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DP/ID/SER. B/654 10 July 1989 ORIGINAL: ENGLISH

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ASSISTANCE FOR THE PRODUCTION OF PLANT DERIVED PHARMACEUTICALS

DP/URT/81/026

TANZANIA

Terminal report*

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

Based on the work of Mr. Shahid Ahmed Pilot Plant Engineer

Backstopping Officer: Dr.R.O.B. Wijesekera, Chemical Industries Branch

United Nations Industrial Development Organization Viewna

^{*} This document has not been edited.

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1. INTRODUCTION

The report under review gives a summary of activities findings and recommendations of the pilot plant Engineer whose duration of mission was 1.5m/m from 20th Feb, 1989 to 4th Apr.1, 1989

The mission was aimed to overcome certain shortcomings which have aroused during the operation of the pilot plant and to expand the operation by trial running of more products. In addition to these the objectives were training on maintenance and some local modifications to make the plant more versatile.

This multipurpose plant under review was installed in 1984 and commissioned in November 1986 comprising of the following:

- 1.1 Chopper or Mill to chop fresh or dried leave, whole herb and bark.
- 1.2 percolator or Extractor to make solvent or aqueous extract from medicinal plants
- 1.3 Soxhlet Extractor to produce solvent extracts
 with undiluted solvents
- 1.4 Distillation still with agitator for steam distillation, solvent rectification and vacuum extraction/concentration
- 1.5 Nutch filter for filtration of extracts by using fabric filter element.

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- 1.6 Condensor to be used in steam distillation, vacuum concentration, soxhlet extraction, solvent rectification etc.
- 1.7 Pecked column to avoid priming during distillation under vacuum
- 1.8 Receiving wessel to collect recovered solvents
- 1.9 Sedimentation tank to remove and decant immisible fluids
- 1.10 Florence flask to be used in steam distillation as separation vessel
- 1.11 Vacuum pump with buffer tank to be used in soxhlet extraction, transfer of solvent and during solvent recovery.

The entire plant was not operational due to various defects in operation. National Management tried to resolve the problems using their own means. They also hired Tanzania Industrial Research and Davelopment Organisation (TIRDO) to repair the plant. TIRDO Engineers identified the problems but nothing was done practically. The Engineer from UNIDO was requested, thereefter, to do the needful.

2. DBJECTIVES AND LUGICS

Keeping in view the technical know-how and facilities available locally the assistance in plant engineering was requested. The following objectives were to be achieved.

- 2.1 Repair the plant so as to make it operational
- 2.2 Modify certain plant's components to improve work flow
- 2.3 Operate at least one batch each of the product to appraise the working of the equipment and removing any shortcoming thereof.
- 2.4 Training of operators and maintenance staff on operation and maintenance functions.

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2.5 Modification of the plant layout

3 ACTIVITIES AND OUTPUTS

Under this, the activities and outputs are discussed with reference to job description so as to appraise the actual quantum of work done and the factors hindering the smooth flow of interlinked activities. The comments are given with each work.

- 3.1 Study present pilot plant on the basis of recommendations of terminal report of the project
 - Dr. M. Alauddin's terminal report covers followings on plant and equipment
- 3.1.1 Some equipment like crushing/grinding machine, vacuum cleaner should be procured
- 3.1.2 Steam supply of 20kg/hr at a pressure of two bars should be ensured.
- 3.1.3 Water supply to the vacuum pump should be directly linked with the mains. To produce Quinine sulphate from Cinchona Bark and other alkaloids isolation, some reactors and digestion vessels are required. The reactors and digestion vessels should be suitably lined to make these acid/alkali resistant.

 The sizes would be determined after assessing the natch size.

The pressure reducing value has been repaired and steam supply of 2 bar to distiller has been ensured. Additionally suitable safety—value has been suggested to release steam whenever the pressure increases from the present limit. The safety—value would be installed with the proposed changes in steam.

the many line which would be in progress soon, the time

It is however recommended for the safety of the equipment that the plant should not be operated without safety valve.

The water to the vacuum pump appears to be sufficient after repair of the PRV as the water requirement to the condensor reduced considerably.

- In addition to the terminal report of Dr. M. Alauddin, the Director of Muhimbili Traditional Medicine Research Unit(T.M.R.U) also sought help from Tanzania Industrial Research & Development Organization (T.I.K.D.O) and following recommendations were made.
- 3.1.4 No water should be added in the still when carrying out steam distillation of essential oils.

 The writerdoes not agree with this recommendation as a general rule. Moistening of some materials before steaming yields better results.
- 3.1.5 The column should be hollow during extraction of essential oils.

 This has already been done, Ways and means to bypass the packed column were also considered so the vepours coming out from the still are condensed immediately. It does not look possible within the plant configuration to do this. As the plant is a multipurpose experimental limit, the effeciency is not a criterion.

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- 3.1.6. The pressure vessel (ie the still) should not be operated without a pressure gauge.
 The pressure gauge is already on the still and in working condition. The gauge has however been tested for proper working.
- temperature of the condensor.

 The water temperature at the inlet of the condensor as an average remains at 30°C. The condensor has been adequately sized to condense vapours. The several trials on steam and vacuum distillation indicated that incoming water supply at 30°C is sufficient enough. It is, therefore, not necessary to cool water to condensor inlet (15 = 20°C) as recommended. The inference from TIRDO as due to faulty pressure reducing valve giving steam at full supply pressure (10 = 15 bars). The valve has been repaired and a safoty valve is to be installed.
- 3.1.8 Defective pressure reducing valve should be replaced.

 The pressure reducing valve has been repaired and now working properly.
- 3.1.9 Routine maintenance should be planned for the unit,
 - A preventive maintenance programme has been written and proper training to the maintenance staff has been imparted.
- 3.1.10 All pressure and temperature gauges should be tested for their correctness and defective gauges should be replaced

Both temperature gauges were found defective. This was due to dry thermal well. A proper thermal fluid was added and readings were taken and compared with laboratory thermometers. The readings were identical which indicated that thermometer are working fine and it was the dry thermal well causing wrong readings. One pressure gauge yielded because of overpressure which has been repaired and recaliberated.

- 3.1.11 percolation unit, vacuum filter and electrostatic precipitation unit require servicing/repair.
 All three units are services/repaired and are now in perfect running condition.
- 3.1.12 The operators of pilot plant should be the chemical engineers.

The writer does not agree to this recommendation of TIRDO.

The present staff is well versed and adequately trained to operate the plant. The Pharmacist Incharge, not only, operate but can also maintain the pilot plant with the present maintenance staff. He has also trained more personnel on the operation side. More training would however be required while scaling up the unit or in the field of new products in medicinal plant processing.

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3.2 Recommended spares, accessories required to render fully operational

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The pilot plant was ordered with sufficient spares. A physical inventory of all these has been taken and a list has been prepared. The plant has been made operational after using these spares. A list for parts, accessories atc has been prepared so as to place an immediate order to the manufacturer.

3.3 Recommend modifications and improvement to increase versatility.

Any major modification to the plant is neither required nor desirable. Some modifications in steam pipework, valves repositioning, providing of temperature gauges etc have been recommended. Chopper, though, requires certain modifications in the end product transfer system, cleaning during change-over for other product and in dust collection.

3.4 Effect any modifications possible with local facilities.

Steam line to the pilot plant is not of proper material and inadequately sized. A steam piping layout has been prepared giving proper regard to safety, instrumentation and energy conservation. The quotations have been called and the job has to be assigned now after UNIDO'S approval. As described earlier the collection method of chopped material will be modified. The cyclone separator was supplied without filter bag causing airborns product dust

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in the environment. Moreover this separator was so positioned that a duct having sharp bends was needed to connect the fan and the cyclone separator. The cyclone separator was removed and a filter bag is installed which retains the material and allows air to secape. To improve the performance further and to ease up cleaning operation, it is recommended to raise the chopper by two feet and keeping the collection point unchanged. Work on this would be started after UNIDO'S approval is received.

3.5 Make recommendations for pilot plant operation for future and list of accessories required.

Complete operating instructions have been prepared with all precautions to be taken and associated safety hazards.

Preventive maintenance schedule has been prepared. The list of accessories/spares required is also completed.

3.6 Establish system of log keeping in pilot plant.

Different formats on operation and maintenance have been prepared \bullet To use these formats some explanations have been given and practically demonstrated.

- ACHIEVEMENT OF OBJECTIVE
 Following in term of objectives are achieved
- 8oth product discharge valves of still have been repaired
 Other leaking valves, faulty prossure and temperature gauges,
 pressure reducing valve, steam traps, gauge glasses and
 vacuum transfer system for solvunt have been repaired
 Leaking Nutsch filter has also been made operational.
 Chopper has been memified and now working.
- 4.2 Modification in certain plants components have been recommended to improve work flow. The modification work would start as soon as the approval from UNICJ is received.
- 4.3 Fallowing products were made after the repair of the plant and no shortcoming was experienced.
 - (i) Cymbopogon citratus oil
 - (ii) Forniculum velgare oil
 - (iii) Coriandrum sativum oil
 - (iv) Matricaria chamomila oil
 - (v) Thymus vulgaries oil
 No other meterial was available to carry on the trial runs.

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- The training has been imparted to the operational and maintenance staff. Operating and maintenance instruction have been documented to help them in future work.
- 4.5 The proposed modifications in the plant would take place immediately after receiving the funds from UNIDO

5. UTILISATION OF PROJECT RESULTS

The project is in the last stages of completion. Number of products have been developed at pilot scale. National Staff has become familiar with the processes involved.

It is therefore imperative to scale up the entire plant so as to utilize the project results. To scale up the plant medicinal/aromatic plants should be cultivated on larger scale and the technology and experience.

already gained should be transferred to farmers. Field distillation units should be installed at appropriate places for initial processing to save cost of transportation. A market strategy should be developed to market the products in national and international markets

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6	REC OMMENDATIONS		
6.1	SHORT TERM		
6,1,1	The completion of piping work should be expedited		
6.1.2	The foundation/platform for chopper should be completed		
6,1,3	Temperature indicator should be installed on condensor		
	water outlet		
6.1.4	preventive maintenance programmed should be adhered to.		
6.1.5	Staff should be diputed for loading/unloading of		
	material and general cleaning of the pilot plant		
6.1.6	More personnel should be trained to operate the plant		
6.1.7	An orientation to other location at the same		
	technological level should be made to the personnel		
	responsible to operate the plant		

6.2 LONG TERM

- 6.2.1 The glass/FRP lined reactors/digestion vessel, vacuum drier and other equipment to isolate alkaloids should be procured
- 6.2.2 The possibility of scaling up the plant should be explored to use the technology and experienced gained from the pilot plant operation.

LIST OF SPARES INSTOCK

NO.		Description	Quantity
1.	Steam v	alve seats	9
2.	Rubber	sizeet 600mm x 600mm x 3	1
3.	Filter (cloth 500mm x 1500mm	1
4.	Chopper	Orive Belts	3
5.	Univers	al coupling with shaft	1
6.	Roller	Bearing	1
7.	Power c	1,6m	
8.	Power ci	1,2m	
9.	PTFE par	cking Half for Florentine Receiver	6
	2580mm : (0.D)	x 2500mm x 25mm (I.D) (thick)	
10.	XP Pluge	s with receptacles	2
11.	Level I	6	
12.	Florence Flask		3
13.	Chopper Blades		9
14.	Level Indicator prismatic glass		3
15.	Sight glass		7
16.	Rubber t	Rubber bushes with wesher	
17.	Steam gasket		
	(i)	140mm × 115mm × 3mm	1
	(ii)	110 × 95mm × 3mm	1
	(111)	110mm × 105mm × 3mm	1
	(iv)	88mm × 72mm × 3mm	2
	(v)	90mm x 55mm x 2mm	2
	(vi)	55mm × 43mm × 3mm	1
	(vii)	90mm × 2mm	4
	(ATTT)	105mm × 2mm	2
	(ix)	115mm x 2mm	7
	(x)	130mm x 2mm	4
	(x1)	180mm × 2mm	2

NO.	Description	<u>Quantity</u>
18.	PTFE gaskets	
	(i) 102mm x 82mm x 1mm	4
	(ii) 85mm × 50mm × 1mm	7
	(iii) 76mm × 42mm × 1mm	19
	(iv) 75mm x 45mm x 1mm	4
	(v) 60mm × 45mm × 1mm	4
	(vi) 55mm x 28mm x 1mm	55
	(vii) 55mm x 35mm x 1mm	8
	(viii) 50mm x 35mm x 1mm	4
	(ix) 45mm x 20mm x 1mm	39
	(x) 55mm x 38mm x 1mm	27
	(xi) 100mm x 85mm x 1mm	27
	(xiii) 65mm x 35mm x 1mm	28
19.	J-Bolts	4
20.	Fan Blades - Cutter	8
21.	Blades packing strips	4
22.	Bolts 12mm dia x 60mm long	31
23.	Bolts 10mm dia x 30mm long	39
24.	Bolts 10mm dim x 70mm long	4

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Spares parts to be ordered

1.	Bearings (set) for chopper	1 set
2.	Searings (set) for vacuum pump	1 set
3.	24 volts XP tube light - lamps	6
4.	24 volts XP tube lights - ballast	6
5.	Temperature Indicator - Capillary type	
	for still 0 - 120°C	1
6.	Temperature Indicator - capillary tube	
	for condensor water injet	1
7.	Pressure gauges for steam 0 - 15 bar	2
8.	Pressure gauges for steam 0 - 6 bar	2
9.	Bearings (set) for worm gear set	
	agigator	1
10.	Repair kit 100 mm. ball valve	3 sets

Operation Instruction I

Steam Distillation

- 1. Close all valves
- 2. Open the hinged cover of the feeding port of the still
- 3. Fill still with chopped meterial Ram the material to compact about 75% of total volume.
- 4. Close the lid and tight the bolts
- 5. Open valves 25, 19, 22, 28 and 39
- 6. Open main steam valve from the boiler
- 7. Check pressure of steam not to exceed 2 bars
- 8. Note temperature rise at still when temperature reaches 85°C open valve 10 and 11 for condensor water. Regulate water to a temperature difference of 5 7°C between water inlet and outlet at condensor
- 9. Oil and water start accumulating in Florence flask
- 10. Drain water layer by opening valve 31 and collect oil

Operating Instruction II

Sexhlet Extraction

- Open soxhlet cover, place chopped and weighted herb in a cloth bag and then in the apparatus basket close the cover tightly
- 2. Close all valves
- 3. Open valves 4, 5, 9, 12, 22 and 23
- 4. Open water valve 6 for vacuum pump
- 5. Start the vacuum pump
- 6. Open valve 30 and put flexible hose attached to it to sc_vent tank to feed required amount of solvent into the still
- 7. Stop vacuum pump and close valves 4, 5, 6, 9, 12, 22 and 23
- 8. Open valves 21, 32, and 29
- 9. Open valves 10 and 11 for water to condensor
- 10. Open steam valves 15 and 16
- 11. Watch the sight glasses, and the condensed solvent would flow through soxhlet and back to still
- 12. Continue the operation until the plant material in soxhlet is exhausted.

Now start concentration of the extract

- 13. Close valves 21 and 32 and open valve 23 and continue distillation of solvent which would be collected in the collection vessel
- 14. When solvent distillation is over close steam and water valves
- 15. Transfer the extract to sedimentation tank by vacuum and allow to settle
- 16. Collect layers by opening valves 33, 34, 35 and 36 as required after filtering through Nutsh filter

Operating Instruction III

<u>Percolation</u>

- 1. Open the cover of percolating filter
- 2. Fill the required amount of chopped and weighted herb and close the cover
- 3. Close all valves
- 4. By means of vacuum, place the solvent in percolating filter by starting vacuum pump and opening valves 1, 3, 4, 5 and 6
- 5. Switch off the pump and close all valves
- 6. Wait till the extraction time is elapsed
- 7. Open valve 7 and collect the mixture of extract and solvent
- 8. Remove the cover and tip the percolating to discharge spent herb
- 9. Transfer the mixture of extract and solvent to distillating still for vacuum concentration/distillation
- 10. Follow the operating instruction IV for vacuum concentration/

Operating Instruction IV Vacuum concentration/Distillation

- 1. Planes the solvent or mixture of extract and solvent in the still
- Start cooling water in condensor by opening valve 10 and
 11
- 3. Open water to vecuum pump valve 6 and start vacuum pump
- 4. Close all velves except 4, 5, 6, 9, 12, 22 and 23
- 5. Start heating the jacket by opening values 15 and 16
- 6. Note the temperature and pressures on still and collection vessel
- 7. Watch the condensation and collection of solvent in the collection vessel
- 8. When distillation is over, stop vacuum pump and steam heating

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9. Gradually open valve 13 to ventilate the plant

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PREVENTIVE MAINTENANCE SCHEDULE

DAILY

- 1. Check for any leakage in the plant
- 2. Observe for the abnormal sound from Agitator, vacuum pump and chopper
- 3. Check for heating, Agitator's bearings, vacuum pump bearing and chopper gearbox by touching with hand
- 4. Check glands of vacuum pump for leakage
- 5. Check working of all pressure and temperature gauges
- 6. Check working of pressure reducing valve, if steam pressure exceeds 2.5 ber. Stop operation
- 7. Blow safety valve manually

WEEKLY

- 1. Perform deily checks
- Check couplings of agitator, chopper and vacuum pump for wear
- 3. Check belt tension of chopper

MONTHLY

- 1. Perform daily and weekly checks
- Grease bearing housing of vacuum pump
- Grease agitator housing

6 MONTHLY

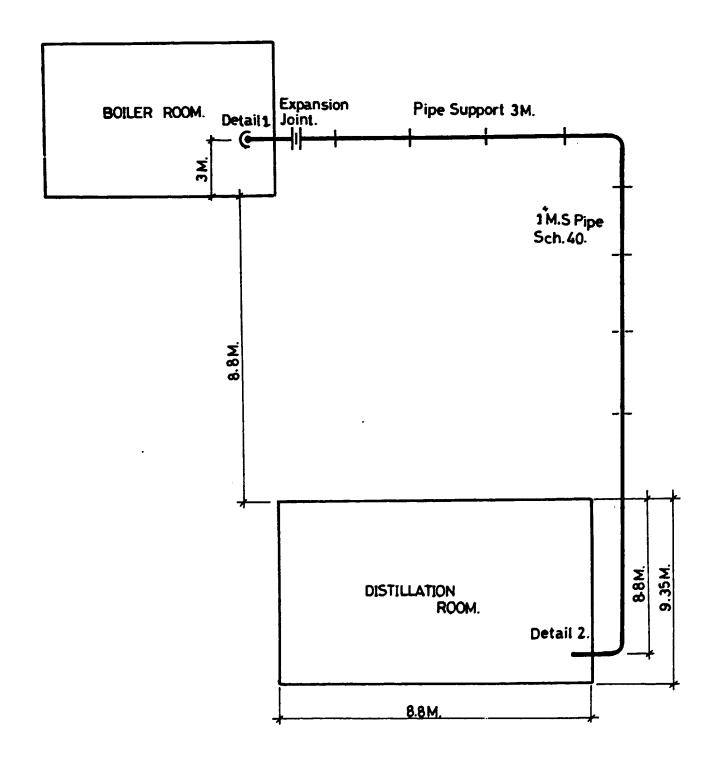
- 1. Perform daily, waskly and monthly check
- 2. Change oil in wurm gear set of agitator
- 3. Change oil in chopper gearbox

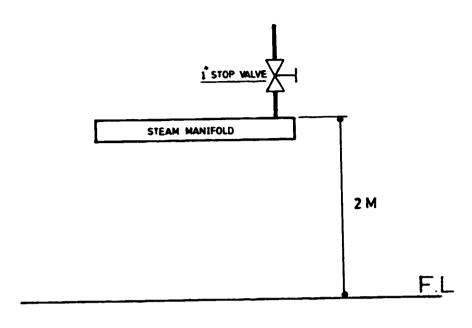
YEARLY

1. Perform daily, weekly, monthly and half yearly checks

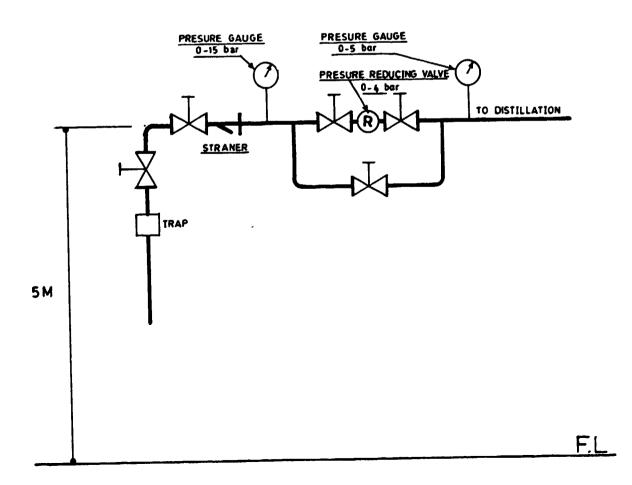
- 2. Open electric motors and replace grease in bearings
- Paint entire plant and supporting structure.

STEAM LAYOUT.





DETAIL: 1



DETAIL: 2

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