OCCASION

This publication has been made available to the public on the occasion of the 50th anniversary of the United Nations Industrial Development Organisation.

DISCLAIMER

FAIR USE POLICY

CONTACT

Please contact publications@unido.org for further information concerning UNIDO publications.

For more information about UNIDO, please visit us at www.unido.org

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION
Vienna International Centre, P.O. Box 300, 1400 Vienna, Austria
Tel: (+43-1) 26026-0 · www.unido.org · unido@unido.org

RESTRICTED

17505

DP/ID/SER.A/1185
7 April 1989
ORIGINAL: ENGLISH

STRENGTHENING OF THE ROYAL DRUGS RESEARCH LABORATORY

DP/NEP/80/003

NEPAL

Technical report: Evaluation and recommendations*

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

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

Backstopping officer: R.O.B. Wijesekera, Chemical Industries Branch

United Nations Industrial Development Organization
Vienna

---

* This document has not been edited.

V.89 54366

Contents

Annexures

Title: Strengthening The Royal Drugs Research Laboratory

Project: DP/NEP/80/003

Station: Kathmandu, Nepal

Duration: January-June 1988

Purpose of Project:

To promote the utilisation of Nepal's existing and potential resources of medicinal and aromatic plants and to enhance self-reliance in the field of Pharmaceuticals.

Objectives

- to check and make all the pilot plants at Godavari operational
- to evaluate/develop processes on bench scale in the utilisation of indigenous medicinal and aromatic plants
- scale-up feasible processes to pilot plant level
- to train technical manpower

Summary

UNDP/UNIDO has created an excellent infrastructure base to conduct applied research on bench scale and on pilot plant with adequate quality control instruments, Vide Project No: DP/NEP/80/003 entitled " Strengthening the Royal Drugs Research Laboratory" a constituent of the Department of Medicinal Plants, HMG of Nepal, whose Director-General DR. Samara Bahadir Malla is the National Project Director.

R and D activities have been under progress since the inception of the project. The UNIDO expert process technology upon his arrival was informed the status of the on-going R and D activities and the problems and short-comings being faced in the refining of Shilajeet, an exudate of high altitute rocks of Himalayan region, processing of Mucuna seeds for L-depa, fluctuations in temperature and unstable conditions in the column in the fractionation of turpentine.

These problems have succesfully been solved and demonstrated in the laboratory, bench scale, pilot plant, level.

An alternate method has been worked out on bench scale to extract diosgenin from hydrolyzed dioscorea deltoidea, an important steroidal hormone base, using ethanol as the solvent. Ethanol is indigenously produced in Nepal.

The ethanol extracted diosgenin is generally, 70-80% pure. Studies were under progress for refining it to 95% pure using suitable solvent, whose consumption is relatively small.

Generally n-hexane is used for the extraction of diosgenin. This solvent is being imported into the kingom.

Helped in getting fabricated locally, a 1000L hydrolyser in wood with FRP lined steam sparger for the hydrolysis of dioscorea tubers. Tested and tried two batches.

Assembled Gallenkamp laboratory model all glass fermenter and succesfully tested 4 batches for about 96 hours each (non stop) with the microorganism available in RDRL. There was no contamination except in one batch.

Reviewed the work done in the distillation of Acorus Calamus, replanned systematic method for its distillation, and executed successfully, in the laboratory, at pilot plant level 100 Kg/Batch and 200 Kg/Batch, established optimum conditions and demonstrated at the premises of M/S HPPCL a Government owned industry. A project report prepared (appended) and submitted to the project counterparts.

Sugandha kokila whole berries are currently being distilled in the industry. The exhausted berries are hydraulically pressed for the recovery of fixed oil. It is a well known fact that the pericarp of berries is rich in essential oil and kernels are rich in fixed oil, which contains a large percentage of lauric ester. Lauric acid is a very valuable industrial raw material.

It is suggested that berries be pre-processed mechanically separating pericarp from kerels and subjecting them separately for the recovery of essential oils and fixed oils respectively. This method when followed is expected to result in:

- higher capacity per unit volume of distillation unit
- higher yields of essential oils
- higher yields of fixed oil
- fixed oil free from contamination.

The fixed oil of Sugandha kokila kernels when recovered commercially will form an industrially important raw material, since it contains more than 50% of lauric acid ester.

Initiated laboratory/bench scale studies for the refirement of :

- Pyrethrum oleoresin
- Belladonna oleoresin
- Synthesis of paracetamol a bulk drug, the total requirement of which is imported into Nepal.

Testing of Pilot Plant

Upon testing, the pilot plant units at Gadovari, all the units at almost all the joints showed leakes, effective steps have been taken to make them leak-proof, a very essential step to prevent fire hazard, since volatile solvents are being used in these units for various developmental studies.

To effectivelly prevent loss of heat, subsquent reduction in the consumption of steam and reduce time of operation, all the pilot plant units, vapour and steam pipes within the pilot plant buildings and the steam lines from the boiler to the pilot plants were properly insulated and water proofed.

During the operation of some pilot plants, a need was felt for additional accessories for almost all the pilot units to make them more flexible(Drawings appended).

Panel discussion

The expert process technology, moderated the panel discussion on "Transfer of Technology from Laboratory to Industry" organized by Nepal Chemical Society, Kathmandu on 16 th March 1988.

UNITED NATIONS

dr: R. Wijesekera/rml

- 4 -

27 June 1986

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

UNIDO

Project for Nepal

JOB DESCRIPTION

DP/NEP/80/003/11-05

Post title	Expert in Process Technology
Duration	6 months (split mission, 2 months each during 1986, 1987, 1988)
Date required	as soon as possible
Duty station	Kathmandu
Purpose of project	To promote the utilisation of Nepal's existing and potential resources of medicinal and aromatic plants and to enhance self-reliance in the field of pharmaceuticals.
Duties	The expert will assist in utilising the pilot plant at Godawari site (supplied by Messrs Tournaire, France and having a capacity of 200 - 250 L) to process and upscale laboratory processes utilising indigenous medicinal plants. The expert will work out protocols giving conditions for each process and will demonstrate the process technology while imparting on-site training of Nepalese counterparts. At the end of each split mission, the consultant will prepare a report (in a fully finalised version) setting out his findings and recommendations. The third of these reports must incorporate the salient features of the preceeding reports and should be in a form ready for reproduction.

..../..

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

V.81-33106

Qualifications

A chemical/mechanical engineer with several years of experience in the pilot-scale processing of medicinal plants and related natural products. The candidate should have experience in process development and in imparting knowledge to those with no previous experience of pilot plant work in this field.

Language

English

Background information

The country has a population of approximately 17 million with an annual growth rate 2.6%. Over 90% of the populace live in rural areas and over 60% of them in the mountain zones. Most of the rural folk utilise plant-preparations for their therapeutic requirements and the traditional system of medicine is very similar and related to the Ayurvedic system prevalent in the Indian subcontinent.

The wealth of medicinal plants can be considered as one of the most important natural resources of the country. The country lies in the Central Sector of the great Himalayas and occupies one third of their total length. The diversity of physiography due to altitudinal and climatic variations has produced a great variety of plant species within the flora of this small country (area - 145,305 sp. km). Much of this flora is used in medicine and the Royal Drugs Research Laboratory (RDRL) is responsible for the R & D efforts leading to the production of pharmaceuticals based on traditional remedies, RDRL is now a well equipped modern research and development institution.

It has been assisted by UNIDO-UNDP for the past two years to enhance its capabilities as an R & D institution, and to provide technical assistance.

A suitably integrated institutional framework already exists in the country for the systematic cultivation, research and development and processing of plant-derived products. The present projects are designed to develop this capability further in order to enhance the Government health-care programme.

ESSENTIAL OIL FROM ACORUS CALAMUS

The plant Acorus calamus (Family: Araceae) is herbaceous, perennial and grows wild on the swampy areas of the temperate zones of Nepal. Its rhizomes measure upto 20 cm long and 2 cm in diameter, longitudinally furrowad on the upper surface and with conspicuous root scars on the lower surface. These on steam distillation yield a volatile oil commercially known as calamus oil. Nepal exported about 11.5 MT during the year 83/84.

The principal producers of calamus rhizomes are Hungary, Poland, Yugaslavia, Bulgaria, USSR, Holland, USA, India, North Korea and Japan.

Calamus oil is used both in flavour and perfumery. It is no longer permitted for use in food under U.S. law since ß-asarone, the main constituent of the oil is reporteo to possess a carcinogenic activity.

The estimated annual world production of calamus oil is about 10 tonnes, the major producer being North Korea.

The main chemical constituent of Nepali calamus oils is ß-asarone (70-90%).

Almost the entire product of the rhizome known as "Bojho" in Napali is exported to India.

Systematic studies have been conducted in the laboratory and in Pilot Plant (100kg/batch) and scaled-up/ 200 kg/batch under varying conditions. The method thus standardised has been succesfully demonstrated on an industrial scale at the premises of M/S HPPCL Kathmandu.

Summary of results are shown in the following tables:

Table-1

Batch no	Quantity Per Batch Kg	State of feed	Distillation method	Cohobation
1.	200	Coarse powder	Live steam at 3 kg per cm^2 at 1.5,2.0 lit/mt	yes
2.	100	Coarse powder	Live steam at 3 kg/cm^2 2 litre/minute	yes
3.	100	Coarse powder	Live steam 3 kg/cm^2 2 litre/minute	no
4.	100	Coarse powder	Live steam 3 kg/cm^2 2 litre/minute	no
5.	100	Coarse powder, passed live steam for 30 mts left for 12 hrs and distilled	Live steam 3 kg/cm^2 2 litre/minute	no
6.	100	Coarse powder, passed live steam for 30 mts left for 12 hrs and distilled	Live steam 3 kg/cm^2 2 litre/minute	no
7.	82.5	Coarse power		no
8.	100	Coarse powder		no
9.	100	Coarse powder		no
10.	97	Coarse powder,passed steam for 30 mts, left for 12 hrs and dittilled		no
11.	200	Coarse powder	Live steam 3 litre/minute	no
12.	200	Coarse powder	Steam	no
13.	200	Coarse powder	Steam	no

Table II

Percentage Yield of oil at every three hours of distillation (Pilot Plant)

Batch no	Batch size (KG)	Oil % (CMFB)	Duration of Distillation (hrs)							
			3	6	9	12	15	18	21	24
1.	200		0.672	1.430	1.90	2.23	2.69	3.19	3.25	3.57
2.	100		1.51	2.96	3.89	4.59	4.41	5.06	5.20	
3.	100		2.30	3.94	4.63	5.09				
4.	100		1.7	2.37	4.31	5.29				
5.	100		2.37	4.31	5.29					
6.	100		2.51	4.47	5.70	6.24	6.53			
7.	82.5		2.59	3.82	4.44	4.70				
8.	100		1.99	3.18	3.85	4.19	4.44			
9.	100		1.84	2.89	3.46	3.78	4.00			
10.	97		1.84	2.56	3.23	3.44	3.60	3.89	260 ml oil recovered during 30mts steaming on the previous day.	
11.	200		2.48	4.39	6.15	6.95	7.58			
12.	200		2.54	5.18	7.32	8.71	9.88			
13.	200		2.66	5.04	6 98	8.15	8.84			

All figures are on Moisture Free Basis

Table III

Batch no	Time of distln.	Ref. Index 20 c	Optical rotation	Solubility in 70% EtOH (20°c) by volume	Asarone content %	Acid no.	Ester no.	Moist. cont. %
1.	3	1.547	(-)0.14	1.6	69.48	0.67	8.66	
	15	1.5517	(+)1.07	1.4	86.48	0.89	6.00	24.1
2.	3	1.5489	(+)2.14	1.3	X	0.82	6.98	
	15	1.5525	(+)0.73	1.3	96.04	0.89	6.30	24.1
3.	3	1.550	(+)2.23	1.3	89.41	0.82	8.20	
	13	1.5516	(+)1.18	1.3	92.35	1.50	6.56	23.95
4.	3	1.5488	(+)2.35	1.25	89.98	1.17	7.87	
	15	1.5495	(+)1.55	1.25	90.49	2.3	5.92	20.3
5.	3	1.5506	(+)1.71	1.4	88.79	0.84	6.23	
	9	1.5510	(+)0.82	1.2	93.05	2.34	5.07	15.5
6.	3	1.5510	(+)1.72	1.4	88.93	0.86	6.23	
	15	1.5511	(+)0.58	1.4	93.97	3.04	4.9	15.6
7.	3	1.5499	(+)1.62	1.3	92.28	1.8	3.45	15.4
	12	1.5509	(+)0.85	1.3	95.64	4.035	1.42	15.12
8.	3	1.5495	(+)1.90	1.25	92.41	1.20	7.95	7.3
	14	1.5500	(+)1.15	1.25	92.45	2.24	5.21	7.6
9.	3	1.5510	(+)1.32	1.04	91.37	1.09	5.57	1.06
	15	1.5506	(+)0.75	1.02	92.45	4.02	8.18	1.07
10.	3	1.5498	(+)1.02	1.08	91.63	1.28	3.18	1.065
	15	1.5495	(+)0.45	1.05	83.33	3.70	5.41	1.064

Table IV

Physico-chemical Properties of Acorus Calamus oil from other countries

Origin of oil	Specific gravity (15°C)	Optical Rotation	Refractive Index (20°C)	Acid No.	Ester No.	Ester No after acetylation	Solubility	Asarone Content (%)	Oil (%)	References
European oil	0.959 to 0.972	(+)9° to (+)31°	1.5028 to 1.5098	up to 3.7	4 to 18	32 to 50	15 vol.of 80% alcohol		1.5 to 4.8	Guenther
Japanese root oil	0.973 to 1.023	(+)7° 20 to (+) 26 °30. In few cases (-) 5° 3€ to (-) 11 ° 25	1.511 to 1.528	up to 2.0	2 to 8	15 to 34	10 vol. of 80% alcohol		up to 5	
Indian root oil I	1.0694	(+) 6° 12	1.5030	1.4	5.1	16.6	1.5 vol. of 70% alcohol	82	2.8	
Indian root oil I	1.076	(-) 1° 30	1.5461	2.4	4.1	15.7	X	82	2.8	
Russian oil	0.952 to 0.955	(+) 12° 48 to (+)15° 0	1.5020 to 1.5031	X	4.7 to 7.5	X	5 vol. of 90% alcohol			

Table V

Laboratory Studies: Percentage Yield of oil

Batch No.	Wt. of sample (grms) Yield wt/wt	Duration of Distillation (hrs)								
		3	6	9	11	12	15	18	21	24
1.	wt. of sample (oil %)	90.12 (2.0)	187 (2.5)	88 (3.86)	-	137 (3.99)	116 (5.7)	107 (5.6)		
2.	"	216 (1.99)	113 (3.4)	91 (4.4)	-	90 (4.8)	88 (5.6)	249 (5.6)	265 (5.7)	311 (5.3)
3.	"	167(4hrs) (2.5)	.	.	.	.	.	.	.	60 (6.2)
4.	"	35(4hrs) (3.4)	.	.	.	.	.	.	.	35 (6.1)
5.	"	34(4hrs) (3.19)	.	.	.	.	.	.	.	.
6.	"	42 (2.14)		46 (4.3)	.	.	.	.	.	.
7.	"	.	.	.	.	.	37.53 (4.19)			
8	"	.	.	.	.	.	49.36 (5.14)			

Table V Contd.

Laboratory Studies: Percentage Yield of oil

Batch No.	Wt. of sample (grms) Yield wt/wt	Duration of Distillation (hrs)								
		3	6	9	11	12	15	18	21	24
9.	wt. of sample (oil %)	-	-	-	57.7 (5.0)					
10.	"	-	71.69 (2.7)	-	-	-	-			
11.	"	-	-	-	-	-	50.14 (3.9)	-	-	
12.	"	-	-	-	-	-	59.93 (4.3)	-	-	-
13.	"	-	-	-	-	-	41.77 (4.98)	-	-	-

Table VI

Batch no	Time of distillation hrs	Ref. Index 20 °C	Optical Rotation	Acid no.	Ester no.	Asarone Content	Moist. Content %
	3	1.5511	(+)1.09	-	-	95.47	
	6	1.5529	(+)0.52	-	-	83.32	
	9	1.550	(+)0.83	-	-	89.83	17.99
1.	12	1.5531	(+)0.44	-	-	96.14	
	15	1.5531	(+)0.33	-	-	-	
	18	1.5537	(+)0.32	-	-	95.98	
	3	1.5515	-		-	94.85	
	6	1.5524	(+)0.82		-		
	9	1.5539	(+)0.51				24.1
	12	1.5539	(+)0.50		-		24.1
2.	15	1.5530	(+)0.34		-		
	18	1.554	(+)0.24			97.33	
	21	1.553	(+)0.26		-		
	24	1.555	(+)0.50			95.07	
	4						
	15						
3.	18						20.3
	24						
	4						
	6						
	10						20.3
3.	15						
(Soaking)	18						
	24					92.78	
4.	4						15.4
4.	4						15.4
(Soaking)							
5.	3						94.09
	9		(+)0.68				-
6.	15	1.5529	(+)0.33	0.6	3.99	93.18	7.3
7.	15	1.5530	(+)0.33	0.59	3.89	89.88	7.6

Table VII

Percentage Yield of oil at different interval of distillation (laboratory)

Batch no	Oil (%) (MFB)	Duration of Distillation (hrs)										
		3	4	6	9	10	11	12	15	18	21	24
1.	Oil %	2.43	.	3.03	4.69	.	.	4.84	6.92	6.90		
2.	"	2.55	.	4.48	5.80	.		6.32	7.38	7.38	7.5	6.98
3.	"		3.13		.	.	.	.	7.4	7.57		
3.(Soaked)	"		4.27	5.94	.	6.66	.	.	7.08	7.15		7.20
4.	"		3.80									
4.(Soaked)	"		3.90									
5.	"	2.6	.		2.34	.		.	.			
6.	"	.	.		.	.	.	.	4.5			
7.	"	.	.		.	.	.	.	5.5			
8.	"	.	.		.	.	5.62	.	.			
9.	"	.	.	2.89	.	.	.	.	.			
10.	"	.	.		.	.	.	.	4.61			
11.	"	.	.		.	.	.	.	5.10			
12.	"	.	.		.	.	.	.	5.86			

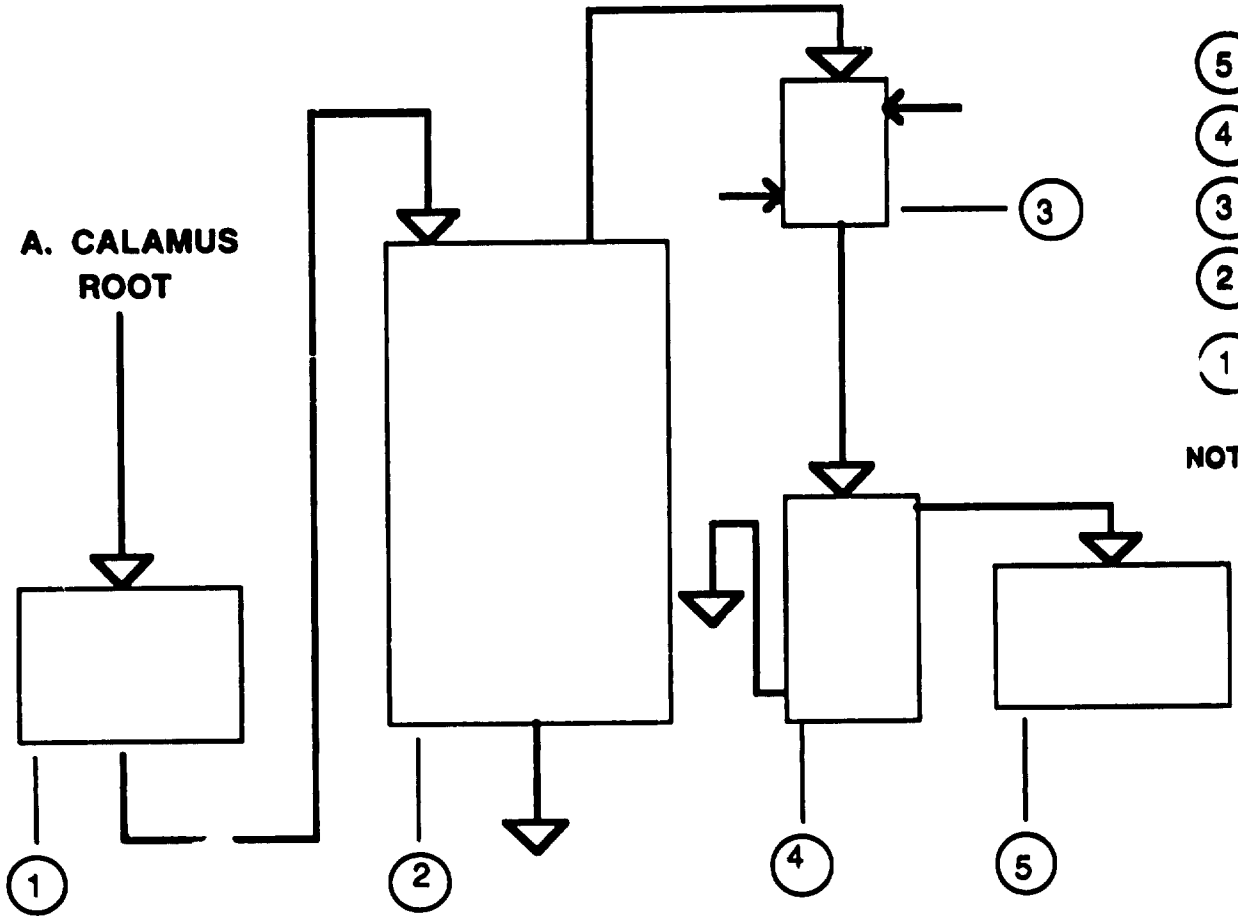
MFB: Moisture Free Basis

Table VIII

Distillation of Marc.

(Residual oil content)

Batch No.	Method of distillation	% oil in Sample (MFB)	%oil in marc.(MFB)
1.	Steam/cohobation	2.7	
2.	Steam/cohobation	3.96	0.04
3.	Steam distillation	4.09	0.8
4.	Steam distillation	3.01	1.17
5.	Steam/soaking	4.34	1.3
6.	Steam distillation	3.7	1.0



5 OIL RECEIVER

4 SEPARATOR

3 CONDENSER

2 STEAM DISTILLATION STILL

1 CRUSHER

NOTE: IN LARGE SCALE OPERATIONS ELEVATOR CONVEYOR SYSTEM COULD BE USED BETWEEN 1 and 2

SCALE: NOT TO SCALE

FLOW SHEET (BLOCK DIAGRAM)

DISTILLATION OF ACORUS CALAMUS

Cost of Calamus Oil Production
(Processing only)

Cost of Steam

N. Rs. 200/ ton

Steam Rate 2Kg/mt : 120 Kg/hr

Water 100 1/hr : N.Rs. 2/1000 1

Cost of Pilot Plant : NC 300,000

3,50,000/-

Cost of Bldg. and land : 50,000

Depreciation (1) PP 10% per annum : 30,000/250x8=15

(2) Bldg 5% per annum : 2500/365x24=0.30

Interest on (1), (2) plus land 15% annum : $350{,}00 \times \frac{15}{100} \times \frac{1}{3655 \times 24} = 6$

Cost per hour

	Rs.
Steam	24.00
Water	2.00
Depreciation	
a) Plant :	15
b) Bldg :	0.30
Interest :	6.00

Plant operation 100/month $\frac{1000}{25 \times 8}$ = Rs. 5/hr

Supervision : 5% of scientists working hour

$= \frac{\text{Rs. } 3000 \times 5}{100 \times 25 \times 8} = 0.75$

Cost of processing per hr. Rs. 53.05/ __ Rs. 55

Cost of calamus per Kg Rs. 968 ____ US $ 44

Basis : 100 Kg charge

Yields of oils worked on MFB (Moisture Free Basis)

Different in yields ..... Litre

	Batch No.						
hrs	4	5	6	7	8	9	10
9	3.036		5.692	4.438	3.847	3.460	3.231
12	3.375		6.242	4.700	4.194	3.734	3.443
15	3.517		6.530		4.442	4.00	3.600
			Yields between 9-15 hrs and 12-15 hrs				
9-12	0.339		0.550	0.262	0.347	0.324	0.212
12-15	0.142		0.288		0.248	0.216	0.159

Operational charges per hour = Rs 70 (Rs 210/3hrs)

Cost of calamus oil at US $ 44.00 per Kg: Rs 968 (a) Rs 22/US $

Conclusion for a batch of 100 Kg

1. Even if 300 ml of oils is recovered in 3 hrs between 9 th and 12 th hours, based on value of oil and cost incurred per additional 3 hrs of distillation, 12 hrs of distillation is considered as optimum period for a batch of 100 Kg
2. Basing the recovery of oil during the first 3 hrs of distillation and taking into consideration optimum utilization of the equipment direct steam distillation is the prefered method.
3. Asarone content of the oil during the first 3 hrs is around 90% reaching to about 95% in the 15 hours of distillation.
4. Rhizomes to be coarsely powdered. Coarsely crushed rhizomes should be well packed before start of distillation.

Conclusion for a batch of 200 Kg

1. Distillation studies scaled up to 200 Kg. at RDRL Pilot Plant. The results are shown in various tables under batch numbers 11,12 and 13.

2. From these data it is concluded that about 80 % of the oil that contained in the feed (M.F.B) could be recovered in a period of 15 hrs.

3. This technology has been demonstrated on a 400 Kg. batch at the factory premises of M/S HPPCL, Kathmandu.

Team Members

Mr. M.B. Narasimha - UNIDO expert.
Dr. S.R. Adhikary
Mr. A.D. Shrestha
Mr. R.R. Prased
Mr. N.P. Shrestha
Mr. D.N. Jha
Miss. S.P. Upadhya
Miss. R. Shakya
Mrs. H.D. Shrestha
Miss. I. Okuda-Japanese Volunteer

Diosgenin from Dioscorea deltoidea

Dioscorea deltoidea known as vyakur in Nepali is a climber belonging to family of Dioscoreacea. It is mostly distributed between 900M-3000M in Nepal.

The dioscin containing tubers which forms the main source of steroids are knotty woody and elongated.

Harvested tubers, cut into 12-25 mm length and field dried are generally supplied. Uncut dried tubers, when supplied for processing are very tough for disintegration. These are therefore soaked in water overnight and then disintegrated in a hammer mill.

The crushed tubers are then hydrolysed with a mineral acid 7-8% for 3-4 hours. This preprocessing is necessary, for converting dioscin contained in dioscorea tubers into diosgenin, which forms a starting material for 16 DPA which is an intermediate compound for progesterone, testosterone, steroids etc.

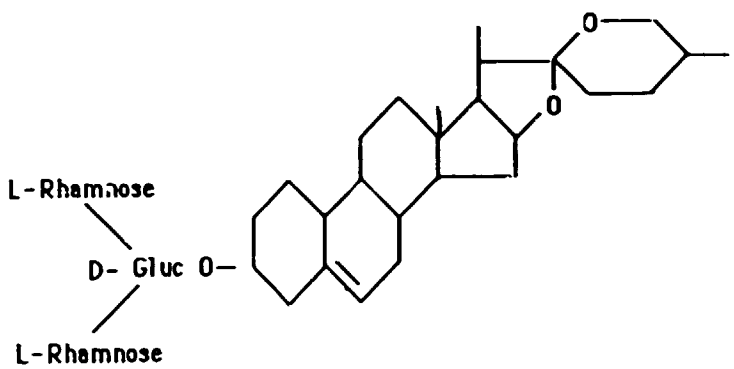
The hydrolysed and dried tubers reduced to about 60% of its original weight are extracted usually with n-Hexane, to obtain diosgenin of about 90-92%, which is further purified before processing into an intermediate compound viz., 16 DPA.

However, n-Hexane is not indigenous to Nepal and has to be imported into it, a landlocked country. It was therefore thought of to explore the possibility of using ethyl alcohol, a produce of Nepal, as a solvent for the extraction and subsequent purification of the crude diosgenin.

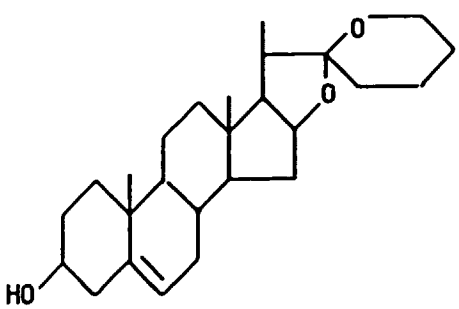
It has been observed that ethyl alcohol was not only a good solvent of diosgenin but also for a lot of impurities present in the hydrolysed dioscorea, and thus necessitates the purification of crude diosgenin.

Another disadvantage of using ethyl alcohol lies in the fact that the alcohol, which gets diluted in the coure of extraction and disolventisation of marc, has to be rectified to make it worthy of its reuse for the purose of extraction.

In the past a few batches of 30 kg each were hydrolysed in Hastealloy reactor, using disintagrated dioscorea, which resulted in fine powder, which was difficult to filter and subsequent extraction



DIOSCIN



DIOSGENIN

(Δ^5 , 25 ⊀ - Spirosten-3ß-ol)

Hydrolysis of precut and field dried tubers, about 150 kg/batch, without further disintegration have been conducted in a wooden vat of 1500 litre capacity.

(The expert in process Technology helped in getting the wooden hydrolyser fabricated locally, with FRP coated pipe steam sparger).

The results obtained in the hydrolyser were promising, and presented no filtration problems.

Constrainsts:

Non-availability of :

- Shelf dryer (under repair)
- Suitable column with reflux distibution arangement for the rectification of dilute alcohoi
- Vapour tight basket-centrifuge with explosion proof electrical fittings
- Reliable analytical method for the analysis of diosgenin

The spent acid which contains in addition to free acid a lot of imprities,which would easily carryover into hydrolysed material when reused. Basing on this fact and the relatively low cost of commercial sulphuric acid, the expert recommends to use dilute commercial acid for one hydrolysis and the spent acid discharged into a pit filled with slaked lime to neutrelize the free acid before letting into the drainage system.

The details have been discussed with the project counterparts.

Table I

Lab/Bench Scale
Hydrolysis

Size of D. deltoidea	%of Moisture	% of Diosgenin (MFB)	Batch Size (gms)	Conc. of H SO %	Time of Hydrolysis (hrs)	Number of Washings	Drying to Bone dry		Wt. of Hyd. Drug (MFB) gms	Diosgenin % in (MFB)
							Time Hrs	Temp °C		
. Powdered	12.7	3.75	100 gms of Dioscorea Powder(MFB)	5	4	10 times with 4L of water	10	100	39.5	10
Small pieces mixe with Powder	12.26	5.03	100 gms of Dioscorea mixed powder(MFB)	5	4	15 times with 5L of water	12	100	38.89	12.60

Pilot Plant

Batch size	Strength of Acid %	Time of Hydrolysis Hrs	Wt of hydrolysd. Drug for estimation(gms)	Diosgenin content %	Strength of spent acid %
1. 150 kg of Powdered	6.32405	4-5	20	12.06	3.90432
2. 100 Kg 25 mm to 50 mm	7.784	4-5	20	5.60	4.508

Table II

Comparative Study of Solvents- Ethyl alcohol and n-Hexane

Batch No.	Batch size	Soxhlet solvent	Time of extraction hrs	Total oleoresin %	Yellow powder of Diosgenin %	Extn. with N-hexane %	Diosgenin crystals %	Purity of Diosgenin %	Melting point °C
1.	250 gms of Hydrolysed Drug	Ethyl alcohol	6	13.7	7.35	6.52	5.32	90.47	202-205
2.	"	n-hexane	6	8.18	5.18	--	4.54	91.78	205-208

Solvent used	Volume of solvent	Time of extn. Hrs	Solvent recovered Litre	Total oleoresin dioscorea %	Material Balance	
					Hydrolysed drug before extraction	
n-Hexane	3 litre	6	1.8	10.2	250 gms of dried Hyd. drug	Marc - 223.2 gms Total extr.- 25.5 gms 248.7 gms Loss in wt= 1.3 gms
Alcohol	3 litre	6	1.79	1.0	250 gms of dried Hyd. drug	Marc -212.23gms Total extr.- 36.5 gms Residue on 1.1 gms filter paper 249.83gms Loss in wt= 0.17 gms
Pilot plant Hydrolysed Dioscorea : Diosgenin = 8.5 % Moisture = 10.0 %						

Table III

Extraction with Different Solvents

Solvent used	Total oleoresin extract %	Dried powder %	Syrupy mass %
Petroleum ether (60-80)	8.18	5.18	2.57
Toluene	9.28	5.81	3.47
Xylene	11.55	4.68	6.90
Ethyl Alcohol	13.7	6.55	7.19

Table IV

Extraction Data
(Hydrolysed Drug)

Batch No.	Batch size (MFB)	Soxhlet Solvent	Time of extraction hrs	Extract concn. after %	Marc wt (MFB) gms	Diosgenin in Marc	
1.	250 gms of Hydrolysed Drug	Ethyl alcohol	6	14.60	212.40	Nil	Note: Hydrolysed drug containing 8.5 % of diosgenin . The same material used in all batches from 1 to 10
2.	250 gms	"	"	18.12	214.40	Nil	
3.	"	"	"	15.11	212.23	Nil	
4.	"	"	"	18.12	214.46	Nil	
5.	"	"	"	14.61	213.46	Nil	
6.	"	"	"	18.90	215.3	Nil	
7.	1 Kg	"	"	13.79	861.20	Nil	
8.	250 gms	Petroleum ether (40-60)	"	7.36	229.40	-	
9.	250 gms	n-hexane	"	10.2	223.2	-	
10.	1 Kg	n-hexane	6	8.18	892.0	-	

The team

1. Mr.M.B. Narasimha
   UNDP/UNIDO Consultant
2. Mrs.Padma Prajapati
3. Mr.Ananda Dev Shrestha
4. Dr. Krishna Ram Amatya
5. Mr.R.R. Prasad

Processing of Crude Shilajit for Ayurvedic use

Shilajeet is one of the most important and prestigeous Ayurvedic drug. It is used for the treatment of many diseases such as hypertension, diabetes, genito-urinary infections, jaundice. According to Charaka, the great practitioner of Ayurvedic system of medicine, "there is hardly any curable disease which cannot be controlled or cured with the aid of shilajeet".

Shilajeet is an exudate on high altitude rocks of Himalayan region and is an exportable item of Nepal.

A small amonnt of it is processed by Singha Durbar Vaidya Khana (SDVK) and other Ayurvedic drug companies of Nepal. These companies employ traditional methods to process crude shilajeet into refined "soft" shilajeet and the period of processing varies from 40 days to 4 months. In view of long processing time, SDVK sought technical help from Royal Drug Research Laboratory to simplify the process and cut down the cost and time.

The aim of the study is:

- To study the traditional technology and develop a suitabl method for processing of crude shilajet
- To evaluate the process and provide samples for evaluation by SDVK.

Raw shilajeet, that is commerically available contains varying proportions of impurities stretching from rock pieces to very fine clay like substances of sub-micron sizes and needs extensive purifications prior to its use.

The main operations in its refinig involve:

- Crushing
- Extraction with water
- Filtration
- Concentration

The physical condition of the raw material available varied from soft to hard lumps. In case of hard brittle lot, a jaw crusher could be used. Since small lots of samples were made available, crushing has been done mannually.

To understand the physical characteristics of its dissolution, filtration and concentration, a few batches of processing have been carried out using water at ambient temperature.

The following problems have been faced during experimental studies both on bench scale and on pilot plant:

- Filtration due to the presence of sub-micron particles
- Severe frothing during concentration.

Both have succesfully been solved at bench scale and at pilot plant scale also.

Table I

	wt. of crude shilajeet Kg	wt. of soft extract Kg
1.	20	9.7
2.	97	36
3.	100	34
4.	50	21.5
5.	35	14

In order to imporve the processing method a few more batches were processed using water preheated to 50°C and maintaing it, stirring the contents mechanically.

Solid to liquid ratio overall 1:4
(Repeatedly extracted thrice at 50°C)

First - 1:2
Second - 1:1
Third - 1:1

The decanted solution was treated with 0.5% filtered aid and filtered through pressure leaf filter. The resulting filtrate was further treated with 0.25% filter aid and filtered through the processure leaf filter with out disturbing the filter bed formed during the previous filtration.The operation was repeated again with 0.1% filter aid and filtered.

This method resulted in filtrate practically free from any solid particles as tested in a laboratory super centrifuge at 15000 rpm.

The resulting filtrate on evaporation under vacuum (temp. 40-45^{o}C) gave very serious frothing problem.

This problem has been studied on bench-scale and successfully solved by carefully controlling vacuum and temperature.

The technique has been scaled-up to pilot plant level without any noticeable frothing problem.

All the operations could be accomplished in two days of 8 hrs. each.

Table II

	Batch Size Kg	Yield of Soft exratct Kg
1.	100	25
2.	100	35
3.	50	21.5
4.	35	12.0

The variations in batch size was due to quantity of raw material available.

The impurities contained in the raw material was not constant, the variations in the yields were expected.

Soft extracts of shilajeet obtained during the above studies were provided to SDVK for evaluation. Some samples were accepted as of good quality and remaining samples were being evaluated at the time of the expert's departure from Kathmandu.

This modified technique is recommended for adoption on an industrial scale.

Team Members

Mr. M.B. Narasimha- UNIDO expert

Dr. S.R. Adhikari

Mr. A.D. Shreshtha

Mrs. Rita Pandey

Mr. N.P. Shrestha

Mr. R.R. Prasad

Production of l-Dopa from mucuna seeds

Mucuna prurita commonly known as cowhage in English and Kauso in Nepali, is a herbacious annual plant belonging to Leguminosae family and grows wild in Terai region of Nepal. The beans of this plant are rich in l-Dopa, which is used in the treatment of Parkinson's disease.

Due to severe allergic quality of furry coat of the pods , some difficulty was experienced in the collection of seeds. It was found, that Brazilian variety has the pods without the allergic furry coat, this variety is reported to be under cultivation in Hetauda herbal farm.

The analysis of six different varieties of mucuna grown wildly in the Terrai region of Nepal showed l-Dopa content from 4-9%. The highest percentage of l-Dopa was obtained from Mucuna Pruriens which contained 9.2% l-Dopa , moisture 12.2% and oil 1.6%.

Twenty batches of 500 grams (each) of seed powder were processed for the extraction of l-Dopa. An average of 4.2% yield of B.P. grade product was obtained.

The results of the study are shown in the Table I.

Table I

	% of l-Dopa in raw material	% yield (crude)	% purity (crude)	% purity (pure)	Oil content		% of l-Dopa in marc	% of l-Dopa in mother liquor	Moisture content in seed	% of acetic acid left		
					l. Powder	marc				Solvent	Mother Liquor	Marc
Seed powder	9.2	..	..	..	1.6 w/w	1.78 w/w	..	..	12.2	..		
Extraction with 1% acetic acid containing SMS	..	4.25	98.18	100-101.2	..		2.7	1.075	..	0.643	0.257	0.0257
Extraction with 1% acetic acid without SMS	..	3.7	93.54	..	..		..	..	..	..		
Extraction with 2% acetic acid	..	2.6	70.45	..	..		..	..	..	..		
Extraction with water (25°C)	..	2.08	86.62	..	..		..	..	..	..		
Successive percolation by 1% acetic acid with SMS	..	3.11	88.06	..	..		..	..	..	..		

Battery system was also used with three glass percolators containing 250 gms (each) of mucuna powder , 1% acetic acid containing 0.2% sodium metabisulphate (anti-oxidant) as the solvent. Th results in Table II:

Table II

Recovery		Yield %	Purity %
Expt. I	1	1.13	86.36
	2	1.9	91.02
	3	1.82	90.44
Expt. II	1	1.72	89.86
	2	1.64	81.69
	3	1.28	96.27

The problem of frothing during concentration under vacuum, could be controlled, by carefully regulating vacuum and temperature.

Team Members

Mr. M.B. Narasimha-UNIDO expert

Mrs Sumitra Singh

Dr. Mrs. Timila Shrestha

Mr. A.D. Shrestha

Annex-1

The Status and Prospect of RDRL Pilot Plants at Godavary

M.B. NARASIMHA

UNDP/UNIDO Exspert in Process Technology

Introduction

Status

UNDP/UNIDO has created an excellent infrastructure base for conducting basic and applied research at RDRL, Thapathali and Godavary vide Project: DP/NEP/80/003, entitled "Strengthening tne Royal Drug Research Laboratory", a constituent of the Department of Medicinal Plants, HMG of Nepal, with a view to exploit the rich and varied flora, indigenous to Nepal.

A pilot section has been created at Godavary, with the following equipment to enable it to successfully develop, demonstrate and transfer, technologies in the industrial utilization of medicinal and aromatic plants:

1. Versatile Extraction Unit: Capacity 250 Litrs.
2. Sohxlet Extractor: Capacity 500 L
3. Percolator: Capacity 300 L
4. Three-Stage Mixer-Settler type Liquid-Liquid Extractor
5. Vacuum Concentrators/Distillation Stills:
   a. Capacity: 500 L witout stirrer
   b. Capacity: 300 L with stirrer
   c. Capacity:100 L with stirrer
6. Reactor (Hastealloy) capacity: 250 L
7. Distillation Still with stirrer: Capacity 1000
8. Pressure leaf filter
9. Rotary vacuum filter

10. Spray Drier

11. Hammer Mill

12. Jaw Crusher

13. Steam Boiler: Capacities 1500 kg/hr from and at 100°C, Oil fired-automatic

All the items, except, item nos. 10 to 13 have been supplied by M/S. Tournaire, France.

In addition to these units, the following equipments installed at the premises of RDRL at Thapathali prior to the commencement of the Project are also avaible:

1. Stainless stell essential oil distillation unit: Capacity: 2000 L
2. Glass-lined reactor with stirrer: Capacity: 100 L
3. Stainless steel reactor with stirrer: Capacity: 250 L
4. Electrically heated distillation unit: Capacity: 200 L
5. Stainless steel basket centrifuge
6. Shelf drier: 24 aluminium trays
7. Disintegrater
8. Steam boiler-oil fired, automatic capacity 300 kg/hr from and at 100°C.

With these combined facilities and the infrastructure base built within the scope of the project at RDRL, R and D work for the development of process technologies for the production of natural products is in full swing.

Shortcomings

However, some shortcomings as enumerated below, have been noticed in some of the units at Godavary:

1. Versatile Extraction Unit

No arrangements exists for refluxing, part of condensate into the packed column, which is very necessary for the rectification of dilute recovered ethanol, thus, restricting the use of this unit for extractions with water immicible solvents only.

2. Percolator has not been provided with a system for the recovery of residual solvent from the marc, thus limiting its use with aqueous solvents only.

3. Three-stage mixer settler type liquid-liquid extractor is incomplete and inoperable in absence of pumping arrangements, to pump miscella and raffinate streams to the distillation stills for the recovery of solvents and extracts.

Suitable action has been initiated to effect modifications to these units, as also minor modifications of the following units to make their use more flexible and broad based.

1. Two vacuum concentrators of 300 L and 100 L Capacities fitted with stirrers, are planned to be converted to act as reactors in addition to their use as vacuum concentrators with minor modifications.

2. Sohxlet Extractor:

This unit takes about 10-15 hours per batch. To cut down the batch time and also with a view to extend its function as a percolator the pipeline conections are planned to be modified, to facilitate circulation of miscella, using the existing solvent pump.

Additional Equipment Suggested

1. Fractional distillation unit of the capacity of about 100 L per batch, equipped with high efficiency internal packings, reflux distributor with electronic timer, vacuum pumps, interconnecting pipes and M.S. structures. Expert in process technology can help in its design and getting it fabricated locally.

2. Multistage centrifugal type liquid-liquid extractor, with provision for separation and clarification bowls with accessories.

Design and Engineering

It is suggested that, while developing technologies for the industrial utilization of indigenous medicinal and aromatic plants, simultaneous development of design and engineering expertise and suitable infrantructure for fabrication of chemical plant and equipment, would not only quicken the process of transfer of technologies from pilot plant level to the user industry, but saves valuable time and foreign exchange and hence a compulsive need of a developing country like Nepal.

These activities may be developed, in a phased manner. In the first phase the expertise development may cover basic design of plant and equipment and have the plants fabricated in the engineering workshops in Kathmandu. At a later stage if found necesarry, fabricational facilities may be built.

Staff

The present staff of Pilot plant consists of four qualified Pharmacists, a physical chemist and two technicians. Considering the nature of developmental work, and suggested augmentation of design and engineering facilities, it is an absolute necessity to have at least a couple of graduate chemical Engineeres on its rolls.

Process-Control at Godavary

The process development and scale up operations are being conducted at Godavary and the samples are being sent to Thapathali for analysis, very essential for process-control, this arrangement is impractical and leading to avoidable delay in the development of technologies. The proposed Process-Control laboratory, for which I understand the funds were approved in the TPR held in 1986 and the equipment ordered subsequently, should be established at the earliest; without this laboratory it would be difficult to operate the pilot plant efficiently.

Prospects

With the suggested modifications to the existing pilot plants, shifting of all the pilot plants from RDRL premises at Thapathali to Godavary, addition of the afore-mentioned equipment and augmentation of technical staff, the pilot plant facilities at Godavary would form the best possible technical base, to generate and transfer technologies not only in the commercial utilisation of broad spectrum of aromatic and medicinal plants of Nepal, but also in the process development of synthetic drugs and pharmaceuticals.

General Remarks

Applied research as compared to basic research is capital intensive, hence prior to undertaking pilot plant studies, the results obtained at bench-scale, should be subjected to indepth evaluation, for technical feasibility by competent scientists and technologists, upon their recommendations for technical feasibility and national priorities, pilot plant

studies are to be undertaken for in-depth studies to establish not only technical feasibility but also economic viability of a new process know-how, and to obtain sufficient data for scaling-up to industrial operations.

Suitable methodology, has to be developed for periodic review, course correction, when necessery, proper checks and controls during the stage of development.

A pilot plant section like the one at Godavary with built-in infrastructure to undertake applied research, is expected to successfully develop, demonstrate and transfer technologies and act as a nerve centre:

- to generate technically feasible and economically viable technologies in the utilisation of natural products.
- to provide R and D facilities to the industry.
- to provide consultancy and advisory services.
- for training and development of technical manpower.

For the above services, a lumpsum premium/charges should be collected from the user industry.

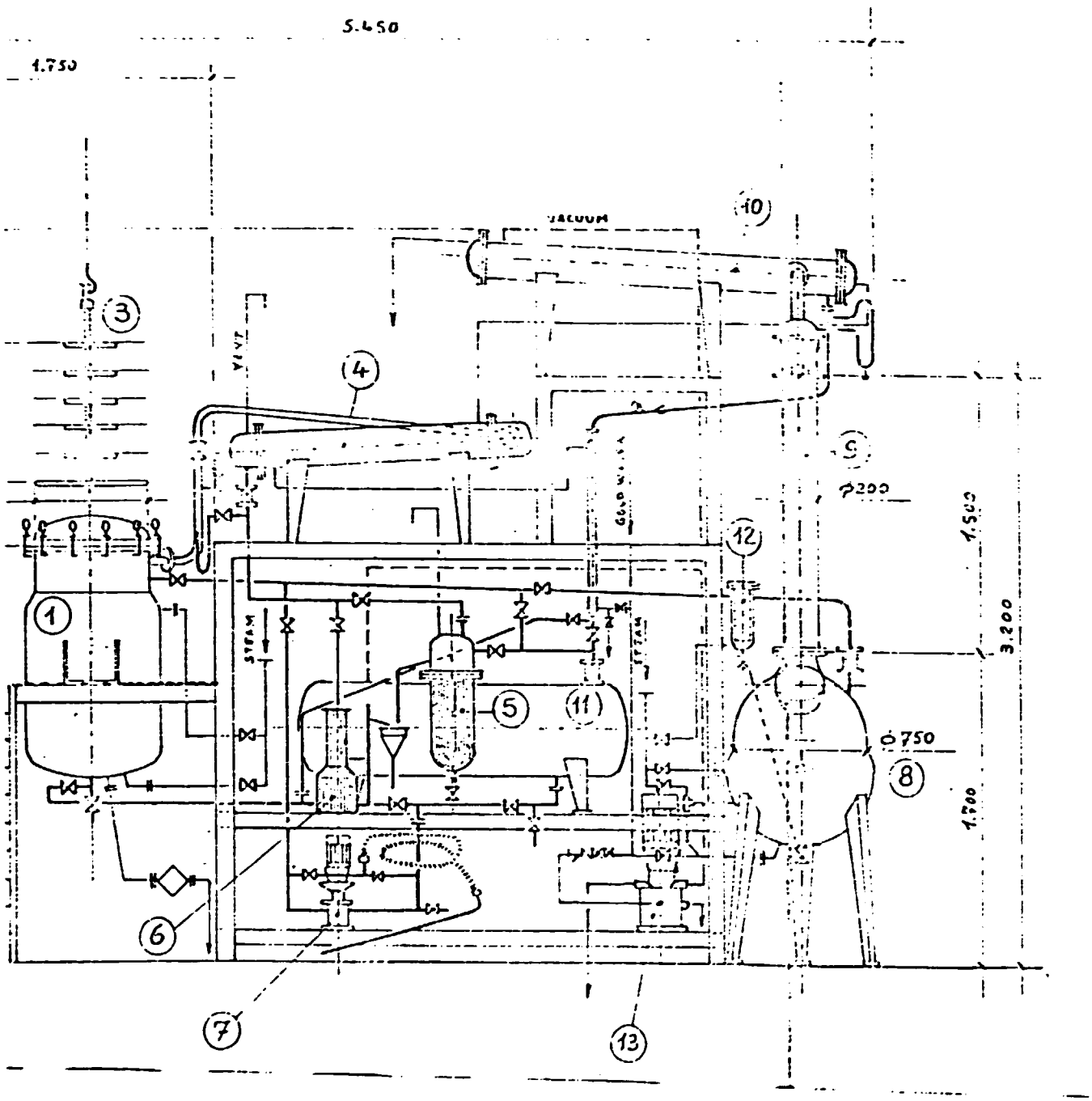
After establishing, credibility of the capacity to develop know-how and competence of its scientific and technical personnel in successfully transfering the know-how in establishing small and medium scale industries, industrial enterpreneurs would come forward to sponsor programmes to generate know-how/technologies.

Annex II

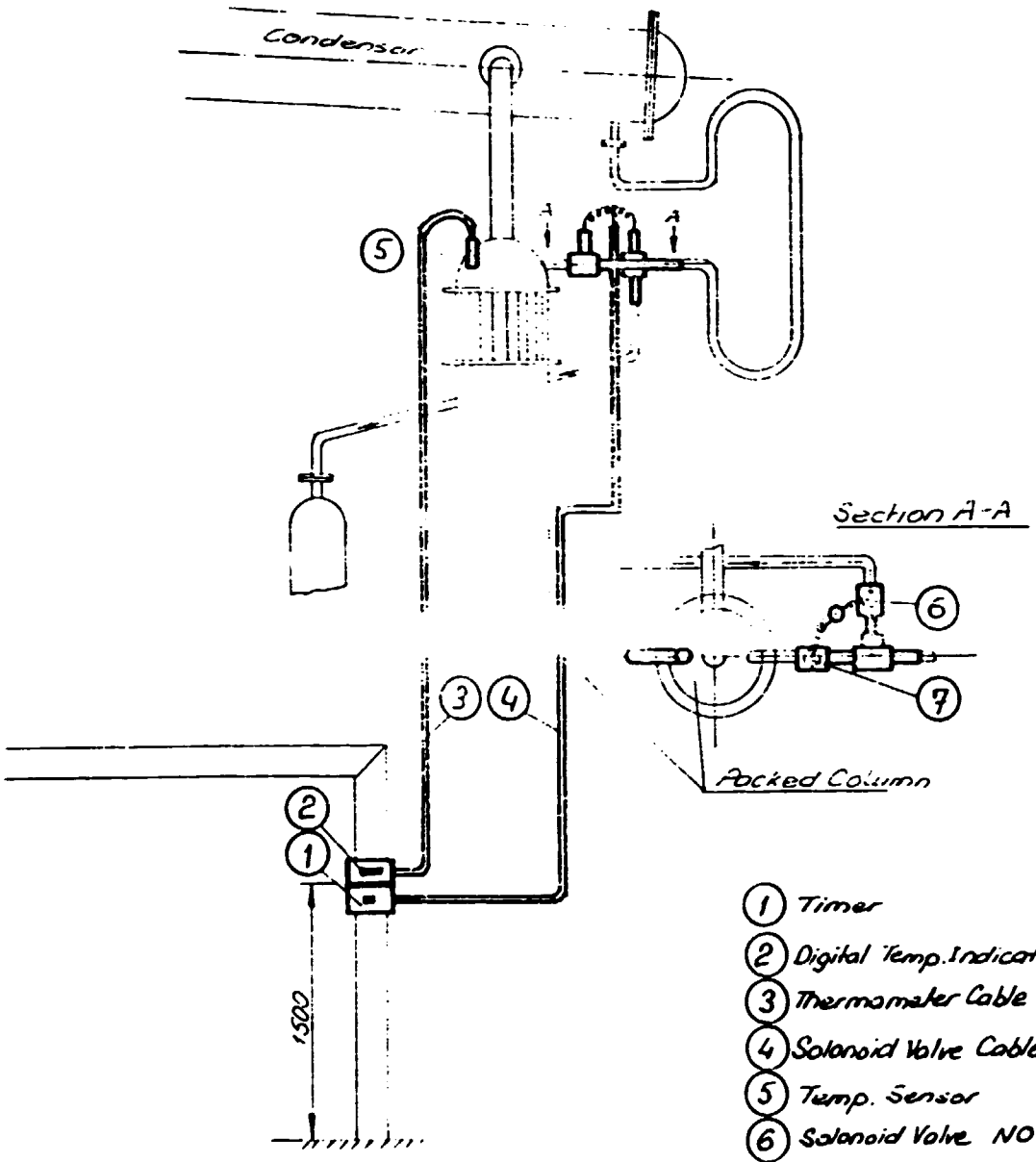
Suggested modifications to pilot plants at Godavari.

With a view to enhance the versatality and operational flexibility of the pilot plants installed at Godavari and to overcome certain short comings as enumerated in a note "The status and prospects of RDRL pilot plants at Godavari" certain modications as detailed in the enclosed drawings are recommended.

Two sets of drawings-showing existing set-up and suggested modifications are enclosed.

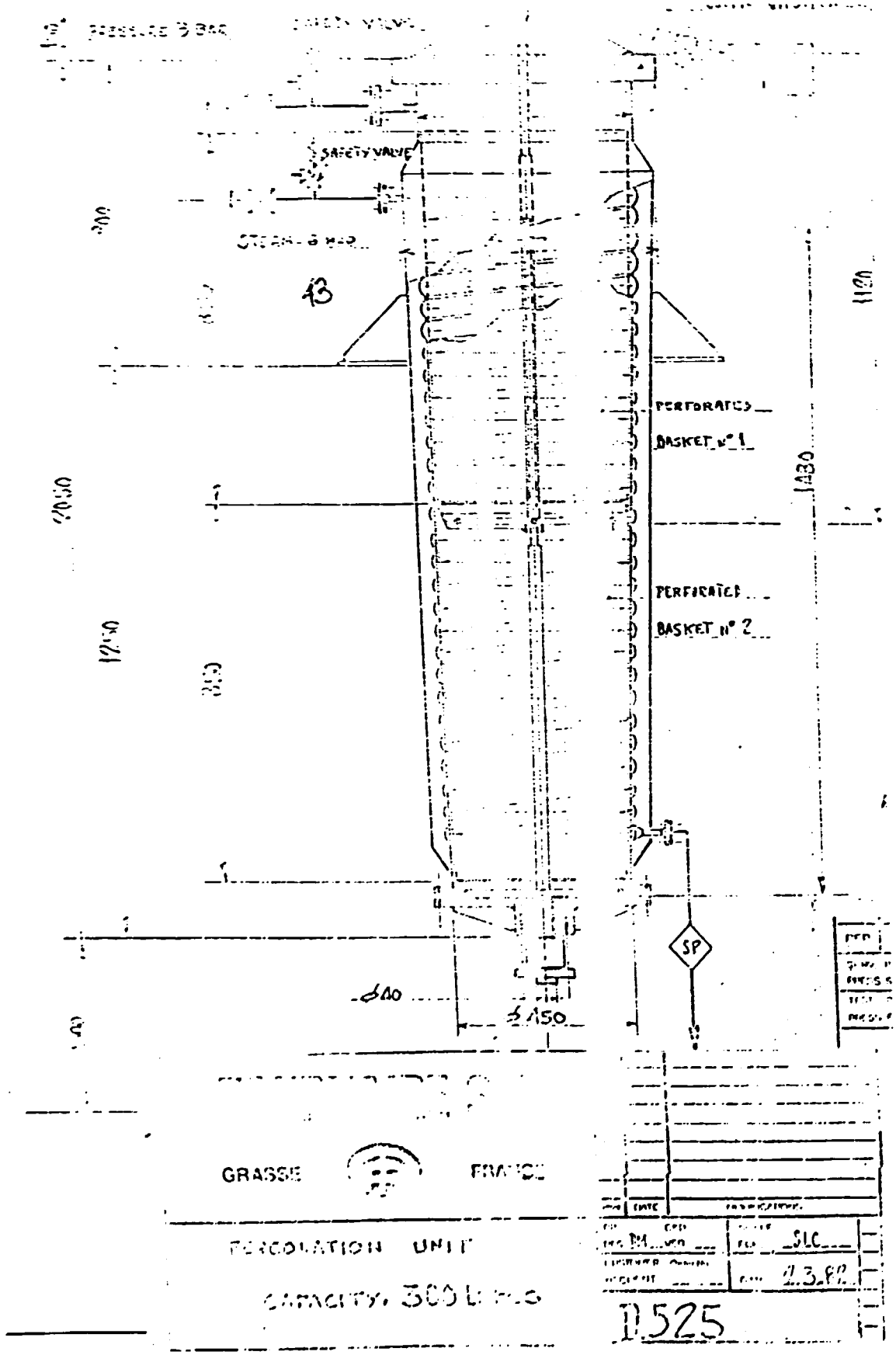


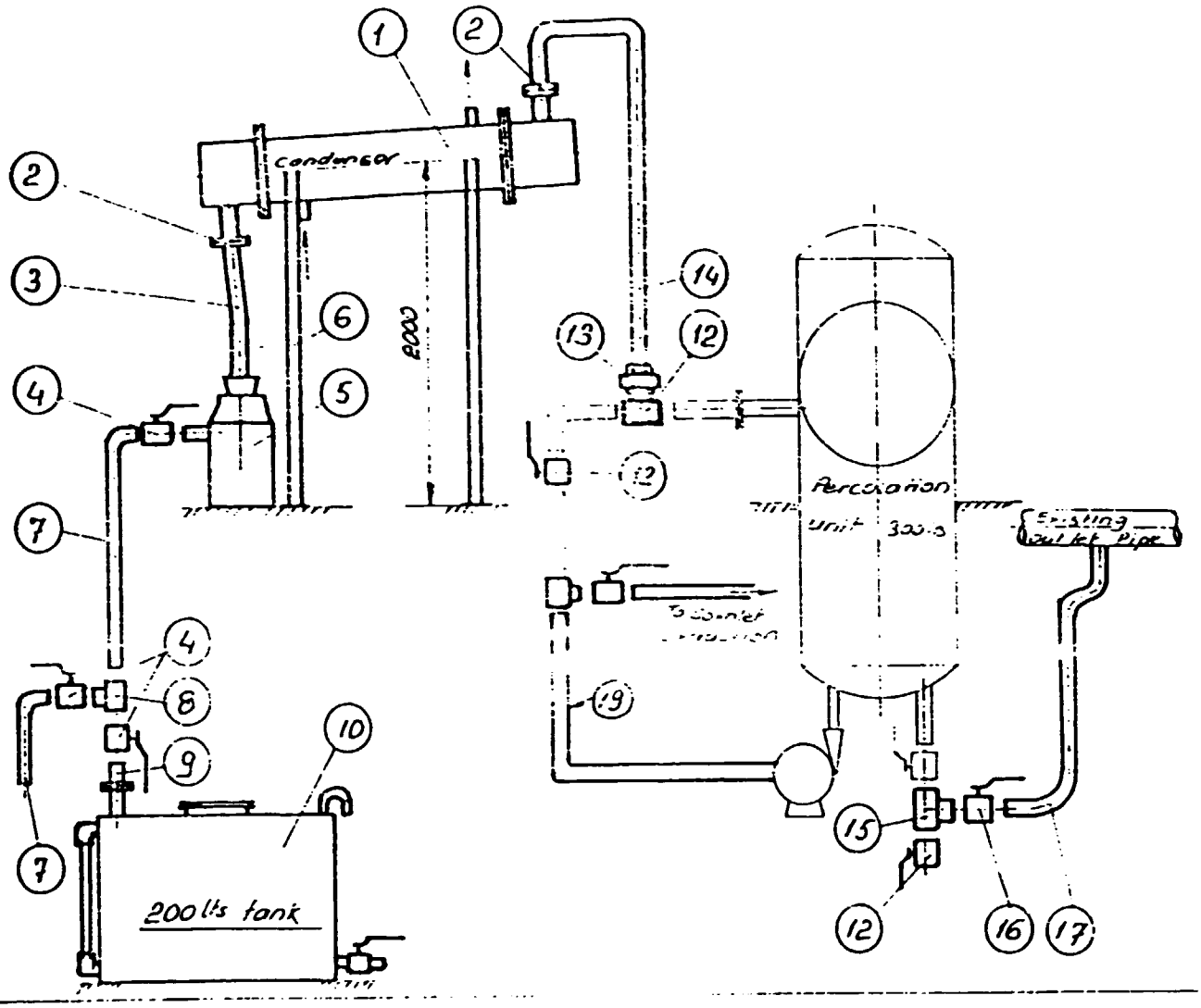
TOURNAIRE S A		GRASSE FRANCE
VERSATILE EXTRACTION PLANT		DATE 30-9-82
		DR M.R



(1)	Timer	1pc
(2)	Digital Temp. Indicator	1pc
(3)	Thermometer Cable Pipe	
(4)	Solenoid Valve Cable Pipe	
(5)	Temp. Sensor	1pc
(6)	Solenoid Valve NO Type	1pc
(7)	Solenoid Valve NC Type	1pc

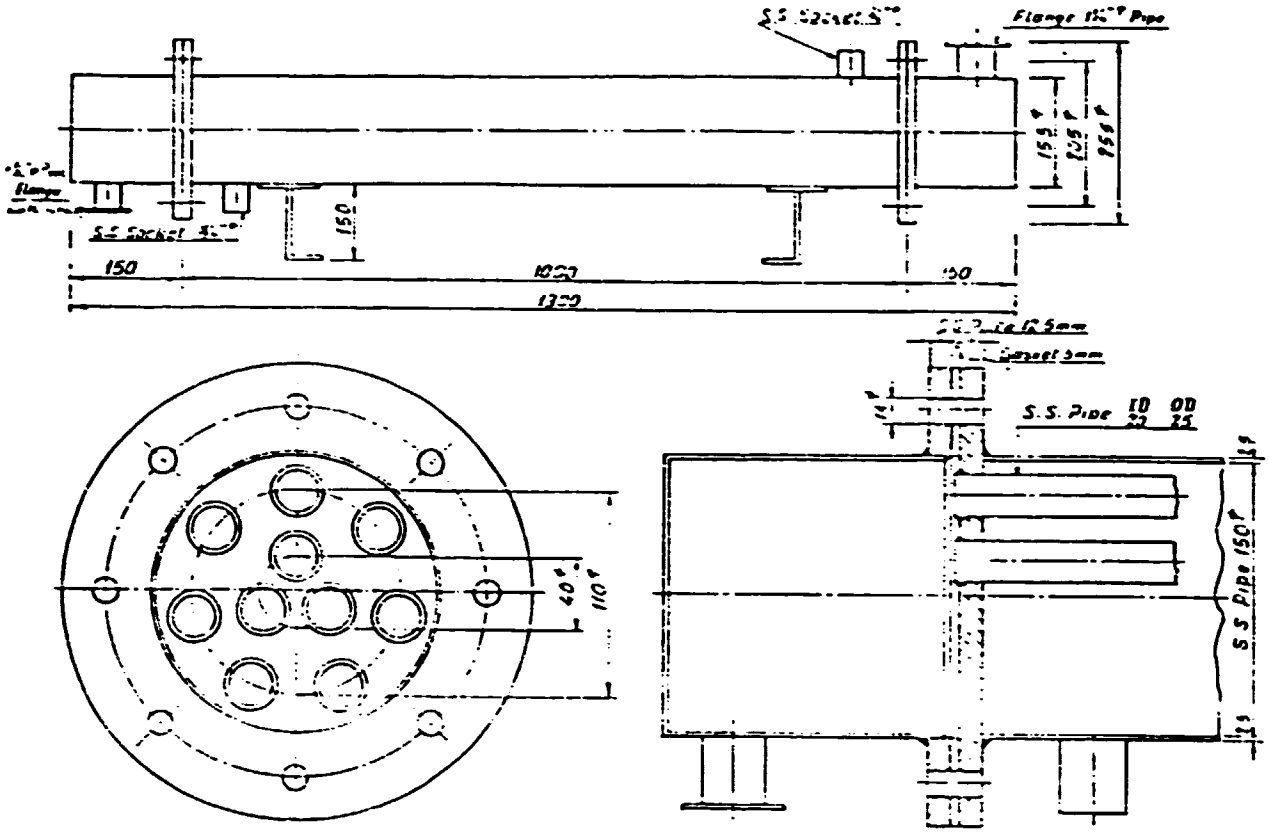
Note: Modification is done acc.to detail provided by Mr. M.B. NARASIMHA
DA/NEP/BO/003/11-05


STEAM 3 BAR
SAFETY VALVE
PERFORATED
BASKET N° 1
PERFORATED
BASKET N° 2
SP
GRASSE
FRANCE
PERCOLATION UNIT
CAPACITY: 300 L P.C.
SLC
2.3.82
D525



(1)	Condenser	1 set	(8)	Tee Joint ø1"	1 pc	(14)	Pipe ø 1¼	
(2)	Flange ø 1¼"		(9)	Nipple with Flange	1 pc	(15)	Tee joint ø1½" to 3/4" ø	1 pc
(3)	Nipple with Flange	1 pc	(10)	Tank	1 pc	(16)	Valve ø3/4"	1 pc
(4)	Valve ø 1"		(11)	Tee joint ø½"	1 pc	(17)	M.S. Pipe	
(5)	Separator	1 set	(12)	Valve ø 1½"	2 pcs	(18)	Tee joint ø1½" to 1" ø	1 pc
(6)	Cond. Stand	1 set	(13)	Union ø 1¼"	1 pc	(19)	Pipe ø1" GI 33 SO 27	– 5 m.
(7)	Pipe ø 1"							

Note: Modification is done acc. to detail provided by Mr. M. B. NARSIMHA DP/NEP/30/003/1105



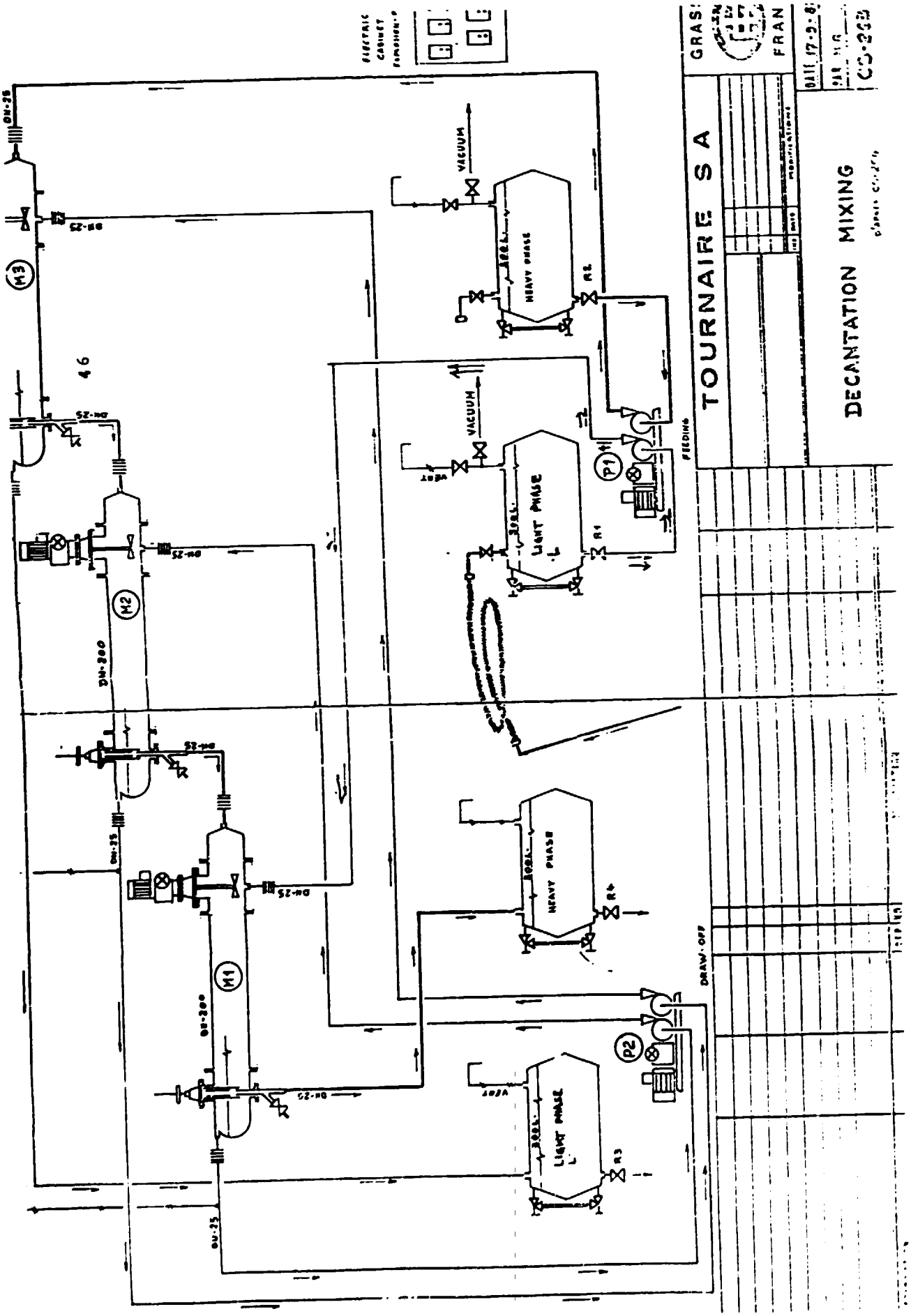
No. of Pipes 10 pcs. (seamless tube)

Tube dia OD 25 ϕ mm

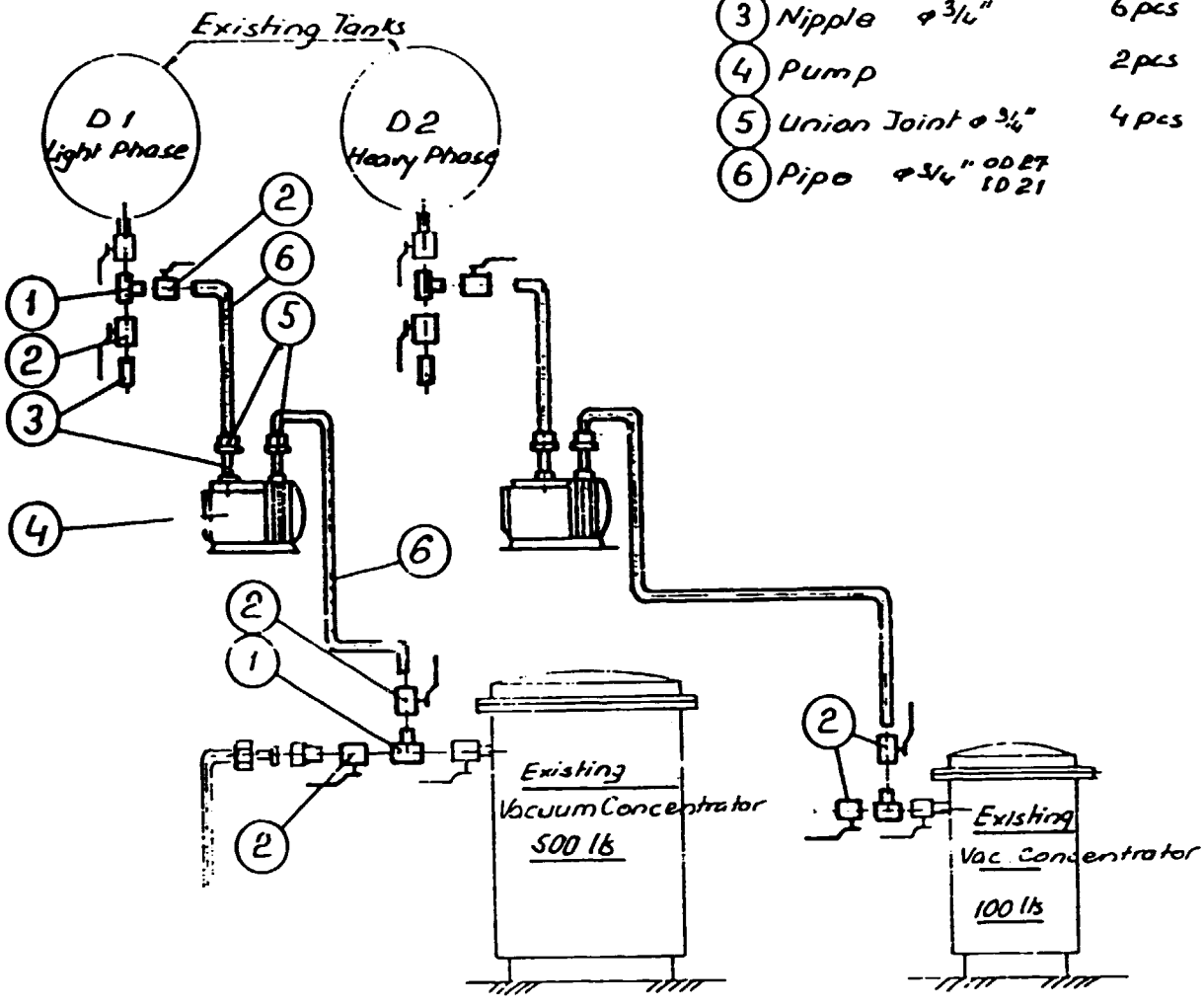
ID 20 ϕ mm

- Material: Stainless Steel 304 / 316
- for Recirculation Unit D525

Suggested By : M. B. NARASIMHA.

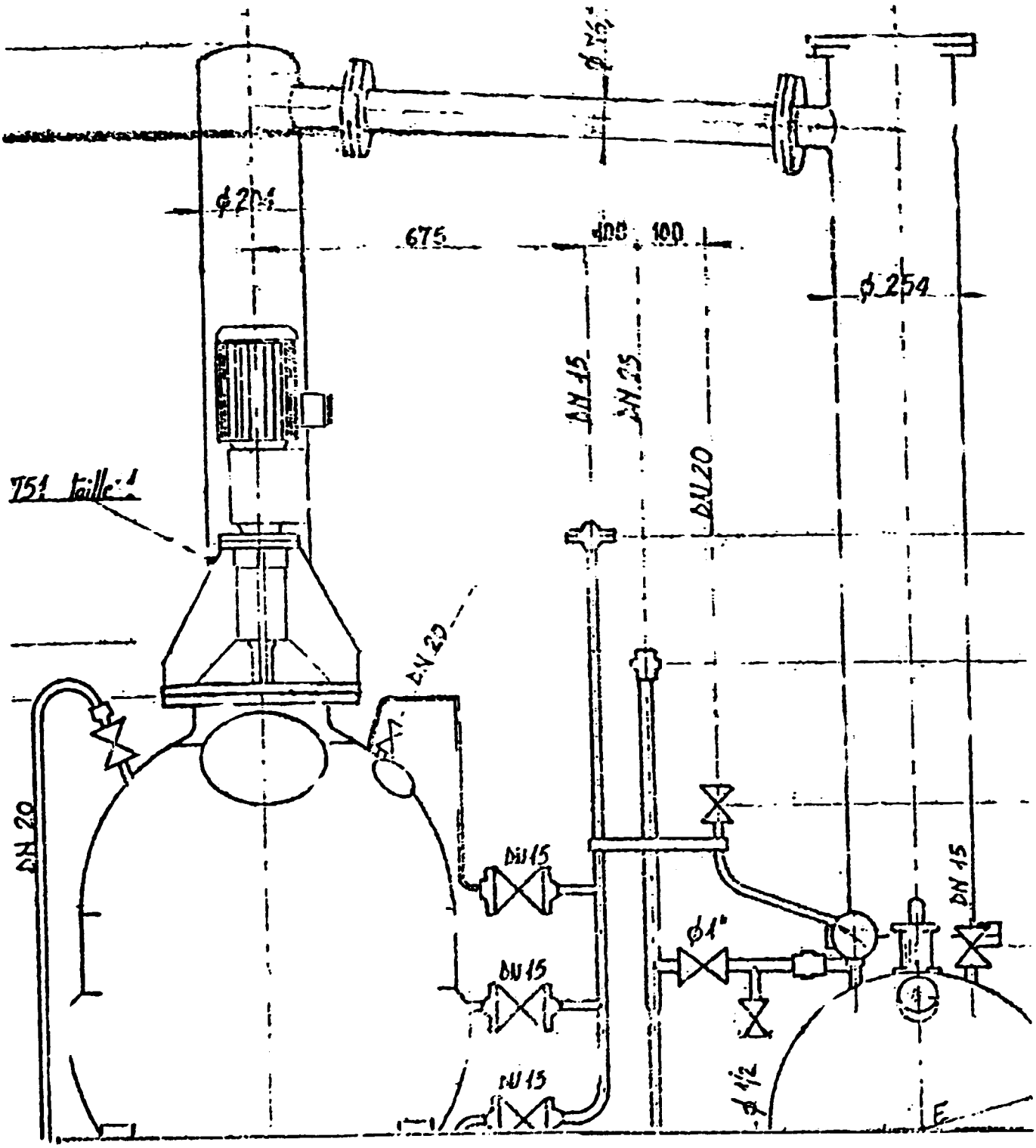

TOURNAIRE S A
DECANTATION MIXING
VACUUM
HEAVY PHASE
LIGHT PHASE
FEEDING
DRAW-OFF
M1
M2
M3
P1
P2
R1
R2
R3
R4
DN-25
DN-200
46

1	Tee Joint ϕ 3/4"	4 pcs
2	Valve ϕ 3/4"	8 pcs
3	Nipple ϕ 3/4"	6 pcs
4	Pump	2 pcs
5	Union Joint ϕ 3/4"	4 pcs
6	Pipe ϕ 3/4" OD 27 ID 21	

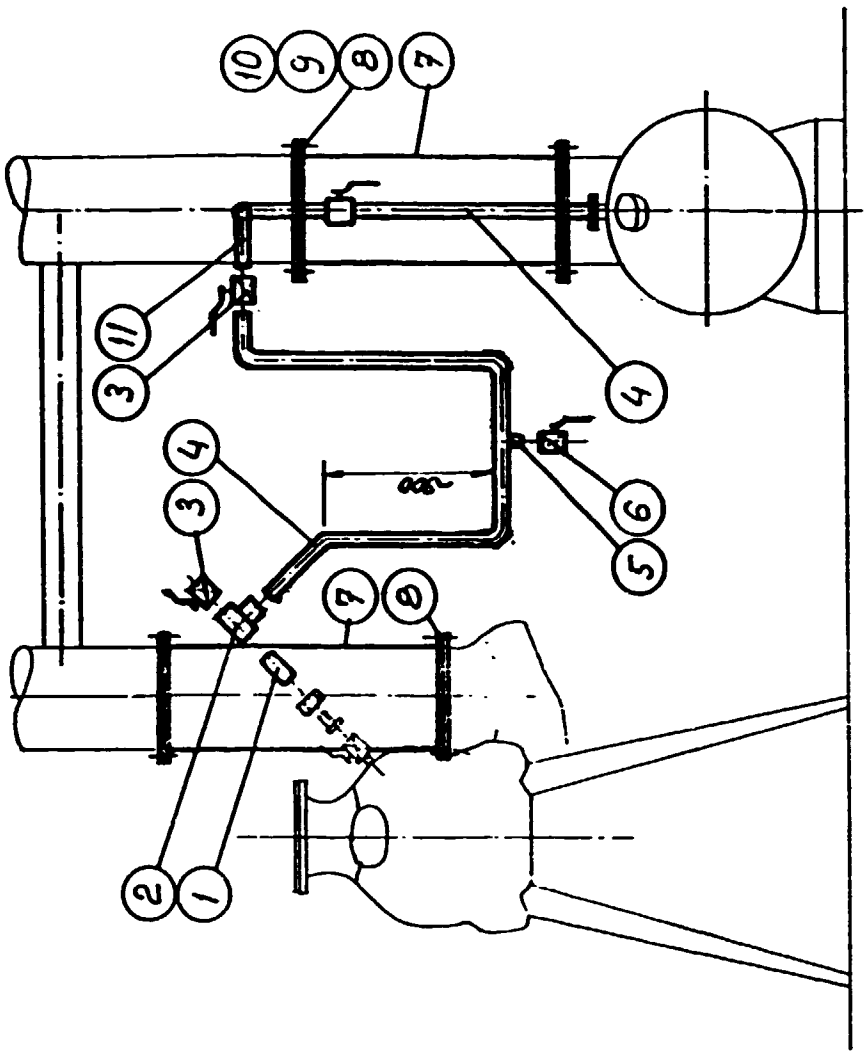


Liquid/Liquid Extractor to Vacuum Concentrator

Note: Modification is done acc. to sketch provided by Mr M B NARASIMHA
DP/NEP/BO/003/11-05



VACUUM CONCENTRATOR
TYPE 507

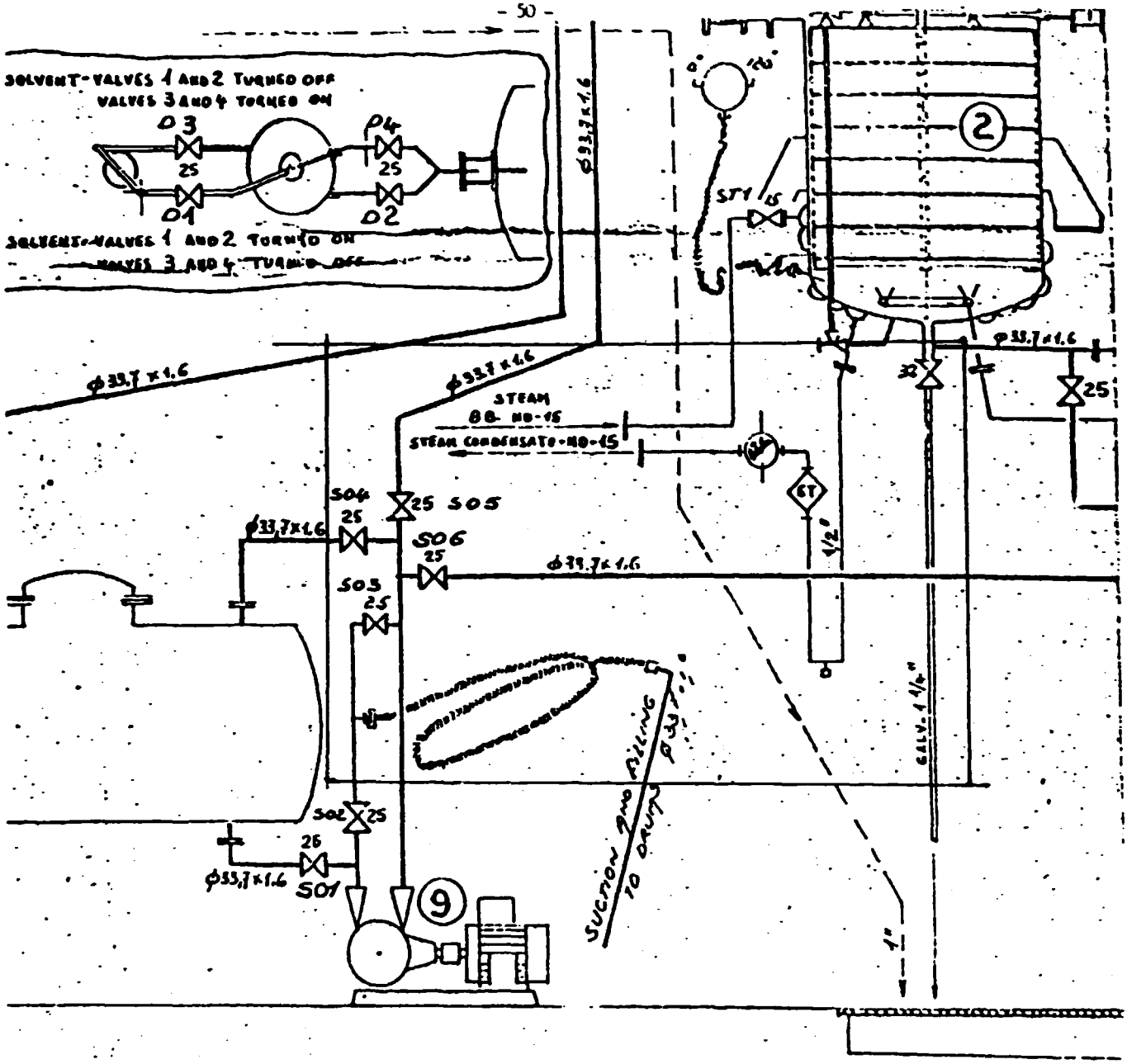


7	Pipe 8'ø OD 20ø ID 19ø	2pcs
8	M.S. Flange ø300	4pcs
9	Fire Fly Gasket 5mm thk.	2pcs
10	M10 Bolt x 50 with Nut	16sets
11	Nipple ø3/4"	1pc

1	Nipple ø3/4"	1pc
2	Tee Joint 3/4"ø	1pc
3	Valve ø3/4"	3pcs
4	Pipe ø3/4" OD 27ø ID 21ø	
5	Pipe Nipple ø1/2"	1pc
6	Valve ø1/2"	1pc

Note: Modification is done acc. to detail provided by Mr. M. B. NARASIMHA
DPINEP/BO/1003/11-05

VACUUM CONCENTRATOR
TYPE 507
Gal Assembly



REP	NB	DESIGNATION	OBSERVATIONS
SERV. PRESS. ____ / PRESS SERV. ____	CAPACITY : ____ GAL / CAPACITE : ____ L		CALCUL SUIVANT CODE ____
TEST PRESS. ____ / PRESS EPREUVE : ____	SURFACE : ____ SQ.FD / SURFACE ____ M²	WT. ____ LB / POIDS : ____ KG	STAMP : ____ / RECEPTION : ____ — DESIGN TEMP. ____ °F / TEMP. DE CALCUL ____ °C

TOURNAIRE S.A.

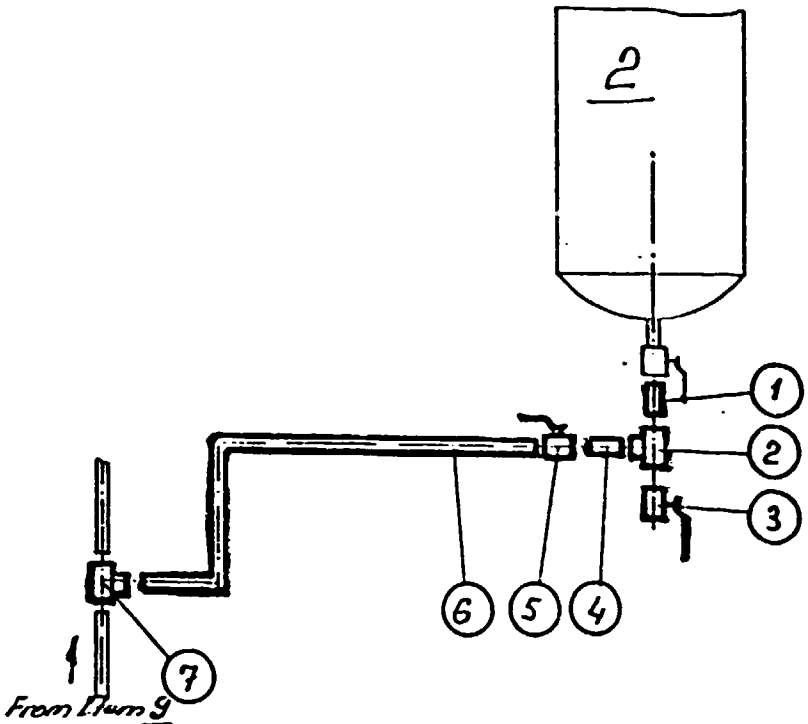
GRASSE FRANCE

A	4/mb	Mise à Jour
INDICE	DATE	MODIFICATIONS

EXTRACTION UNIT TYPE SOXHLET

DR CKD. / DES ____ VER. ____	SCALE / ECH. : 1 ____
CUSTOMER Order No / N° CLIENT : ____	DATE 2-11-82

Soxhlet Extraction



(1)	Nipple ϕ1"½"	1pc
(2)	Tee joint ϕ1"¼ to 1"	1pc
(3)	Valve ϕ1"½"	1pc
(4)	Nipple	1pc
(5)	Valve ϕ1"	1pc
(6)	Pipe ϕ1" OD 33 SD 27	
(7)	Tee joint ϕ1"	1pc

TOURNAIRE S.A.

GRASSE FRANCE

A	11/86	Mise à Jour
marks	DATE	MODIFICATIONS

EXTRACTION UNIT TYPE SOXHLET

FLOW-SHEET

DR CKD
DES VER.
CUSTOMER Order No
N° CLIENT
SCALE
ECH. : 1
DATE 2-11-82

T-437 A

List of required materials for Godavari Pilot Project

1. Stainless steel 316/304
   a. seamless pipes
   b. Nipples
   c. Unions
   d. Tee Joints
   e. Plain sheet

2. Stainless steel Ball valve, flanges type.
   Teflon sealing with lever operated S.S.RB. 7210

3. Centrifugal Pump. Positive suction Head 5 to 10m
   Flame proof motor
   Flame proof starter
   Pipe size ø 3/4"
   Capacity 1/2 HP

4. Timer switch adjustable.
   Time setting ration. 0:60, 5:55, 10:50, 15:45
   20:40,25,35, 30:30, 35:25
   40:20,45:15, 50:10, 55:5
   60:0

   All electrical fittings controls should be flame proof.

5. Digital temp. indicator, with extension cable 4m long.

6. Temperature sensor, range 0^0 - 150^0c

7. Soloniod valves
   Energize to close type
   Energize to open type
   AC Volt 220V
   Working pressure 3 bar
   Valve pipe ø 3/4"

   - 2 nos. Solenoid valve
   - 1 no. each
     1. stainless steel
     2. vacuum 20mm absolute

8. The items for Percolation Unit will be fabricated at BYS as per your requirement out of stainless steel sheets 316/304.

9. a. Seperator will be made as per your provided sample.
   b. 200 lts tank will be made as per BYS sketch 3 out of 3,15mm sheet.
   c. Condensor will be made as per BYS sketch 2, out of seamless pipe, 12,5mm S.S. flange with cover of 2,5mm sheet.

Annex-III

A note on Safety Precautions in Solvent (Volatile) extraction plants.

Solvent extraction is an unit operation employed to extract solutes from solute bearing materials with a solvent. The solvent diffuses into the cells of the material and dissolves the solute, resulting in miscella. The miscella when subjected to simple distillation, separates into its constituents-solvent and solute.

The most commonly used solvent at every stage-bench scale, pilot plant and in Industry is, n-Hexane, which is higly inflammable. Due to hazards of inflammablity it is imperative that the design of solvent exraction plants should aim at elimination of any hazard and make it safe for operation.

For any solvent to catch fire, it is necessary that a certain proportion of solvent vapour and air should exist and enough heat to start the combustion reaction.

It has been reported that flammable limits (n-Hexane) is 1.2% minimum and 6.9% maximum. The mixtures below and above these concentrations are considered safe. The combustion temperature is 260^{o} C.

Precautionery Steps:

1. All outlet connections of condensers, heat exchangers should be lead though a single cold trap or breather.
2. The vent of gases must be located at a height of 6 m. above the plant. The venting lines should be of sufficient size and with out valves.
3. The buildings housing the plants should be well ventilated.

It is recommended that the commerical extraction plants should be open-air type, just sufficient roofing to protect against climatic conditions.

4. All heating operations in the plant are to be carried by steam only.
5. Bulk solvent storate tank should be iocated at least 10 m from the plant, dwellings and thoroughfares and should be buried under the ground and fenced all around.
6. All electrical appliances such as motors, starters, light fittings, cables should be of flame proof type.

Visit to M/S KOSKA Helvacisi
İSTANBUL

M.B. NARASIMHA
Prof. Dr. K.Hüsnü Can BAŞER
Chem. Eng. Temel ÖZEK

Persons contacted
1. Mr. Nevzat Dindar
   Owner
2. Mr. Cevat Pehlivan
   Partner

Extn. Method followed:

50 kg of crushed *Gypsophila* boiled with 200 litres of water for 3 hours in an open pan fitted with a perforated plate covered with wire mesh at the bottom. About one half of the extractor in placed in a brick furnace which is oil fired.

The extract thus obtained is concentrated in another unit similar to the one as above for 3 hrs. This is then used in making helva.

The solution analysed at TBAM gave about 13.57 % solid content and density d_{20}= 1.05

Improved Method:

50 kg of crushud *Gypsophila* boiled with 150 litres of water for 2 hours in an open pan. The extract thus obtained is designated as A. Subsquently it is extracted twice with 150 liters of water for 2 hours each, the extracts obtained aredesignated as B and C. The marc is discarded.

A fresh batch of 50 kg of *Gypsophila* is extracted with the extract "A" to obtain concentrated extract "H1", followed by extractions with B,C and fresh water respectively. The resulting extracts designated as A1,B1 and C1. The marc is discarded. The procedure is repeated with fresh solid of *Gypsophila*.

The procedure is indicated below.

	1. Boiling	2. Boiling	3. Boiling	4. Boiling
1. Batch	W ==> A	W ==> B	W ==> C	----
2. Batch	A ==> H_1	B ==> A_1	C ==> B_1	W ==> C_1
3. Batch	A_1 ==> H_2	B_1 ==> A_2	W ==> B_2	W ==> C_2
4. Batch	A_2 ==> H_3	B_2 ==> A_3	W ==> B_3	W ==> C_3
5. Batch	A_3 ==> H_4	B_3 ==> A_4	W ==> B_4	W ==> C_4

This procedure demonstrated of the Company Premises at İstanbul has resulted in the following advantages.

- Concentrated extract obtained is directly used in helva making.
- Additional operation of evaporation, evaporator, energy and labour are avoided, thus affecting substantial savings.
- The battery system demonstrated has the additional advantage of thoroughly exhausting the *Gypsophila*, before being discarted.
- M/S Koska Helvacisi is following this procedure regularly.

7. To avaid genention of static electricity, the electrical continuity between any two points in an extraction plant should be ensured and earthed (the entire plant) at least two places.
8. Suitable instrumments should be installed at strategic places to measure, monitor, record, concentration levels of solvent vapour in the plant area.
9. Lightening arresters should be installed at suitable points and properly grounded to earth.
10. Suitable and adequate fire-fighting equigment at strategic points in and around the plant, should be provided.