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## DEVELOPMENT OF INDUSTRIAL PRODUCTION OF ESSENTIAL OILS, ARONAS AND FLAVOURS

DP/VIE/84/010/11-54

THE SOCIALIST REPUBLIC OF VIET NAM

Technical report: Processing of Vietnamese essential oils and related natural products for local use as well as export\*

Prepared for the Government of
the Socialist Republic of Viet Nam
by the United Nations Industrial Development Organization,
acting as executing agency for the United Nations Development Programme

Based on the work of Y. P. Talwar, menthol technologist

Backstopping Officer: T. De Silva, Chemical Industries Branch

United Nations Industrial Development Organization Vienna

<sup>\*</sup> This document has not been edited.

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#### INTRODUCTION

The National Centre for Scientific Research of Vietnam is working on the project on "Production of crystal menthol for export purposes from Vietnamese Mint oil" (DP/VIE/84/010/11-54) for the last about two years. The project is being supported by UNDP and the executing agency is UNIDO.

The project has been undertaken with the main object to manufacture crystal Menthol of international standard from Vietnamese Mint oil.

The activity requires the assistance of a consultant on the production of crystal Menthol from Vietnamese Mint oil, their quality control, improve crystal purity, size, colour and flavour ofcrystallisation.

The duties assigned in the job description are as follows:

- 1. Carry out parameters optimisation of the production of crystal Menthol from <u>Mentha</u> <u>arvensis</u> oil by chilling process.
- 2. Improve crystal purity, size, colour and flavour by recrystallisation or other means.
- 3. Upgrade free Menthol content of raw material prior to processing for crystals.
- 4. Advise on standards for international market, quality control and export packing and storage related matters.
- 5. Recommended any requirements for further development of Menthol production.

In addition, the consultant will beexpected to prepare a final report on his mission, setting out his findings

and recommendation on further action to be taken. However, during various meetings with the CTA and the National Project Director regarding programming of the whole work, it was decided to convert powder menthol to bolder crystals of Menthol. First, followed by upgrading of Mint oil and matching the prouduct with international standards for export purposes.

### Tough task of making crystal Menthol from Vietnamese Mint oil

The expert who had many years of experience for making bold crystals of Menthol from standard variety of Mentha arvensis as raw materials or the Shivalik variety of Mentha arvensis as raw materials was faced with the Vietnamese raw material of an entirely different composition characterized by very low free Menthol content (52.0%) and high Menthone and Terpenes content. The expert was therefore confronted with the task of developing a news technological approach for producing crystal menthol suited to the poor quality of Vietnamese raw material. In view of very short time at the disposal of the expert, the job of developing a new process suited to Menthol production to Vietnamese Mint oil was carried out successfully, first by demonstrating the principle involved in the commercial process and then developing a step wise production process technology.

## II. FACILITIES AVAILABLE AT THE PROJECT SITE FOR CARRYING OUT THE ASSIGNED WORK

The Enteroil premises the main facility for preparation of Menthol crystals from Mint oil was in the form of

- 1. A walkin chilling chamber with temperature control in the range of + 15 to 5 ° C.
- 2. A walkin freezing chamber with temperature control in the range of 0 to  $-25^{\circ}$  C.
- 3. A stainless steel centrifuge of 50 cm diameter for separation of crystals from mother liquor.

The expert did not have access to the use of multilitre glass or stainless steel mixing, refluxing or dissolving type steam jacket reaction vessels which were needed for recrystallisation of small and impure crystals to make bold crystals. In the absence of this facility, the expert had to improvise use of open buckets and manual stirring for recrystallisation related dissolving work and also improvise heating systems.

# III. THE PRINCIPLE INVOLVED IN COMMERCIAL MANUFACTURE OF MENTHOL FROM MINT OIL

On the global scale, Menthol crystals are made from high menthol containing varieties of Mentha arvensis commercially called Japanese Mint. The standard varieties grown in Brazil and China contains 65 to 70% free Menthol content besides 10-15% additional Menthol in the form of Menthyl acetate. Some newly developed varieties have grown in recent years in India, China and Korea, which still gives higher percentage of Menthol content and corresponding the lesser percentage of Menthyl acetate. The commonly grown latest Indian variety of Mentha named Shivalik contains 82 to 84% of free Menthol and practically traces of Menthyl acetate and low quantity of menthone. The standard method for making Menthol from the above

varieties of Mentha arvensis is based on following basis:

Step wise chilling and freezing to induce crystal formation without any other treatment followed by recovering of small crystals by suitable centrifuging.

This is followed by solvent crystallisation for converting the crystals into bold crystals for higher purity and size. The solvent used for crystallisation can be petroleum solvents such as Pet. Ether 80-100°C or food grade Hexane. Caution has to be exercised that such solvent donot contain odorous, impurity, which effect the Menthol flavour adversely. In case of the standard varieties of Mentha arvensis, which were traditioonally grown in Brazil, China and India. It was customery in some factories to subject the Mint oil to Alkaline hydrolysis in order to convert Menthyl acetate to Menthol. This results in increase in free Menthol content of the oil and consequently higher recovery and ,ield of Menthol. In the recent years, the availability of new varieties with oil having high free Menthol content with no Menthyl acetate, ester hydrolysisj step is not necessary. The example is the high Menthol Shivalik variety of Mentha arvensis.

However, the method to produce bold crystals of Menthol with high yield and purity cannot be carried out from Mentha arvensis of Vietnamese Mint oil, because of the very low Menthol content and higher percantge of Menthone and higher Terpenes in the oil. This difficulty can be approached by comparison of the three types of Mentha oil givxen in the table below:

	Standard Mentha arvensis	Shivalik var.	Vietnam var.
Free Menthol	65%	847	5 <b>2%</b>
Menthyl acetate	10 to 12%	47	47
Menthone	18%	47	247
Terpenes	8 to 10%	87	12%
Higher Terpenes	_	-	87

Please also see the G.L.C. Chart in Fig.1 for composition of Vietnam oil.

WORK ASSIGNED TO THE CONSULTANT

Iv. In consultation with the CTA, and National Project Director, the following jobs were decided to be completed during one month mission of the Consultant.

- i) Production of bolder crystals of Menthol of international standards.
- 11) Parameter optimisation of the production of crystal Menthol from Vietnamese Mint oil.
- iii) Improve crystal purity, size colour and flavour by recrystallisation
- iv) Upgrading free Menthol content of Mint oil prior to processing for crystals.
- v) Advise on standards on international markets, quality control export packaging and storage related matters.
- vi) Recommendations for further development of Menthol production

### V. WORK CARRIED OUT BY THE CONSULTANT

The Consultant was successful in producing bold crystals from inferior grade powder Menthol already in production by the Enteroil contains fairly large quantity of adhering oil and that makes the crystal look very bad in respect of colour, size, purity and flavour. Thus the main job for the Consultant was to prepare bold crystals from powder Menthol. The term bold crystals is

referred to shinning white, good size, dried, easily separable with other crystals, long storage life, purity and flavour. The comparative characteristics of Menthol crystals prepared by Enteroil and that of Consultant are shown in table below:

Characteristics	Enteroil Page 1	Consultant	International standards
1. Melting point	Less than 40°C	44°C	42-44°C
2. Colour, size purity	powder, sticky	Shinning white	100% by GLC
	impure 95% by GLC	100% by GLC	
3. Non-volatile residue	More than 0.05%	Not more than 0.05%	Not more than 0.05%
4. Optical rotation	Not maching	-49 <b>°</b>	-49 to -51

The respetive G.L.C. charts of powder Menthol of Enteroil and bolder crystals of Menthol prepared by the Consultant along with photographs are attached.

Fig. 1 GLC CHART	Vietnam Mint oil
Fig. 2 GLC CHART	Powder Menthol Entercil
Fig. 3 GLC CHART	Bold crystals of Menthol prepared by Consultant.
Fig. 4 PHOTOGRAPH	Showing crystals of Menthol Enteroil Consultant
Fig.5 GLC CHART	The GLC diagram of enriched Menthol fraction

BAC H Start	A .ONG NGUYET		1. IG.1		G.L.C. Cha (Vietnames	ert se Mint oil)	
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5	4.367	1986	٧		8.8497		7
3	4.567	32	٧		8.8252		
4	4.875	397	V		0.309	*********	
5 6	5.822	2848	Y		1.5871	MONOTERPENES	
7	5.392 5.9	63 4578	V V		0.0492 3.5625	1	
8	6.447	18	Ÿ		9.8141		
9	6.7	15			8.8117	)	
i 6	7.183	496			0.3857	series de la con	
11	8.487	31339			24.3862	MENTHÓNE	
12 13	- 8.727 9.878	5314 67171	Y		4.135 52.2686	MENTHOL	
14	9.9	120	Ÿ		8.0932		
15	10.497	3988	¥		3.0971		
16	10.817	5738	٧		4.4589	MENTHYL SCETATE	
17 18	11755	385	Y		0.2374		
19	11.988 12.427	3390 133	V		2.6379 0.1835		
29	13.2	193	Ÿ		8.1582		
21	13.55	76	¥		0.059		
22	14.883	241	u		0.1872		
23 24	15.2 15.748	49 1975	V		0.0381 0.8365		
25	16.3	21	•	•	8.016		
26	16.75	125	٧		0.0975		
27	17.212	347	٧		0.2783		
28 29	17.565 18.05	46 189	٧		9.0357		
38	18.553	199	V		0.078 0.0145		
		• •	•		0,0140		

#### POWDER MENTHOL ENTEROIL FIG.2 GLC CHART

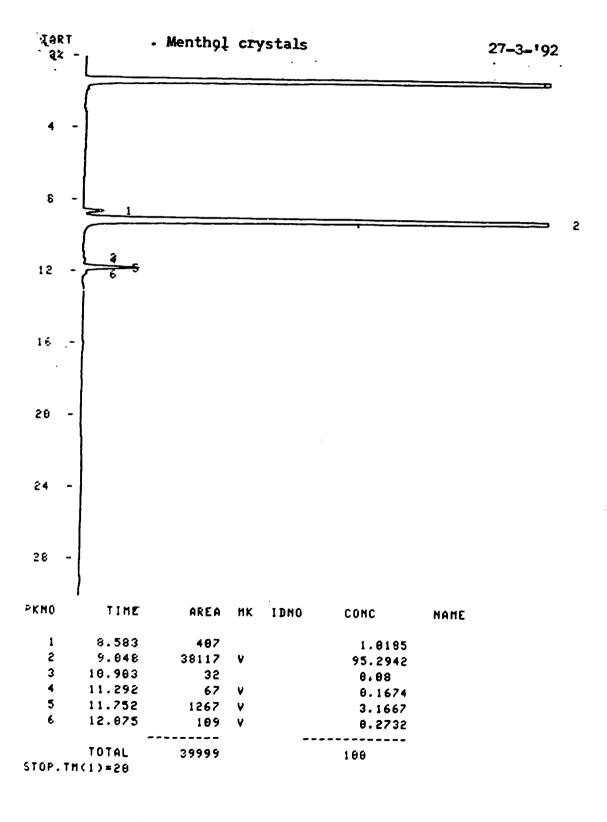


FIG.3 GLC CHART BOLD CRYSTALS OF MENTHOL PREPARED BY CONSULTANT

221-25412

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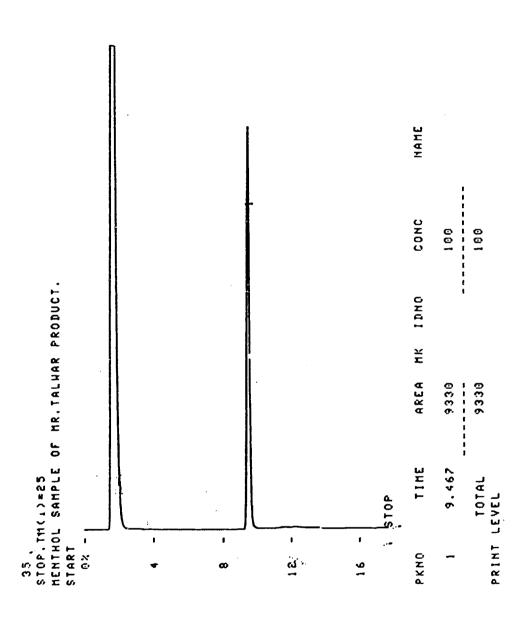
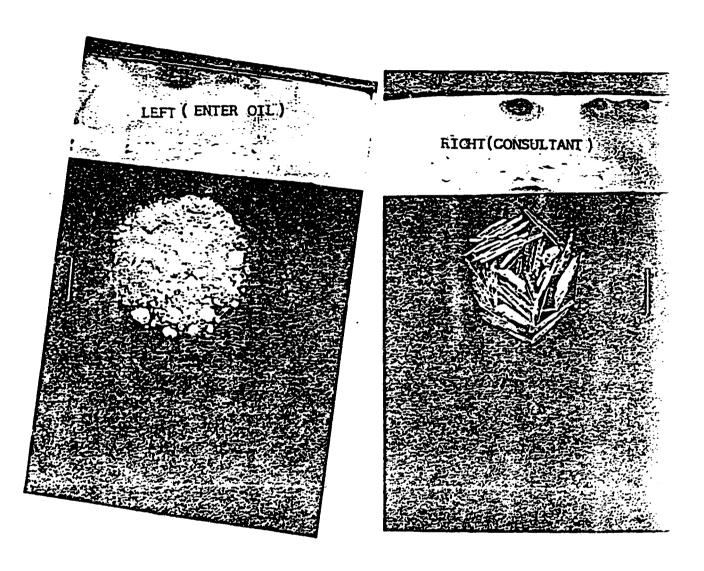
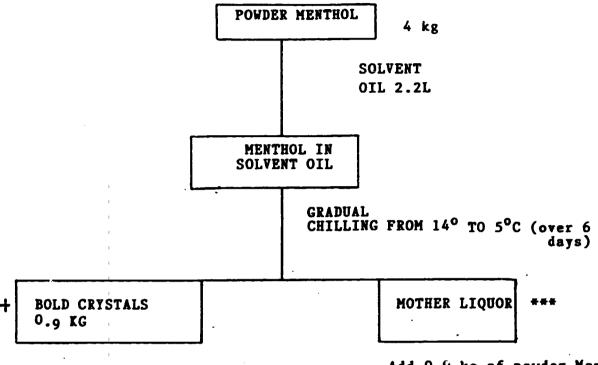


FIG. 4

PHOTOGRAPHS OF CRYSTAL MENTHOL PREPARED BY THE ENTER OIL
AND CONSULTANT



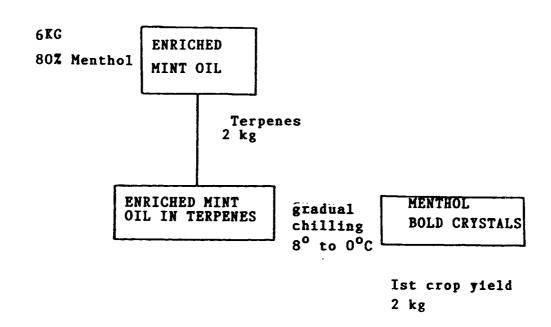
### VI. FLOW SHEET FOR PRODUCTION OF BOLD CRYSTALS FROM POWDER MENTHOL



Add 0.9 kg of powder Menthol
to replenish 0.9 kg bold
crystals recovered at +
Repeat this cycle many
times, each time,
replenish menthol
by adding 0.9kg powder
crystal.

- \* Obtained by drastic stepwise chilling of V.N. oil to subzero temperatures.
- \*\* Petroleum ether 80°C or food grade Hexane solvent
- \*\*\* Recycle mother liquor by adding calculated quantity of powder Menthol followed by chilling + 14° to +5°C for recovering further crops of bold crystals.

# VII. FLOW SHEET FOR THE PRODUCTION OF BOLD CRYSTALS FROM ENRICHED VIETNAM MINT OIL



MOTHER LIQUOR

\* Terpenes fraction obtained by fractional distillation of Vietnam Mint oil. This solvent is a mixture of monoterpene hydrocarbons.

### VIII. MATERIAL BALANCE OF 4 KG LOT OF POWDER MENTHOL

- i) Took 4 kg powder Menthol
- ii) Solvent oil 2.2 litre V/W
- iii) Ist crop 0.9 kg
- iv) Remaining mother liquor Recycled
- v) Added powder Menthol 9.9 kg
- vi) Recovery of Menthol 2nd crop 1.6 kg
- vii) Total powder Menthol used 4.9 say 4.6 kg being 95.07
- viii) Total Menthol recovered 2.5 kg
- ix) Total Menthol left in 2.1 kg mother liquor
- x) Loss of Menthol Nil
- xi) Loss of solvent oil in 0.6 litre two batches
- IX. OPERATING PROCEDURES FOR PRODUCTION OF MENTHOL CRYSTALS BOLD FROM POWDER MENTHOL
- I. Production of bold crystals from powder crystals
- 1. Solvent Petrol ether, 80 C (b.p.)
- 2. Preparing the solution for crystallisation:
  - Cleaning of powder: put 2.2 kg of solvent and 4 kg of powder, dissolve the powder crystals into solvent filtering-transparent, clear solution- put to the chilling chamber.

### 3. Crystallisation step

- 3.1 Gradual crystallisation: 16°C (start temperature)
- 3.2 Velocity of chilling:

Day 1 2 3 4 5 6

Temperature  $16^{\circ}_{\cdot}^{\circ}$ C  $14^{\circ}$ C  $12^{\circ}$ C  $10^{\circ}$ C  $9^{\circ}$ C  $6\pm1^{\circ}$ 

- 3.3 Chilling time: 6 days
- 3.4 Centrifuging the first crystals ( $\underline{A}_{kg}$ )
- 3.5 Put more A kg powder crystals to mother liquor and start again chilling chamber from  $16^{\rm o}{\rm C}$  with the same chilling velocity.
- 3.6 After 5 times (cycles), chill the mother liquor to get powder.
- 3.7 Put powder to new prepared solvent
- 3.8 Start from point 3.1

Production of bold crystal from enriched Mint oil

Refer flow sheet page = 11

## X. PRODUCTION OF MENTHOL CRYSTALS FROM MINT OIL OF LOW MENTHOL CONTENT

### 1. UPGRADING OF THE OIL

Vietnamese Mint oil is poor in Menthol content. It has the following compositions:

Monoterpenes6.8 %Menthone24.4 %Menthol52.3 %Menthyl acetate4.5 %

Sesquiterpenes and other components 12.0 %

The GLC diagram of the oil is also demonstrated in GLC chart Fig.1. It is important to solve the problem of its upgrading before crystallisation. The process of upgrading is by fractionation in the vacuum distillation column with dia 100 mm (ID), height 4m, packed with high flux packings. 42.5 kg of the oil was fractionated into 4 fractions

·Ist fraction(monoterpenes)	4.2 kg	9.9 %
2nd fraction ( menthone)	4.9 kg	18.6 %
3rd fraction (enriched menthol)	23.9 kg	5 <b>4.</b> 5 <b>%</b>
4th bottom residue	5.4 kg	12.7 %
5th loss due to rectification water in oil and polymerization	1.8 kg	4.2 %
	42.5 kg	100 %

# Composition of upgraded 3rd fraction Vietnamese Mint oil

### TABLE - II

Menthone	11.5 %
Menthol	79.2 %
Menthyl acetate	3.2 %
Other components	6.1 %

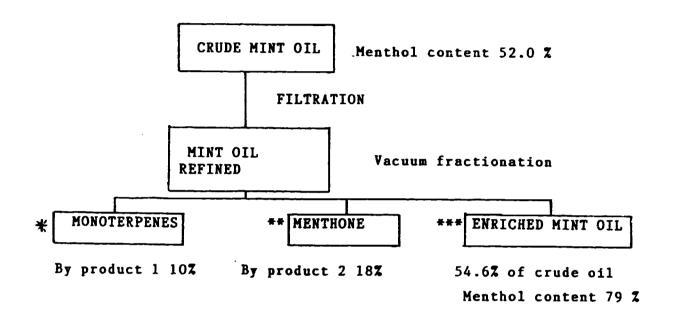
The GLC diagram of enriched menthol fraction is shown in Fig. 5

### FIG.5 - THE GLC DIAGRAM OF ENR. HED MENTHOL FRACTION

TEST SLOP MENT STAR	ING E HOL	50 sec 31.2001 - PKT.				Upgraded	Mint	oil
દુ	- {	·			MEI	NTHONE		
	(							MENTHOL
ie	- (		MENTHY	L AC	ETATE	•		
	-							
16	- }	6 7						
20	-							
24	-							
ekno	1	TIME ,	AREA	nĸ	IDNO	conc	NAME	
1		8.922	6269			11.5193		
ż		9.458	43062	V		79.2414		
3		10.382	1827			3.3621		
4		11.188		V		3.2368		
5		12.305	1240			2.2813		
€.		15.14	80			0.1472		
7		16.008	115			6.212		
		-	54949			106		

P P

### FLOW SHEET FOR THE UPGRADING OF VIETNAMESE MINT OIL



This forms the starting material for Menthol production

- \* By product 1 use as solvent for crystallisation
- \*\* By product 2 saleable in Tobacco flavour industry and cheap blending agent.
- \*\*\* For detailed composition see Table- IV page 18

### TABLE-III

Monoterpenes	6.8 %
Menthone	24.4 %
Menthol	52.3 %
Menthyl acetate	4.5 %
(4.76) Sesquiterpenes and other	
heavy components	12.0 %

### TABLE -- IV

# COMPOSITION OF UPGRADED VIETNAM MINT OIL UPGRADING BY RECTIFICATION

Menthone	11.5 %
Menthol	79.2 %
Menthyl acetate	3.2 %
Other components (Isomenthol, Sesquiterpenes)	6.1 %

### TABLE-V

# BY-PRODUCTS RECOVERED DURING UPGRADING VIETNAM MINT OIL

*Terpenes	4.2 kg	9.8 %
* Menthone	7.9 kg	18.6 %

<sup>\*</sup> To be used as solvent for bold crystals when petroleum solvents are not available.

<sup>\*</sup> Menthone saleable as cheap flavour and tobacco blending

### FINAL RECOMMENDED FLOW SHEET FOR PROCESSING VIETNAMESE MINT II. OIL Vietnamese Mint oil Menthol = 52 %Stepwise 100 kg Chilling-to Sub Zero Temperature Menthol crystals Dementholised oil 75 kg 36% Menthol= 27 kg 25 kg Fractionation Terpenes Enriched DMO Menthone 45 kg 60% Menthol = 27 kg 10 kg 20 kg Add 55 kg fresh Vietnamese Mint oil Total 45+55 = 100 kgFinal composition Menthol = 55.6 kg

Feed for second cycle

= 44.4 kg

Rest

Note: The overall process losses are expected to be maximum 5%.

### XII. RECOMMENDATIONS

The following steps will be desirable in Vietnam and Enteroil desired to become a big producer and exporter of Menthol.

- a) There is a strong need to produce better quality raw material that is variety giving Mint oil with at least 70% free Menthol or a variety in which the total Menthol and Menthyl acetate content is at least 75%. The presently Mint oil produced in Vietnam has average 52.0% Menthol only.
- b) It appears that the distillation technology at field is primitive because it is largely based on hydrodistillation. It is felt that by switching over to steam distillation the existing variety of the oil is likely to be upgraded from 52.0 to 65.0% free Menthol content. Steam distillation requires larger initial capital investment but the results obtained and cost benefit analysis justify adoption of steam distillation as a means of high quality oil in higher yield and greater Menthol recovery.
- c) At the Enteroil premises the following additional modifications and new equipments should be added in order to carry out Menthol recovery and bold crystallization properly.
- i) The basket centrifuge should be refrigerated or installed in a cold room at 2°C. It is possible to install a cooling system in the room in which the centrifuge is installed.
- ii) A stainless steel reaction vessel provided with a stirrer and reflux condenser is necessary as an additional input for the Menthol production unit. This vessel should have a bottom discharge

opening. This vessel will be used for dissolving powder Menthol in solvent oil for making bold crystals. (Drawing enclosed Appendix-1)

- iii) All electrical fittings in crystallisation and filteration room should be spark proof to prevent an chance of fire due to presence of solvent and no smoking sign should be strictly observed.
- iv) If in future better quality raw material that is Mint oil with large proportion of Menthyl acetate is available for processing, the hydrolysis of esters may be carried out as a pretreatment prior to chilling process. This hydrolysis can be carried out in stainless steel vessel proposed under (ii).
- v) Menthone which is going to be a sizeable by-product of Vietnamese Mint oil can be sold at a low price for tobacco blending and pharmaceutical industry. However, it is desirable to convert it into high value products by converting it into a mixture of l-Menthol and liquid Menthol by Sodium ammonia reduction or suitable catalytic hydrogenation. This mixture is saleable at higher price than Menthone. Recovery of crystal menthol from the mixture is technically feasible, but not economically viable on the production scale, at which Enteroil is dealing with.
- vi) The expert has demonstrated the unit step involved in Menthol technology. The expert has also applied these steps involved in order to obtain data from Vietnamese low quality Mint oil for production of bold crystals.

The expert has further processed the Vietnamese Mint oil in more than one way to produce crystal Menthol in order to arrive

at conclusion which forms the basis of the proposed technological approach for production of bold crystals from Vietnamese low Menthol content raw material. Temperature, time and conditions of crystallisation has been optimized for the major crop of the crystals. Optimal yield will be obtained by Enteroil by the judicious recycling of the mother liquor. This approach of the recycling has been outlined in the recommended process.

### XIII CONCLUSION

- 1. Because of high content of Menthone and higher Terpenes in Vietnamese Mint oil, the procedures for bold crystals of Menthol production has to be in some differences from Indian oil.
- 2. Results in crystallisation shows that it is possible for the production of bolder crystals of Menthol from inferior grade powder Menthol already in production by the process stepwise chilling to match the international standards.
- 3. Upgrading the Vietnamese oil after getting 25% of the crystalMenthol by stepwise chilling is likely to be possible solution for the production of crystal Menthol.

### XIV. DAILY DIARY OF THE CONSULTANT

7th March, 1992	Arrival in Hanoi
8th March, 1992	Sunday
9th March, 1992	Had detailed briefing with CTA regarding programming of the whole work.
10th March,1992	Production of bold crystals from powder Menthol
11th March,1992	Gradual chilling to optimise the conditions
12th March, 1992	Continuing
13th March, 1992	Fractionation of Vietnamese Mint oil for upgrading continuation
14th March, 1992	Continuation
15th March, 1992	Centrifuging of bold crystals
16th March,1992	Recycling of bold crystals
17th March, 1992	Determination of international standards of the bold crystals
18th March, 1992	Continued
19th March, 1992	Continuation of gradual chilling for optimisation of bold crystals
20th March, 1992	Continued
21st March, 1992	Continued
22nd March, 1992	Recovery of bold crystals
23rd March, 1992	Production of bold crystals
	from enrichewd Mint oil using
	monoterpenes as solvent

Gradual chilling for getting 24th March, 1992 bold crystals 25th March, 1992 Production of bold crystals from powder Menthol (new lot) Continuation of gradual chilling 26th March, 1992 Continued 27th March, 1992 Continued 28th March, 1992 Continued 29th March, 1992 30th March, 1992 Preparation of final report to 2nd April, 1992 Departure for Jammu, India 3rd April, 1992

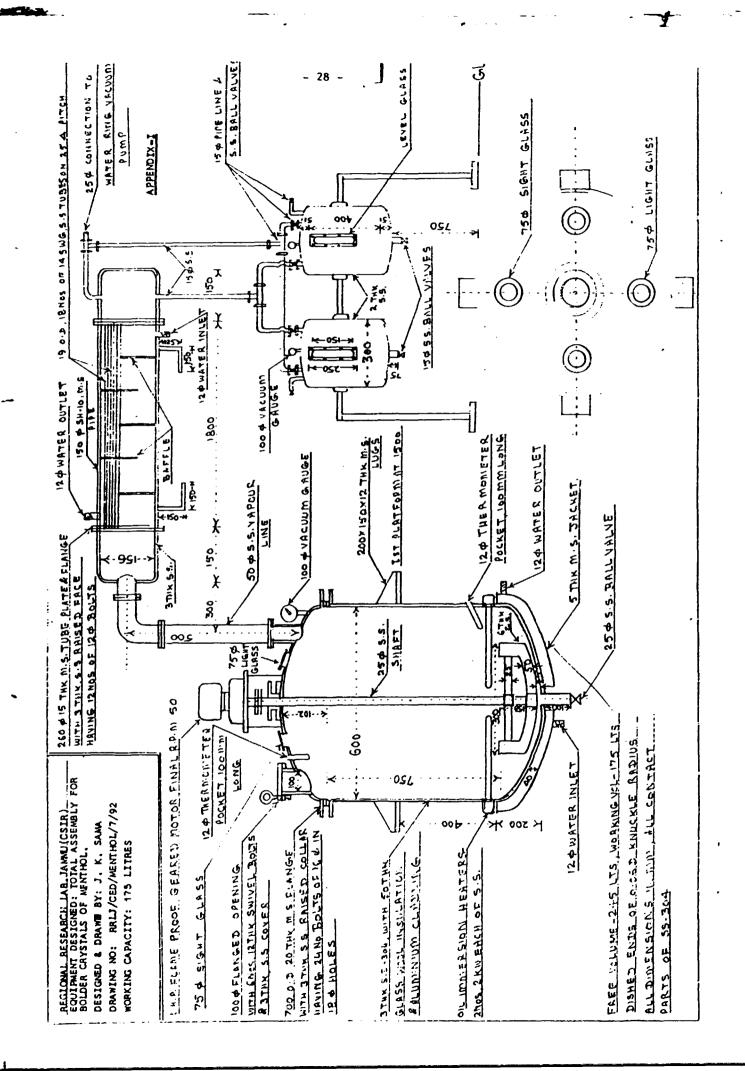
### <u>ACKNOWLEDGEMENTS</u>

During the time of my work as specialist in Essential Oils Enterprise (Enteroil), I have got very good cooperation from CTA, Dr. C.K.Atal and help and co-operations from Enteroil General Director, Mr. Le Trong Vong, the National Project Director, Dr. Nguyen Quyet Chien and other Vietnamese colleagues, Dr. Dang Xuan Hao, Dr. Luong Si Binh, Mr. Van Ngoc Danh. My thanks to all these people.

I am also thankful to Mr. Nguyen Nha Duc who was very co-operative with me in solving difficulties and who helped in secretarial assistance.

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- 8. Mr. Nghiep Tinh Dau, National Centre for Scientific Research of Vietnam Hanoi, Vietnam.



### Backstopping Officer's Technical Comments based on the work of Mr. Y.P. Talwar DP/VIE/84/010/11-54

The report contains in detail the work carried out by the Consultant. The Consultant has accomplished his task successfully and suggested improvements to enhance the yields of menthol. He has given the design for a reaction vessel which could be fabricated in Vietnam. The inferior quality mint oil has to be enriched as suggested by the Consultant. The need to improve field processing of mint using steam distillation should be considered as a measure to increase the yields. Suggested introduction of a good variety has to be persued as a future investment. The other recommendations of the Consultant in terms of safety requirements, air conditioning of the room and chilling of oil should be implemented on a priority basis. The technology for production of bold crystals from menthol powder as well as from enriched oil has been transferred. The more economic use of the by-products and the mother liquor has also been suggested.