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PROCESSING OF AROMA CHEMICALS AND FRAGRANCE MATERIALS

DP/VIE/86/033/11-53

VIET NAM

Technical report: Aroma chemicals and perfume blending\*

Prepared for the Government 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 S. Jain, aroma chemicals and  
perfume blending expert

Backstopping Officer: T. De Silva  
Chemical Industries Branch

United Nations Industrial Development Organization  
Vienna

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### ABSTRACT

The mission took place over a period between 22nd November 1993 and 14th January 1994 during which time the expert was attached to The Vietnam Fragrance and Flavour and Personal Product Co., situated near Ho Chi Minh City.

As per Job Description of the assignment, the expert functioned in collaboration with the National Project Coordinator and the counterpart staff, to fulfil the following specific functions:

- transferred technology for the production of methyl ionone.
- advised on the production of other aroma chemicals and isolates from locally available essential oils.
- trained counterpart staff members in the development of process parameters for the production of aroma chemicals and isolates.
- formulated perfume blends for incorporation into local soaps and detergents.
- supervised and directed incorporation of the said perfume blends in local soaps and detergents.
- evaluated test marketing and recommended market strategies for the locally produced soaps and detergents.
- assisted in setting up the sensory evaluation laboratory.
- recommended additional developments and processes for new products.

In addition to the aforementioned work performed as per the Job Description, the following work was done at the request of the Backstopping Officer:

- evaluated the functioning of all items of pilot plant equipment supplied to the project.
- detected defects in construction and faults in operation of the said pilot plant equipment.
- evaluated the know how transfer with respect to what had been included in the purchase order.
- advised on safety requirements.

Further, at the request of UNDP/UNIDO, Hanoi, the following work was also done during the mission:

- assisted the counterpart in preparation of contract for purchase of know how package from Prof. Qui of Ho Chi Minh University.
- assisted the counterpart in preparation of list of raw materials required to demonstrate the processes included in the know how package.
- assisted the counterpart in the preparation of specifications for the soap machinery required for the pilot scale production and test facility.

## INTRODUCTION

The project DP/VIE/86/033, " Processing of Aroma Chemicals and Fragrance Materials" funded as part of an agreement between the Vietnamese Government and UNDP/UNIDO has enabled the General Department of Chemistry through the Vietnam Fragrance and Flavour and Personal Product Co. (VIFF) to greatly expand its activities and take up the manufacture of Aroma Chemicals and Fragrance Materials in addition to its present activities.

Vietnam has been a producer of natural essential oils but in recent times this industry has suffered through want of Research and Development backup and modern marketing initiatives. To remedy this situation, the Government through the agency of the UNDP and UNIDO has provided the industry with Research and Development backup in the form of a project titled, "Processing of Vietnamese Essential Oils and Related Natural Products", DP/VIE/84/010. Since new prospective essential oils will soon become available and a dire need for fragrance materials exists, the Government again through the agency of the UNDP and UNIDO, wishes to set up this second project, directed towards fractionation of locally produced essential oils and production of a basic array of aroma chemicals in order to formulate and compound fragrance materials. These fragrance materials are to be produced first on a bench scale and once the technological problems are surmounted, produced on a pilot scale to serve the needs of an ongoing soap and toileteries industry. Export possibilities will also be assessed to provide a potential foreign exchange earning capacity.

The expert was expected to work in collaboration with the National Project Coordinator and counterpart staff members and was to be responsible for transferring technology for the manufacture of Aroma Chemicals, prepare perfume blends for use in soaps and detergents, conduct test marketing, evaluate the pilot plant and prepare terms of reference for purchase of technology from a National Expert and decide specifications for Soap Machinery to be purchased by the project authorities.

The main duties as per the job description were as follows:

- transfer technology and demonstrate the production of methyl inonone on a pilot scale if the requisite chemicals are supplied by the counterparts.
- advise on the production of other aroma chemicals and isolates from locally available essential oils.
- train counterpart staff members in development of process parameters for the production of aroma chemicals and isolates.
- formulate perfume blends for incorporation into local soaps and detergents.

- supervise and direct incorporation of locally blended perfumes in soaps and detergents.
- evaluate test marketing methods and recommend market strategies for the locally produced soaps and detergents.
- assist in setting up the sensory evaluation laboratory.
- recommend additional developments and processes for new chemicals.

The expert was expected to furnish a complete and fully processes terminal report at the completion of his mission.

Please refer to Annexure 1 for Job Description

The following additional duties were assigned by the backstopping officer during the course of the briefing with reference to the pilot plant supplied to the project:

- evaluate the functioning of all items of equipment supplied.
- detect any defects in construction and faults in the operation of the equipment.
- evaluate the know how transfer with respect to what has been included in the purchase order.
- advise on safety requirements.

Please refer to Annexure 2 for official letter on the subject.

After the terminal TPR meeting of this project, an agreement was reached between SPC, UNDP and UNIDO to field the expert to perform the following additional duties during the course of his mission:

- to assist the counterpart in the preparation of contract for the purchase of know-how package from Prof. Qui of Ho Chi Minh City University.
- to assist the counterpart in the preparation of list of raw materials required for demonstrating the processes included in the aforementioned know how package.
- to assist the counterpart staff in preparing the specifications for soap machinery for the test scale soap production facility.

Please refer Annexure 3 for communication from UNIDO, Hanoi on the subject.

**FINDINGS, OBSERVATIONS AND WORK PERFORMED****1. Technology of Methyl Ionone:**

It was found that all the required chemicals necessary to demonstrate the production of this aroma chemical were not available with the project authorities.

The full list of the chemicals required had been provided to Mr. Nguyen Van Quy, Deputy Director, Vinarom and Dr. C.K. Atal, CTA during the course of their visit to the factory of the expert during 20 January to 23 January 1992 to discuss this subject. Subsequently, the list of materials required for methyl ionone was faxed to the backstopping officer in Vienna on 23.9.93 who in turn transmitted the information to the project authorities. The project authorities informed the expert as per their fax dated 11/13 October 1993 that two chemicals, namely, Benzul Chloride and Triethyl amine were not available. The expert upon receiving this information again requested that the materials in question be arranged so that a practical demonstration of the full process may be given. This was however not done.

Consequently, during the briefing the expert received permission from the backstopping officer to transfer the technology to the project authorities and forego its practical demonstration if the requisite chemicals were not made available by the counterparts.



Upon arrival at the project site at the commencement of the mission, it was found that two essential chemicals, namely benzyl chloride and triethyl amine were not available. Consequently the practical demonstration of the pilot scale production process of methyl ionone could not be done. In lieu thereof, the detailed process technology including all procedures, parameters and precautions was transferred to the project authorities/counterparts.

For details of the process, see Annexure 4.

## 2. Production of Other Aroma Chemicals from Locally Available Essential oils:

It was found that the following essential oils are locally available in large quantities:-

- a. Citronella java oil
- b. Eucalyptus citriodora oil
- c. Litsea cubeba oil
- d. Turpentine oil
- e. Sassafras oil

These essential oils could be used to make the following aroma chemicals and isolates:-

- |                                |   |
|--------------------------------|---|
| i. Citronella java oil:        | Geraneol<br>Citronello<br>Dimethyl Octanol<br>Geranyl Acetate<br>Geranyl Formate<br>Geranyl Butyrate<br>Citronellyl Acetate<br>Citronellyl Formate<br>Citronellyl Butyrate<br>Citronellyl Valerate<br>Geraneol for Soap<br>Citronella java oil Terpenes |
| ii. Eucalyptus citriodora oil: | Hydroxycitronellal<br>Citronellol<br>Isopulegol<br>Esters of Citronellol<br>Eucalyptus citriodora oil Terpenes  |
| iii. Litsea cubeba oil:        | Citral<br>Ionone pure or 100%<br>Ionone Alpha<br>Methyl Ionone<br>Citronellol   |

Esters of Citronellol  
Citral Nitrile

iv. Turpentine oil:

Terpeneol  
Terpenyl Acetate  
Pine oil  
Isobornyl Acetate  
Isoborneol  
Camphor  
Dipentene  
Pine Tar

v. Sassafras oil:

Safrole  
Heliotropin

The fact that such a large variety of aroma chemicals and isolates could be produced from locally available essential oils was explained to the project authorities. It was emphasised during the discussions that from a long term point of view, the counterparts should not depend on purchased or given know how but should develop their own research and development facilities to develop process technology.

It was also explained to them that the aroma chemicals to be manufactured should be chosen on the basis of their captive requirements and that inhouse demand for aroma chemicals should be met first.

As the first step towards enlarging the variety of aroma chemicals that the counterparts are able to make, it has been decided subject to approval by UNDP/UNIDO to purchase know how from a Vietnamese expert who is a Professor of Chemistry in HCM University.

It has been explained to the counterparts that a consultant/adviser relationship may be continued with this scientist so that he may be able to advise them on technical matters on a day to day basis and help them to set up a proper research and development facility.

### 3. Training of Counterpart Staff Members:

One of the primary objectives of the project is the production of Aroma chemicals from Vietnamese essential oils. To this end, a pilot plant consisting of reactors and distillation equipment has been set which is supposed to be used for the manufacture of Aroma Chemicals and also for the development of new technologies in the same field.

It was found that this pilot plant had not been properly commissioned by the suppliers of the equipment and as such was not being used for the production of aroma chemicals as originally intended.

In the absence of functional production facilities, recourse was taken to explanation of the process parameters in detail to the counterpart staff with each point being explained on the pilot plant. It was explained to the counterpart staff that the engineering aspect of any chemical plant had a critical bearing upon the eventual success or failure of any process. The critical design parameters of the plant were co-related to the process parameters of the aroma chemicals in which the counterpart staff were interested.

It was also explained to the counterpart staff that from a long term point of view it was necessary to develop in-house research and development facilities so as to sustain the anticipated growth in the production and consumption of aroma chemicals.

#### 4. Formulation of Perfume Blends for Incorporation into Local Soaps and Detergents:

It was found that the main item of production for the project authorities was detergents and that in future it was planned to go in for production of toilet soaps on a large scale.

Since no fragrances are produced locally in Vietnam, the entire requirement has to be imported resulting in financial as well as logistical difficulties for the consumers. It was felt that if technology could be developed for the formulation of perfume blends in Vietnam then the industry would be able to derive a competitive edge from the new technology. It would also of course result in the establishment of a new industry in Vietnam.

With these objectives in mind it was decided to formulate a number of soap blends and to transfer the technology for the same to the project authorities.

The personnel trained by the expert in sensory evaluation and perfume blending during the the course of his last mission to the project were available and using these personnel and the aroma chemicals and essential oils procured by the project authorities, a proper sensory evaluation and perfume blending laboratory was set up in the premises provided by the project authorities.

The laboratory was duly equipped with the following:-

- samples of raw materials
- weighing balances of proper accuracy
- magnetic stirrer and heater
- working tables with proper shelves
- efficient exhaust and ventilation system
- wash basin
- conical flasks, beakers, pipettes, droppers and funnels
- detached smelling room free from odours for odour evaluation

Using this laboratory and with the help and co-operation of the counterpart staff, the following soap fragrances were successfully created:-

1. SANDALWOOD SOAP COMPOUND
2. LUX FRAGRANCE TYPE 1
3. LUX FRAGRANCE TYPE 2
4. PALMOLIVE TYPE SOAP COMPOUND
5. LUX SOAP COMPOUND TYPE 3
6. CAMAY SOAP COPMOUND
7. SANDALWOOD SOAP COMPOUND TYPE 2
8. PALMOLIVE SOAP COMPOUND TYPE 3
9. PALMOLIVE SCAP COMPOUND TYPE 4
10. LEMON - EAU DE COLOGNE TYPE
11. LEMON - FLORAL TYPE
12. JASMINE SOAP COPMOUND - FRSH FLORAL TYPE
13. JASMINE SOAP COMPOUND - SWEET TYPE

5. Incorporation of Locally Blended Perfumes in Soaps and Detergents:

It was found that the project authorities did not have small scale production and test equipment for soap such as a triple roller mill, a mixer-blender and a plodder which are essential for testing fragrances in soaps.

As a stop gap measure, the project authorities provided a domestic grating and shredding machine which was used for mixing the fragrance with the soap although not in the best possible manner. Because of the inadequacy of the equipment, the results obtained were at best approximate.

The mix of soap and fragrance was stamped into soap tablets in a hand operated stamping machine.

The soap tablets thus produced had the locally blended soap perfume incorporated in it and these soap tablets were then evaluated and the formulations modified till satisfactory results were obtained.

For final formulations. please see Annexure 5

6. Evaluation of Test Marketing Methods and Recommendation of Market Strategies:

It was found that the concepts of test marketing were not very well established and as such a short seminar was conducted at the premises of The Saigon Cosmetic Company and the same was attended by the personnel of The Saigon Cosmetic Company as well as the counterpart staff.

In this seminar, the topics covered were as under:-

- market survey
- assessment of market requirement
- design of the product to suit the requirement
- in-house evaluation of the product design
- release of product in a limited area to conduct test marketing
- evaluation of data generated during test marketing
- finalisation of product design
- national launch of product
- importance of advertising
- importance of standardisation

The concepts explained were in accordance with modern management practices and it was explained to the participants that in developing market conditions of Vietnam it was essential to follow these practices to survive in the market in the face of national and international competition.

**7(a) Evaluation of the Functioning of All Items of Equipment(pilot plant) Supplied:**

It was found that a complete pilot plant for the manufacture of aroma chemicals had been procured by the project authorities as a part of the project. It was found that this pilot plant, although designed to be of universal application, had been supplied specifically to produce two aroma chemicals, namely, Citronellol and Terpineol. It was further found that the said pilot plant was not in a functional condition. As per the information provided by the project authorities, the pilot plant had in fact not been satisfactorily commissioned by the suppliers of the equipment and the processes contracted for had not been satisfactorily implemented on the pilot plant.

A technical evaluation was made of the said pilot plant by comparing the design requirements dictated by the processes to be implemented on the pilot plant and the actual design of the pilot plant.

**7(b) Detection of defects in construction and faults in operation of the equipment:**

It was found during the technical evaluation of the equipment that certain design deficiencies were apparent in the equipment on the basis of standard chemical engineering practices and these were in turn leading to the inefficient operation of the equipment. The same were studied carefully and have been duly elaborated and enumerated in the technical evaluation.

**7(c) Evaluation of Know-how Transfer with Respect to the Purchase order:**

It was found that there were some deviations from the purchase order in respect of the equipment and know-how as mentioned in the purchase order and as actually supplied to the project authorities. These were duly studied and have been elaborated and enumerated in the technical evaluation.

A detailed and comprehensive report of the technical evaluation covering all the aforementioned aspects was made and submitted to the project authorities.

Please see Annexure 6, for the detailed report.

**8. Assistance to Counterparts in Preparation of Terms of Reference for Purchase of Know-How package from National Expert:**

It was found that during the terminal TPR meeting of this project, based on the request of the Ministry of Heavy Industry and the State Planning Committee, the meeting agreed to the project counterpart to carry out the following activities in order to complete the project in a successful manner:

- to sign a contract with a competent local university or institution to carry out the transfer of the know how package (preparation of two aroma chemicals on the pilot plant) which was contracted to the plant supplier but the supplier has not yet started with the work.

- to sign a local contract to develop the technologies for preparation of three aroma chemicals on the pilot plant and to train project staff in these technologies. These tasks were planned to be conducted by three international experts. The experts have remained selected for a long time but their missions could not be realised due to the incompleteness of the pilot plant.

It was found that the project authorities had nominated as the local expert, Prof. Tran Kim Qui, Director of The Research Centre for Applied Chemistry, HCM City to fulfill the aforementioned tasks and as such at the request of UNIDO/UNDP, HANOI, a job description/terms of reference for the purchase of know-how package covering the transfer of know how for the following aroma chemicals was prepared and submitted to the project authorities:-

- heliotropin ex sassafras oil
- citronellol ex citronella java oil/ eucalyptus citriodora oil
- geraneol ex citronella java oil
- terpineol ex turpentine oil
- methyl ionone ex litsea cubeba oil
- hydroxy citronellal ex eucalyptus citriodora/citronella java oils

The job description/terms of reference covered the following aspects:-

- transfer of technology and demonstration of production
- production of commercially acceptable qualities
- provision of raw materials
- training of counterpart staff

Subsequently a quotation was received from Prof. Tran Kim Qui and the same was forwarded to UNIDO/UNDP, HANOI by the project authorities.

For Job Description/Terms of Reference, please see Annexure 7

9. Assistance to Counterparts in the Preparation of List of Raw Materials Required to Demonstrate the Processes Included in the Know-How package:

At the request of UNIDO/UNDP, HANOI, the list of raw materials required for the demonstration of the commercial processes for citronellol, geraneol, heliotropin, terpenol, methyl ionone and hydroxy citronellal was discussed with Prof. Tran Kim Qui and finalised and forwarded to UNIDO/UNDP HANOI. The project authorities obtained quotations from two local suppliers and these were forwarded by them to UNIDO/UNDP, HANOI.

Subsequently, a detailed explanation regarding the value and quantities of raw materials mentioned in the list was submitted to UNIDO/UNDP, Hanoi.

It was felt by the UNIDO/UNDP, HANOI and the State Planning Committee, Hanoi that the list was excessive and accordingly a revised list containing only 60% of the previous quantities was submitted to the State Planning Committee, Hanoi with copy to UNIDO/UNDP, HANOI.

For list of raw materials and related papers, please see Annexures 8, 8A, 8B

10. Assistance to Counterparts in Preparation of Specifications for Soap Machinery Required for Test Scale Soap Production Facility:

It was found that in the terminal TPR meeting of this project, based on the request of the Ministry of Heavy Industry and the State Planning Committee, the meeting agreed for the project counterparts to purchase locally one plodder and one presser(stamper) for the test scale soap production facility in order to complete the project in a successful manner.

At the request of UNIDO/UNDP, HANOI, a detailed specification for both the plodder and the stamper was drawn up. Various possible sources of local supply were examined but none was found to be suitable except The Saigon Cosmetic Company which is itself manufacturing soap using machinery made in its own workshops. The working of these machines was observed and found to be acceptable. Thereafter, quotations were obtained from this party by the project authorities and forwarded to UNIDO/UNDP, Hanoi. Subsequently a detailed explanation was submitted to UNIDO/UNDP, Hanoi regarding the machinery detailing therein the various functions required to be fulfilled by the machinery as well as the configuration in which it would be installed and operated.

The State Planning Committee wished for some amendment to be made in the existing quotation which was arranged by the project authorities and submitted to the authorities.

For specifications of soap machinery and quotation. please see Annexure 9 and 9A.

## CONCLUSIONS AND RECOMMENDATIONS

### CONCLUSIONS:

#### 1. Production of Methyl Ionone:

It was concluded that although the technology for the commercial production of methyl ionone has been transferred to the project authorities, its practical demonstration could not be carried out for the following reasons:-

- non-availability of critical raw materials required for the preparation of the catalyst, namely, benzyl chloride and triethyl amine
- the pilot plant not being in a functional condition

It was further concluded that if the manufacture of aroma chemicals is to be a regular activity in the project, then the following inputs have to be organised on a professional basis by the project authorities:-

- the logistics of being able to obtain basic chemicals as and when required to serve as raw materials for the manufacture of aroma chemicals



- sufficient knowledge about chemical engineering and process technology so as to overcome the various problems that occur during any regular production programme and cannot always be foreseen by the consultant or technical adviser.

## 2. Production of Other Aroma Chemicals from Local Essential Oils:

It was concluded that a very useful variety of natural essential oils is available in Vietnam which can be utilised for the production of aroma chemicals required in the manufacture of perfumes/fragrances.

It was also concluded that know-how for only a small number of aroma chemicals would be transferred to the project authorities under the on-going project and therefore in order to be able to make the full range of aroma chemicals possible from the locally available essential oils, know-how would have to be developed within the project.

It was therefore concluded that for the long term viability of the project and for the realisation of the desired outputs, a proper research and development facility devoted to the development of process technology for aroma chemicals would have to be created as a logical extension of the technical inputs already provided by UNIDO/UNDP.

## 3. Training of Counterpart Staff:

It was concluded that to become fully competent in the manufacture of aroma chemicals, the technical personnel of the project would have to be trained under practical conditions in the various aspects of the industry i.e. process technology, chemistry, engineering and quality control.

It was concluded that in order to achieve this, the pilot plant would have to be made operational so that the production of some aroma chemicals could be initiated. At the same time, arrangements would have to be made to consume the aroma chemical/s being produced. It is thus further concluded that in addition to the aforementioned training aspects, training would also have to be given further so as to enable the staff to independently formulate useable fragrances using the in-house aroma chemicals to the maximum extent.

It was concluded that under the on-going UNIDO/UNDP programme, substantial training has already been given which is fundamental in nature and that now to function in an industrial environment, hands-on training would have to be given under practical production conditions.

#### 4. Formulation of Perfume Blends:

It was concluded that it was a feasible proposition to formulate perfume blends locally using the technology provided by the expert so as to create soap and detergent perfumes useable in the local products.

It was also concluded that from a long term point of view, the staff already trained in the art of perfume blending would have to be trained further so as to enable them to create fragrances independently. It was concluded that to achieve this the following must be done:-

- raw materials, both natural and synthetic, from all the major perfumery companies should be available in the perfumery creation laboratory
- the staff should become familiar with the usage of the materials by conducting trials and using the guidelines provided by the manufacturers of raw materials
- independent creation of fragrances should then be attempted using at first the formulae provided by the expert as a guideline and later trying new and original combinations

It was concluded that thereafter, it would be possible to make fragrances for in-house use as well as for sales to other customers.

#### 5. Incorporation of Locally Blended Perfumes in Soaps:

It was concluded that without proper test scale soap production machinery, it was not possible to conduct proper trials of locally blended perfumes in soaps.

It was also concluded that since the manufacture of toilet articles and personal products was the stated commercial aim of the company, it would be necessary to create an applications laboratory to fulfill the following functions:-

- test the fragrances from in-house laboratory in the article for which they are destined
- test fragrances from other suppliers in a similar way
- develop new toilet articles and personal products using ideas and technology provided by the suppliers of raw materials

It was concluded that after the creation and proper utilisation of these facilities, the company would be able to exploit the market to the maximum extent.

## 6. Test Marketing Methods and Marketing strategies:

It was concluded that Vietnam is a fast developing market for consumer products of various kinds especially for toilet articles and personal care products and that as more and more multinational companies set up manufacturing facilities in the country, the market share of the domestic companies would come under pressure. It was concluded that in order to maintain the market share in the face of foreign competition, constant touch with the needs of the consumer would have to be maintained and products developed to suit the consumer.

It was concluded that to achieve this end, the domestic companies which have hitherto been used to practically captive markets would have to establish facilities to fulfill the following functions:-

- survey the market to assess consumer needs
- design products as per the consumer needs
- test market the newly designed products
- market the product after consumer approval

## 7. Technical Evaluation of the Pilot Plant:

It was concluded that the pilot plant as supplied to the project authorities was not in an optimal functional condition due to certain design defects in the original design of the equipment as ordered and also due to some deviations from the purchase order itself.

It was concluded upon examination of these defects that certain steps would have to be taken to make the pilot plant fully functional. These steps could be undertaken in two stages as follows:-

- making alterations to the existing plant to remove basic defects in the reactors to make them fit for use as per the process requirements
- addition of some essential equipment, such as a thermic fluid heater and vacuum pumps of sufficient capacity, in addition to the aforementioned alterations, to make the pilot plant fully functional on a long term basis.

It was concluded that the alterations to the reactors could be carried out by the project authorities with only nominal expenditure and in point of fact would have to be carried out before any processes could be demonstrated on the pilot plant.

## RECOMMENDATIONS

### 1. Production of Methyl Ionone:

It is recommended that:-

- all the requisite raw materials be procured by the project authorities so as to be able to initiate the production of this aroma chemical
- the pilot plant be made functional by carrying out all the necessary repairs/alterations and additions
- for future production of other aroma chemicals, the procurement of the raw materials be organised in a professional manner so that at the time of need all items are available, since the lack of even one chemical can prevent a process from being implemented
- the technical personnel acquire sufficient knowledge and training regarding the engineering principles underlying the production of aroma chemicals so that day to day problems can be handled by them after the project is completed

### 2. Production of Other Aroma Chemicals:

It is recommended that:-

- the maximum utilisation of the indigenous essential oils be made to make the largest possible variety of aroma chemicals.
- proper research and development facilities be set up to develop process technology for these aroma chemicals, if possible under the guidance of an experienced technical expert

### 3. Training of Counterpart Staff:

It is recommended that:-

- the technical staff of the project be trained under actual and practical production conditions so that they can become familiar with the technology of manufacture of aroma chemicals as well as the techniques used therein

### 4. Formulation of Perfume Blends:

It is recommended that:-

- a comprehensive collection of perfumery raw materials be made in the perfume creation laboratory of the project

- the perfumers of the project make themselves completely familiar with these raw materials so as to be able to know their characteristics and use them accordingly

- after familiarising themselves with these raw materials, the perfumers of the project begin to attempt perfume creations independently and carry on with the process till they gain the requisite proficiency in the art

#### 5. Incorporation of Locally Blended Perfumes in Soaps:

It is recommended that:-

- proper test scale soap production machinery be procured by the project to enable them to test properly the fragrances created as a result of the technical inputs provided to the project

#### 6. Test Marketing Methods and Market Strategies:

It is recommended that:-

- divisions be created in the project to conduct market surveys, create new product designs, evaluate the new product designs, conduct test marketing, evaluate the results of test marketing and formulate marketing strategies accordingly

- these activities be supported by a properly equipped applications laboratory wherein new products can be designed, formulated and tested

#### 7. Technical Evaluation of the Pilot Plant:

It is recommended that:-

- that the pilot plant be made functional at the earliest by carrying out the work recommended in the report on the captioned subject, in two phases if necessary by first modifying the reactors and the adding equipment such as a thermic fluid heater, vacuum pumps and a rubberlined centrifuge.

**ANNEXURE 1****JOB DESCRIPTION****DP/VIE/86/033/11-53**

- Post Title:** Aroma Chemicals and Perfume Blending Expert
- Duration:** 1.5 w/m
- Date Required:** ASAP
- Duty Station:** Ho Chi Minh City
- Purpose of Project:** Utilization of indigenous essential oils to develop suitable fragrance materials and formulations for local industry as well as export.
- Duties:** The expert will work in collaboration with the National Project Coordinator and counterpart staff members to perform the following specific duties:
- Transfer technology and demonstrate the production of methyl ionone on a pilot scale if the requisite chemicals are supplied by counterparts.
  - Advice on the production of other aroma chemicals and isolates from locally available essential oils.
  - Train counterpart staff members in development of process parameters for the production of aroma chemicals and isolates.
  - Formulate perfume blends for incorporation into local soaps and detergents.
  - Supervise and direct incorporation of locally blended perfumes to soaps and detergents.
  - Evaluate test marketing methods and recommend market strategies for the locally produced soaps and detergents.
  - Assist in setting up the sensory evaluation laboratory.
  - Recommend additional developments and processes for new products.

The expert will furnish a complete and fully processed terminal report at the completion of his mission outlining the findings of his mission and his recommendations for follow-up action.



## ANNEXURE 2

**UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION**


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VIENNA INTERNATIONAL CENTRE

P.O. BOX 300, A-1400 VIENNA, AUSTRIA

TELEPHONE: 211 310 TELEGRAPHIC ADDRESS: UNIDO VIENNA TELEX: 135612 uno a FAX: 232156

23 November 1993

Dear Dr. Quong

 Subject: DP/VIE/86/033 - Processing of Aroma Chemicals and  
Fragrance Materials

Please assist Mr. Sudhir Jain to carryout the following activities as his report will assist us in finalizing the payments to be made to the supplier of pilot plant equipment.

- 1) Evaluate the functioning of all items of equipment supplied.
- 2) Detect any defects in construction and faults in operations of equipment.
- 3) Evaluate the know-how transfer with respect to what has been included in the Purchase Order.
- 4) Advice on safety requirements.

This work will be done in addition to incorporation of perfumes into soaps and market testing. His work will be limited to the available chemicals and what we have now ordered. He will also transfer the know how on methyl ionone but may not be able to demonstrate pilot production due to lack of essential chemicals.

Please get permission to procure the soap making accessories and conduct the workshop planned.

Thank you very much.

With kind regards.

Sincerely yours,

  
 T. De Silva  
 Special Technical Adviser  
 Chemical Industries Branch  
 Department of Industrial Operations

Dr. Quong  
Vice NPD

cc. Mr. R. Morey  
UNDP HANOI

## ANNEXURE 3

United Nations  
Development Programme

World Development

## F A C S I M I L E

|  |                                     |   |
|--|-------------------------------------|---|
| <b>TO: MR. NGUYEN XUAN THUAN</b><br><b>DIRECTOR</b><br><b>LIAISON OFFICE. (HCMC)</b><br><b>FOR: MR. DO LINH CUONG</b><br><b>DNPD OF VIE/86/033</b> | <b>Date: November 29, 1993</b>      |   |
|  | <b>Message No. /93</b>              |   |
|  | <b>Fax No.: 231834</b>              |   |
| <b>From: P. HJORTLUND</b><br><b>UNIDO FIELD OFFICER</b><br><b>HANOI, VIETNAM</b>   | <b>Account:</b><br><br><b>UNIDO</b> | <b>File: VIE/86/033</b><br><b>(GEN)</b> |
|  | <b>Drafted by: NKT/BH</b>           |   |
| <b>Subject: VIE/86/033, AROMA CHEMICALS</b>  |                                     | <b>Total Page(s): 03</b>                |

MESSAGE:

TOP PRIORITY

Please find attached one copy each of the letter of Dr. De Silva and our fax relating to the coming mission of Mr. Dudhir Jain.

According to the conclusion of the recent terminal TPR meeting and our following discussion with Mr. Gautier, Senior Deputy Resident Representative, please most urgently prepare with Mr. Jain Terms of Reference for the local purchase orders for the know-how package; the transfer of the technologies for the 3 other fragrances and the soap equipment.

With best regards.

URGENT





## ANNEXURE 4

PREPARATION OF METHYL IONONE  
COMMERCIAL PROCESSSTEP 1: PREPARATION OF THE CATALYST( BENZYL TRIETHYL AMMONIUM  
HYDROXIDE )

THE CATALYST IS PREPARED IN SITU FOR THE FIRST STEP OF THE PROCESS  
IE PRODUCTION OF PSEUDO METHYL IONONE.

THE RAW MATERIALS REQUIRED ARE AS UNDER:-

1. METHANOL, anhydrous.
2. BENZYL CHLORIDE, min 99% purity
3. TRIETHYL AMINE, min 99% purity
4. POTASSIUM HYDROXIDE, min 85% purity, commercially pure grade

THE EQUIPMENT REQUIRED IS AS UNDER:-

A HEATED STAINLESS STEEL REACTOR WITH EFFICIENT STIRRING AND  
PROVISION FOR ADDITION OF REACTANTS, MEASUREMENT OF TEMPERATURE,  
MAINTAINING REFLUX AND A BOTTOM OUTLET AND DISTILLATION OF SOLVENTS  
AND UNREACTED REACTANTS.

A 50 LITRE FLASH DISTILLATION UNIT

A 50 LITRE FRACTIONATION UNIT

## PROCEDURE:

IN THE STAINLESS STEEL REACTOR, ADD UNDER STIRRING, ONE BY ONE,  
CAREFULLY WEIGHED QUANTITIES OF THE FOLLOWING CHEMICALS AT ROOM  
TEMPERATURE:-

1. 30 lits METHANOL
2. 1750 g BENZYL CHLORIDE
3. 1750 g TRIETHYL AMINE

AFTER THE ADDITION WAS COMPLETE, STIRRING WAS CONTINUED AND HEATING  
WAS STARTED. THE RATE OF HEATING WAS CONTROLLED SO AS TO FIRST  
INITIATE AND THEN MAINTAIN REFLUX. THE REFLUX TEMPERATURE WAS 66 DEG  
CENTIGRADE

IF THE HEAT SOURCE IS STEAM, THEN THE TEMPERATURE IS CONTROLLED BY  
OPENING OR CLOSING THE STEAM INLET VALVE TO THE DESIRED EXTENT TO  
CONTROL THE FLOW OF STEAM AND HENCE THE TRANSFER OF HEAT TO THE  
REACTOR.

IF THE HEAT SOURCE IS HOT OIL/THERMIC FLUID, THEN THE TEMPERATURE  
IS CONTROLLED IN TWO STAGES:-

- i) FIRST THE THERMOSTAT AT THE HOT OIL/THERMIC FLUID HEATER IS SET  
AT A TEMPERATURE OF 30-35 DEG CENTRIGRADE ABOVE THE TEMPERATURE

DESIRED IN THE REACTOR AND THE HEATERS ARE SWITCHED ON. THESE HEATERS WILL NOW OPERATE AUTOMATICALLY AND SWITCH THEMSELVES ON OR OFF THROUGH THE THERMOSTAT WHILE MAINTAINING THE DESIRED TEMPERATURE IN THE HOT OIL SYSTEM.

ii) SECOND THE FLOW OF OF HOT OIL/THERMIC FLUID IN THE LIMPET COIL IS CONTROLLED BY OPENING OR THROTTLING THE HOT OIL/THERMIC FLUID INLET AND OUTLET VALVES TO THE DESIRED EXTENT SO AS TO ATTAIN THE DESIRED TEMPERATURE IN THE REACTOR. IT IS USUAL TO OPEN THE VALVES TO THE FULL EXTENT IN THE BEGINNING AND THEN TO ADJUST THEM ONCE THE DESIRED TEMPERATURE HAS BEEN ACHIEVED.

THE REFLUX WAS CONTINUED UNDER STIRRING FOR HALF AN HOUR.

IN THE MEANTIME A SOLUTION OF 1000 g OF POTTASSIUM HYDROXIDE WAS MADE IN 15 lits OF METHANOL UNDER HEATING AND STIRRING AND KEPT READY FOR ADDITION IN THE HOT OR AT ROOM TEMPERATURE AS MAY BE CONVENIENT ACCORDING TO THE EQUIPMENT IN HAND.

AFTER THE REFLUX FOR HALF AN HOUR WAS OVER, THE AFOREMENTIONED KOH/METHANOL SOLUTION WAS ADDED TO THE REFLUXING MASS UNDER STIRRING. HEATING IS STOPPED DURING THE COURSE OF THE ADDITION AND IS STARTED AGAIN AFTER THE ADDITION IS COMPLETE, SO AS TO RESTART THE REFLUX. THE REFLUX IS NOW CONTINUED FOR 5 HOURS WHILE MAINTAINING STIRRING AT THE SAME TIME.

NOTE: THE QUANTITY OF POTTASSIUM HYDROXIDE TAKEN HERE IS ON THE BASIS OF 85% PURITY OF THE COMMERCIAL GRADE. IF A MORE PURE GRADE IS BEING USED, THE QUANTITY IS TO BE ADJUSTED ACCORDINGLY.

AS THE REFLUX IS CONTINUED FOR FIVE HOURS, POTTASSIUM CHLORIDE SEPARATES AS A PRODUCT OF THE REACTION AND REMAINS SUSPENDED AS A SALT IN THE SOLUTION. AFTER THE REFLUX IS OVER, THE HEATING AND STIRRING ARE STOPPED AND THE REACTION MASS IS LEFT OVERNIGHT.

## STEP 2: PREPARATION OF PSEUDO METHYL IONONE

NEXT DAY, TO THE SAME BATCH A FURTHER QUANTITY OF 1000 g OF POTTASSIUM HYDROXIDE FLAKES ARE ADDED UNDER STIRRING AND STIRRING IS CONTINUED TILL THE KOH DISSOLVES IN THE REACTION MASS.

THEREAFTER 50 KILOS OF METHYL ETHYL KETONE IS ADDED UNDER STIRRING AND WHILE CONTINUING THE STIRRING, THE TEMPERATURE IS RAISED TO 50 DEG CENTIGRADE.

NOW AT THE ELEVATED TEMPERATURE OF 50 DEG CENTIGRADE, 20 KILOS OF FRESHLY REDISTILLED LEMONGRASS OIL/ LITSEA CUBERA OIL (STABILISED WITH 0.5% HYDROQUINONE) CONTAINING NOT LESS THAN 77% CITRAL IS ADDED QUICKLY UNDER STIRRING WITHIN A PERIOD OF 15-30 MINUTES.

AS THE ADDITION OF THE LEMONGRASS/LITSEA CUBEBA IS CONTINUED, THE TEMPERATURE BEGINS TO RISE AND TOWARDS THE COMPLETION OF THE ADDITION IT SHOOTS UP TO 63 DEG CENTIGRADE.

HEATING IS CONTINUED FURTHER AT THIS STAGE AND THE TEMPERATURE IS RAISED TO 65-67 DEG CENTIGRADE AND MAINTAINED AT THIS LEVEL FOR 15-30 MINUTES.

TO THE AFOREMENTIONED REACTION MASS IS NOW ADDED , UNDER STIRRING BUT AFTER DISCONTINUING HEATING, 2000 ml OF GLACIAL ACETIC ACID SO AS TO BRING THE pH OF THE ENTIRE REACTION MASS DOWN TO 6.THE pH IS CHECKED AFTER STIRRING THE ENTIRE REACTION MASS WELL AFTER THE ADDITION OF THE GLACIAL ACETIC ACID SO AS TO GIVE A TRUE READING OF THE pH.

THE UNIT IS NOW GOT READY FOR DISTILLATION.

THE EXCESS METHANOL AND THE METHYL ETHYL KETONE IN THE REACTION MASS IS NOW DISTILLED OFF.THE STIRRING IS CONTINUED AND THE POT TEMPERATURE IS RAISED TO 70 DEG CENTIGRADE.THE BOILING POINT OF METHANOL IS 65-66 DEG CENTIGRADE AND DISTILLATION BEGINS AT THIS POINT.THE HEATING IS CONTINUED SUCH THAT THE POT TEMPERATURE GRADUALLY RISES AND THE DISTILLATION IS CONTINUED AND MAINTAINED.THE RECOVERY OF THE METHANOL AND THE METHYL ETHYL KETONE IS CONTINUED BY DISTILLATION TILL A POT TEMPERATURE OF 95 DEG CENTIGRADE AT WHICH TEMPERATURE THE RECOVERY IS COMPLETE FOR ALL PRACTICAL PURPOSES AS THE BOILING POINT OF METHANOL IS 65-66 DEG AND THE BOILING POINT OF METHYL ETHYL KETONE IS 80 DEG CENTIGRADE.THE RECOVERY DISTILLATION IS DONE UNDER ATMOSPHERIC PRESSURE.

THE TOTAL RECOVERY OF METHANOL PLUS METHYL ETHYL KETONE IS 92.5 KILOS.

THE CONTENT OF METHYL ETHYL KETONE OF THE RECOVERED MATERIAL IS ESTIMATED BY THE HYDROXYLAMINE HYDROCHLORIDE METHOD. THE MIXTURE OF METHANOL AND METHYL ETHYL KETONE IS FRACTIONATED SEPARATELY TO GET PURE CUTS OF METHANOL AND METHYL ETHYL KETONE AND THE OVERLAPS ARE SET ASIDE FOR FRACTIONATION ALONGWITH THE RECOVERED MATERIAL FROM THE NEXT BATCH.

THE RESIDUAL PRODUCT REMAINING BEHIND IN THE REACTOR IS NOW TRANSFERRED TO THE WASH VESSEL AND WASHED AS FOLLOWS TILL NEUTRAL:-

FIRST WASH:- BY 50 LITS OF PLAIN NEUTRAL DEMINERALISED WATER. THE QUANTITY OF WATER REQUIRED FOR THE WASH IS PREWEIGHED AND ADDED TO THE MATERIAL TO BE WASHED UNDER STIRRING.FOR PROPER WASHING TO TAKE PLACE THE WASH VESSEL MUST BE EQUIPPED WITH AN EFFICIENT STIRRER.AFTER THE ADDITION OF THE WATER UNDER STIRRING, THE STIRRING IS CONTINUED FOR 10-15 MINUTES AND THE ORGANIC AND THE AQUEOUS LAYERS ARE THEN ALLOWED TO SETTLE FOR 30 MINUTES BY STOPPING

THE STIRRING.AFTER THE LAYERS HAVE SETTLED, THE AQUEOUS LAYER IS REMOVED FROM THE BOTTOM AND SET ASIDE FOR SOLVENT EXTRACTION IF NECESSARY.THE ORGANIC LAYER IS CARRIED FORWARD TO THE NEXT WASH.

THIS STIRRING AND SETTLING PROCEDURE IS TO BE FOLLOWED WITH EVERY WASH.

SECOND WASH:- BY 50 LITS OF 8-10% COMMON SALT(SODIUM CHLORIDE SOLUTION)

THIRD WASH:- BY 25 LITS OF PLAIN NEUTRAL DEMINERALISED WATER.

THIS PLAIN WATER WASH IS REPEATED IF THE MATERIAL IS NOT NEUTRAL AFTER THE THIRD WASH

AFTER THE WASHES HAVE BEEN COMPLETED AS DETAILED ABOVE, THE CRUDE PSEUDO METHYL IONONE OBTAINED IS WEIGHED.THE WEIGHT OF THE CRUDE SHOULD BE 26.750 KILOS.

### STEP 3: DISTILLATION OF CRUDE PSEUDO METHYL IONONE

THE CRUDE METHYL IONONE OBTAINED ABOVE IS CHARGED INTO A 50 LITRE SHORT COLUMN DISTILLATION UNIT FOR THE PURPOSES OF FLASH DISTILLATION.

TO CONDUCT THE DISTILLATION, PROCEED AS FOLLOWS:-

AFTER THE CHARGING OF THE CRUDE PSEUDO METHYL IONONE INTO THE DISTILLATION UNIT IS COMPLETED, COMMENCE THE HEATING OF THE FLASK/REBOILER GRADUALLY TILL IT REACHES 60-70 DEG CENTIGRADE.AT THIS POINT APPLY VACUUM TO THE DISTILLATION UNIT THROUGH THE WATER RING VACUUM PUMP GRADUALLY TILL IT REACHES THE MAXIMUM THAT THE WATER RING VACUUM PUMP CAN DELIVER IE 710mm.NOW START RAISING THE TEMPARATURE AGAIN SLOWLY. AS THE TEMPARATURE CROSSES 70-75 DEG, THE RESIDUAL MOISTURE IN THE SYSTEM WILL BEGIN TO BE RECOVERED ALONGWITH SOME ORGANIC MATERIAL, MAINLY METHANOL AND METHYL ETHYL KETONE. RAISE THE TEMPARATURE SLOWLY TO 80-85 DEG WHILE MAINTAINING THE VACUUM AND CONTINUING THE RECOVERY/DISTILLATION OF MOISTURE AND LOW BOILERS.

THE TOTAL RECOVERY OF MOISTURE AND LOW BOILERS AT THIS STAGE IS 2.900 TO 3.000 KILOS.

AFTER THE RECOVERY OF MOISTURE AND LOW BOILERS UNDER THE GIVEN CONDITIONS HAS BEEN COMPLETED, NO MORE MATERIAL WILL DISTILL. AT THIS POINT, THE REFLUX TEMPARATURE WILL BEGIN TO FALL.NOW, START RAISING THE TEMPARATURE OF THE FLASK/REBOILER AND AT THE SAME TIME DISCONNECT THE WATER RING VACUUM PUMP FROM THE SYSTEM AND CONNECT THE MAIN VACUUM PUMP THROUGH THE THREE WAY BALL VALVE INSTALLED FOR

THIS PURPOSE. SLOWLY INCREASE THE VACUUM IN THE SYSTEM WHILE INCREASING THE TEMPERATURE ALSO TILL THE PRESSURE IN THE UNIT IS REDUCED TO 10-20mm AND THE TEMPERATURE IN THE FLASK/REBOILER RISES TO 110 DEG. AT THIS POINT DISTILLATION OF LOW BOILERS WILL COMMENCE. CONTINUE TO INCREASE THE VACUUM IN THE SYSTEM TILL THE PRESSURE IS REDUCED TO 1-2 mm AND ALLOW THE TEMPERATURE TO RISE SO AS TO MAINTAIN THE DISTILLATION. THE ENTIRE DISTILLATE IS TO BE COLLECTED AS A SINGLE CUT. THE DISTILLATION WILL CONTINUE TILL A REBOILER TEMPERATURE OF 180 DEG AT 1-2 mm PRESSURE.

THE RESIDUE OF DISTILLATION AT THIS STAGE WILL AMOUNT TO 2.800 TO 3.000 KILOS AND IS TO BE SET ASIDE FOR A SECOND FLASH DISTILLATION SO TO COMPLETELY RECOVER ALL PSEUDO METHYL IONONE.

THE WEIGHT OF THE DISTILLATE RECOVERED AFTER THE FLASH DISTILLATION IS 20.000 TO 21.000 KILOS EXCLUDING THE WEIGHT OF THE MOISTURE AND REBOILERS.

THIS DISTILLATE IS NOW TAKEN FOR FRACTIONATION TO OBTAIN THE PSEUDO METHYL IONONE. THE PROCEDURE IS AS UNDER:-

THE FLASH DISTILLED CRUDE PSEUDO METHYL IONONE IS CHARGED INTO A 50 LITRE FRACTIONATION UNIT FITTED WITH A 2 METER X 100 mm COLUMN PACKED WITH KNITTED WIRE MESH OR BETTER STILL SULZER PACKING.

THE HEATING OF THE FLASK/REBOILER IS STARTED AND WHEN THE TEMPERATURE REACHES 75-80 DEG, THE MAIN VACUUM PUMP IS STARTED AND THE PRESSURE GRADUALLY REDUCED TO 10mm.

AT THIS REDUCED PRESSURE AND REBOILER TEMPERATURE OF 110 DEG, REFLUX WILL COMMENCE. THE COLUMN IS NOW ALLOWED TO STABILISE IE ACHIEVE THE MINIMUM REFLUX/VAPOUR TEMPERATURE AT THE GIVEN TEMPERATURE AND PRESSURE.

THE FRACTIONS ARE NOW TAKEN OUT ONE BY ONE AND AS EACH FRACTION IS TAKEN OUT, THE VACUUM IN THE SYSTEM IS INCREASED AS WELL AS THE TEMPERATURE. WHEN 25% OF THE DISTILLATE HAS BEEN TAKEN OUT, THE VACUUM IS RAISED TO THE MAXIMUM. THE PRESSURE AT THIS STAGE IS 1-2 mm PROVIDED THE VACUUM PUMP IS WORKING AT FULL EFFICIENCY. HOWEVER IN PRACTICE THIS IS NEVER THE CASE AND TEMPERATURES HIGHER THAN THE IDEAL ARE ALWAYS REQUIRED TO CONDUCT THE DISTILLATION.

THE PATTERN OF THIS FRACTIONATION WILL BE AS UNDER:-

| FRAC.NO. | REBOILER TEMP | VAPOUR TEMP | WT. OF FRAC. | TYPE OF FRAC |
|----------|---------------|-------------|--------------|--------------|
| 1        | 110-128 DEG   | 40-85 DEG   | 2.500 KILOS  | TOPS         |
| 2        | 128-143 DEG   | 85-107 DEG  | 2.750 KILOS  | TOPS         |
| 3        | 143-146 DEG   | 107-110 DEG | 1.150 KILOS  | REWORKS      |
| 4        | 146-146 DEG   | 110-125 DEG | 1.100 KILOS  | REWORKS      |

|   |             |             |              |         |
|---|-------------|-------------|--------------|---------|
| 5 | 146-148 DEG | 125-130 DEG | 1.350 KILOS  | REWORKS |
| 6 | 148-180 DEG | 130-140 DEG | 11.850 KILOS | MAINS   |

THE TOPS CONSIST PRIMARILY OF TRACES OF METHYL ETHYL KETONE ALONGWITH TERPENES OF LEMONGRASS OIL/LITSEA CUBEBA OIL.

THE REWORKS CONSIST OF UNREACTED CITRAL ALONGWITH OVERLAPPING PSEUDO METHYL IONONE AND ARE TO BE RECYCLED IN SUBSEQUENT BATCHES.

THE MAINS CONSIST OF PSEUDO METHYL IONONE AND ARE TO BE USED FOR THE CYCLISATION REACTION TO PRODUCE METHYL IONONE.

#### STEP 4: CYCLISATION OF PSEUDO METHYL IONONE

##### RAW MATERIALS REQUIRED:-

- a) PHOSPHORIC ACID 83%
- b) BENZENE
- c) PSEUDO METHYL IONONE

##### EQUIPMENT REQUIRED:-

SAME AS IN PSEUDO IONONE, AND

A 20 LITRE SHORT COLUMN FLASH DISTILLATION UNIT .

A 20 LITRE FRACTIONATION ASSEMBLY

##### PROCEDURE:-

TAKE IN THE REACTOR 9.000 KILOS OF BENZENE AND COOL TO 13 DEG TO 18 DEG CENTIGRADE. ADD TO THIS UNDER STIRRING 9.000 KILOS OF DISTILLED PSEUDO METHYL IONONE AND MAINTAIN THE TEMPERATURE AT 13 DEG TO 18 DEG CENTIGRADE. TO THIS MIXTURE ADD AT THE SAME TEMPERATURE, 75.000 KILOS OF PHOSPHORIC ACID 83% UNDER STIRRING, SLOWLY AND STEADILY WITHIN 45 MINUTES, WHILE MAINTAINING THE SAME TEMPERATURE.

CONTINUE STIRRING AND MAINTAIN THE TEMPERATURE FOR A FURTHER PERIOD OF 30 MINUTES TO 45 MINUTES AT 15 DEG TO 20 DEG CENTIGRADE.

THEREAFTER ADD UNDER STIRRING, 11.000 KILOS OF BENZENE. CONTINUE STIRRING FOR 15 MINUTES AND THEN ADD UNDER STIRRING 20.000 KILOS OF NEUTRAL DEMINERALISED WATER.

STIR FURTHER FOR 30 MINUTES TO 45 MINUTES WHILE MAINTAINING THE TEMPERATURE AT 20 DEG CENTIGRADE.

STOP THE STIRRING AND ALLOW THE LAYERS TO SETTLE.

THE LOWER LAYER OF ACID IS TAKEN OUT AND SET ASIDE FOR CONCENTRATION AND RECYCLING IN SUBSEQUENT BATCHES.

**STEP 5: WASHING OF CRUDE METHYL IONONE**

THE ORGANIC LAYER IS WASHED NEUTRAL AS FOLLOWS:

a) FIRST WASH:- WITH 40 LITRS OF OF PLAIN NEUTRAL DEMINERALISED WATER, WHICH IS ADDED TO THE MATERIAL TO BE WASHED AT ROOM TEMPARATURE UNDER STIRRING AND THEREAFTER STIRRING IS CONTINUED FOR 15 MINUTES AND THEN THE LAYERS ARE ALLOWED TO SETTLE.THE AQEOUS LAYER IS CUT AND CHECKED FOR PRESENCE OF METHYL IONONE.IF PRESENT, THE AQEOUS LAYER IS EXTRACTED WITH BENZENE.

b) SECOND WASH:- WITH 40 LITRES OF 5% SALT SOLUTION, THE PROCEDURE BEING THE SAME AS ABOVE.

c) THIRD WASH:- WITH 40 LITERS OF 3% SODA BICARB SOLUTION, THE PROCEDURE BEING THE SAME AS ABOVE.

d) FOURTH WASH:- WITH 40 LITRES OF PLAIN NEUTRAL DEMINERALISED WATER, THE PROCEDURE BEING THE SAME AS ABOVE.THE AQEOUS WASHES ARE CONTINUED IF THE ORGANIC MATERIAL IS NOT NEUTRAL.

AFTER THE WASHING IS OVER, THE UNIT IS READIED FOR DISTILLATION.

THE TEMPARATURE OF THE REBOILER IS RAISED TO 80-90 DEG CENTIGRADE AND BENZENE IS RECOVERED AT ATMOSPHERIC PRESSURE.THE TOTAL QUANTITY OF BENZENE RECOVERED IS 18.000 TO 20.000 KILOS.THIS IS KEPT ASIDE AND RECYCLED IN SUBSEQUENT BATCHES.

AFTER RECOVERY OF BENZENE THE CRUDE METHYL IONONE IS TAKEN OUT AND WEIGHED.THE WEIGHT OF THE CRUDE OBTAINED IS 8.25 KILOS.

**STEP 6: DISTILLATION OF CRUDE METHYL IONONE**

THE CRUDE METHYL IONONE IS FLASH DISTILLED AND THEN FRACTIONATED IN 20 LITRE ASSEMBLIES AS PER PROCEDURE GIVEN UNDER PSEUDO METHYL IONONE.

THE PATTERN OF FRACTIONATION IS AS UNDER:-

| FRAC. NO. | REBOILER TEMP. | VAPOUR TEMP | WT OF FRAC  | TYPE OF FRAC |
|-----------|----------------|-------------|-------------|--------------|
| 1         | 145-150 DEG    | 105-140 DEG | 1.250 KILOS | REWORKS      |
| 2         | 150-155 DEG    | 140-143 DEG | 1.350 KILOS | REWORKS      |
| 3         | 155-155 DEG    | 143-145 DEG | 2.550 KILOS | MAINS        |

|   |             |             |                      |
|---|-------------|-------------|----------------------|
| 4 | 155-175 DEG | 145-147 DEG | 1.350 KILOS MAINS    |
| 5 | 175-200 DEG | 147-147 DEG | 0.750 KILOS MAINS    |
| 6 |             |             | 0.550 KILOS RESIDUES |

THE REWORKS ARE BULKED TOGETHER AND KEPT ASIDE. WHEN SUFFICIENT QUANTITY IS AVAILABLE THEY ARE REFRACTIONATED AND IN THIS WAY THE PROCESS OF REDISTILLATION OF REWORKS IS CONTINUED.

THE MAINS ARE BULKED TOGETHER AND AREATED BY BUBBLING NITROGEN THROUGH THE BULKED MATERIAL FOR TWO DAYS. A CONTAINER FULL OF THE BULKED MATERIAL IS KEPT ASIDE FOR 3 WEEKS DURING WHICH TIME THE MATERIAL IS PHYSICALLY AGITATED FOR HALF AN HOUR EVERY DAY. THE METHYL IONONE SETTLES DOWN AFTER THIS TIME AND BECOMES OLFACATORILY ACCEPTABLE.

#### REFINEMENT OF USED PHOSPHORIC ACID TO MAKE IT FIT FOR RECYCLING

1. TAKE 100 KILOS OF SPENT PHOSPHORIC ACID IN A STAINLESS STEEL REACTOR.
2. ADD TO IT UNDER STIRRING, 5 KILOS OF ACTIVATED CHARCOAL.
3. HEAT THE CONTENTS UNDER STIRRING TO 60 DEG CENTIGRADE AND CONTINUE HEATING AND STIRRING FOR 30 MINUTES.
4. IF THE SOLUTION REMAINS HIGHLY COLOURED, REPEAT STEP 3 AND INCREASE STIRRING TIME TO 60 MINUTES.
5. FILTER THE CHARCCALED MATERIAL THROUGH A BAG FILTER.
6. TRANSFER THE FILTERED MATERIAL TO AN EVAPORATION PAN AND BOIL OFF THE EXCESS WATER SUCH THAT THE CONCENTRATION OF THE ACID INCREASES TO 83%. AT THIS TIME IT WILL BECOME FIT FOR RECYCLING AGAIN.



## ANNEXURE 5

**FORMULATIONS OF PERFUME BLENDS FOR INCORPORATION INTO  
LOCAL SOAPS AND DETERGENTS**

**1. SANDALWOOD SOAP COMPOUND**

|                                |     |
|--------------------------------|-----|
| SANDELA                        | 150 |
| SANDALORE                      | 150 |
| ALDEHYDE C-11                  | 40  |
| SANDAL MYSORE CORE             | 40  |
| BACDENOL                       | 200 |
| GALAXOLIDE                     | 100 |
| GERANIUM OIL                   | 200 |
| ROSE CRYSTALS                  | 30  |
| DIPHENYL OXIDE                 | 150 |
| CETONE V                       | 20  |
| BACCARTOL                      | 100 |
| ADOXAL                         | 30  |
| JASMINE SOAP COMP-FRESH FLORAL | 100 |
| MUSK AMBRETTE                  | 50  |
| MUSK KETONE                    | 80  |
| METHYL IONONE                  | 80  |
| BENZYL SALICYLATE              | 180 |
| YLANG OIL                      | 70  |
| CAMPHOR                        | 70  |
| DIHYDRO ROSE OXIDE             | 20  |
| ROSE OXIDE                     | 10  |
| DAMASCONE ALPHA 10% IN DPG     | 40  |
| LILLIAL                        | 100 |
| PTBCHA                         | 150 |
| IONONE ALPHA                   | 30  |
| EUGENOL                        | 15  |
| LAVENDIN OIL                   | 70  |
| DIPROPYLENE GLYCOL             | qs  |

## 2.LUX BASE NO.1

|                              |     |
|------------------------------|-----|
| BENZYL ACETATE               | 40  |
| PHENYL ETHYL ACETATE         | 5   |
| STYRALLYL ACETATE            | 10  |
| PHENYL ETHYL ALCOHOL         | 30  |
| ALDEHYDE C 10 10% IN DPG     | 10  |
| ALDEHYDE C 12 LAURIC         | 5   |
| ALDEHYDE C 12 MNA            | 5   |
| ALDEHYDE C 15                | 5   |
| AMYL CINNAMIC ALDEHYDE       | 25  |
| ALDEHYDE C 11                | 15  |
| MYRAC ALDEHYDE               | 5   |
| ALLYL AMYL GLYCOLATE         | 5   |
| AURANTINE EXTRA              | 10  |
| SANDALORE                    | 15  |
| CITRONELLOL                  | 15  |
| CIVET                        | 2   |
| COUMARIN                     | 5   |
| METHYL COUMARIN              | 1   |
| DIMETHYL HYDROQUINONE        | 1   |
| DAMASCONE ALPHA 1% IN DPG    | 15  |
| DIHYDRO MYRCENOL             | 20  |
| SANDELA                      | 10  |
| EVERNYL 1% IN DPG            | 15  |
| PEARLIDE                     | 20  |
| ISO EUGENOL                  | 5   |
| MUSK KETONE                  | 20  |
| IONONE ALPHA                 | 10  |
| BENZYL SALICYLATE            | 40  |
| DIHYDRO ROSE OXIDE 1% IN DPG | 200 |
| EUGENOL                      | 5   |
| ALDEHYDE C 20                | 1   |
| TRIPLAL                      | 2   |
| HYDROXYCITRONELLAL           | 15  |
| ROSE CRYSTALS                | 30  |
| LILLIAL                      | 40  |
| LINALOL                      | 10  |
| METHYL IONONE                | 20  |
| MUSK XYLOL                   | 10  |
| ROSE OXIDE 1% IN DPG         | 100 |
| RASPBERRY 10% IN DPG         | 10  |
| ALDEHYDE C 14                | 5   |
| AMYL SALICYLATE              | 5   |
| CYCLAMEN ALDEHYDE            | 10  |
| VERTOFIX                     | 10  |
| ETHYL VANILLIN               | 1   |
| PTBCHA                       | 50  |
| VANILLIN                     | 5   |
| YARA YARA                    | 10  |
| ORANGE OIL                   | 10  |

|                               |    |
|-------------------------------|----|
| PATCHOULY OIL                 | 15 |
| YLANG OIL                     | 25 |
| FIXOLIDE                      | 30 |
| HEDIONE                       | 1  |
| SANDAL MYSORE CORE            | 2  |
| ALDEHYDE C 13-13              | 2  |
| ALDEHYDE C 11-11              | 5  |
| BOIS AMBREINE FORTE           | 1  |
| ALLYL CYCLOHEXANYL PROPIONATE | 2  |
| DIPROPYLENE GLYCOL            | qs |

## LUX FRAGRANCE TYPE 1

|                    |     |
|--------------------|-----|
| LUX BASE           | 550 |
| DIHYDRO MYRCENOL   | 10  |
| TDM 75             | 30  |
| MYRAC ALDEHYDE     | 2   |
| CITRALVA           | 1   |
| TRIPAL             | 1   |
| ALDEHYDE C 13-13   | 1   |
| ALDEHYDE C 11-11   | 3   |
| CEDROL             | 20  |
| VERTOFIX           | 10  |
| ISO E SUPER        | 5   |
| DIPROPYLENE GLYCOL | qs  |

## 3. LUX FRAGRANCE TYPE 2

|                         |     |
|-------------------------|-----|
| BENZYL ACETATE          | 175 |
| PHENYL ETHYL ACETATE    | 15  |
| STYRALLYL ACETATE       | 25  |
| STYRALLYL ALCOHOL       | 10  |
| PHENYL ETHYL ALCOHOL    | 110 |
| ALDEHYDE C 10           | 3   |
| ALDEHYDE C 12 LAURIC    | 15  |
| ALDEHYDE C 12 MNA       | 4   |
| ALDEHYDE C 18           | 2   |
| HEXYL CINNAMIC ALDEHYDE | 75  |
| ALDEHYDE C 11-11        | 9   |
| ALDEHYDE C 13-13        | 9   |
| MYRAC ALDEHYDE          | 10  |
| ALLYL AMYL GLYCOLATE    | 15  |
| AMBROXAN 1% IN DPG      | 10  |

|                             |     |
|-----------------------------|-----|
| METHYL ANTHRANILATE         | 20  |
| AURANTINE EXTRA             | 30  |
| SANDAL MYSORE CORE          | 5   |
| BACDANOL                    | 15  |
| CITRONELLOL                 | 70  |
| BACCARTOL                   | 50  |
| CITRALVA                    | 5   |
| INDOLE                      | 5   |
| BENZYL PHENYL ACETATE       | 10  |
| PARA CRESYL PHENYL ACTATE   | 15  |
| COUMARIN                    | 50  |
| METHYL COUMARIN             | 10  |
| DIMETHYL HYDROQUININE       | 5   |
| DAMASCONE ALPHA 1% IN DPG   | 100 |
| DIHYDRO MYRCENOL            | 150 |
| EVERNYL 1% IN DPG           | 10  |
| GALAXOLIDE                  | 70  |
| ISOEUGENOL                  | 10  |
| LILLIAL                     | 90  |
| LINALOL                     | 50  |
| METHYL IONONE               | 40  |
| MUSK XYLOL                  | 50  |
| ROSE OXIDE 10% IN DPG       | 10  |
| DIHYDRO ROSE OXIDE          | 10  |
| RASPBERRY KETONE 10% IN DPG | 5   |
| ALDEHYDE C 14               | 5   |
| AMYL SALICYLATE             | 30  |
| MUSK KETONE                 | 40  |
| BENZYL SALICYLATE           | 100 |
| VERTOFIX                    | 20  |
| VANILLIN                    | 10  |
| ETHYL VANILLIN              | 2   |
| PTBCHA                      | 50  |
| YARA YARA                   | 5   |
| CLOVE OIL                   | 10  |
| ORANGE OIL                  | 100 |
| PATCHOULY OIL               | 30  |
| YLANG OIL                   | 30  |
| HEDIONE                     | 5   |
| FLOROTONE                   | 30  |
| GARDINOLENE                 | 45  |
| DIPHENYL OXIDE              | 150 |
| HELIOTROPIN                 | 2   |
| ESTRAGOLE                   | 5   |
| ACETOPHENONE                | 2   |
| DIPROPYLENE GLYCOL          | qs  |

## PALMOLIVE TYPE SOAP FRAGRANCE

|                          |      |
|--------------------------|------|
| COUMARIN                 | 15   |
| METHYL COUMARIN          | 5    |
| DIMETHYL HYDROQUINONE    | 2    |
| HELIOTROPIN              | 5    |
| ANISIC ALDEHYDE          | 5    |
| CITRONELLOL              | 70   |
| PHENYL ETHYL ALCOHOL     | 70   |
| ROSE CRYSTALS            | 30   |
| DIPHENYL OXIDE           | 150  |
| BACCARTOL                | 100  |
| BENZYL ACETATE           | 50   |
| FLOROTONE                | 20   |
| GARDINOLENE              | 20   |
| ALLYL AMYL GLYCOLATE     | 70   |
| DIHYDROMYRCENOL          | 70   |
| ALDEHYDE C 13-13         | 20   |
| ALDEHYDE C 14            | 10   |
| ALDEHYDE C 18            | 5    |
| LILLIAL                  | 70   |
| STYRALLYL ACETATE        | 5    |
| LINALOL                  | 20   |
| ALDEHYDE C 9             | 5    |
| ALDEHYDE C 11            | 10   |
| ADOXAL                   | 5    |
| DIHYDRO ROSE OXIDE       | 10   |
| HEDIONE                  | 5    |
| LINALYL ACETATE          | 20   |
| VANILLIN                 | 5    |
| ETHYL VANILLIN           | 1    |
| LYRAL                    | 30   |
| DAMASCONE ALPHA 10%      | 10   |
| MUSK KETONE              | 30   |
| METHYL IONONE            | 30   |
| BENZYL SALICYLATE        | 60   |
| LAVANDIN OIL             | 30   |
| SANDALWOOD SOAP COMPOUND | 1000 |
| DIPROPYLENE GLYCOL       | qs   |

## LUX SOAP COMPOUND TYPE 3

|                             |     |
|-----------------------------|-----|
| VERTOFIX                    | 30  |
| ISO E SUPER                 | 5   |
| LILLIAL                     | 50  |
| LYRAL                       | 15  |
| DIHYDROMYRCENOL             | 100 |
| PHENYL ETHYL ALCOHOL        | 50  |
| BACCARTOL                   | 30  |
| BENZYL ACETATE              | 40  |
| ALDEHYDE C 11-11            | 15  |
| ALDEHYDE C 13-13            | 20  |
| ADOXAL                      | 5   |
| HELIONAL                    | 2   |
| AMBROXAN 1% IN DPG          | 10  |
| ALDEHYDE MANDARIN 1% IN DPG | 20  |
| MYRAC ALDEHYDE 10% IN DPG   | 5   |
| CITRALVA                    | 2   |
| HEDIONE                     | 1   |
| TRIPAL                      | 1   |
| ALLYL AMYL GLYCOLATE        | 5   |
| FLOROTONE                   | 40  |
| GARDINOLENE                 | 50  |
| MUSK XYLOL                  | 30  |
| METHYL IONONE               | 3   |
| ALDEHYDE C 12 MNA           | 5   |
| GALAXOLIDE                  | 20  |
| ALDEHYDE C 10               | 2   |
| FIXOLIDE                    | 30  |
| SANDELA                     | 50  |
| DIPROPYLENE GLYCOL          | qs  |

## CAMAY SOAP COMPOUND

|                        |     |
|------------------------|-----|
| BENZYL ACETATE         | 200 |
| PHENYL ETHYL ACETATE   | 50  |
| STYRALLYL ACETATE      | 40  |
| STYRALLYL ALCOHOL      | 10  |
| TERPENYL ACETATE       | 60  |
| PHENYL ETHYL ALCOHOL   | 200 |
| AMYL CINNAMIC ALDEHYDE | 100 |
| HELIOTROPIN            | 10  |
| ANISIC ALDEHYDE        | 30  |
| HELIONAL               | 10  |
| ALDEHYDE C 8           | 10  |
| ALDEHYDE C 9           | 5   |
| ALDEHYDE C 10          | 10  |

|                                     |     |
|-------------------------------------|-----|
| ALDEHYDE C 11                       | 20  |
| ALDEHYDE C 12 LAURIC                | 5   |
| ALDEHYDE C 12 MNA                   | 5   |
| MYRAC ALDEHYDE                      | 5   |
| PHENYL ACETALDEHYDE 50%             | 5   |
| PHENYL ACETALDEHYDE DIMETHYL ACETAL | 5   |
| CITRONELLOL                         | 90  |
| COUMARIN                            | 60  |
| METHYL COUMARIN                     | 10  |
| DIMETHYL HYDROQUINONE               | 5   |
| ISO E SUPER                         | 10  |
| GERANEOL                            | 50  |
| INDOLE                              | 10  |
| LINALOL                             | 60  |
| METHYL IONONE                       | 60  |
| MUSK KETONE                         | 60  |
| BENZYL SALICYLATE                   | 120 |
| ROSE OXIDE 10% IN DPG               | 100 |
| ALDEHYDE C 14                       | 5   |
| ALDEHYDE C 18                       | 3   |
| TERPENEOL                           | 150 |
| IONONE ALPHA                        | 20  |
| METHYL HEPTINE CARBONATE 1% IN DPG  | 70  |
| LILLIAL                             | 30  |
| CYCLAMEN ALDEHYDE                   | 10  |
| LYRAL                               | 20  |
| ETHYL VANILLIN                      | 1   |
| PROPENYL GUAETHOL                   | 1   |
| PTBCHA                              | 150 |
| VERTOFIX                            | 100 |
| ARMOISE OIL                         | 10  |
| MAHAGONATE                          | 20  |
| TIMBEROL                            | 20  |
| SANDELA                             | 20  |
| GERANIUM OIL                        | 60  |
| CLOVE OIL                           | 10  |
| LAVENDIN OIL                        | 60  |
| PATCHOULI OIL                       | 30  |
| YLANG OIL                           | 50  |
| VETIVER OIL                         | 10  |
| FIXOLIDE                            | 80  |
| CIVET                               | 5   |
| CITRONELLYL ACETATE                 | 15  |
| BOIS AMBREINE FORTE                 | 1   |
| ALDEHYDE C 13-13                    | 5   |
| ALDEHYDE C 11-11                    | 5   |
| JASMACYCLATE                        | 5   |
| DAMASCONE ALPHA 10% IN DPG          | 30  |
| DIMETHYL OCTANOL                    | 20  |
| EVERNYL 1% IN DPG                   | 200 |
| VANILLIN                            | 5   |

|                         |    |
|-------------------------|----|
| CETONE V                | 5  |
| DIHYDRO MYRCENOL        | 50 |
| UNDECAVERTOL 10% IN DPG | 5  |
| DYHDRO ROSE OXIDE       | 5  |
| DIPROPYLENE GLYCOL      | qs |

## SANDALWOOD SOAP COMPOUND TYPE 2

|                       |      |
|-----------------------|------|
| SANDELA               | 1000 |
| SANDALORF             | 100  |
| ALDEHYDE C 11         | 30   |
| SANDALMYSORE CORE     | 20   |
| BACDANOL              | 100  |
| FIXOLIDE              | 100  |
| GERANIUM OIL          | 100  |
| ROSE CRYSTALS         | 20   |
| DIPHENYL OXIDE        | 120  |
| VERTOFIX              | 100  |
| VANILLIN              | 5    |
| HELIOTROPIN           | 15   |
| CETONE V              | 20   |
| BACCARTOL             | 100  |
| ADOXAL                | 20   |
| LAVANDIN OIL          | 60   |
| TERPENYL ACETATE      | 30   |
| CIVET 10% IN DPG      | 15   |
| RESINOID OAKMOSS      | 70   |
| PATCHOULY OIL         | 150  |
| RESINOID BENZOIN      | 30   |
| MUSK AMBRETTE         | 50   |
| COUMARIN              | 20   |
| METHYL COUMARIN       | 5    |
| DIMETHYL HYDROQUINONE | 5    |
| RESINOID LABDANUM     | 10   |
| MUSK KETONE           | 100  |
| METHYL IONONE         | 100  |
| BENZYL SALICYLATE     | 200  |
| ALDEHYDE C 14         | 5    |
| ALDEHYDE C 18         | 5    |
| VETIVER OIL           | 120  |
| CAMPHOR               | 50   |
| EVERNYL 1% IN DPG     | 100  |
| EUGENOL               | 10   |
| LILLIAL               | 50   |
| DIPROPYLENE GLYCOL    | qs   |



## PALMOLIVE SOAP COMPOUND TYPE 3

|                             |     |
|-----------------------------|-----|
| BENZYL ACETATE              | 110 |
| DMBCA                       | 15  |
| STYRALLYL ACETATE           | 60  |
| CINNAMIC ALCOHOL            | 15  |
| CINNAMYL ACETATE            | 5   |
| PHENYL ETHYL ALCOHOL        | 100 |
| ANISIC ALDEHYDE             | 15  |
| HELIOTROPIN                 | 10  |
| HELIONAL                    | 5   |
| ANISYL ACETATE              | 5   |
| ALDEHYDE C 8 10% IN DPG     | 1   |
| ALDEHYDE C 9                | 3   |
| ALDEHYDE C 10               | 3   |
| ALDEHYDE C 12 LAURIC        | 5   |
| HEXYL CINNAMIC ALDEHYDE     | 55  |
| ALDEHYDE C 11               | 6   |
| ALDEHYDE C 11-11            | 6   |
| METHYL ANTHRANILATE         | 3   |
| BACDANOL                    | 10  |
| METHYL BENZOATE             | 5   |
| CITRONELLOL                 | 20  |
| COUMARIN                    | 30  |
| METHYL COUMARIN             | 5   |
| DIMETHYL HYDROQUINONE       | 5   |
| EUGENOL                     | 10  |
| GALAXOLIDE                  | 80  |
| GERANEOL                    | 20  |
| BACCARTOL                   | 30  |
| CITRALVA                    | 5   |
| INDOLE                      | 1   |
| LILLIAL                     | 60  |
| LINALOL                     | 20  |
| DIHYDROMYRCENOL             | 40  |
| METHYL IONONE               | 90  |
| MUSK KETONE                 | 90  |
| BENZYL SALICYLATE           | 200 |
| ROSE OXIDE 10% IN DPG       | 10  |
| ALDEHYDE C 14               | 15  |
| PHENYL ETHYL PHENYL ACETATE | 15  |
| BENZYL PROPIONATE           | 5   |
| ROSE CRYSTALS               | 30  |
| AMYL SALICYLATE             | 30  |
| ISO BUTYL SALICYLATE        | 30  |
| SANDALORE                   | 40  |
| TERPENEOL                   | 25  |
| VERTOFIX                    | 40  |
| TONQUITONE                  | 15  |
| ETHYL VANILLIN              | 3   |
| VANILLIN                    | 5   |

|                      |     |
|----------------------|-----|
| COUMARIN             | 10  |
| PTBCHA               | 100 |
| VETYVENAL            | 70  |
| CUMIN OIL 10% IN DPG | 5   |
| ORANGE OIL           | 30  |
| PATCHOULY OIL        | 10  |
| YLANG OIL            | 50  |
| DIPROPYLENE GLYCOL   | qs  |

PALMOLIVE SOAP COMPOUND TYPE 4

|                                 |      |
|---------------------------------|------|
| SANDELA                         | 60   |
| SANDALORE                       | 60   |
| ALDEHYDE C 11                   | 40   |
| SANDAL MYSORE CORE              | 10   |
| BACDANOL                        | 100  |
| GALAXOLIDE                      | 200  |
| GERANIUM OIL                    | 100  |
| ROSE CRYSTALS                   | 40   |
| DIPHENYL OXIDE                  | 200  |
| CETONE V                        | 30   |
| BACCARTOL                       | 100  |
| ADOXAL                          | 30   |
| JASMINE SOAP- FRESH-FLORAL TYPE | 200  |
| MUSK AMBRETTE                   | 50   |
| MUSK KETONE                     | 60   |
| METHYL IONONE                   | 60   |
| BENZYL SALICYLATE               | 120  |
| YLANG OIL                       | 100  |
| CAMPHOR                         | 50   |
| DIHYDRO ROSE OXIDE              | 20   |
| ROSE OXIDE                      | 10   |
| DAMASCONE ALPHA 10% IN DPG      | 30   |
| LILLIAL                         | 150  |
| PTBCHA                          | 150  |
| IONONE ALPHA                    | 40   |
| EUGENOL                         | 5    |
| LAVENDIN OIL                    | 20   |
| ALDEHYDE C 9                    | 10   |
| ALDEHYDE C 12 LAURIC            | 15   |
| ALDEHYDE C 12 MNA               | 25   |
| ALLYL AMYL GLYCOLATE            | 30   |
| DIPROPYLENE GLYCOL              | 2000 |

## LEMON--EAU DE COLOGNE TYPE

|                      |      |
|----------------------|------|
| ALLYL AMYL GLYCOLATE | 110  |
| HEDIONE              | 5    |
| ALDEHYDE C 10        | 10   |
| TRIPAL               | 10   |
| MYRAC ALDEHYDE       | 15   |
| LYRAL                | 100  |
| LILLIAL              | 100  |
| NEROL                | 30   |
| DIHYDROMYRCENOL      | 120  |
| ALDEHYDE C 13-13     | 15   |
| ALDEHYDE C 11-11     | 15   |
| MUSK XYLOL           | 30   |
| MUSK KETONE          | 30   |
| MUSK AMBRETTE        | 30   |
| METHYL IONONE        | 30   |
| BENZYL SALICYLATE    | 60   |
| DIPHENYL OXIDE       | 100  |
| ALDEHYDE C 14        | 15   |
| ALDEHYDE C 18        | 5    |
| BENZYL ACETATE       | 75   |
| CETONE V             | 10   |
| BACCARTOL            | 30   |
| ORANGE OIL           | 100  |
| CITRALVA             | 100  |
| ALDEHYDE C 16        | 5    |
| ALDEHYDE C 20        | 5    |
| ALDEHYDE C 12 LAURIC | 20   |
| ALDEHYDE C 12 MNA    | 20   |
| DIPROPYLENE GLYCOL   | 1200 |

## LEMON -- FLORAL TYPE

|                          |     |
|--------------------------|-----|
| HEDIONE                  | 10  |
| ALDEHYDE C 10            | 10  |
| TRIPAL                   | 10  |
| MYRAC ALDEHYDE           | 15  |
| LYRAL                    | 100 |
| LILLIAL                  | 200 |
| NEROL                    | 60  |
| DIHYDROMYRCENOL          | 150 |
| ALDEHYDE C 13-13         | 30  |
| PHENYL ACET ALDEHYDE 50% | 20  |
| MUSK AMBRETTE            | 100 |
| BENZYL SALICYLATE        | 100 |

|                      |      |
|----------------------|------|
| IONONE ALPHA         | 20   |
| DIPHENYL OXIDE       | 100  |
| ALDEHYDE C 14        | 20   |
| BENZYL ACETATE       | 100  |
| CETONE V             | 10   |
| BENZYL PROPIONATE    | 20   |
| BACCARTOL            | 30   |
| ALDEHYDE C 12 MNA    | 30   |
| ALDEHYDE C 12 LAURIC | 20   |
| CITRALVA             | 100  |
| DMBCA                | 30   |
| YLANG OIL            | 30   |
| DIPROPYLENE GLYCOL   | 1200 |

**JASMINE SOAP COMPOUND-FRESH FLORAL TYPE**

|                                     |      |
|-------------------------------------|------|
| BENZYL ACETATE                      | 1000 |
| BENZYL PROPIONATE                   | 100  |
| BENZYL PHENYL ACETATE               | 50   |
| AMYL CINNAMIC ALDEHYDE              | 200  |
| ALLYL AMYL GLYCOLATE                | 40   |
| AURANTINE EXTRA                     | 30   |
| ALDEHYDE C 14                       | 20   |
| METHYL HEPTINE CARBONATE 10% IN DPG | 10   |
| BENZYL SALICYLATE                   | 100  |
| INDOLE 10 % IN DPG                  | 20   |
| HEDIONE                             | 20   |
| HEXYL CINNAMIC ALDEHYDE             | 200  |
| METHYL IONONE (ISORALDEINE)         | 50   |
| LILLIAL                             | 30   |
| LINALOL                             | 10   |
| PARA CRESYL PHENYL ACETATE          | 30   |
| ALDEHYDE C 18                       | 10   |
| CIVET                               | 30   |
| YLANG OIL                           | 40   |
| DIMETHYL BENZYL CARBINYL ACETATE    | 40   |
| MUSK XYLOL                          | 50   |
| MUSK AMBRETTE                       | 70   |
| METHYL IONONE                       | 10   |
| DIHYDRO MYRCENOL                    | 60   |
| JASMACYCLATE                        | 20   |
| ALLYL CYCLOHEXYL PROPIONATE         | 20   |
| BACCARTOL                           | 50   |
| LINALYL ACETATE                     | 40   |
| DPG                                 | qs   |

## JASMINE SOAP COMPOUND - SWEET TYPE

|                                  |      |
|----------------------------------|------|
| BENZYL ACETATE                   | 1000 |
| BENZYL PROPIONATE                | 100  |
| BENZYL PHENYL ACETATE            | 50   |
| AMYL CINNAMIC ALDEHYDE           | 200  |
| ALLYL AMYL GLYCOLATE             | 20   |
| AURANTINE EXTRA                  | 30   |
| ALDEHYDE C 14                    | 20   |
| ALDEHYDE C 16                    | 20   |
| BENZYL SALICYLATE                | 100  |
| BACDANOL                         | 30   |
| HEDIONE                          | 20   |
| HEXYL CINNAMIC ALDEHYDE          | 200  |
| ISORALDEINE                      | 50   |
| LILLIAL                          | 30   |
| PHENYL ACETIC ALDEHYDE           | 10   |
| PARA CRESYL PHENYL ACETATE       | 30   |
| ALDEHYDE C 18                    | 10   |
| CIVET                            | 20   |
| YLANG OIL                        | 20   |
| DIMETHYL BENZYL CARBINYL ACETATE | 20   |
| MUSK XYLOL                       | 60   |
| MUSK AMBRETTE                    | 60   |
| DIHYDRO MYRCENOL                 | 50   |
| JSMACYCLATE                      | 20   |
| ALLYL CYCLO HEXYL PROPIONATE     | 20   |
| RASPBERRY KETONE                 | 10   |
| BACCARTOL                        | 30   |
| LINALYL ACETATE                  | 30   |
| DPG                              | qs   |

TECHNICAL EVALUATION OF PILOT PLANT EQUIPMENT

DP/VIE/S6/033 - PROCESSING OF AROMA CHEMICALS AND FRAGRANCE MATERIALS

WORK DONE BY MR SUDHIR JAIN, UNIDO EXPERT AS PER LETTER DATED 23.11.93 OF DR. T DE SILVA, SPECIAL TECHNICAL ADVISER, ADDRESSED TO DR CUONG, VICE NPD

1. EVALUATE THE FUNCTIONING OF ALL ITEMS OF EQUIPMENT SUPPLIED
2. DETECT ANY DEFECTS IN CONSTRUCTION AND FAULTS IN OPERATIONS OF EQUIPMENT
3. EVALUATE THE KNOW HOW TRANSFER WITH RESPECT TO WHAT HAS BEEN INCLUDED IN THE PURCHASE ORDER
4. ADVISE ON SAFETY REQUIREMENTS

The evaluation has been done on a unit wise basis and along with the findings the conclusions and recommendations are also mentioned for the sake of easy and quick reference.

FINDINGS

1. FRACTIONATING COLUMN
  - a. The limpet coil of the reboiler is connected to steam and the inside coil is connected to thermic fluid.
  - b. The thermic fluid(hot oil) heating system is inadequate in terms of both design and capacity. This results in insufficient heat being supplied to the reboiler resulting in incomplete distillations since the temperatures required to boil off the higher boiling fractions are not attained.
  - c. The thermic fluid(hot oil) heating system is electrically heated, with electric heating being limited to 12 kw of which only 9 kw is actually connected. This wattage is insufficient to attain the temperatures required in the fractionating column.
  - d. At the top of the column, provision has been made for control of reflux ratio through a solenoid valve/electromagnetic timer, which is correct. At the same time contingency provision has also been made for manual control of reflux ratio but no provision has been made for the visual control of the actual rate of reflux thus rendering the additional facility difficult to operate. Visual control of reflux ratios is essential as in some complex mixtures such as essential oils where many components are present, mechanical control of reflux ratios is not sufficient for obtaining optimum results.

- e. The 2 vacuum pumps which have been provided are of too small a capacity for the size of the fractionating unit. Each pump is of 600 lpm capacity which under industrial conditions is the size recommended for 20l units whereas the fractionating column here is of 150 litres capacity. Since the pumps are of an inadequate capacity, insufficient vacuum is generated in the system leading to higher than normal temperatures of distillation which coupled with an inadequate heating system results in incomplete distillations.
- f. The vacuum pumps destined for the fractionation unit are also connected to other units for the purposes of charging and filtration which is wrong as the pumps being used for fractionation should not be used for any other purpose as it leads to unnecessary wear and tear of the pump and contamination of the oil leading a fall in the efficiency of the pump and consequently improper distillations.
- g. No service line of vacuum has been provided to carry out ancilliary functions such as charging, filtration and removal/distillation of moisture/low boilers/solvents.

#### HYDROGENATION VESSEL

- a. The stirring provided in the hydrogenation vessel is quite inadequate for a 3 phase reaction. In all reactions of this type, it is necessary to ensure that the stirring is vigourpus enough to create a vortex in the centre around the shaft of the stirrer.
- b. The hydrogenation vessel has been provided with a sparger to purge hydrogen into the system which is of the cross and circle type. This type of design is undesirable in a reactor where a 3 phase reaction is to carried out as it effectively creates a reactor within reactor in the sense that the dynamics of the motion of the reactants below and above the sparger become quite different with the sparger acting as a barrier to the free movement of the catalyst which must intermingle intimately with the reactants to enable the reaction to proceed ahead.
- c. There is no gauge provided on the vessel to check the pressure/vacuum during the reaction. Although hydrogenation is a pressure reaction, the required pressure being 2-3 kg/cm<sup>2</sup>, it is necessary to have a facility to read vacuum also. This is so because the progress and continuity of the reaction is judged by pressurising the vessel with hydrogen after the catalyst has been duly added and then shutting off the hydrogen supply. The progress of the reaction/consumption of hydrogen is indicated by a gradual fall in the pressure in the vessel and eventual

creation of vacuum in the vessel as all the hydrogen is consumed, thereby indicating that the reaction is going on.

- d. The limpet coil of the vessel should be connected to hot oil as limpet coils are unsuitable for use for heating by steam unless and until dry/super heated steam is available. As per standard practice in design of chemical equipment, vessels to be heated with hot oil are provided with limpet coils and vessels to be heated with steam are jacketed.
- e. The filtration unit of the hydrogenator is connected through the storage vessel to the main vacuum pump thus rendering it unsuitable for use for fractionation.
- f. The hydrogenator vessel should be provided with a hydrogen pressure gauge and safety release valve.
- g. The outlet of the hydrogen pressure release valve should be taken to ground level and led into a large bucket or tank so as to contain the splashing that will occur in case of emergency release of pressure.

#### GLASS LINED REACTOR

- a. The shaft of the glass lined vessel is worn out at the top where it fits into the housing assembly. Although there is no play in the circular movement of the shaft, yet the worn out condition indicates that it is an old unit.
- b. The enamel/glass lining at the bottom of the stirrer shaft and at the bottom surface of all the three blades is worn out with the steel being clearly exposed. The lower end of the thermowell is also similarly worn out. The inner surface of the valve fitted to the bottom outlet is also similarly worn out.
- c. The gasket fitted between the top lid and the main body of the vessel is exposed thereby rendering it susceptible to attack by the reactants.
- d. The bottom outlet of the glass lined reactor and the outlet of the two way bottom valve of the same vessel is too narrow for the slurry to flow out. In fact, in the two way valve the bore has been throttled thus preventing the slurry from flowing under gravity.



CENTRIFUGE

- a. For the filtration of the Terpin Hydrate after the reaction is completed, a rubber lined centrifuge should have been provided because the material to be filtered is a highly acidic slurry. In fact, a plain stainless steel centrifuge has been provided which cannot be used for this purpose.

ACID WASHING TANK

- a. The washing tank provided for terpin hydrate is of an inadequate design and instead of a separate filtration device, a false bottom has been provided inside the wash tank, through which the liquid of the slurry is supposed to pass. This vessel should have been provided with lead lining or glass lining. Instead only a fibreglass lining has been provided which is unacceptable from the technical point of view. Also no method of compressing the slurry has been provided leading to incomplete filtration and therefore a poor quality of the final product.
- b. The fibreglass lining of the wash tank is brittle and likely to crack under constant use due to the dissolution action of organic chemicals on the resins used to bind the composite.
- c. The positioning of the wash tank is incorrect as the slurry is supposed to flow under gravity from the reactor to the wash tank and the wash tank has been placed some distance away from the hydration unit making the transfer of the slurry difficult to achieve.

DEHYDRATOR

The dehydration of terpin hydrate is based upon the reaction between a fixed quantity of terpin hydrate and a dilute organic acid. This equation is not required to be disturbed during the reaction and that is why the water from the distillate is fed back into the reactor to maintain the proportion. The distillate contains terpeneol also, as the top organic layer and the same is collected as the product till the end of the reaction. The exact quantity of the water produced as a result of the dehydration is removed at the end of the reaction so as to maintain the concentration of acid in the system.

In the system supplied for carrying out the dehydration reaction, there are the following faults:-

- i) Provision has been made for injecting steam into the vessel through a sparger, the idea being to co-distill the terpeneol with the steam. This concept however is wrong because the vapours pass through an unheated column which effectively acts as a condenser and prevents the total steam injected from going out of the reactor, resulting in flooding of the reactor.
- ii) The limpet coil of the reactor has been connected to steam whereas it should have been connected to a hot oil system because of the heat required to distill water which is necessary requisite of this stage of the reaction.

CONCLUSIONS AND RECOMMENDATIONS

1.

FRACTIONATING COLUMN

a. The inner coil of the reboiler should be connected to hot oil system, thermic fluid heater to provide proper heat input to the system. The inside coil of the reboiler for the present may be connected to the hot oil system to provide additional transfer of heat. Under normal circumstances, the inside coil is to be connected to steam to distill off low boilers and solvents etc which are normally encountered in the beginning of any fractionation or distillation

b. A proper thermic fluid heating system of the following type should be provided:-  
Fully automatic, coil type, oil fired, forced draught, packaged type thermic fluid heater of capacity 100,000 k. cal./hr. It should have the following accessories:

- Thermic fluid circulating pump
- Deareator cum expansion tank
- Thermic fluid charging pump
- Dust proof prewired control panel housing with necessary starter, fuse, contactor, relays etc.
- All requisite piping, mountings, fittings, instrument controls and safeties. It should be complete with all inter-connecting pipelines, valve fittings. instrumentation including the lagging of the main body.

c. Till such time that a proper thermic fluid heater can be arranged for, some modifications may be carried out in the existing electrically heated hot oil system to make it operational on a temporary basis. These are:-  
The number of immersion heaters fitted to the hot oil tank may be increased so as to increase the connected load to a minimum of 24 kw. This is of course space being physically available to carry out the modification. The total electrical heating of 24 kw will provide just sufficient heat for the fractionation unit to distill oils such as Citronella, Citriodora, Litsea cubeba and Turpentine.  
The entire pipeline of the thermic fluid as well as the main body of the heater should be properly insulated to minimise heat losses.

d. A glass visioer should be provided at the reflux back point to facilitate the visual observation/ control of actual reflux rates.  
This will enable the operator of the fractionating column to exercise precise control over the distillation.

- e. The existing vacuum pumps which are of 600 lpm/2HP capacity should be replaced with pumps of a capacity of 2000 lpm/5HP to provide sufficient suction capacity so as to generate the desired vacuum levels in the fractionating column. It must be borne in mind that under practical operating conditions, the efficiency of the vacuum pumps falls very quickly with usage and as such an overcapacity has to be created in suction to facilitate normal working.
- f. The vacuum pumps destined for fractionation should be disconnected from other units and should not be used for the purposes of charging and filtration or any other ancilliary function.
- g. A Water-ring Vacuum Pump of the following specification should be installed to carry out all ancilliary functions such as charging, filtration, removal/distillation of moisture/low boilers /solvents:-  
One no. water ring vacuum pump with flame proof motor of the following capacity:-
- Maximum suction (with open suction): 220 cu. mts (5500 lpm)
  - Maximum vacuum (with close suction): 710 mm of Hg
- the pump should be fitted with all standard accessories.
- h. Till such time that the larger capacity vacuum pumps can be procured, the following measures should be taken to optimise the efficiency of the existing pumps:-
- the oil of the vacuum pumps should be checked regularly and should be changed as soon as some odour of aromatics is detected in the oil.
  - the vacuum pumps connected to the fractionation unit should be disconnected from all other units to which they are connected to carry out ancilliary functions and should be used for fractionation alone.
  - the two pumps, each of 600 lpm capacity should be run in parallel so as to effectively double the capacity of suction.
  - alongwith this, the electrical heating of the hot oil system should be increased from 9 kw to 24 kw.
- i. The pressure/vacuum regulators fitted to the vacuum pumps should be disconnected as they are not operational and unnecessary. The control of vacuum should be exercised by regulating the air leak.  
The aforementioned changes if incorporated should result in the fractionation unit operating satisfactorily.

## 2. HYDROGENATION VESSEL

- a. The stirring provided in the hydrogenation vessel should be modified in the following manner:-
  - the rpm should be raised to 200-250 by changing the gears in the gearbox or the gearbox itself.
  - the impellor of the stirrer is required to be modified by increasing the length of the blades from the present approx. 50mm to 125/150mm.Both the aforementioned changes should result in the stirring taking place in such a manner that a vortex is created in the centre of the vessel around the stirrer shaft. Some minor changes in the rpm and blade dimensions may be necessary as the exact dimensions cannot be arbitrarily decided and must be finalised under actual working conditions.
- b. The sparger design is required to be modified such that the circle and the cross of the sparger are removed, leaving only a thin tube leading to the very bottom of the vessel, just below the centre of the stirring shaft.
- c. A guage should be provided to check the pressure/vacuum during the reaction.
- d. The limpet coil of the vessel should be connected the packaged type thermic fluid heater when it is obtained.
- e. The filtration unit of the hydrogenator should be connected through the storage vessel to the ancilliary water ring vacuum when it is obtained.
- f. The hydrogenator vessel should be provided with a hydrogen pressure guage and safety release valve.
- g. The outlet of the hydrogen pressure release valve should be taken to ground level and led into a large bucket or tank so as to contain the splashing that will occur in case of emergency release of pressure.

## GLASS LINED REACTOR

- a. Since the condition of the reactor indicates that it has been used prior to supply, ideally the reactor should be replaced. At the very least the following parts of the reactor which are clearly damaged should be replaced:-
  - the stirrer including the shaft and the blades
  - the thermowell
  - the bottom outlet valve

- b. Till such time that the replacements can be effected, the reactor should be used with epoxy being applied over the worn out and damaged parts. Epoxy resins of various grades are now available and an acid resistant grade should be chosen.
- c. The gasket fitted between the top lid and the main body of the vessel should be refitted in a proper manner such that it is not exposed. If the construction of the vessel prevents this from happening then the vessel should be replaced.
- d. The outlet of the two way bottom valve should be at least as wide as the bottom outlet of the vessel itself so as not to throttle the path through which the slurry will flow out. This point should be borne in mind while replacing this valve.
- e. Till such time that these replacements are effected, the slurry should be taken out in a plastic barrel whose top has been cut away by dumping the entire slurry into it after removing the bottom valve.

#### CENTRIFUGE

- a. A new rubber lined centrifuge should be procured. As per the purchase order a rubber lined centrifuge was supposed to have been supplied. However in actual fact the centrifuge that has been supplied is a plain stainless steel centrifuge and therefore totally unsuitable for filtration of a slurry which contains dilute inorganic acids. As such in accordance with the purchase order, the existing centrifuge should be replaced and a new rubber lined centrifuge supplied in its place.
- b. Till such time that this replacement is effected, a gravity bag filter made by suspending a bag made of polypropylene filter cloth in a plastic barrel whose top has been cut away should be used with the residual liquid being removed by applying some compression with a wooden paddle.

#### ACID WASHING TANK

- a. The false bottom provided inside the wash tank is to be removed and under all circumstances the filtration of the slurry containing terpin hydrate is to be conducted outside this vessel either in a centrifuge or in a gravity bag filter as the case may be.
- b. The existing lining of fibre glass is to be removed and replaced with lead lining so that materials acidic with inorganic acids may be washed therein.

- c. Once lined with lead this tank should be used for washing terpin hydrate neutral.
- d. The filterations required after each wash are again to be conducted either in the rubber lined centrifuge or the gravity bag filter.
- e. The wash tank should be repositioned as close to the glass lined reactor as possible so as to minimise the distance required to be travelled by the slurry. Ideally it should be placed just beside the materials charging pump. The charging of the acid washing vessel should be done through the wide man hole so that in case the slurry becomes thick it can be charged manually into the acid washing tank.
- f. Till such time that the vessel is lined with lead, it may be on a temporary basis be lined with stainless steel and used for washes provided the inorganic acids have been filtered to the maximum extent prior to washing.

#### DEHYDRATOR

- a. The sparger installed for the injection of steam into this unit is to be removed, alongwith the false bottom fitted over the sparger. The terpeneol must co-distill with the steam produced from the water already present in the reactants and an equivalent quantity of condensed water must be returned to the dehydrator through the oil/water separator.
- b. Since the latent heat of water is very high, a lot of energy is required to distill it. In this case, the limpet coil of the dehydrator has been connected to steam and since a limpet coil has a restricted volume, the amount of heat transfer that takes place is not sufficient to distill the water. Therefore, in order to make the reaction go forward, a higher input of heat is necessary which can be achieved either by connecting the limpet coil of the dehydrator to a hot oil system operating at a temperature much higher than the boiling point of water or by jacketing the vessel and connecting it to dry/ super heated steam.

DEVIATIONS FROM THE PURCHASE ORDER IN THE EQUIPMENT1. SUB-UNIT A

In this unit there are no faults in fabrication. However from the operational point of view there are some mistakes that have been made in the steam/hot oil/vacuum connections and the same have been duly pointed out in the findings/recommendations.

2. SUB-UNIT B

- a. Unless and until specifically understood between the buyer and the seller, all equipment that is supplied is supposed to be new/ unused equipment. In this case the condition of the glass lined reactor clearly indicates that it had been under use prior to supply. Furthermore the condition in which the equipment has been supplied is such that the equipment is unusable for the purpose for which it was supplied.
- b. From the operational point of view there are some mistakes that have been made in the steam/hot oil/vacuum connections and these have been duly pointed out in the findings/recommendations.

3. ACCESSORIES

- a. As per the the purchase order, 1 no. basket centrifuge, all contact parts rubber lined suitable for both acids and alkalies was to have been supplied. In actual fact a plain stainless steel unlined centrifuge has been supplied.



KNOW HOW TRANSFER

The procedures provided for the manufacture of aroma chemicals are sketchy and not detailed and precise so as to ensure strict compliance and repeatability.

The following are the itemwise comments:-

**a. CITRONELLOL.....EXPERIMENTAL WORK AND TECHNOLOGY DEMONSTRATION**

i) The first method suggested for the production of citronellol ie Aluminium Isopropoxide Reduction is never used in the industrial production of citronellol. Citronellol is always produced by the hydrogenation of citronellal in the presence of Raney Nickel Catalyst.

The suggested method ie aluminium isopropoxide reduction method is a valid method only for the production of Cinnamic Alcohol from Cinnamic Aldehyde and has only theoretical validity in the case of citronellol.

ii) The second method suggested for the production of citronellol ie Catalytic Hydrogenation of Citronellal with Raney Nickel is the correct method. However wholly inadequate details have been provided. In place of an exact operating procedure, only a brief outline of the method has been given which is not sufficient for somebody wishing to make citronellol by this method.

**b. TERPENEOL.....EXPERIMENTAL WORK AND TECHNOLOGY TRANSFER**

The method suggested for the production of Terpeneol ie via the Terpin Hydrate route is the correct method but wholly inadequate details have been provided. In place of an exact operating procedure only a brief outline of the method has been given which is not sufficient for somebody wishing to make terpeneol by this method.

LIST OF THE REPAIR WORK/ALTERATIONS ALREADY CARRIED OUT BY THE  
PROJECT AUTHORITIES IN THE PILOT PLANT

1. INSULATION

Additional glass wool was applied to all the units including the service lines to increase the thickness of the existing insulation. The resultant glass wool insulation was then cladded in glass wool to protect it and extend its efficiency and life.

As reported by the project authorities the units and the service lines as received from and erected by the suppliers had insufficient insulation and no cladding or covering thereon.

2. THERMOMETERS

The probes of the thermometers supplied were too short and did not reach the bottom of the vessels thereby giving inaccurate readings.

The short probes have been replaced by long probes and wherever necessary the probes have been introduced directly into the vessels without the thermowells to give more accurate readings.

New temperature controllers/thermostats have been connected along with all temperature indicators. These were not supplied with the original equipment.

3. VACUUM GAUGES

The vacuum gauges received from the suppliers of the equipment were not working properly as per the report of the project authorities and as such all the existing gauges have been changed with new Japanese gauges.

4. HEATING OF FRACTIONATION UNIT

The limpet coil of the reboiler of the fractionation unit was originally connected to steam which provided inadequate heating for distillation. This has now been disconnected from steam and connected to the hot oil system thereby increasing the efficiency of the system .

5. STIRRING OF THE HYDROGENATOR

The rpm of the unit as received from the suppliers was only 50. This has been increased to 150 by changing the gear ratios in the gear box.

#### 6. ACID WASING VESSEL

The existing fibreglass lining has been replaced with stainless steel lining making it temporarily useable. All plastic connecting pipes from the glass lined reactor to the acid washing vessel have been replaced with stainless steel pipes.

#### 7. CONTROL PANEL

The starter buttons of all the units have now been put near the units for ease of operation instead of the original fitting on the main control panel.

UNITED NATIONS DEVELOPMENT PROGRAMME  
PROCESSING OF AROMA CHEMICALS AND FRAGRANCE MATERIALS  
DP/VIE/86/033  
HO CHI MINH CITY VIETNAM

Mr. Preben Hjortlund,  
Officer-in-charge,  
UNIDO Office,  
HANOI (Viet Nam)

11 DEC 1993

Dear Sir,

File ref: DP/VIE/86/033

Please refer to copy of our fax dated 6 Dec 1993 addressed to Dr T Desilva, Special Industrial Adviser, Chemical Industries Branch, UNIDO, Vienna regarding the technical evaluation of the pilot plant installed at the project site.

I have prepared an extract of the earlier detailed report, highlighting therein the amendments/alterations and repair works that can be carried out by the project authorities themselves so as to facilitate the immediate commissioning of the said pilot plant and make it operational on a temporary basis pending the execution of the other recommendations which concern the overall improvement in the working of the plant from a more long term point of view.

The cost involved in these repair works/alterations/amendments is USD 11,000/- including USD 1500/- for contingency expenses and the itemwise details have been provided in the enclosed report.

It is recommended that these funds be made available to the project authorities to enable them to commission the pilot plant and commence production trials.

Thanking you,

yours truly

SUDHIR JAIN, UNIDO EXPERT

RECOMMENDATIONS FOR ESSENTIAL CORRECTION OF FAULTS IN THE PILOT  
PLANT TO FACILITATE IMMEDIATE COMMISSIONING

The full set of findings, conclusions and recommendations regarding the pilot plant has been submitted as a separate report and this particular set of recommendations is concerned only with those repairs and alterations which can be carried out locally so as to facilitate immediate commissioning of the pilot plant by the project personnel themselves.

The estimated cost of each item/repair has been mentioned with the item and the same is subject to reconfirmation from local mechanics/workshops.

1. FRACTIONATING COLUMN

- a. The limpet coil of the reboiler should be connected to hot oil system to provide proper heat input to the system. The inside coil of the reboiler for the present may be connected to the hot oil system to provide additional transfer of heat. Under normal circumstances, the inside coil is to be connected to steam to distill off low boilers and solvents etc which are normally encountered in the beginning of any fractionation or distillation

NO COST OF MATERIALS IS INVOLVED AS ONLY CONNECTIONS ARE TO BE CHANGED. WORK CAN BE DONE BY TECHNICIANS AVAILABLE IN THE PROJECT.

- b. Till such time that a proper thermic fluid heater can be arranged for, some modifications may be carried out in the existing electrically heated hot oil system to make it operational on a temporary basis. These are:-

The number of immersion heaters fitted to the hot oil tank may be increased so as to increase the connected load to a minimum of 24 kw. This is subject to space being physically available to carry out the modification. The total electrical heating of 24 kw will provide just sufficient heat for the fractionation unit to distill oils such as Citronella, Citriodora, Litsea cubeba and Turpentine.

THE NUMBER OF NEW HEATERS REQUIRED IS FOUR AND EACH HEATER OF 3 KW EACH SHOULD COST APPROX. USD 125/-. THE ADDITIONAL ELECTRICAL WIRING AND SWITCHGEAR REQUIRED SHOULD COST APPROX. USD 1000/-. THEREFORE, TOTAL EXPENDITURE ON THIS ITEM IS USD 1500/-.

The entire pipeline of the hot oil system as well as the main body of the heater should be properly insulated to minimise heat losses.

THIS HAS ALREADY BEEN DONE AND AS SUCH NO EXPENSE IS REQUIRED TO BE INCURRED ON THIS ACCOUNT.

- c. A glass visioer should be provided at the reflux back point to facilitate the visual observation/ control of actual reflux rates.  
This will enable the operator of the fractionating column to exercise precise control over the distillation.

THIS IS A PRECISION JOB AND CAN ONLY BE CARRIED OUT IF GOOD GLASS BLOWING FACILITIES ARE AVAILABLE LOCALLY. THE TOTAL EXPENSE INCLUDING THE COST OF THE GLASS VISIOER AND THE GLASS TO METAL FLANGES SHOULD BE APPROX. USD 750/-.

- d. The vacuum pumps destined for fractionation should be disconnected from other units and should not be used for the purposes of charging and filtration or any other ancilliary function.

ONLY SOME CONNECTIONS ARE TO BE REMOVED IN THIS CASE AND NO EXPENSE IS NECESSARY. THE WORK CAN BE DONE BY THE TECHNICIANS AVAILABLE IN THE PROJECT.

- e. Till such time that the larger capacity vacuum pumps can be procured, the following measures should be taken to optimise the efficiency of the existing pumps:-  
-the oil of the vacuum pumps should be checked regularly and should be changed as soon as some odour of aromatics is detected in the oil.

THIS IS A RECURRING EXPENDITURE AND PART OF THE OVERHEAD EXPENSES OF RUNNING THE PLANT.

-the vacuum pumps connected to the fractionation unit should be disconnected from all other units to which they are connected to carry out ancilliary functions and should be used for fractionation alone.

ONLY SOME CONNECTIONS ARE TO BE REMOVED IN THIS CASE AND NO EXPENSE IS NECESSARY.

-the two pumps, each of 600 lpm capacity should be run in parallel so as to effectively double the capacity of suction.

AS THE CONNECTIONS ARE ALREADY THERE . NO EXPENSE IS NECESSARY.

- f. The pressure/vacuum regulators fitted to the vacuum pumps should

be disconnected as they are not operational and unnecessary. The control of vacuum should be exercised by regulating the air leak.

ONLY SOME CONNECTIONS ARE TO BE MADE AND NO EXPENSE IS NECESSARY ON THIS ACCOUNT. THE WORK CAN BE DONE BY THE TECHNICIANS AVAILABLE IN THE PROJECT.

The aforementioned changes if incorporated should result in the fractionation unit operating satisfactorily.

2. HYDROGENATION VESSEL

- a. The stirring provided in the hydrogenation vessel should be modified in the following manner:-  
-the rpm should be raised to 200-250 by changing the gears in the gearbox or the gearbox itself.

THE COST OF REPAIRING THE GEAR BOX SO AS TO CHANGE THE RPM OF THE STIRRING SYSTEM SHOULD BE APPROX. USD 1000/- INCLUSIVE OF LABOUR CHARGES.

-the impellor of the stirrer is required to be modified by increasing the length of the blades from the present approx. 50mm to 125/150mm.

THE COST OF THESE REPAIRS SHOULD BE APPROX. USD 250/-.

Both the aforementioned changes should result in the stirring taking place in such a manner that a vortex is created in the centre of the vessel around the stirrer shaft. Some minor changes in the rpm and blade dimensions may be necessary as the exact dimensions cannot be arbitrarily decided and must be finalised under actual working conditions.

- b. The sparger design is required to be modified such that the circle and the cross of the sparger are removed, leaving only a thin tube leading to the very bottom of the vessel, just below the centre of the stirring shaft.

SOME EXISTING FITTINGS IN THE GIVEN VESSEL ARE REQUIRED TO BE REMOVED ALONGWITH SOME CHANGE IN CONNECTIONS. ONLY A MINOR ADDITION IS TO BE MADE TO THE VESSEL.  
THE WORK CAN BE DONE BY THE TECHNICIANS AVAILABLE IN THE PROJECT AND THE TOTAL EXPENSE SHOULD BE APPROX. USD 500/-.

- c. A guage should be provided to check the pressure/vacuum during the reaction.

THE COST OF THOS ADDITION SHOULD BE USD 250/-.

- d. The limpet coil of the vessel should be connected to the hot oil system.

SOME CONNECTIONS ARE REQUIRED TO BE CHANGED AND ADDITIONAL PIPELINE WILL HAVE TO BE LAID FOR THE HOT OIL.  
THE TOTAL EXPENSE INCLUDING THE COST OF THE PIPELINE AND THE INSULATION SHOULD BE USD 500/-.

- e. The filtration unit of the hydrogenator should be disconnected from the main vacuum pumps used for fractionation/distillation.

ONLY SOME CONNECTIONS ARE REQUIRED TO BE CHANGED AND NO EXPENSE IS REQUIRED.

- f. The hydrogenator vessel should be provided with a hydrogen pressure guage and safety release valve.

THE COST OF THESE ADDITIONS SHOULD BE USD 500/-.

- g. The outlet of the hydrogen pressure release valve should be taken to ground level and led into a large bucket or tank so as to contain the splashing that will occur in case of emergency release of pressure.

THE COST OF THIS AMENDMENT WILL BE USD 250/-.

#### GLASS LINED REACTOR

- a. Till such time that the replacement of defective parts can be effected, the reactor should be used with epoxy being applied over the worn out and damaged parts. Epoxy resins of various grades are now available and an acid resistant grade should be chosen.

THE COST OF THIS REPAIR SHOULD BE USD 250/-. THIS WILL HOWEVER BE A RECURRING EXPENDITURE AND PROVISION SHOULD BE MADE FOR USD 1000/- IF THE PILOT PLANT IS TO BE USED REGULARLY.

- b. Till such time that the bottom outlet is replaced, the slurry should be taken out in a plastic barrel whose top has been cut away, by dumping the entire slurry into it after removing the bottom valve.



NO EXPENSE IS NECESSARY AS A USED BARREL CAN BE UTILISED. HOWEVER IF A PROPER PLASTIC TANK IS PROVIDED THEN THE COST WILL BE APPROX. USD 250/-.

#### CENTRIFUGE

- a. Till such time that this equipment is replaced, a gravity bag filter made by suspending a bag made of polypropylene filter cloth in a plastic barrel whose top has been cut away should be used with the residual liquid being removed by applying some compression with a wooden paddle.

THE COST OF RIGGING UP THIS FILTRATION DEVICE WILL BE USD 500/- AND THE WORK CAN BE DONE BY THE TECHNICIANS AVAILABLE IN THE PROJECT.

#### ACID WASHING TANK

- a. The false bottom provided inside the wash tank is to be removed and under all circumstances the filtration of the slurry containing terpin hydrate is to be conducted outside this vessel either in a centrifuge or in a gravity bag filter as the case may be.

NO EXPENSE IS NECESSARY AND THE WORK CAN BE DONE BY THE TECHNICIANS AVAILABLE IN THE PROJECT.

- b. The filtrations required after each wash are again to be conducted either in the rubber lined centrifuge or the gravity bag filter.
- c. The wash tank should be repositioned as close to the glass lined reactor as possible so as to minimise the distance required to be travelled by the slurry. Ideally it should be placed just beside the materials charging pump. The charging of the acid washing vessel should be done through the wide manhole so that in case the slurry becomes thick it can be charged manually into the acid washing tank.
- d. Till such time that the vessel is lined with lead, it may be on a temporary basis be lined with stainless steel and used for washes provided the inorganic acids have been filtered to the maximum extent prior to washing.

THE COST OF THIS AMENDMENT WHICH REQUIRES PRECISION WORKING AND GOOD QUALITY STAINLESS STEEL, WILL BE USD 1500/- AND THE WORK WILL HAVE TO BE DONE BY A PROFESSIONAL WORKSHOP.

DEHYDRATOR

- a. The sparger installed for the injection of steam into this unit is to be removed, alongwith the false bottom fitted over the sparger. The terpeneol must co-distill with the steam produced from the water already present in the reactants and an equivalent quantity of condensed water must be returned to the dehydrator through the oil/water separator.

ONLY SOME CONNECTIONS AND FITTINGS ARE TO BE REMOVED AND THE WORK CAN BE DONE BY THE TECHNICIANS AVAILABLE IN THE PROJECT. NO EXPENSE IS NECESSARY.

- b. Since the latent heat of water is very high, a lot of energy is required to distill it. In this case, the limpet coil of the dehydrator has been connected to steam and since a limpet coil has a restricted volume, the amount of heat transfer that takes place is not sufficient to distill the water. Therefore, in order to make the reaction go forward, a higher input of heat is necessary which can be achieved by connecting the limpet coil of the dehydrator to a hot oil system operating at a temperature much higher than the boiling point of water.

THE COST OF LAYING THE NEW SECTION OF THE HOT OIL SYSTEM AND INSULATING IT WILL BE USD 750/- AND THE WORK CAN BE DONE BY THE TECHNICIANS AVAILABLE IN THE PROJECT.

ASPER THE AFOREMENTIONED RECOMMENDATIONS, THE TOTAL COST OF THE REPAIRS AND ALTERATIONS ARE ESTIMATED TO BE USD 9500/- .THE RELEVANT DETAILS ON AN ITEMWISE BASIS ARE GIVEN BELOW:-

|                         |                         |
|-------------------------|-------------------------|
| a) FRACTIONATING COLUMN | USD 2250.00             |
| b) HYDROGENATION VESSEL | USD 3250.00             |
| c) GLASS LINED REACTOR  | USD 1250.00             |
| d) CENTRIFUGE           | USD 500.00              |
| e) ACID WASHING TANK    | USD 1500.00             |
| f) DEHYDRATOR           | USD 750.00              |
|                         | -----                   |
|                         | TOTAL USD 9500.00       |
| h) CONTINGENCY EXPENSES | USD 1500.00             |
|                         | -----                   |
|                         | GRAND TOTAL USD11000.00 |

IT IS RECOMMENDED THAT FUNDS BE MADE AVAILABLE TO THE PROJECT AUTHORITIES TO CARRY OUT THESE ESSENTIAL REPAIRS AND MAKE THE PILOT PLANT OPERATIONAL.

## ANNEXURE 7

30 Nov. 1993

## JOB DESCRIPTION

DP/VIE/S6/033

POST TITLE: AROMA CHEMICALS EXPERT

DURATION : 2 months

DATE REQUIRED : ASAP

DUTY STATION : HO CHI MINH CITY

PURPOSE OF PROJECT : Utilization of indigenous essential oils to develop suitable fragrance materials and formulation for local industry as well as export.

DUTIES : The expert will work in collaboration with the NPD and counterpart staff members to perform the following specific duties :

a/ Transfer technology and demonstrate the production of the following aroma chemicals on a pilot scale :-

Citronellol from Java citronella oil / Eucalyptus citriodora oil

Geraniol from Java citronella oil .

Hydroxy citronellal from Eucalyptus citriodora oil/ Java citronella oil.

Terpineol from turpentine oil.

Heliotropine from sassafras oil.

Methyl ionone from Litsea cubeba oil.

b/ The demonstration will include all processes including fractionation, reaction, hydrogenation, hydration, dehydration, oxidation, cyclisation, isomerization, etc. as may be necessary for each of the aroma chemicals mentioned above .

c/ The processes demonstrated are required to produce aroma chemicals of a commercially acceptable quality both in terms of purity as well as olfactory quality.

d/ The required raw materials will be provided by the expert to the project authorities prior to the commencement of the assignment.

e/ The repeatability of the processes will be demonstrated on the equipment provided by the project authorities.

f/ The counterpart staff will be trained in the methods of production so as to enable them to undertake the same independently.

QUOTATION AS PER JOB DESCRIPTION  
Submitted by Prof. TRAN KIM QUI  
Director of The RESEARCH CENTER FOR APPLIED CHEMISTRY

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Items / Aroma chemicals included  
in know-how package : HELIOTROPIN, CITRONELLOL,  
GERANIOL, TERPINEOL, METHYL  
IONONE, HYDROXYCITRONELLAL.

Raw material sources  
( indigenous essential oils) : HELIOTROPIN Ex Sassafras  
oil.  
CITRONELLOL Ex Citronella  
Java oil/ Eucalyptus  
citriodora oil.  
GERANIOL Ex Citronella Java  
oil.  
TERPINEOL Ex Turpentine oil  
METHYL IONONE Ex Litsea  
Cubeba oil  
HYDROXYCITRONELLAL Ex  
Citronella Java oil/  
Eucalyptus citriodora oil.

Scale of operations: Pilot plant scale. The  
know-how will be demonstrated on the pilot plant installed in the  
project premises if the same is functional. Otherwise it will be  
demonstrated on bench scale in the laboratory of the project. Each  
process will be repeated three times to demonstrate and establish  
the process and its yield.

Training of project personel:

The counterpart staff will be trained in the methods of  
production so as to enable them to undertake the production  
independently. This training aspect is part and parcel of the know-  
how transfer package.

Raw materials required: Citronella Java oil  
( Min. 30% citronellal content)  
Diethanolamine tech.

Sulfuric acid Min. 95% purity.  
Toluene  
Caustic soda.  
Sodium chloride  
Benzene.  
Potassium fluoride.  
Sassafras oil.

Aluminium oxide.  
 Sodium dichromate/ Chromic oxide.  
 Glacial acetic acid.  
 Sodium hydrogen phosphate  
 Raney nickel  
 Hydrogen gas.  
 Turpentine oil.  
 Non-ionic emulsifier.  
 Phthalic anhydride.  
 Sodium hydroxide.  
 Oxalic acid.  
 Litsea cubeba oil.  
 Benzyl chloride.  
 Triethylamine.  
 Methanol.  
 Potassium hydroxide.  
 Methyl ethyl ketone.  
 Phosphoric acid.(min.85%)  
 Sodium bicarbonate.  
 Sodium carbonate.

The quantity of materials required will be specified by the contractor providing the know-how package depending upon the scale of operations finalized by the project authorities. The contractor will organize the purchase/procurement of the raw materials if so requested by UNDP/UNIDO/Project authorities.

Performance guarantee: The aroma chemicals produced as the result of the transfer/purchase of the know-how package will conform to standard acceptable commercial qualities used in the international trade.

Know-how fees:

|                      |             |
|----------------------|-------------|
| Heliotropine:        | USD 3,000   |
| Citronellol :        | USD 3,000.  |
| and Geraniol         |             |
| Terpineol :          | USD 3,000.  |
| Methyl ionone:       | USD 3,000.  |
| Hydroxy citronellal: | USD 3,000.  |
|                      | -----       |
| Total :              | USD 15,000. |

Payment : 50% of the total amount after signing the contract and the balance 50% after successful transfer of know-how package demonstration of processes and training of personnel.

## ANNEXURE S

KNOW-HOW PACKAGE OFFERED BY PROF. TRAN KIM QUI

List of raw materials required for demonstration of commercial processes for CITRONELLOL/GERANIOL, HELIOTROPINE, TERPINEOL, METHYL IONONE and HYDROXY CITRONELLAL

1. RAW MATERIALS REQUIRED FOR CITRONELLOL and GERANEOL

| ITEMS  | QTY/BATCH | TOTAL QTY | VALUE    |
|--|-----------|-----------|----------|
| a. Citronella java oil<br>(min 85% alcohols) | 300 kilos | 900 kilos | \$ 5,400 |
| b. Raney Nickel Catalyst<br>(ready to use)   | 5 kilos   | 30 kilos  | \$ 750   |
| c. Caustic Soda                              | 10 kilos  | 60 kilos  | \$ 120   |
| d. Caustic Potash                            | 2 kilos   | 12 kilos  | \$ 25    |
| e. Acetone                                   | 5 kilos   | 30 kilos  | \$ 75    |
| f. Hydrogen gas                              | 50 kilos  | 300 kilos | \$ 200   |
| -----  |           |           |          |
| Total  |           |           | \$ 6,570 |

2. RAW MATERIALS REQUIRED FOR METHYL IONONE

| ITEMS                                   | QTY/BATCH | TOTAL QTY | VALUE    |
|---|-----------|-----------|----------|
| a. Methanol                             | 100 lit.  | 300 lit.  | \$ 300   |
| b. Benzyl chloride                      | 5 kilos   | 15 kilos  | \$ 50    |
| c. Triethylamine                        | 5 kilos   | 15 kilos  | \$ 150   |
| d. Caustic potash                       | 5 kilos   | 15 kilos  | \$ 30    |
| d. Methyl ethyl ketone                  | 120 kilos | 360 kilos | \$ 720   |
| e. Litsea cubeba oil                    | 100 kilos | 300 kilos | \$ 2,100 |
| f. Glacial acetic acid                  | 5 kilos   | 15 kilos  | \$ 75    |
| g. Common salt                          | 50 kilos  | 150 kilos | \$ 150   |
| h. Phosphoric acid<br>(min. 85% purity) | 250 kilos | 750 kilos | \$ 1,500 |
| g. Benzene                              | 60 kilos  | 180 kilos | \$ 200.  |
| h. Sodium bicarbonate                   | 50 kilos  | 150 kilos | \$ 150.  |
| -----                                   |           |           |          |
| Total :                                 |           |           | \$ 5,425 |

3. RAW MATERIALS REQUIRED FOR HYDROXY CITRONELLAL:

| ITEMS                        | QTY/BATCH | TOTAL QTY | VALUE     |
|------------------------------|-----------|-----------|-----------|
| a. Eucalyptus citriodora oil | 150 kilos | 450 kilos | \$ 2,700. |
| b. Diethanol amine           | 60 kilos  | 360 kilos | \$ 1,080. |
| c. Sulfuric acid             | 15 kilos  | 100 kilos | \$ 100.   |
| d. Common salt               | 60 kilos  | 360 kilos | \$ 300.   |
| e. Benzene                   | 60 kilos  | 360 kilos | \$ 400.   |
| f. Caustic soda              | 12 kilos  | 75 kilos  | \$ 150.   |
| g. Sodium bicarbonate        | 50 kilos  | 300 kilos | \$ 300.   |
| Total:                       |           |           | \$ 5,030  |

4. RAW MATERIALS REQUIRED FOR TERPENEOL:

| ITEMS               | QTY/BATCH | TOTAL QTY | VALUE   |
|---------------------|-----------|-----------|---------|
| a. Turpentine oil   | 100 kilos | 300 kilos | \$ 150. |
| b. Sulfuric acid    | 35 kilos  | 105 kilos | \$ 105. |
| c. Emulsifier       | 2 kilos   | 6 kilos   | \$ 120. |
| d. Oxalic acid      | 1 kilos   | 3 kilos   | \$ 10.  |
| e. Sodium carbonate | 5 kilos   | 15 kilos  | \$ 15.  |
| Total:              |           |           | \$ 400. |

5. RAW MATERIALS REQUIRED FOR HELIOTROPINE:

| ITEMS                       | QTY/BATCH | TOTAL QTY | VALUE     |
|-----------------------------|-----------|-----------|-----------|
| a. Potassium fluoride       | 40 kilos  | 120 kilos | \$ 240.   |
| b. Sassafras min. 92%       | 50 kilos  | 150 kilos | \$ 900.   |
| c. Aluminium oxide          | 200 kilos | 600 kilos | \$ 600.   |
| d. Chromic oxide            | 20 kilos  | 60 kilos  | \$ 150.   |
| e. Acetic acid glacial      | 20 kilos  | 60 kilos  | \$ 300.   |
| f. Sodium dihydro phosphate | 10 kilos  | 30 kilos  | \$ 60.    |
| Total :                     |           |           | \$ 2,250. |



UNITED NATIONS DEVELOPMENT PROGRAMME  
PROCESSING OF AROMA CHEMICALS AND FRAGRANCE MATERIALS  
DP/VIE/S6/033  
HO CHI MINH CITY VIETNAM

Mr. Preben Hjortlund.  
Officer-in-charge.  
UNIDO Office,  
HANOI (Viet Nam)

11 DEC 1993

Dear Sir.

File ref: DP/VIE/S6/033

Please refer to our fax dated 4 Dec 1993 regarding the list of raw materials required for the demonstration of the processes whose know how will be transferred to the project by Prof. TRAN KIM QUI.

Please note the following explanations:-

- a) the quantities of raw materials mentioned by us represent the quantities required for a minimum of three to a maximum of six batches depending on the steps involved in completing each process.
- b) in each case the quantities have been rounded off so as to eliminate odd figures, the rounding off being done on the higher side.
- c) in the case of Citronella java oil specifically, double the required quantity has been taken because when the pilot plant is commissioned, material will be consumed in standardising the operation of each unit which finally may or may not be recovered as an ACCEPTABLE finished product.
- d) it is envisaged that the actual demonstration of the processes should start after standardisation of the operation of the pilot plant.
- e) since training the staff of the project is a necessary requirement of the know how supply package, the consumption of raw materials is envisaged to be more than the requirement dictated by the process as certain steps may have to be repeated for the sake of training.

f) since in the international market, all the chemicals that are required by us are traded in minimum lots of 1000 kilos each, except for the essential oils which are all of indigenous origin, the prices that will be charged by the suppliers for the relatively small quantities required by the project will be substantially higher than the ruling international prices for which provision has been made by calculating prices on the higher side.

g) the raw material prices that have been mentioned are the estimated raw materials and actual quotations will have to be obtained from suppliers prior to placement of purchase orders.

We hope that this has clarified all the points.

Kindly arrange to do the needful in the matter and oblige.

thanking you,

yours truly

SUDHIR JAIN, UNIDO EXPERT

REPORT REGARDING KNOW HOW PACKAGE OFFERED BY PROF. TRAN KIM QUI  
OF UNIVERSITY OF HO CHI MINH CITY

The process technologies for the following 6 Aroma Chemicals have been discussed with Prof. Tran Kim Qui:-

1. Heliotropin
2. Citronellol
3. Geraneol
4. Terpeneol
5. Methyl Ionone
6. Hydroxycitronellal

It was found that the processes as explained by Prof Qui are the normal processes used in the industry.

As such it is felt that given the required inputs of raw materials and the proper equipment, he will be able to demonstrate the processes to the project authorities in a satisfactory manner.

The raw material requirements have also been discussed with Prof. Qui and the quantities of raw materials required by him have been conveyed to the project authorities. The raw material requirement specified is sufficient for 3 batches of each Aroma Chemical. Only those chemicals which are actually required in the processes have been included in the list.

Quotations for raw material supply have been obtained from two sources which are however subject to the normal scrutiny before placement of orders.

The processes will be demonstrated on the pilot plant if the same is an operational condition at the time of demonstration of the processes. If not, then the processes will be demonstrated on the bench scale.

It is necessary to mention here that Prof. Tran Kim Qui was the nominated expert of the project authorities and as such no other offer for purchase of similar knowhow package has been obtained from any other possible expert.

The know how package offered by Prof. Qui is subject to Performance Guarantee and Training of Personnel of the Project, as mentioned in the Terms of Reference.

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ANNEXURE 9

QUOTATION FOR SOAP MACHINERY

| ITEM   | PRICE/UNIT  | NO./UNITS | TOTAL                          |
|--|-------------|-----------|--------------------------------|
| 1. Soap Plodder of plodding capacity min. 500 kilos of soap powder mixed with fragrance per working shift of 8 hours; unit supplied will be complete with requisite gears, motors, hopper and 3 nos. extrusion dies to make bar soap in sizes 150g, 80g, and 20/25g. Main cylinder and screw will be made in stainless steel and the body of the main cylinder will be water cooled.   | US\$ 5000/- | 2 nos.    | US\$10,000/-                   |
| 2. Soap Stamping machine, mechanised; capacity min. 4000 bars of 150g each & 6000 bars of 80g each subject to manual feeding of single pieces of cut extruded soap bars and pedal operated mechanised movement of the stamping die to exert sufficient pressure to produce a clean well formed bar of soap; will be supplied with 3 nos. stamping dies as per the design of the customer in the three specified sizes ie 150g 80g and 25g. | US\$2500/-  | 2 nos.    | US\$5000/-                     |
| 3. Requisite accessories and spare parts   | US\$1000/-  | 1 set     | US\$1000/-                     |
|  |             |           | -----<br>US\$16,000/-<br>----- |

**TERMS OF BUSINESS**

1. THE PRICE QUOTED IS FOR DELIVERY EX-STOCK SUBJECT TO PRIOR SALE.
2. THE PRICE QUOTED IS VALID FOR 30 DAYS FROM THE DATE OF THE QUOTATION.
3. THE PRICE QUOTED IS C&F FACTORY SITE OF THE BUYER IN HCM CITY AND IS INCLUSIVE OF INSTALLATION AND COMMISSIONING CHARGES AND LOCAL TAXES IF ANY ARE PAYABLE.
4. THE FULL PAYMENT AS PER THE QUOTATION WILL BECOME DUE IMMEDIATELY AFTER SATISFACTORY INSTALLATION AND COMMISSIONING OF THE QUOTED ITEMS AS CERTIFIED BY THE BUYER.

## SAIGON COSMETIC FACTORY

1099 TRAN HUNG DAO STR. DIST 5 HOCHIMINH CITY VN  
 PHONE : 350181 - 356893 - TELEX : 811270 PBT LX VT - FAX : 84.8.298540

## QUOTATION FOR SOAP MACHINERY HOCHIMINH CITY DEC 9 ,L993

| <u>ITEM</u>   | <u>PRICE/UNIT</u> | <u>NO/UNITS</u> | <u>TOTAL</u> |
|---|-------------------|-----------------|--------------|
| 1. Soap Plodder of plodding capacity min 500 kilos of soap powder mixed with fragrance per working shift of 8 hours; unit supplied will be complete with requisite gears, motors, hopper and 3 nos. extrusion dies to make bar soap in sizes 150 g, 80g, and 20/25g. Main cylinder and screw will be made in stainless steel and the body of the main cylinder will be water cooled.  | USD 5,000/-       | 2 nos           | USD 10,000   |
| 2. Soap Stamping machine, mechanised, capacity min. 4000 bars of 150 g each, 6000 bars of 80g each subject to manual feeding of single pieces of cut extruded soap bars and pedal operated mechanised movement of the stamping die to exert sufficient pressure to produce a clean well formed bar of soap; will be supplied with 3 nos. Stamping dies as per the design of the customer in the three specified sizes ie 150g 80g and 25g | USD 2500/-        | 2 nos           | USD 5,000    |
| 3. Requisite accessories and spare parts  | USD 1000/-        | 1 set           | USD 1000/-   |

Total value : USD 16,000.00

TERMS OF BUSINESS :

1. The price quoted is for delivery ex-stock subject to prior sale
2. The price quoted is valid for 30 days from the date of the quotation
3. The price quoted is C,F factory site of the buyer in HCM city and is inclusive of installation and commissioning charges and local taxes if any are payable .
4. The full payment as per the quotation will become due immediately after satisfactory installation and commissioning of the quoted items as certified by the buyer .

SAIGON COSMETIC COMPANY  
 DIRECTRESS,



*Nguyen Thi Lien*

UNITED NATIONS DEVELOPMENT PROGRAMME  
PROCESSING OF AROMA CHEMICALS AND FRAGRANCE MATERIALS  
DP/VIE/S6/033  
HO CHI MINH CITY VIETNAM

Mr. Preben Hjortlund,  
Officer-in-charge,  
UNIDO Office,  
HANOI (Viet Nam)

11 DEC 1993

Dear Sir,

File ref: DP/VIE/S6/033

Please refer to our fax dated 9 DEC 1993 regarding the quotation of soap machinery from SAIGON COSMETIC COMPANY.

We submit herewith the following explanations:-

- a) as per the requirements of the project, a duplex plodder was required to fulfill the twin functions of pelletization and extrusion in the same machine in two successive steps.
- b) such duplex plodders are available in the international market and are used as standard equipment in the soap industry.
- c) because of the short time available to the project to source such plodders, it was decided to buy such a machine from a Vietnamese supplier.
- d) upon investigation in the local market, it was found that the local fabricators were only making what are called simplex plodders which means that they can fulfill only one function at a time in the machine and after fulfilling the first function of pelletization, the pelletized material has to be rerun through the machine a second time after making some alterations to it to get the extruded soap which is ready for stamping or pressing into the required shape.
- e) since from the industrial point of view this is not considered a feasible method of working, it was decided to buy two simplex plodders instead of one duplex plodder and connect them, effectively, in series so as to enable us to feed the pelletized material from the first machine manually into the second machine immediately thereafter to get the extruded soap ready for stamping.

f) so for all practical purposes, two simplex plodders combined together are equivalent to one duplex plodder and hence the request for two plodders.

g) to balance and effectively use the extrusion capacity of the aforementioned combination of plodders, it is necessary to use two stamping machines as the machines requested by us depend on manual feeding of the extruded soap and as such are not as efficient as the fully automatic machines used as standard equipment internationally in which the extruded soap is fed automatically into the stamping machine.

h) in the machines requested for by us, the extruded soap has to be cut manually into single pieces roughly equivalent to the size of the soap and then fed into the stamping machine piece by piece and hence the request for two stamping machines.

We hope that this has clarified all the points.

kindly do the needful in the matter and oblige.

thanking you,

yours truly

SUDHIR JAIN, UNIDO EXPERT

## REPORT REGARDING PURCHASE OF SOAP MACHINERY

The requirement was discussed with the project authorities and accordingly various sources of supply in HCM City were examined.

It was found that only The Saigon Cosmetic Company which is manufacturing soap itself was in a position to supply dependable equipment as they are using the same in their own factory to produce good quality soap.

Accordingly the possibility of buying machinery from them was discussed and quotation obtained for one complete set of machinery to fulfill the functions of pelletisation, extrusion and stamping.

In the short time available it was not found possible to search for other sources of supply and as such only one quotation has been obtained.

On the basis of our experience in the industry and the observations made in the factory of The Saigon Cosmetics Factory regarding the quality of machines offered and the quality of the product being produced, the prices quoted by the supplier appear to be reasonable.



**Backstopping Officer's Technical Comments  
based on the work of Mr. S. Jain  
DP/VIE/86/033/11-53**

The comprehensive report submitted by the consultant contains details of activities carried out by him including the additional work assigned to him by the UNDP and project authorities. The job description of the consultant was modified during briefing in Vienna in order to get him to technically evaluate the pilot plant equipment supplied from India. Furthermore the fact that demonstration of technology for methyl ionone was not possible due to lack of requisite reagents was taken into account in modifying the job description. Eventhough the duration of the mission of the expert was reduced due to decisions taken at the Tripartite Meeting to conclude all activities by 31 December 1993, the expert successfully completed all activities assigned to him.

The technology for the production of methyl ionone has been transferred without actual demonstration. The project authorities are expected to conduct trial runs once the reagents are available.

The recommendations of the expert as for production of aroma chemicals and isolates and the know how transferred in formulating perfume blends and their incorporation into soaps and detergents should be fully exploited to develop the local perfumery and soap industries. As marketing strategies have been outlined by the expert, market promotion has to be continued to export products to the countries in the region.

The additional work carried by the expert has been very useful in evaluating the equipment supplied and the extent to which know how has been transferred by the supplier of equipment. This permitted the sub contracting of the know how development and transfer from a local expert. The requirements of

accessories for the pilot soap making machine were drawn up by the expert so that a fully functional soap making machine would be available for the project follow up work. The expert has very successfully completed his obligations.

**We regret that  
some of the pages  
in the microfiche  
copy of this report  
may not be up to  
the proper legibility  
standards, even  
though the best  
possible copy was  
used for preparing  
the master fiche**