel structure
- Crusher.

The installation of this unit was accomplished with the assistance of an engineer from the suppliers, M/s. Tournaire, Grasse, France.

On initial testing of this unit, a number of short-comings were noticed:

Crusher

- emited unbearably high pitch noise
- produced very fine powder, choking the crusher room with very fine gypsophila root powder
- the resulting fine powder when subjected to ethanol extraction in the pilot plant in turn resulted in the following disadvantages:
  - poor percolation of solvent
  - higher periods of extraction
  - formation of "islands"
  - fine dust in miscella; and
  - poor desolventisation - loss of more ethanol.

On close observation it was noted that the rpm of the crusher was about 6000. Suitably modified to reduce the r.p.m. to about 3400. After modification, 300 kgs. gypsophila roots and 300 kgs. red peppers were crushed and observed that it:

- emited considerable less noise
- practically free of floating dust - consequently clean surroundings
- produced coarse materials - suitable for extraction purposes.

Rectification Column

A packed column with ceramic packings, fitted over distillation still, meant for the rectification of dilute ethyl alcohol obtained by the extractions in the plant, could not be used for this purpose, in the absence of reflux distribution arrangement.

Suitable modifications have been worked out, a sketch prepared and submitted to the suppliers through UNIDO.

Drawings - Annex-IV

Vacuum Filter

Some plant extracts viz., Gypsophila, when concentrated and on subsequent cooling, precipitate separates out, needing filtration. In the absence of a suitable filtration unit in the supply, designed a vacuum filtration unit of 600mm diameter and 750mm height and got the same fabricated locally.

Installation

The following all glass equipment imported from Europe, to reinforce the Project Infrastructure have been installed and commissioned·

1] A general purpose "Buchi" all glass "Chem Reactor CR 100" of 100 litre capacity, consisting of stirred reactor, heat exchangers with refluxing arrangements, receivers, interconnecting glass tubing, glass fittings, and the G.I. tubular structure. When completely assembled, the unit measured about 0.7m x 2m x 3.5m.

This unit has not been provided with separate vacuum pump, however, suitable flexible connections, have been made to connect it to the vacuum system of Tournair Pilot Plant.

2] "Buchi" Rotavapor - 1501/E"; capacity: 10 litres/batch.

3] "Buchi" Rotavapor R - 151/Standard"; capacity: 10 lit./hour.

4] Sohxlet Extractor 12 K SS.316;
Capacity: 12 kg/batch,
supplied by: M/s. Armand Deprest
Belgium.

DESIGN & ENGINEERING:

The project has diversified itself with the assistance and full time participation of this expert, into extremely useful area of developing, expertise in the design and engineering.

It is suggested that while developing technologies, for the industrial utilization of indigenous medicinal and aromatic plant materials, simultaneous development of design and engineering expertise and suitable infrastructure for the fabrication of chemical plant and equipment, will not only quicken the process of transfer of technologies developed at the centre [MPRC] to the industry, but can save valuable time and foreign exchange and hence a compulsive need of the developing countries.

With these considerations in view a beginning has been made with the design and fabrication of the following equipment:

1] 2 M^2 essential oil distillation unit, consisting of tapered [to facilitate easy removal of Marc] still in mild-steel lined with thin stainless steel. Aluminium shell and tube condenser, a suitable separator. A pair of "Spiders" have been provided for easy removal of exhausted marc from the distillation still. For this purpose the still has been installed in such a way that the manually operated hoist provided with Taurnaire Pilot Plant could also be utilized for this purpose.

A larger still has become necessary to distill comparatively large amounts of green, semi-dried or dried aromatic bearing plants, containing various percentages of oil, to obtain reliable distillation parameters, appropriate for scale-up to commercial plants.

In addition, sufficiently large amounts of essential oils could be obtained for further R & D efforts to obtain terpeneless oils and specific "cuts".

Drawings - Annex-I

2] To facilitate further R & D efforts on fractionation of essential oils, a fractional distillation unit with a batch capacity of about 150 litres with a suitable clumn, packed with the latest internals, heat exchangers, electro-magnetic reflux distributor with electronic timer, receivers, traps, all in stainless steel and supporting structure in mild steel, have been designed and got it fabricated locally.

An annexe to the Pilot plant building is being built by the project authorities with adequate height for its installations and also to accommodate additional equipment viz., Battery-type aqueons percolators, spray drier etc.

Drawings - Annex-II

3] A three stage counter current percolator system, suitable for use with aqueous solvents.

A three stage counter current Percolators, suitable for cold/ hot aqueous extractions has been designed and fabricated using suitably modified,discarded, jet-plane exhaust pipes, measuring 750mm bottom diameter, 950mm top diameter and height of 1745mm salvaged from Air Force Junk yards.

Locally purchased stainless steel pumps, stainless steel pipes, sheets, have been used in its fabrication in the University work-shop.

This unit when installed and commissioned, will be able to process about 500 kg. of Glycyrrhiza Glabra per batch.

The aqueous extract produced in this unit, can be further processed in the 500 lit. evaporator of Tournair pilot plant to produce liquorice syrup or spray dried in spray drier [when made available].

Drawings: Annex-III

Medicinal plants processing units generally use, highly volatile solvents for extraction purposes, as a preventive measure, all the electrical fittings in the pilot plant buildings have been replaced with explosion-proof fittings.

However, suitable fire-fighting facilities have been provided to the pilot plants and the project area as a whole.

Liaison with Industry - Solution to Industrial Problems

The expert[Chemical Technology] accompained by the Project Director, visited some plant based industrial units, in Izmir, Marmaris, and rose oil distillation units, in Isparta.

As a result of these visits, the management of M/s. Gulbirlik, Isparta, a major producer of Rose oil in Turkey, about 80% of Turkey's and 40% of world's rose oilwas produced by this firm, brought to the notice of the team, that the technology of rose oil distillation, remained the same since its establishment in mid fifties and the quality of oilwas poor when compared to other major European producers and requested to study the plant and update the technology.

The team conducted a detailed and systematic study supported by its own laboratory equipment from 10th to 13th June, 1987 stretching from 12 to 18 hours a day, improved the processing capacity of the plant by about 50% [flowers], without any modifications or additions to the plant and enhanced the quality of oil considerably.

Mr. Muzaffer Tuleman R., General Manager of M/s. Gulbivlik verbally acknowledged that the project team's work in his factory, would save for his firm, about 300 million Turkish Lira [about US$ 3,600,000] in one season alone.

A copy of report in Turkish and its English translation.

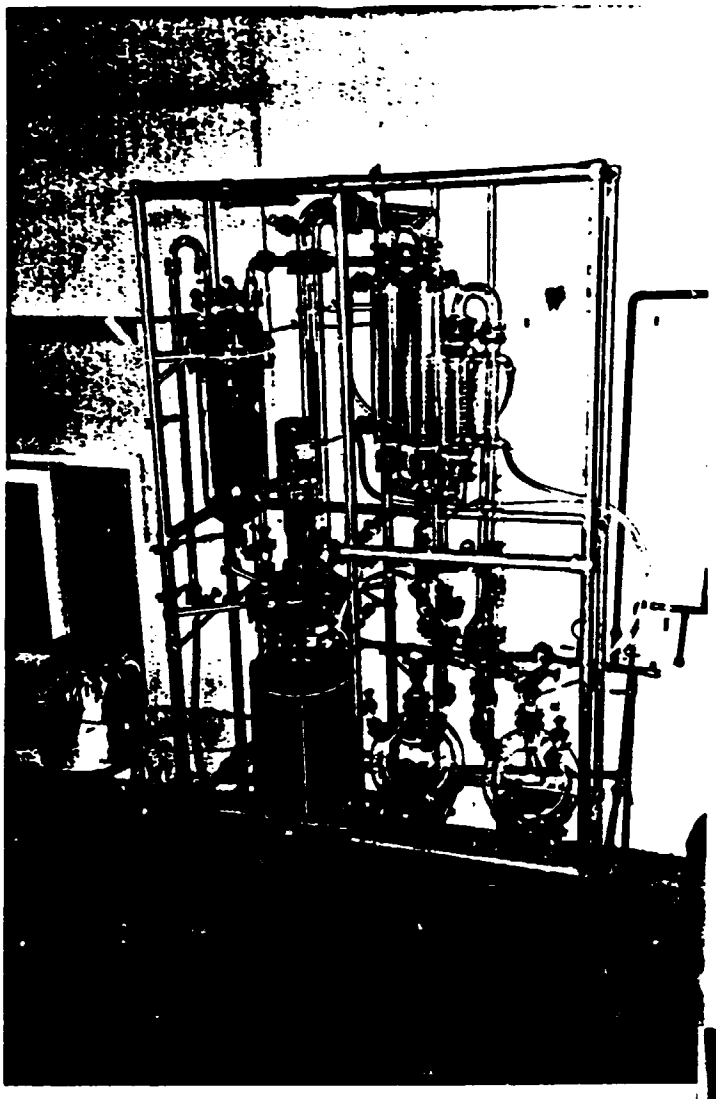
Drawings - Annex-V

1. A party in Istanbul sponsored a project to refine crude storax for use in hair shampoos and toilet soaps.

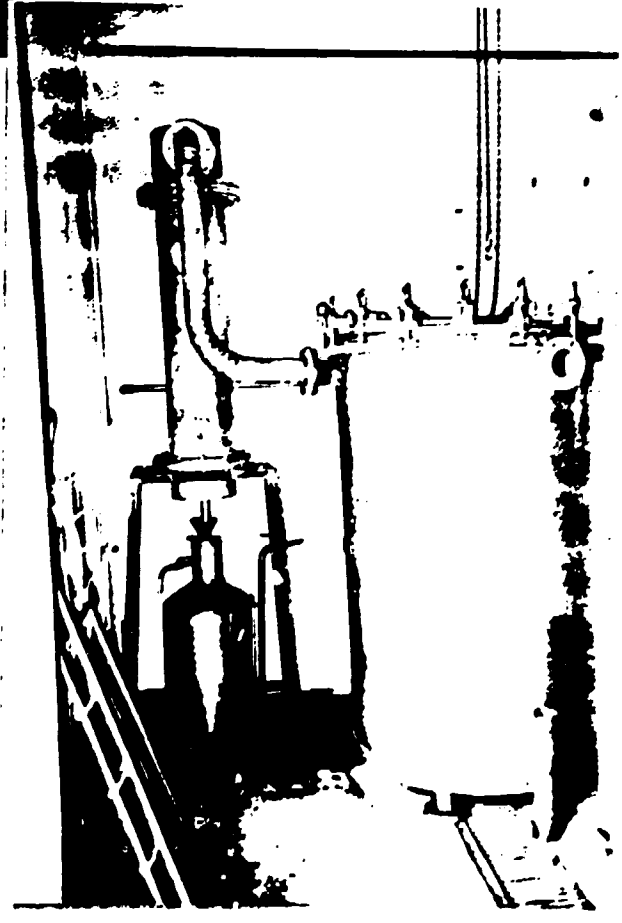
Laboratory investigations have almost been completed scale-up studies remained to be worked out.

Results Reported under-Bench scale and Pilot plant Experimental Results:

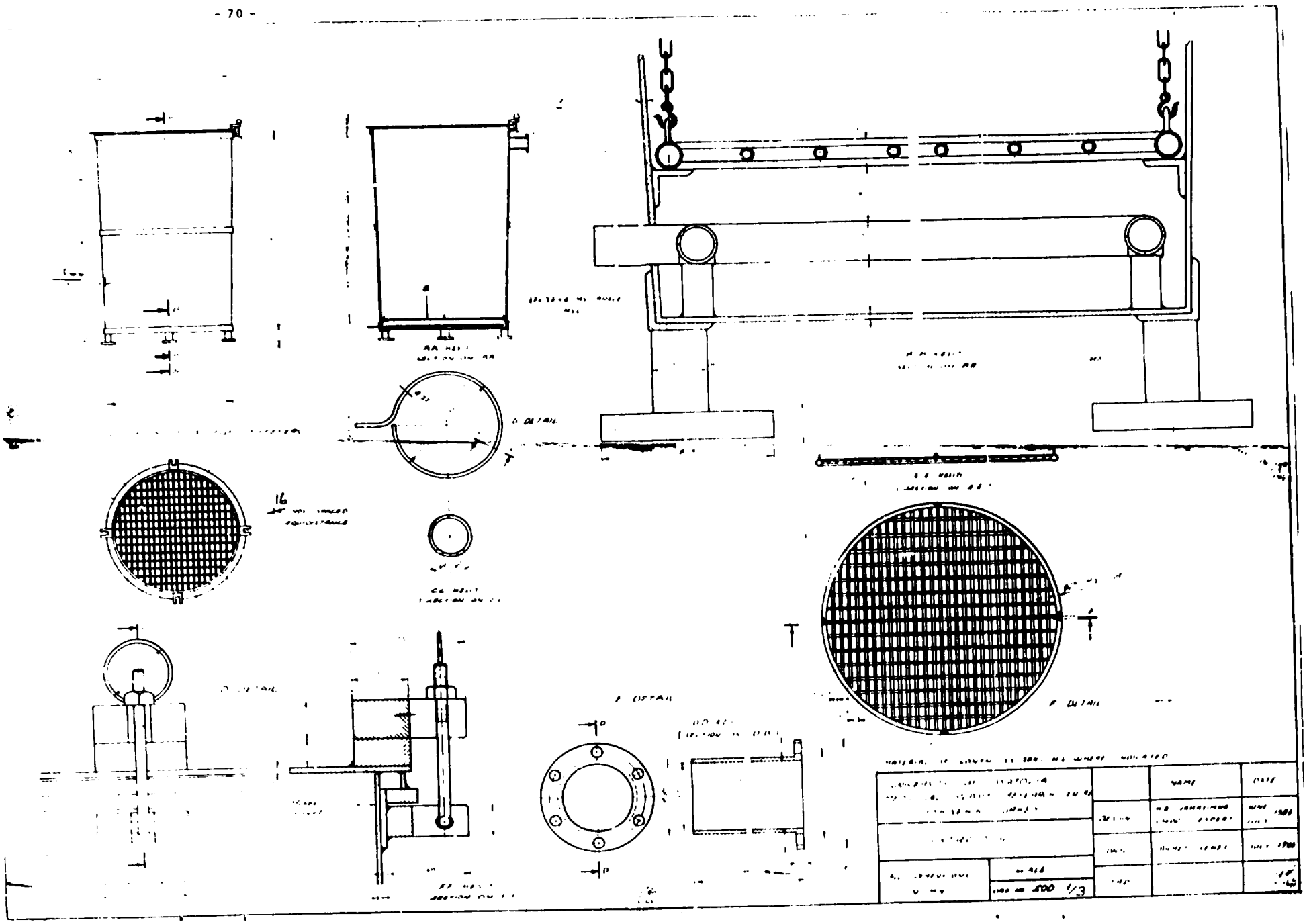
2. Hops Growers Society in Bilijek, showed keen interest in establishing a commercial Rose oil plant, on turn-key basis. This society has sufficiently large area under the cultivation of Rose Domascena.

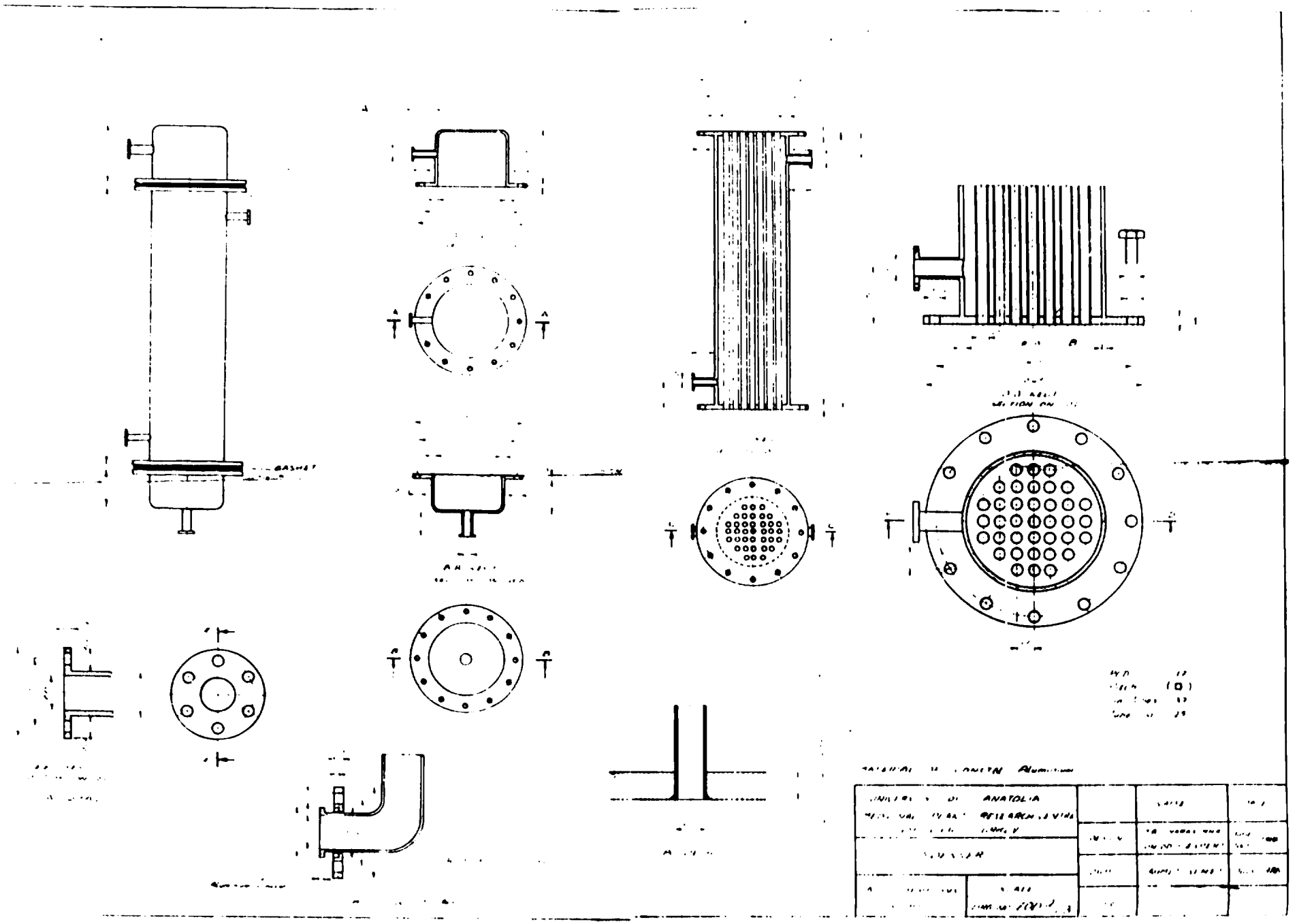


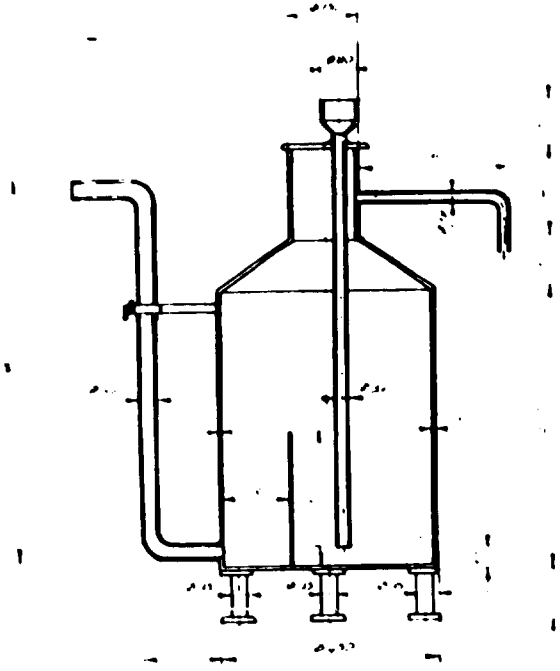
Buchi all glass
"Chem Reactor",
100 litre capacity.



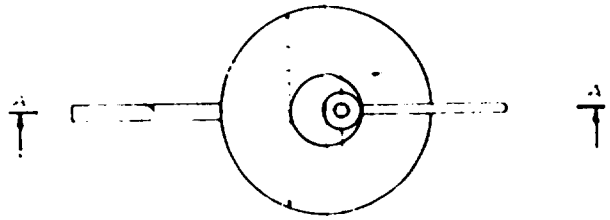
Essential Oil Distillation
Unit;
2000 litre capacity
[Designed & Fabricated]




ANATOLIA

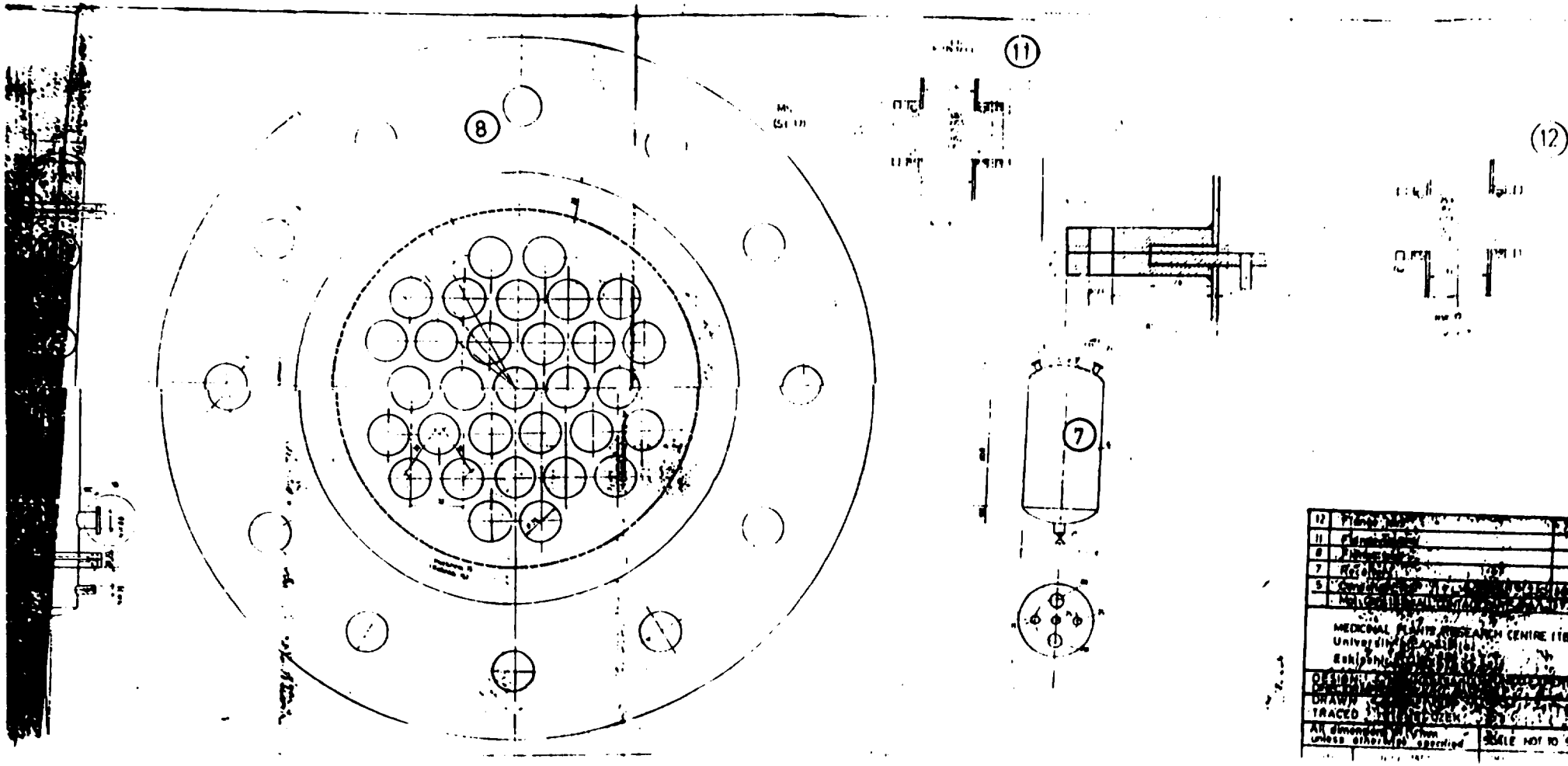


AA RESULT



MATERIAL OF CONSTN Aluminium

		NAME	DATE
SEPARATOR			
SCALE			


8
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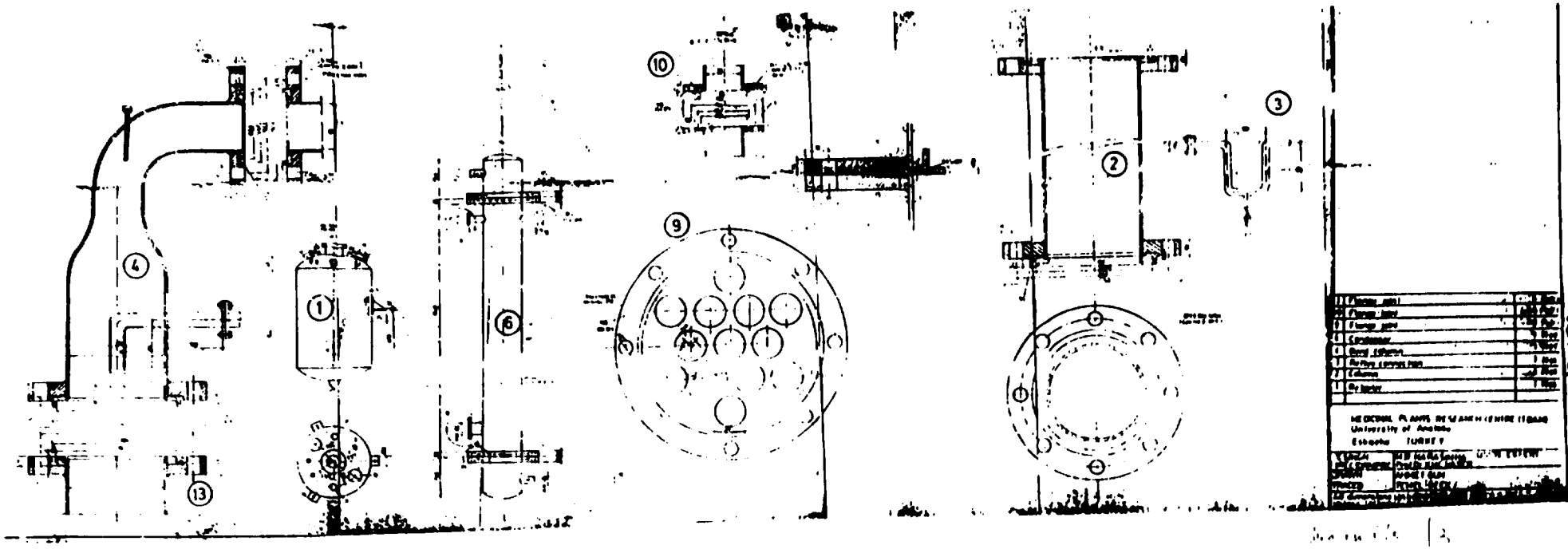
MEDICINAL PLANTS RESEARCH CENTRE (TIBAH)
SCALE NOT TO SCALE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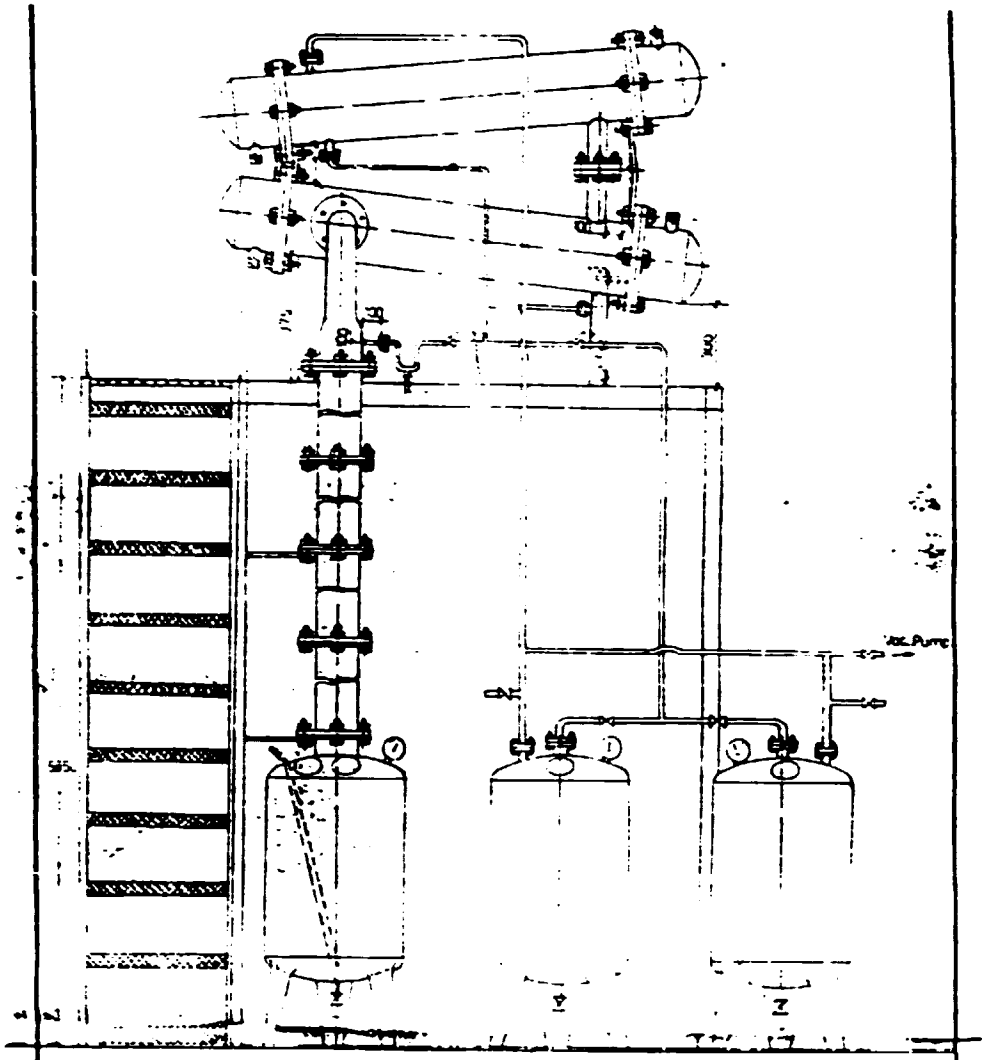
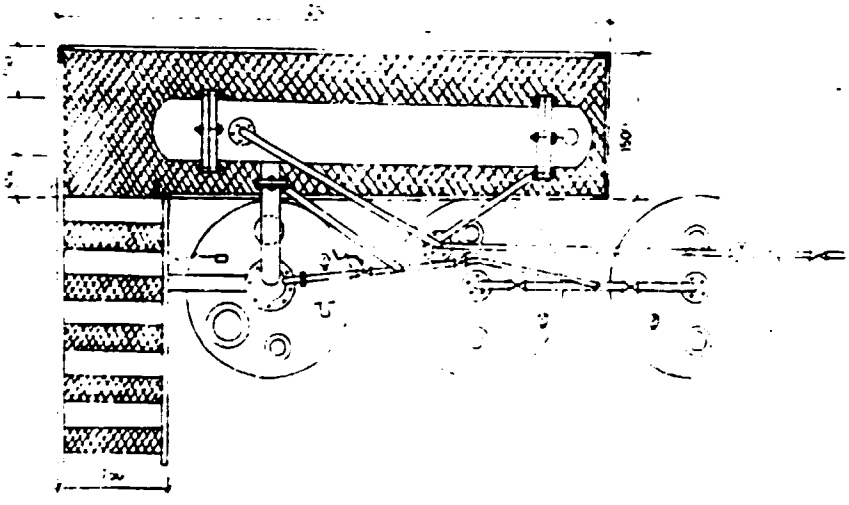
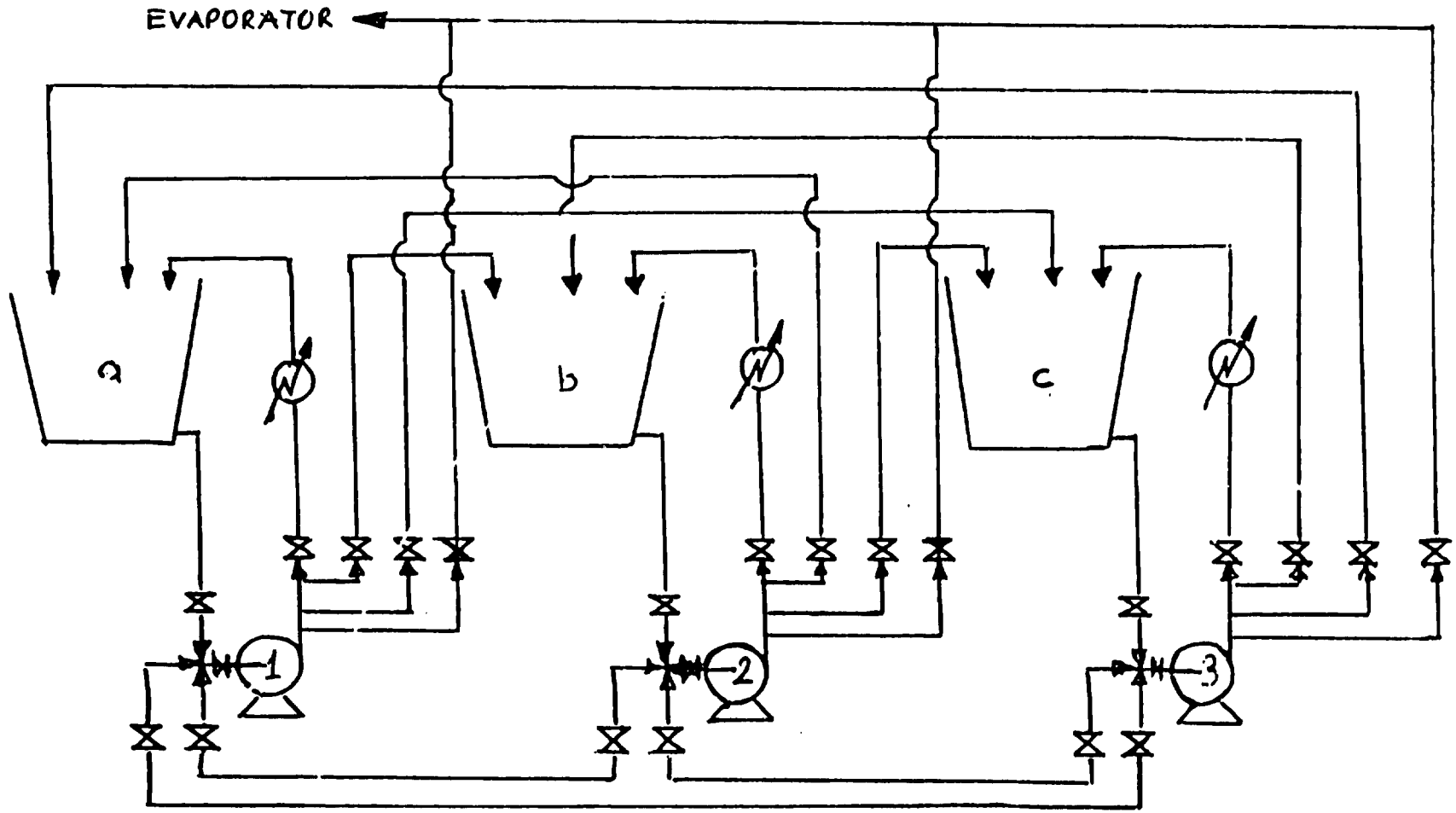




MEDICINAL PLANTS RESEARCH CENTRE (TBAM)
University of Anatolia
Eskişehir, TURKEY

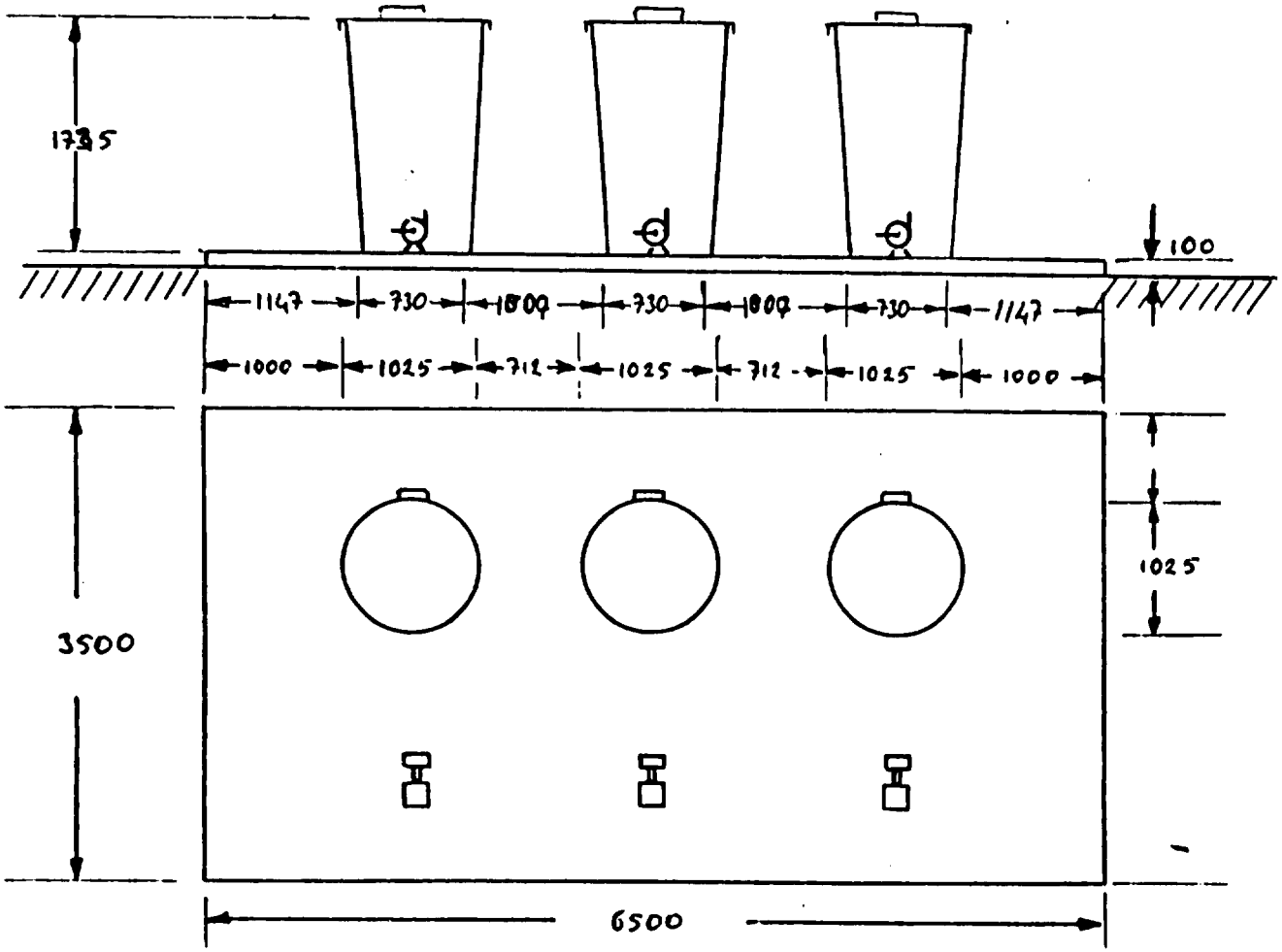
DESIGN DIRECTOR MPRC	M B NARASIMHA UNIDO EXPERT Prof.Dr.K.HC. BASER	
DRAWN TRACED	AHMET GUN TEMEL OZEK	
All dimensions in mm unless otherwise specified		SCALE NOT TO SCALE
Date	June 1987	DRG NO Fc 3/3

ANNEX-III/1



BATTERY OF PERCOLATORS

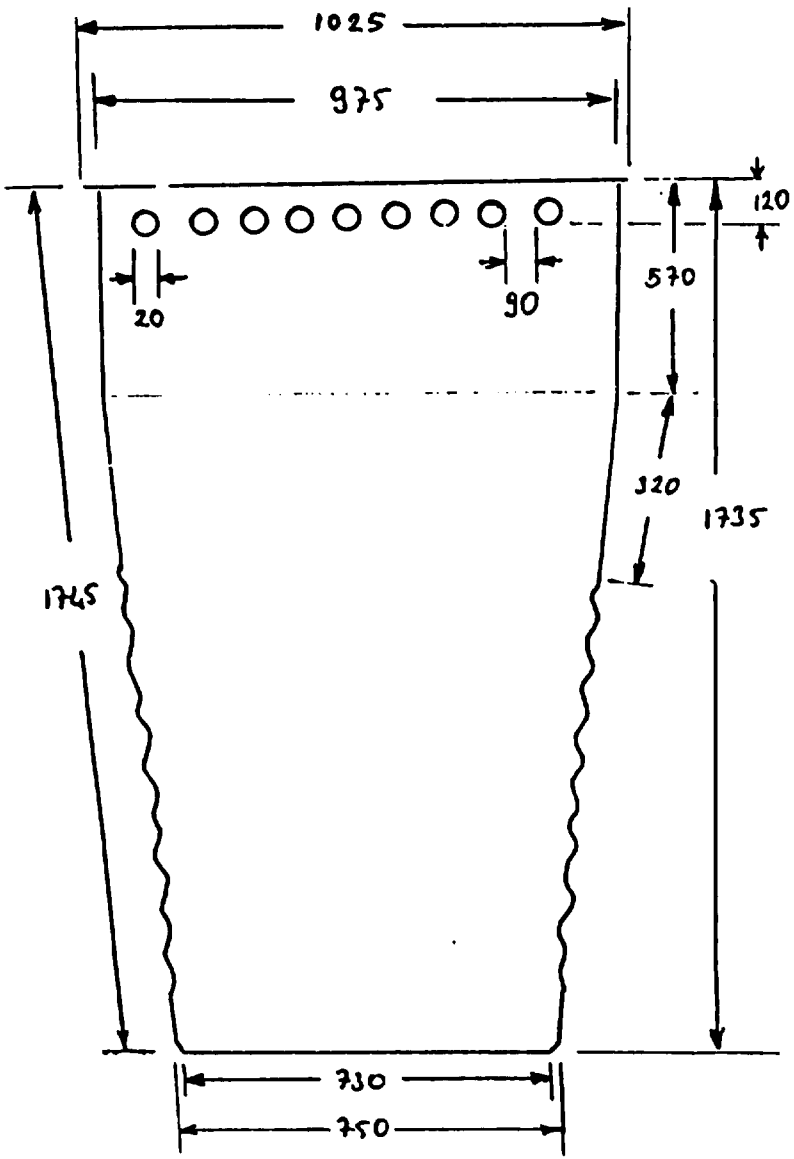
M. B. Narahari



BATTERY OF PERCOLATORS USING DISCARDED AEROPLANE EXHAUST PIPE

DIMENSIONS IN mm

ANNEX-III/3



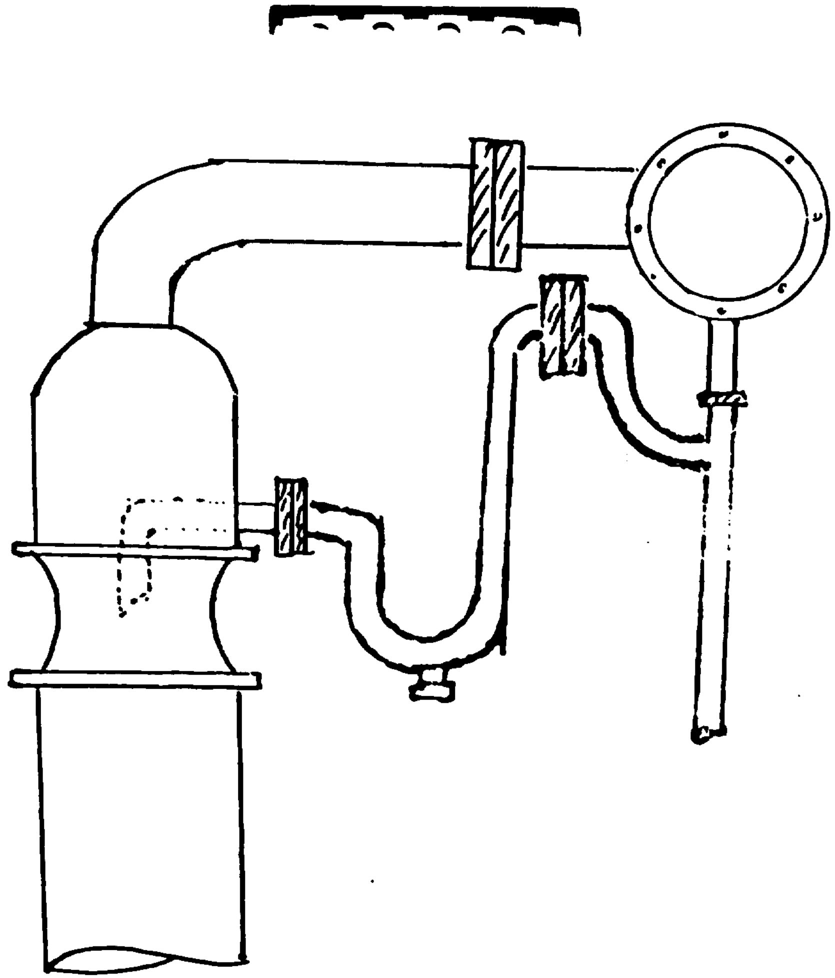
VOLUME : 1.095 m^3

Dimensions in mm

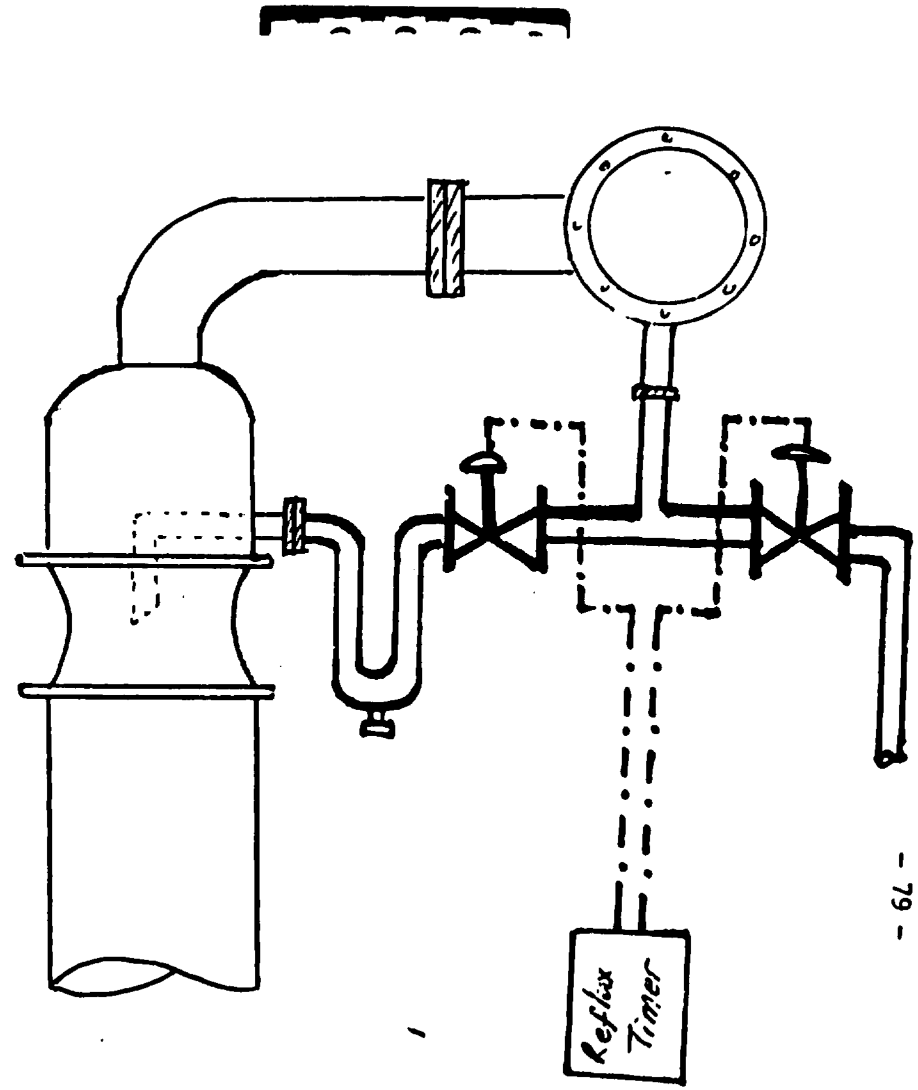
DETAILED SKETCH OF DISCARDED PLANE EXHAUST PIPE

M. B. Nanaphinly

ANNEX-IV/1



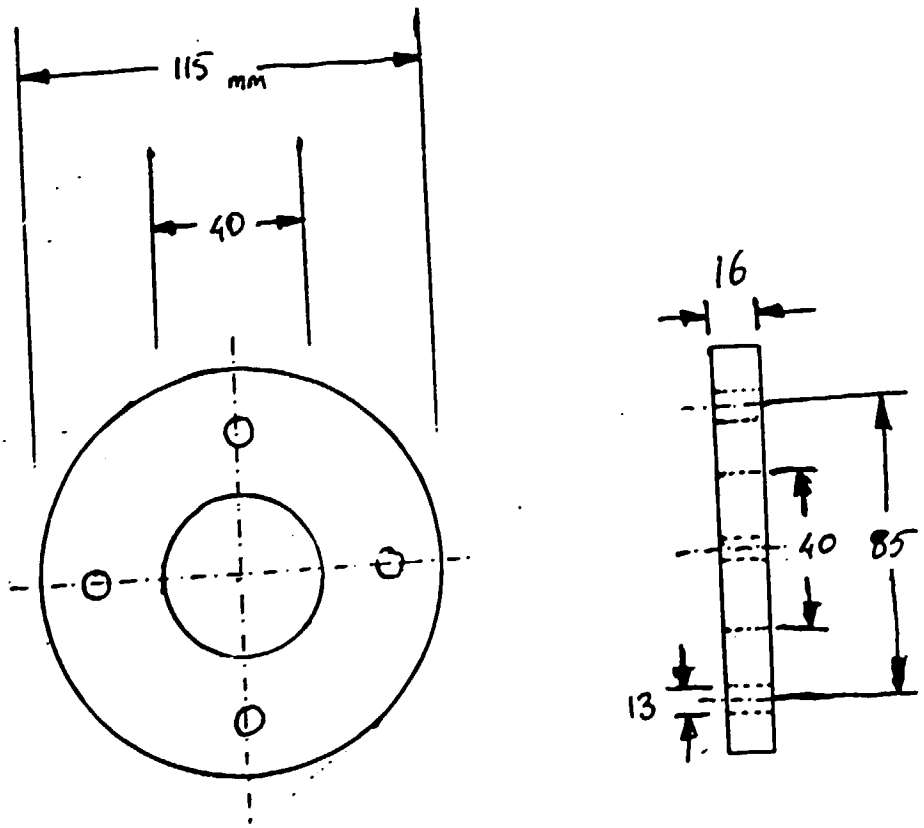
Existing Set Up



Proposed Set Up

List of additional items required:

1. Electromagnetic Values : 2+2 (spare)
2. S.S. pipe : one length 6 m. 40mm dia.
3. Reflux timer 1+1 (spare)
4. Flanges : size as per sketch 3 pairs
5. S.S. union Joint : one set



ANNEX - V

English translation of the report submitted to M/s. Gulbirlik, Ispanta.

At the request of M/s. Gulbirlik, Prof. Dr. K.H.C. Baser, Director of Medicinal Plants Research Centre, University of Anatolia, Eskisehir and Mr. M.B. Narasimha, UNIDO Expert, accompanied by MPRC Colleagues conducted intensive studies of Rose oil distillation at their factory, between 10th and 13th June, 1987 working 12 to 16 hours a day.

A brief summary of the team's observations and recommendations are enclosed. A detailed report shall be submitted if the company management so desires.

OBSERVATIONS:

Harvesting Period

Harvesting is being done upto 3 P.M., where as the regulation limit upto 11 A.M. only.

During hot sunny days with strong low wind, oil content of roses collected would be low, additionally the extended collection would upset, the processing in the plant with the dumping of additional flowers, leading to partial fermentation with the resulting poor quality and lower yields of oil.

- No proper scientific methods were being followed in the distillation of flowers;

- Records were not maintained to indicate the percentage of oil obtained in the first and second waters and the amount of rose water, still-wise;

- First water obtained from distillation of flowers were directly distilled in the "oil stills". It leads to inefficient recovery and poor quality of oil.

- All the distillation stills were so linked, that checking, performance of individual still, was not possible.

- No facilities exists to check and monitor quality control of rose oil.

- Physical condition of the stills seemed to be fine. However, the performance of condensers were observed to be erratic, may be due to the formation of scales.

- All the distillation stills were being operated without proper insulation. This lead to higher consumption of steam, prolonged period of distillation, and poor quality of oil.

In addition to this, the surroundings, were lead into very uncomfortable working conditions.

- Spenk flowers were let into the nearby stream.

- Boilers were never serviced after insta lation in 1976.

- Water-softening plants, procured along the boilers, were not commissioned.

- Very heavy investments were made in the establishment of the factory, buildings, and infrastructure. These facilities were being utilized for a couple of months in a year only.

- Number of labour seemed to be much more than necessary, leading to avoidable confusion.

Recommendations

1] Flowers : [a] Spread on clean floor and turn frequently.

[b] Filling in bags and stack near the stills for not more than 2 hours.

2] Insulation: [a] All the Stills

[b] Steam pipes

[c] Hot water lines.

3] Explore suitable methods to increase the capacity of each still at peak periods. Systematic studies have been successfully conducted to increase the production capacity of each still from 500 kg/batch to 750 kg batch, without any additional investment in equipment and manpowers.

4] The Rose oils at each factory was filtered through a filter paper, the oils may still contain some fine drops of water. Recommend to set-up at a central place, a super centrifuge, facilities for the removal of fine suspended solids and fine drops of water.

5] Cohobation equipment after appropriate studies using water of the plant at Medicinal Plants Research Centre, University of Anatolia Eskisehir, Turkey.

6] Lack of R & D backing to update the technology. Recommend that the services of MPRC be retained for this purpose.

7] Lack of quality control and measurement. Recommend that services of MPRC be retained for this purpose.

8] Heat exchanger tubes have never been cleaned, since their installation, resulting inefficient functioning. Recommend cleaning immediately after every season.

9] Same goes with Boilers.

10] Explore the possibility of using spent flowers as farm composte.

11] Periodic training of technical manpower in production techniques and labour management.

Sd/-
[M.B. NARASIMHA]

Sd/-
[K.H.C. BASER]