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PRODUCTION OF PHARMACEUTICALS FROM MEDICINAL AND AROMATIC PLANTS  
(PHASE II)

DP/TUR/88/001

TURKEY

Technical report: Findings and recommendations\*

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

Based on the work of Marla B. Narasimha  
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United Nations Industrial Development Organization  
Vienna

\* This document has not been edited.

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## 1. INTRODUCTION

As noted by the consultant in his earlier reports, the Anadolu University's Medical Plants Research Centre, Eskisehir has grown from its humble beginnings to the present state with the assistance of UNIDO/UNDP and its consultants, and is considered to be the nodal centre in Turkey in the field of medicinal and aromatic plants research. It has an excellent infra-structure with modern pilot plants, sophisticated analytical instruments and trained technical and scientific man-power.

M P R C is now able to undertake R and D in the efficient utilisation of medicinal and aromatic plants, to develop viable technologies, to design and fabricate chemical plant and equipment, to conduct diagnostic studies of sick industries and their successful rehabilitation - these activities by MPRC with UNIDO assistance are considered first of its kind in the field of pharmaceutical industries in Turkey.

The following have already been completed.

- Laurel Oil Factory in Silifke  
successfully rehabilitated under SI.TUR.88.803.
- Sistag Liquorice Factory  
Diagnostic Studies.
- Ortas (Turpentine) Factory, Edrenit  
Diagnostic Studies.
- Status report of Alcohol Factory  
Preliminary Studies.

## 2. SUMMARY

The consultant arrived in Turkey on 14th September, 90 and left on 4th November, 90. During his stay he held detailed discussions with Dr. Kamal Hussein UCD in Ankara and Prof. Dr. K.H.C.

Baser, NPD in Eskişehir. Keeping in view their broad guidelines and suggestions, he drew-up a broad working plan.

Executed the following :

2.1 Assistance in conducting TRUMAP 1990 Programme as a resource person.

2.2 Conducted diagnostic studies of a sick privately owned company reported to be the only factory in Turkey and one of the seven such factories in the world to produce :

- Rosin
- Turpentine
- Pine Oils
- Pine Tar

from pine stumps by solvent extraction method.

2.3 Prepared preliminary study of the sick Eskişehir Alcohol Factory.

2.4 Development of drug technology for producing Sulphamethoxazole.

2.5 Initiated R & D plans :

2.5.1 for hydration of  $\alpha$ -Pinene rich turpentine to  $\alpha$ -terpeneol.

2.5.2 to refine sulphated turpentine, a by-product of paper industry.

2.5.3 mixed fatty acids from Laurel berries.

2.6 The programme under General Engineering covered the following :

- 2.6.1 Assistance in indigenous procurement, including manufacture of S.S coils for packing, installation and commissioning of all-glass fractionation column, with high precision controls for heating, reflux and vacuum.
- 2.6.2 Modifications to Belgium-supplied stainless steel sohxle extraction unit of 12 kg/Batch capacity to make its operations flexible and to make it possible to use high boiling solvents.
- 2.6.3 Commissioning of all glass 1 litre capacity German supplied sohxlet cum liquid-liquid extractor.

#### DETAILED REPORT

##### 3.1 TRUMAP 1990

The consultant as a resource person assisted the National Project Director in conducting "Training Programme on the utilization of Medicinal and Aromatic plants in Pharmaceutical and Related Industries" organized by UNIDO in collaboration with the Government of Turkey and conducted by MPRC at Eskişehir from 10 to 29 September, 1990, (Please see Annexe - II).

During this training programme, as in the past, more emphasis was laid on "Hands on" training covering from bench scale experiments to scale-up operations on pilot plant scale, quality control and analytical techniques using modern sophisticated instruments and visits to phyto-chemical industries in and around Eskişehir.

He also conducted the trainees on a study tour to a sugar and an alcohol factory and prepared and distributed the background papers on the distillation of essential oils and a note on development and transfer of technologies.

The following eight trainees from developing countries participated in this programme :

- |    |                                       |                   |
|----|---------------------------------------|-------------------|
| 1. | Mr. A.B. Anwar Hussain                | - Bangladesh      |
| 2. | Mr. Getachew Assefa                   | - Ethiopia        |
| 3. | Mr. Aschelew Hunde                    | - Ethiopia        |
| 4. | Mr. Boahen Opoku Yaw                  | - Ghana           |
| 5. | Mr. Majeed Ameer Poor                 | - Iran            |
| 6. | Dr. Mrs. Sally Saad El-Din El Nakkady | - Egypt           |
| 7. | Ms. Olufolake Ganiyat Sule Odu        | - Nigeria         |
| 8. | Mr. Mohammed Kasim Nejym              | - Yemen Republic. |

A trainee from Ghana has in a private communication mentioned that " I have actually benefited from the training programme and I have been able to impart some of the knowledge and experience to my colleagues here in Ghana. We have also started looking at some of the aromatic plants of Ghana with the hope of going into the pilot scale extraction ".

An interesting feature of this programme was organising a " Panel discussion ". A very interesting and relevant topic "Development and transfer of technology " was chosen. All the trainees and technical staff of the MPRC participated very enthusiastically. The panel discussion was chaired by NPD and the consultant acted as a Moderator. (Please see Annexe - III).

The trainees were so impressed by this panel discussion, that they unanimously expressed at the round table conference that such

panel discussions should be conducted in future and repeated at every week-end with different topics.

### 3.2 DIAGNOSTIC STUDIES

3.2.1 M/s. Ortaş located at Edremit, Turkey, a privately owned company, is the only factory in Turkey and reported to be one of the seven factories in the world to produce rosin, turpentine and pine oils from pine stumps by solvent extraction method. Commissioned in 1975, the plant had the designed capacity of about 30 tpd pine stump chips. It was in a running condition but due to several technical and operational deficiencies, the company was incurring heavy losses.

As a part of general policy of Dr. Kamal Hussein UCD in Ankara, to extend technical support in diagnostic studies of sick units and their rehabilitation, and with the co-operation of Dr. Başer NPD and approval of the company management, the consultant accompanied by a counterpart Chemical Engineer visited the factory, and conducted preliminary studies and submitted a report, which is appended. (Please see Annexe - IV).

#### 3.2.2 Eskişehir Alcohol Factory

In Turkey there are thirty Sugar Mills, using locally cultivated sugar beet as the raw material. Molasses obtained as a by-product in these mills, containing about 50% non-crystallisable sugar is used in the production of ethyl alcohol in four factories in Turkey.

Eskişehir alcohol factory was established in the year 1938, with an installed capacity of 60,000 lpd. It



was closed down in May, 1990 due to very serious environmental problems.

The consultant and the National Project Director visited the factory for preliminary study and reported the team's observations and recommendations, and the same is appended. (Please see Annexe - V)

### 3.3 Development of drug technology - Sulphamethoxazole.

Pharmaceutical drugs (synthetic bulk drugs) are not presently manufactured in Turkey. The consultant has come across one company in Istanbul manufacturing bulk drugs in a small way. Almost all the multi-national pharmaceutical companies import bulk drugs from their parent companies, formulate and market them in Turkey.

To give an impetus to develop indigenous technology for the manufacture of bulk drugs in the country, an improved five-step process to synthesis sulphamethoxazole, an important broad-spectrum antibiotic has been worked out on a laboratory scale and demonstrated to the counter part scientists. The product obtained was found to be at par with USP grade. Suitable analytical methods were being worked out by the counter-part scientists to assess and evaluate the quality of the product at the end of each step. Suitable modalities were indicated to scale-up the processes to pilot plant level.

It is contemplated that during the early stages, some of the raw materials required for its manufacture may have to be imported, but given proper encouragement and promptings MPRC can form an important centre for the growth of ancillary units for the

manufacture of basic raw materials and bulk drugs.

4. R and D Plans were initiated for ;

4.1 Hydration of  $\alpha$ -Pinene rich turpentine to  $\alpha$ -turpeneol.

As reported earlier, M/s. Ortaş is reputed to be one of the seven factories in the world to solvent extract pine resin from pine stumps and roots. Its one of the products is a turpentine fraction containing  $\alpha$ -pinene as high as 80-85%.

Laboratory studies have been completed for hydrating  $\alpha$ -Pinene contained in this fraction to  $\alpha$ -turpeneol. This when isolated finds extensive use in perfume industries and in the manufacture of value-added industrial chemicals.

4.2 Refining of sulphated turpentine

Sulphated turpentine, a by-product of SEKA, State owned paper mills in Turkey, is abnoxious-smelling and when refined will form a very valuable industrial raw material.

About 40-50 tons of this chemical is reported to be available per month.

M/s. Ortaş brought this to the notice of the consultant, during his visit to the factory.

Laboratory studies have been initiated in consultation with NPD in MPRC for refining of sulphated turpentine.

#### 4.3 Mixed fatty acids from laurel berries

This is a follow-up action plan of the programme prepared during the previous mission of the expert for the preparation lauric acid/ester from laurel berries. This programme is linked with the revival of the second phase of "Laurel oil factory in Silifke. The capacity of the fixed oil plant is 5000 tpa. The essential oil part of the plant of the capacity of 2000 tpa has been successfully commissioned in March, 1989.

As mentioned else where the consultant was associated with the revival of essential oil plant (SI/TUR/88/803).

#### 5.0 General Engineering Services

- 5.1 Assisted in the indigenous procurement of an all-glass fractionation column - laboratory size, its installation and commissioning. Helped a local engineering company to manufacture very fine stainless steel coils using S.S wire, which were used as internal packings in the fractionation column.

The laboratory model fractionation unit has been fitted with high precision controls for heating, refluxing and vacuum.

The steel tubular structure necessary for the installation of the fractionation column has been fabricated in the University's work shop.

- 5.2 Modifications to the Werkhuizen Armand Deprest Belgium supplied SS Soxhlet extraction unit of 12 kg/Batch capacity.

The Belgium-supplied unit is meant for the solid-liquid extraction of medicinal plants.

The arrangement was for the evaporation (electrical) of the solvent to about 70°C, thus restricting its use with light boiling solvents only. To improve its usefulness to include high boiling solvents, the heating system was suitably modified, discarding the electrical heating system and introducing steam in its place using solenoid valves for control.

The unit was tested after modifications and found working satisfactorily with high boiling solvents (both polar and non-polar).

### 5.3 Commissioning of all-glass (5 litre capacity) West German-supplied solid-liquid cum liquid-liquid extractor.

Commissioned liquid-liquid extractor and the following observations were noted :

- The solvent distillation flask of the capacity of 2.5 l and the electrical heater were not provided with the bottom outlet to facilitate easy discharge of the flask.
- Provision to charge about 2.5 l solvent per batch, whereas the capacity of the solvent distillate receiver is only 250 ml.
- Dismantling and reassembly of the whole unit necessary for every change of raw material.

## 6.0 Recommendation

The consultant assisted MPRC in the rehabilitation of Laurel oil factory in Silifke during Nov. 88 and March 89 under UNIDO Project SI/TUR/88/803. This plant and all the essential services viz., Power, Steam and Water are fully functional. However, next to this plant there is another semi-installed plant whose accessories and essential parts lay scattered all over the plant area.

This plant is a continuous type 25 tpd meant for extraction of fixed oil from laurel berries. The fixed oil was found to contain about 60-65% lauric ester, a very important constituent of Shampoo industry. A small percentage of these berries are currently used for the extraction of oil on cottage scale by a crude and very inefficient method of boiling in water over burning wood.

The resulting oil used in making crude washing soap.

It is an irony that a partly installed plant of the capacity of 25 tpd was allowed to rust and laurel berries grown abundantly in the coastal regions of Turkey, which is known to contain highest concentration of very valuable lauric ester are being allowed to go waste, while meeting the entire needs of the lauric ester in the country by imports.

It is strongly recommended for the speedy rehabilitation of this invaluable plant, before it is rusted beyond reclamation point, since almost all the parts of this plant are made of

mild steel, which is amenable to rusting when exposed to humid air and more quickly when moist air is laden with common salt a common occurrence in the coastal places.

#### Acknowledgement

I would like to place on record my grateful thanks to Dr. R.O.B. Wijesekera, Special Technical Adviser UNIDO, Vienna, Dr. M. Kamal Hussein, UCD in Ankara and Prof. Dr. K.H.C. Baser, NPD Project DP.TUR.88.001, for encouragement and advise.

My thanks are also due to the members of staff of UNDP Ankara and the youthful & enthusiastic staff members of MPRC Eskisehir for their cooperation.

ANNEX - I



UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

## JOB DESCRIPTION

DP/TUR/88/001/11-01

Post title	Chemical Technologist
Duration	1.2 months
Date required	September 1990
Duty station	Eskisehir Turkey
Purpose of project	Industrial utilization of medicinal and aromatic plant resources for the production of pharmaceuticals.
Duties	<p>The expert is expected to work in the Medicinal Plants Research Centre, University of Anatolia, and guide and assist the Director and his staff in the pilot-scale processing of pharmaceuticals from Medicinal and Aromatic Plants.</p> <p>The preparations from medicinal plants are for therapeutic use within the country as well as for export. Essential oils from aromatic plants could also be for export purposes. The expert will be specifically required to accomplish the following:</p> <ol style="list-style-type: none"> <li>a. Initiate the development of suitable process technology for successful products based on work done in Phase I.</li> <li>b. Train local counterparts in chemical technology related to the production of plant-derived pharmaceuticals and take part in training programmes by delivery of lectures etc.</li> <li>c. Compile a short list of candidate-products for experimental production in the following years, and develop draft process-protocols, and techno-economic parameters.</li> </ol> <p>The expert will also be requested to complete a terminal report, before he arrives for debriefing, in which he is expected to discuss his findings and outline his recommendations as regards the future of the project.</p>

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Applications and communications regarding this Job Description should be sent to  
 Project Personnel Recruitment Section, Industrial Operations Division  
 UNIDO, VIENNA INTERNATIONAL CENTRE, P.O. Box 300, Vienna, Austria

## ANNEX - II

## TRUMAP

Training Programme on the Utilization of Medicinal  
and Aromatic Plants in Pharmaceutical and Related  
Industries, 10-29 September, 1990, Eskisehir, Turkey

10 September Monday

- |               |  |
|---------------|--|
| 9.30 - 10.30  | Opening Ceremony   |
|               | - Prof. Dr. K.H.C.Başer,<br>Programme Director.  |
|               | - Prof. Dr. K. Hussein, SIDFA, UNIDO   |
|               | - Prof. Dr. S.Öz-Alp, Deputy-Rector<br>Anadolu University.                                       |
| 11.00 - 12.00 | - Introduction to the Training Programme<br>Prof. Dr. K.H.C. Başer                               |
| 14.00 - 15.00 | - Prof. Dr. K.H.C.Başer,<br>Assoc. Prof. Dr. E.Yeşilada<br>From Plants to Products - An overview |
| 15.00 - 15.30 | -  |
| 15.30 - 18.00 | - Visit to the University Campus   |
| 19.00 - 21.00 | - Reception  |

11 September Tuesday

- |              |   |
|--------------|---|
| 8.30 - 9.00  | - Prof. Dr. K.H.C.Başer,<br>Assoc. Prof. Dr. E.Yeşilada<br>A brief lecture on laboratory<br>techniques.   |
| 9.00 - 18.00 | - Prof. Dr. K.H.C.Başer,<br>Assoc. Prof. Dr. E.Yeşilada,<br>Assist. Prof. Dr. N.Kirimer,<br>Assist. Prof. Dr. M.Ogutveren<br>Laboratory experiments |



Group A  
Extraction of a medicinal plant

Group B  
Essential oils

- Distillation
- Concrete extraction

\* Facilities have been provided for conducting experiments individually.

12 September Wednesday

- 9.00 - 18.00 - Experiments for Groups (A) and (B) will be interchanged.

13 September Thursday

- 8.30 - 9.00 - Prof. Dr. M. Tunçel  
A brief lecture on analytical techniques.

- 9.00 - 18.00 - Prof. Dr. M. Tunçel,  
Assist. Prof. Dr. N. Kirimer  
Laboratory experiments.
- Refractometry
  - Polarimetry
  - Gas Liquid Chromatography
  - TLC - densitometry
  - Gas Chromatography -  
Mass Spectrometry (GC-MS)

14 September Friday

- 9.00 - 18.00 - Prof. Dr. M. Tunçel,  
Assist. Prof. Dr. N. Kirimer  
Laboratory experiments.
- High Pressure Liquid Chromatography (HPLC)
  - UV spectrophotometry
  - IR spectrophotometry
  - Potentiometry
  - Polarography



- 22 September Saturday Visit to Rose Oil Distilleries and stay in Isparta.
- 23 September Sunday Sight seeing in Antalya and back to Eskisehir.
- 24 September Monday
- 9.00 - 10.00 - Mr. Z.Komur  
Rose oil production in Turkey
- 10.30 - 12.00 - Prof. Dr. E.Sezik  
Essential oil bearing plants of Turkey.
- 13.30 - 15.00 - Prof. Dr. E.Sezik  
Assays on essential oils
- 15.15 - 17.00 - Assoc. Prof. Dr. Y.Öztürk  
Pharmacological screening of medicinal plants for industrial use.
- 17.00 - 18.00 - Technical Film  
Rehabilitation of Silifke Laurel Oil Factory.
- 25 September Tuesday
- 9.00 - 18.00 - Mr. M.Muderrisoglu,  
Assist. Prof. Dr. Y.Yazan  
Cosmetic formulations  
Theory and practice
- 26 September Wednesday
- 9.00 - 18.00 - Prof. Dr. E.Güler,  
Assist. Prof.Dr.Y.Yazan  
Pharmaceutical formulations  
Theory and practice.

27 September Thursday

9.00 - 17.00

Visit to Beet root sugar and  
by-product recovery factory

28 September Friday

9.00 - 12.00

Round Table Discussion

14.00 - 16.00

Evaluation Meeting

Note : Arrangements are being made for entertainment during the  
course of Training Programme.

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ANNEXE - III



Training Programme in the Utilization of Medicinal and  
Aromatic Plants in Pharmaceutical and Related Industries  
10-29 September 1990, Eskişehir, Turkey



PANEL DISCUSSION

Topic : Development and Transfer of Technology  
Date : 20th Sept 1990  
Time : 3.00 P.M.  
Venue : TBAM, Eskişehir

Chairman : Prof.Dr.K.Hüsnü Can Başer, Director, TBAM  
Moderator : Mr .M.B.Narasimha, UNIDO Expert

ANNEXE - IV

**Preliminary Report**

visit to

**ORTAŞ FACTORY Edremit**

By

M. B. Narasimha  
UNIDO Consultant

**Present status:**

Ortaş at Edremit Turkey a privately owned factory is presently running at loss.

All the plant operations are arbitrary some modifications are needed in the plant. The quality of produce is poor and inconsistent fetching poor returns. It is an uneconomical operation.

To quote the company's Director General "They do not know what products and to what quality are being produced in the factory".

Turpentine produced contains 70-85%  $\alpha$ -pinene. The pitch produced during the fractionation is burned in the boiler.

The price of imported resin, a major product of the factory is TL 2300/kg and the labour cost alone to produce a Kg of it in the factory is about 2500 - 3000 TL, sometimes it goes as high as 4000/kg (according to the T.D.).

Most of the equipment seems to be in good working condition, but needs some modifications.

Operations are carried out to no particular procedures and standards and products are of inconsistent quality and value.

The factory has only one chemical engineer, who is the technical director and have no qualified chemists.

**Recommendations:**

It is a fit case for UNIDO to support to rehabilitate a privately owned sick but working factory, reported to be the only factory in Turkey and one of the seven such factories in the world, for:

- in depth study
- to introduce optimum conditions in all its operations
- to modify plant and equipment wherever necessary
- to effect production of consistent quality products
- to effect optimum utilization of its installed capacity
- to train counterpart engineers and production staff in the use of new technique to produce value added and consistent quality products.

### Factory set-up

M/S Ortaş, Edremit, Turkey, owned by a private company, is the only factory in Turkey and reported to be one of the seven such factories in the world to produce resin, turpentine and pine oils from pine stumps by solvent extraction process.

Work began to establish the factory in 1972 with the Italian technology and plant and machinery. Due to some unknown reasons the Italian Collaborators, withdrew from their contract during the middle of the project. The plant was commissioned in 1975.

The designed capacity of the factory is 30 tonnes of pine stump chips per day.

Broadly the plant consists of

- 1- Raw material preparation section
- 2- Solvent extraction section
- 3- Rosin and turpentine section
- 4- Fractionation section
- 5- Packing section
- 6- Quality control section
- 7- Steam generation and maintenance section

The stumps of pine trees pulled out from the ground in the forest and transported to the plant site are first sawed into large logs in the three band saw mills each fitted with 20 HP motors.

Feeding of the stumps and transporting the cut logs to the nearby primary chipper is done manually. These logs are again manually fed into the primary chipper which is driven by a 160 HP motor.

The chips are pneumatically carried on to the overhead vibratory screens. The oversizes are automatically fed by gravity through a chute into the primary chipper. Undersizes are stored separately and mixed with the chips of secondary chipper. The middles coarse chips are further chipped in the secondary chipper (45 HP) and conveyed directly into the extractors by means of elevator conveyor system.



### Extraction section

A rotocell continuous type solid liquid extractor with meal desolventizer is found installed right in the middle of extraction section, since resin content varies widely (10-25%) in the stumps, it is not being used for the last ten years and is replaced by six M.S. vertical percolators of 3 tons (each) capacity.

Chips contain

Moisture: 10-25%

Resin : 10-25% Av 16-17%

Solvent : n-hexane 60-70°C obtained from İzmit refinery

Extraction temp: 30°C, very approximate. No measurement possible with the present set-up, solvent recovery- not available. Residual solvent in marc - not available. Concentration of resin in miscella not available.

At any time four percolators are used for extraction counter currently. Of the remaining two, one for charging with fresh chips and the other for discharging the marc.

One percolator occupies for 8-10 hrs after charging and before discharging.

Miscella is pumped into the storage tank and subsequently into recovery section.

Fresh solvent or weak miscella is sprayed on the top of the percolator and discharged from the bottom into another percolator through a pump.

Heating of the percolator is done through the jacket provided throughout the height of the percolator.

Miscella is stored in a 10 m<sup>3</sup> tank. Concentration of miscella is supposed to be around 20 % of resin.

Miscella is preheated (to what temperature is not known) and flows into the evaporator. Batch operation. Original design seems to be for continuous operation.

The evaporator during the final stages is heated to 120°C, to make sure that no solvent remains in the resin.

Solvent vapors are first condensed in shell and tube condenser. The outlet gases are passed through a breather and freon cooled trap before letting out to the atmosphere.

The resin at 120 °C is pumped through a preheater, heated with steam at 7-8 atm pressure to 150 °C into resin distillation still.

This still was designed for continuous operation, but switched over to batch operation. The reason for switch over as mentioned by the technical director is that when resin distilled continuously, the rosin obtained has considerable low melting point, may be due to insufficient stripping of volatile matter from rosin.

This still is operated under vacuum of about 100 mm-Hg (fluctuates). The volatile matter is condensed and stored in 5m<sup>3</sup> storage tank.

This condensed volatile matter is fractionated once or twice in a week. Spare capacity available for fractionation.

The reboiler capacity is 5m<sup>3</sup> per batch. Fractionation is done under vacuum of about 100 mm-Hg, (under very widely fluctuating vacuum). It is heated by hot oil to a maximum temperature of 200-250°C. This is done in a Wanson (Belgium made) oil heater. The heating medium of this preheater is the pitch obtained from the fractionating column, admixed with a volatile component of the factory produce.

It appears that this pitch when produced to certain specifications (not known to the management) is used in the rubber industry.

Since the pitch produced in this factory is not up to standard, a local industry is importing the pitch from abroad.

The fractionation column, diameter 950 mm and height 11.5 m is packed with ceramic rasching rings.

There is no proper refluxing management and other controls.

Top cuts are taken very arbitrarily and are of not consistent quality.

Three cuts are taken from the top. The first cut is turpentine having about 80%  $\alpha$ - pinene, light oil and heavy oil.

No quality control measurement. The laboratory is not equipped to undertake such analysis.

Quality control lab is poorly equipped and not commensurate with the factory's requirements. After visiting the factory and the quality control laboratory, the expert was not surprised when the management informed him that "they do not know what products and to what quality are being produced in the factory". This in fact is the essence of the status of the Ortaş factory at Edremit.

The desolventized marc of the solvent extraction plant is conveyed pneumatically to the boiler site and is used in the boiler as fuel. No other fuel is used in the boiler.

There is a small maintenance room adjacent to the boiler house with some tools and welding machine.

The expert accompanied by the company's technical director then visited a few local work shops and hardware shops, to acquaint himself with the facilities available in and around Edremit.

#### Suggested activities

-To make systematic studies of all the operations in each section and establish optimum conditions

Time

Temperature

Concentration

Number of cycles

Reflux (fractionation)

- To work out design deficiencies

- Modifications where required

- Additional parts and equipment required

-To establish conditions for full utilization of installed capacity to produce products of specific and consistent qualities

-Train counterpart engineers and production staff in the use of new techniques in the production of value added products.

After visit to the factory, I had a meeting with the following

1. Mr. Emin Pars Director General
2. Mr. Çetin Çolak Technical Director
3. Mr. Sedat Hakkı Beis TBAM

During the course of the meeting, Mr. Emin Pars has indicated his priorities

1. Purification of sulphate turpentine

About 40-50 tons of sulphate turpentine is available as a by-product from SEKA, State owned paper mills in Turkey. This is foul smelling. The management desires on priority to purify this in the first instance.

The company has spare capacity for fractionation.

The company management also desires to improve the colour of rosin. I brought a litre of this turpentine and initiated trials in TBAM.

TBAM is well equipped to undertake such studies.

2. To stream-line the factory production operations so as to make productions economical.
3. Installation of Polish made new factory equipment.

New equipment costing about (according to Mr. Emin Pars, Director general of the factory) US 1.65 Million ("book value") purchased by a Turkish company in 1980 from Poland.

This plant is for the extraction of rosin and turpentine from pine stumps. The plant remained with in its original packages.

M/S Ortaş purchased it from them in 1989 paying 80% of its price and dumped them at the premises of Ortaş factory without even opening them.

Even some sensitive equipment presumably containing electrical equipment with written instructions on the boxes to store them in covered dry place, are also dumped in the open exposed to sun and rain. This was brought to the notice of the D.G.

Due to paucity of funds required for the construction of building to house this plant, they would like to take it up at a later date.

**Acknowledgement**

My grateful thanks to Dr. M. Hussein UCD Ankara for suggesting to take up the diagnostic studies and encouragement. Prof.Dr. K.H.C. Başer for enthusiastic cooperation and Mr. Sedat Hakkı Beis the bright young chemical engineer of TBAM for his help in the studies.

**ANNEXE - V**

**Preliminary Report**

**Status of Eskişehir Alcohol Factory**

By

**M. B. Narasimha**  
**UNIDO Consultant**

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In Turkey there are thirty sugar mills using locally cultivated sugar beet as raw material. Molasses obtained as a by-product in these mills (this contains about 50 % sugar, mostly non-crystallisable) is used in the production of ethyl alcohol in the following four factories:

	Location	Year of Start	Production Capacity LPD
1.	Eskişehir	1938	60,000
2.	Turhal	1956	50,000
3.	Malatya	1974	40,000
4.	Erzurum	1980	75,000

The Eskişehir factory has been closed-down in May 1990 due to very serious environmental problem.

We visited this factory, installed adjacent to the sugar mills, for study.

This report in brief gives our observations and recommendations.

#### Raw material :

Molasses containing 50-52 % sugar (85 Brix) pH 6.8 is diluted with water to 12 % sugar (12 Brix) and filtered and adjusted the pH to 5.5.

#### Preparation of raw mash :

The diluted molasses solution is normally sterilized and yeast grown at 5.5 pH. This is not practised at this factory. Instead the pH of the solution is lowered to about 3.8 by addition of 60% H<sub>2</sub>SO<sub>4</sub> to suppress bacterial action.

Ammonium phosphate and magnesium sulphate are added as nutrients.

Completion of this operation is indicated by the reduction of sugar content to 6-7 %.

#### Preparation of sweet mash :

Molasses are diluted to 20-24 Brix pH 6.8 and sterilized with live steam. The concentration of sterilized solution remains around 14-15 % sugar. This solution is known as sweet mash.

#### Preparation of yeast : [yeast (growth) station]

Yeast from stock is added to sour mash and its volume increased to 50 L by gradually adding sour mash.

This solution is transferred to 250 L yeast growth tank. To this 50 L sour mash is added. It is left to ferment for 1 - 3 hr. During fermentation solid matter content drops from 9-10 Brix to 6.5 Brix. After 3 hr period additional 100 L of sour mash is added to it, with this addition solid matter goes up to 9-10 Brix. Fermentation is continued till solid

content drops down to 6.5 Brix in 3 hr time.

The following is list of equipment in this station :

250 L Fermenters	3 Nos
1000 L Fermenters	4 Nos
4000 L Fermenters	4 Nos

The final yeast growth station, which is called " VORGER " has:

25 M <sup>3</sup> Fermenters	3 Nos
35 M <sup>3</sup> Fermenters	1 No
60 M <sup>3</sup> Fermenters	1 No

#### Fermentation :

Fermentation of sugar to ethanol by yeast is an anaerobic process. This process is followed in this factory.

About 17-18 M<sup>3</sup> of yeast solution from VORGER is pumped into the fermenter, and sweet mash is charged into it gradually, in such a way that at the end of 10 hr period 72 M<sup>3</sup> of sweet mash is charged, bringing the overall ratio of yeast to sweet mash to 1:4.

After complete addition of sweet mash, it is allowed to ferment anaerobically for another 8-10 hrs. Maintaining temperature between 32-35 oC, by circulating cool water. The warm water is cooled in a cooling tower and recirculated. The heat of fermentation is not recovered.

#### After fermentation :

pH	: 5.5
Alcohol content	: 7 %
Unconverted sugar	: 0.02-0.2 % (sometimes as high as 1.0 %)
Concentration of higher alcohol	: not measured
CO <sub>2</sub>	: not recovered

Fermented broth is centrifuged to separate the liquors from yeast.

The yeast separated from the broth contains 20 % solids. pH is adjusted to 2.0 and left to stand for 1-2 hr to destroy bacteria. It is then diluted to 6 Brix and reused. Liquors from centrifuge is subjected to 3 stage fractional distillation operation for the recovery of ethyl alcohol, and fusel oil (containing ethyl, propyl, butyl and amyl alcohols- about 50-55 % of it is composed of amyl alcohol.) as a by product.

In the first stage 85 M<sup>3</sup> of liquors distilled in two bubble cap columns made of copper. The top product from these columns produces ethanol of 50% concentration admixed with aldehydes.



The bottoms, known as "slop" or "spillage", this spillage is the major cause of air pollution in Eskisehir, due this reason the factory had to be shutdown.

On an average 1000 L of liquors when processed in the bubble cap columns yields 140 L of ethanol (50%) and 860 L of spillage.

The dried spillage contains

Organic solids	6.5 %
Nitrogen matter	0.43 %
Potassium oxide	1.07 %
Ash	2.1 %

Details of bubble cap columns

	Column I	Column II
Diameter (metres)	1.5	1.5
Height (metres)	8.0	8.7
Number of trays	16	18
No of bubble caps in each tray	10	10
Number of condensers	3	3

Surface area of the condensers

1.	50 M <sup>2</sup>	50 M <sup>2</sup>
2.	40 M <sup>2</sup>	40 M <sup>2</sup>
3.	20 M <sup>2</sup>	20 M <sup>2</sup>

The feed to the column is used as a coolant in the condensers, the temperatures reached in the condensers being 50°C and at the final condenser 70°C.

Dilute alcohol of 50% concentration containing aldehydes at about 65°C-70°C with hot water at 100°C is fed (second stage) with the column (Bubble cap also made of copper) at the point where diameter of the column is reduced from 1.4 M to 1.0 M.

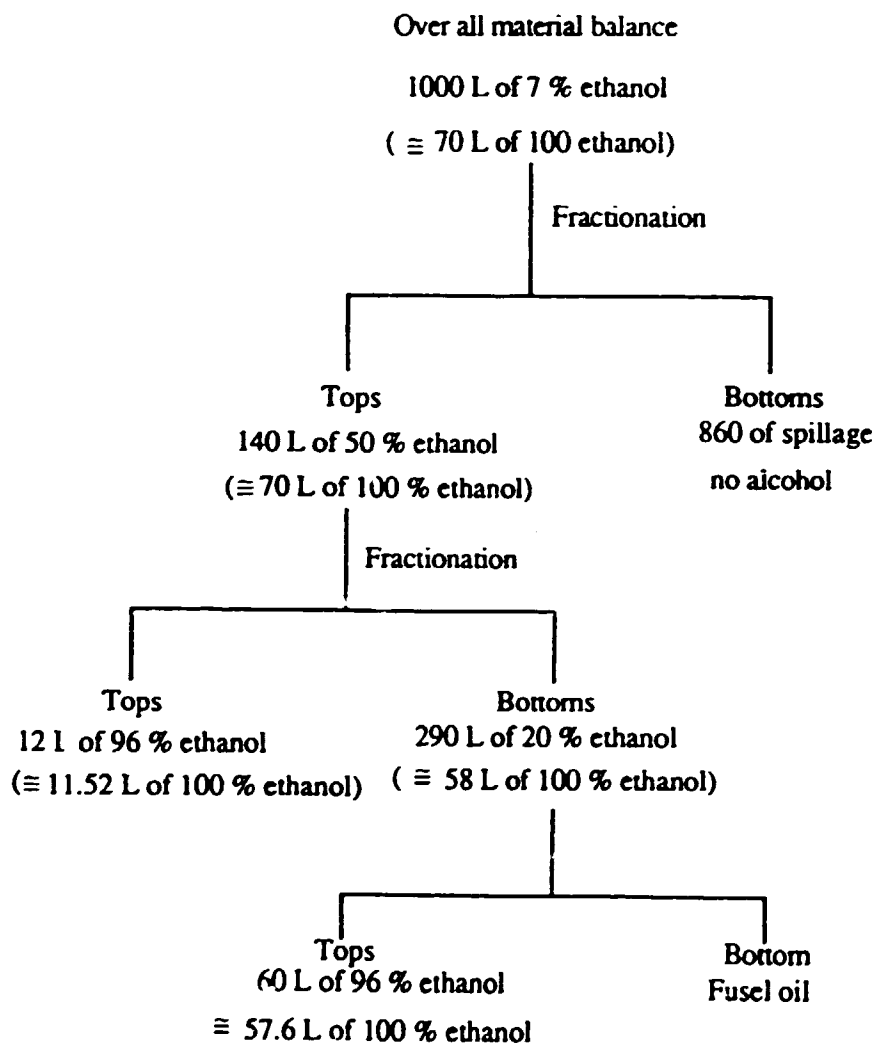
This column of diameter 860 mm, height 10.3 M, 62 trays containing 50 bubble caps in each tray. Two condensers of 20 M<sup>2</sup> and 9 M<sup>2</sup> surface area respectively are fitted.

Every 300 litres of feed into the column yields 12 liters top product containing alcohol (95% conc.) and aldehydes and 287 litres as bottoms, ethanol (20% conc.) and fusel oil, but free of aldehydes.

In this third stage dilute ethanol (20% conc.) and fusel oil mixture is fractionated in another column also made of copper with 17 trays.

Top product from this column is fusel oil free ethanol of 96 % conc. (60 L) and fusel oil as the bottoms.

Fusel oil goes into the another column of 700 mm  $\phi$ , 11.6 M height with 64 trays, equipped with three condensers of 20 M<sup>2</sup>, 20M<sup>2</sup> an 3M<sup>2</sup> surface area respectively for further recovery of alcohol of 96 % conc., which is added to alcohol obtained in the preceding column.



## A.

**Possible methods for the elimination of obnoxious / polluting spillage**

The residue of the first distillation unit known as "spillage" is reported to be the main cause for the environmental problem.

It is suggested to study in detail each and every operation and equipment that are employed in the production of ethanol from molasses - a by product of sugar industry based on beet root.

Complete analysis of "spillage" and on stream products, identify the substances causing environmental pollution.

**Some Suggestions:**

- To sterilise sour mash and sweet mash to avoid bacterial contamination and maintain pH of raw mash at 5.5 or as near pH 7.00 as possible to reduce acid concentration to facilitate easy handling and disposal of "spillage" without creating environmental problem.

- Additionally, efforts should be made to eliminate polluting chemicals totally or reduce them to tolerable levels by

1. Adsorption using locally available adsorbents
2. Ion exchange resins
3. Chemical treatment

## B.

The following are some of the suggestions

- The plant and technology should be updated if not totally replaced
- Each operation should be studied in detail - time and motion studies.
  - Replace obsolete equipment
  - Introduce optimum utilizational and operational conditions
  - Replace copper bubble cap columns with packed columns (Copper has the resale value)
  - There is tremendous scope to effect steam efficiency and overall steam economy
  - It has been reported that the cost of production of ethanol is more than twice when compared to the last year's production cost after due weightage provided for general inflation in the country.

By careful diagnostic studies and effecting judicious changes/, modifications/ optimisations in operations, equipment and by enhancing steam economy the obnoxious pollution problem may be overcome, cut down production cost and rehabilitate a sick commercial factory which is presently shut down. By rehabilitating this vital factory export of molasses and partial import of ethanol could be stopped.