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DP/ID/SER.A/1591
27 July 1992
ORIGINAL: ENGLISH

i, 37p. diagrams Illus

PROCESSING OF AROMA CHEMICALS AND FRAGRANCE MATERIALS

DP/VIE/86/033/11-55

THE SOCIALIST REPUBLIC OF VIET NAM

Technical report: Building modifications plant foundation water power distribution system and designing of commercial fractional distillation unit*

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

Based on the work of C. L. Tikoo, Plant engineer

Backstopping Officer: T. De Silva, Chemical Industries Branch

United Nations Industrial Development Organization
Vienna

* This document has not been edited.

C O N T E N T S

I. INTRODUCTION

Vietnam has been a producer of aromatic essential oils for several decades. These natural essential oils are being exported at fairly low price to other countries. Against this low price oil Vietnam is importing fairly large quantities of aroma chemicals and fragrance materials for their internal use in soap and cosmetic industries. It does not have the capacity and technology to process part of these natural oils to obtain perfumery grade purified isolates and aroma chemicals of suitable olfactory quality which may then be formulated and compounded into fragrances and cosmetic products. The growing demand of isolates and aroma chemicals from natural essential oils in presence of scarce foreign exchange has led Government of Vietnam to set up some organised sector to produce these aroma chemicals which would meet the internal demand thereby saving considerable foreign exchange and also be in a position to export Aroma Chemicals in future in international market.

The South Vietnam Detergent Company is a union which runs a complex of factories which produce soaps toiletries and cosmetics have their research and development centre known as Vinarom. A project funded by UNDP and UNIDO being the executing agency has been envisaged for Vinarom to meet the objective of developing technologies on bench and pilot scale for aroma chemicals which would make South Vietnam Detergent Factory Principal beneficiary.

The ofjective of the project is to create.

1. Laboratory and bench scale fascilities to develop technologies for producing isolates and aroma chemicals from natural essential oils.
2. To scale up the processes on Pilot scale.
3. Training to Vietnamese staff for running of Pilot Plants and create capabilities to adopt new technologies.
4. Training to handle G.C, U.V, IR instruments for quality control.
5. Training abroad to some Vietnam staff members to learn separation techniques and synthesis of aroma chemicals.

Under the Project apart from other objectives the most important ofjective is to set up a multifunctional Pilot Plant for processing natural essential oils to produce variety of isolates and aroma chemicals.

The Pilot Plant has been devided into two Sub units.

1. Sub unit A for fractional distillation for producing isolates.
2. Sub unit B for producing aroma chemicals.

The unit A consists of a 150 litre per batch fractional distillation unit having a packed column using multinit packing to give about 40 theoritical plates. It is provided with a

shell and tube condenser, a product cooler, receivers, vacuum pumps, vapour trap, and a chilling plant. The reboiler of the unit has provision for working both on steam and hot oil system.

The unit has been provided with necessary instrumentation to control the process conditions. It can handle variety of essential oils to produce different isolates from wide range to very close range boiling points.

The sub unit "B" consists of the following

1. Hydrogenator with agitator and reflux condenser.
2. Catalyst filteration unit.
3. Oil Service tank.
4. Glass lined reaction vessel with S.S. column and packed with multinit packing. This has provision for both heating and cooling.
5. F.R.P. acid washing tank.
6. F.R.P. acid receiving tank.
7. Basket centrifuge.
8. Dehydrator with condenser.
9. Oil separator.
10. Product cooler.
11. Product receiver.
12. Liquid transfer pump.
13. Cooling overhead tank.

The sub unit B is a multifunctional Pilot unit which can handle varieties of raw material to produce aroma chemicals both of synthetic and natural origin.

W O R K D O N E

The consultant arrived HoChiMinh City on 6th of Nov 1992. He had to carry out two assignments one four month mission on Project DP/VIE/85/001 (Dyes and Pigments) and one two month mission on Project DP/VIE/86/033 (Aroma chemicals) under common CTA. The consultant was briefed by the CTA and the NPD of the Aroma Chemicals Project and was asked to carryout the following jobs relating preparatory to receive pilot plant equipment during his two month mission.

A. Building modifications to accomodate pilot plant equipment.
B. Design of water distribution system.
C. Design of power distribution system.
D. Civil foundations of aroma chemical plant.
E. Training to Vinarom staff on the design of fractional distillation unit.
F. Design of a commercial scale fractional distillation unit.

A work plan was drawn which is given in activity chart Annexure I.

A. BUILDING MODIFICATIONS

Vinarom has a large building shed. It was decided to locate portion of this shed to house pilot plant equipment and its auxilary units. A space of 12 mts x 6.5 mts was selected. The total height of the shed being 7.2 mts can not house the unit as the clear height requirement for plant is 8 mts. In addition to this some head space has also to be provided. Since the clear height of 8 mts is required for fractionating column, it was decided by the consultant to raise the central portion of the roof top in stead of raising the whole shed which would involve huge expenditure.

To achieve the abjective the consultant designed building modifications which would involve minimum cost.

The shed was raised by 400 mm from the side with bricks and the central portion 4 mt x 6.5 mt upto 9 mts height. The sides of the central portion have been covered with G.I. sheets.

The building modification is shown in Annexure II. The task has been accomplished and the modified building is ready to receive the Pilot Plant equipment.

B. DESIGN OF WATER DISTRIBUTION SYSTEM:

There is an inadequate supply of water to Vinarom complex. In order to run the Pilot Plant about 15000 litres of water per hour is required. The complex has a water under ground tank of 50,000 litres capacity. Need was therefore felt that the water should be recycled back from the utilities to the main sump to meet the demand. The consultant designed a water distribution system which comprises, construction of 6000 litre mesonary overhead tank at a height of 9 mts, a water supply line of 42 mm ID from underground tank to the overhead tank, a water supply line of 49 mm ID from the overhead tank to the Pilot Plant building and a 49 mm water return line from condensers to the underground tank. A pumping set of 20,000 litre per hour capacity has been provided to pump the water to overhead tank with level controler to monitor the pump.

The design of the system is given in Annexure III. The task has been accomplised.

C. DESIGN OF POWER DISTRIBUTION SYSTEM:

The existing Power cable from Viso factory to Vinarom complex was inadequate to bear the power load of Pilot Plant equipment. The consultant recomended to change this power cable to 150 kw power cable by providing 3 wire and neutral power cable of 70 mm which would enter the main switch board of the Vinarom. This board shall house one 400 Amps switch and two ACB's of 50 Amps and 200 Amps. The 50 Amps ACB shall cater the load of test laboratories and the 200 Amps ACB shall take

care of Pilot Plant load. A cable has been drawn from this ACB to pilot plant building. This board houses one isolating iron clad switch of 300 Amps, one ACB of 30 Amps and one ACB of 200 Amps. The board is also provided with phase indicators, single phase preventer volt and ampemeter. The complete power distribution and electric circuit has been shown in Annexure IV, V, VI and VII.

D. TRAINING TO VINAROM STAFF ON DESIGN ENGINEERING OF FRACTIONAL DISTILLATION UNIT.

The consultant conducted a training course of ten days to inmpart training to Vinarom staff on engineering design of fractional distillation unit. During training course the following aspects were dealt with.

1. Principles of fractionation.
2. Equipment required for fractionation of essential oils.
3. Different types of columns.
4. Selection of right type of column.
5. Details of packed column.
6. Details of packing materials used in packed column.
7. Specifications and Physical characteristics of packing material.
8. Process data and process parameters required for the design of column.
9. Calculations for finding out vapour load for determing diameter of the column.
10. Calculations for finding out number of theoritical plates and height of theoritical plate to find out column height.
11. Design of reboiler.
12. Design of condenser.
13. Design of product cooler.
14. Design of product receivers.

15. Design of vapour trap.
16. Selection of material of construction.
17. Interconnecting pipe lines valves and instrumentation required for the column.
18. Selection of material of construction.
19. Mechanical Design of the column i.e. thickness of vessels, Flanges, supports etc.
20. Fractional distillation layout.
21. Some aspects on fabrication practices and codes used.
22. Thermal design to work out heating loads, heating systems and optimal insulation of the column.
23. Specification of vacuum pump and chilling system.
24. Operation of distillation unit.

This training course has given a foundation to Vinarom staff as to how distillation columns can be designed under defferent process conditions but the designing capabilities to design an efficient unit shall depend more on practice which Vinarom engineers can achieve by working on pilot plant to generate more process and engineering data.

E. CIVIL FOUNDATIONS OF AROMA CHEMICAL PLANT.

The civil foundations of the steel structure for aroma chemical plant have been completed. The plant layout and the details of the civil foundations are given in Annexure VIII, IX and X.

F. The consultant has designed a commercial scale 500 litres per batch fractional distillation unit. Detailed engineering drawings of components of the unit have been prepared along with P.I. diagram, plant layout, plant assembly and also the isometric view of the plant. Detailed designed details and engineering drawings along with specifications of the auxilary components have been given in separate chapter attached to this report.

C O N C L U S I O N S

1. Civil modifications of the building have been completed.
2. Civil foundations of the steel structures have been completed.
3. Water overhead tank and water distribution system have been completed.
4. Power distribution system of the pilot plant building has been completed.
5. Boiler has been installed and the steam piping upto pilot plant building has been completed.
6. Training to Vinarom staff on design of fractional distillation unit have been completed.
7. A 500 litre batch capacity semi commercial fractional distillation unit has been designed by the consultant and the detailed engineering drawings. P.I. diagram and specifications have been prepared.
8. All pre installation requirements have been completed and Vianrom is ready to receive the Pilot Plant equipment and machinery for its installation and commissioning.

R E C O M M E N D A T I O N S

1. All water inter connecting pipes from water header to different equipment have to be provided and fixed by the subcontractor.
2. All internal electrical network wiring from power board to different equipment have to be provided and connected by the subcontractor.
3. All steam pipe lines from the steam header to different equipment have to be supplied and fixed by the contractor.
4. All insulation material for vessels and steam pipes with aluminium cladding have to be provided by the contract. However Vinarom management have to provide skilled people for insulation work.
5. Vinarom has to provide skilled labour, a welder and a welding machine to assist installation team from the subcontractor during installation.
6. There is power shedding due to the shortage of power (three to four days in a week) which can be deterimental to the progress of installation and commisioning work. Necessary arrangement should be taken well in advance by the management to overcome this difficulty.
7. A separate drain connection to be provided to the equipment handling corrosive liquids like acid and alkalies and these corrosive liquids to be collected

in a rubber lined sump where it can be neutralised and be allowed it to go into the main drain. This aspect has been discussed by the consultant with Vinarom management and they should take necessary steps in this regard.

WEEKLY DIARY OF THE CONSULTANT

4 Nov 91	Left New Delhi for Bangkog
6 Nov 91	Left Bangkok for HoChiMinh City.
6 Nov 91	Arrived HoChiMinh.
4 week Nov 91	Discussion with CTA and NPD of the Project regarding tasks to be completed preparatory to Pilot Plant installations.
1 and- 2 week Jan 92	Design of building modification, water distribution system and training to Vinarom staff.
4 week Jan 92	Design of power distribution system.
2 week Feb 92	Building modification carried out
4 and 5 week Feb 92	Designing of fractionating column.
3 week March 92	Checking of building modification and marking of civil foundations for steel structur.
4 week April 92	Preparation of report.
5 May 92	Left for India.
7 May 92	Reached Jammu (India)

ACKNOWLEDGEMENTS

The consultant greatly acknowledges the thanks for

1. The guidance and co-operation provided by Dr. C. K. Atal CTA of the Project.
2. The excellent cooperation provided by Mr. Dinh Van Thin NPD of the Project.
3. The excellent cooperation from Mr. Do Linh Cuong.
4. Commedable assistance provided by Mr. Nguyen Van Quy, Deputy Director.
5. Excellent cooperation from Mr. Giao, Civil Engineer.
6. The excellent assistance from the staff members of Vinarom.

The Consultant is also greatful to Mr. Le Khac Tho and Miss Huong Secretary and the typist of the project who were of great help in typing and compiling of this report.

file: RE-TKOackOC

Annex 1

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

JOB DESCRIPTION

DP/VIE/86/033/11-55

Post Title: Plant Engineer

Duration: 2 w/m

Date Required: February - March 1992

Duty Station: Ho Chi Minh City

Purpose of Project: Utilization of indigenous essential oils to develop suitable fragrance materials and formulations for local industry as well as export.

Duties: The expert will work under the joint supervision and direction of the UNIDO Chief Technical Adviser and the National Project Coordinator and will have particular responsibility for assisting in the optimisation and use of the pilot plant equipment.

The expert will also perform the following specific duties:

1. Upscale the laboratory processes developed on a bench-scale.
2. Make bench-scale runs to produce aroma chemicals for trial use
3. Train local staff in the proper use, maintenance and upkeep of processing equipment.
4. Guide local staff in engineering and fabrication drawings to design a Commercial plant for essential oils.
5. Carry out any other function assigned by the CTA.

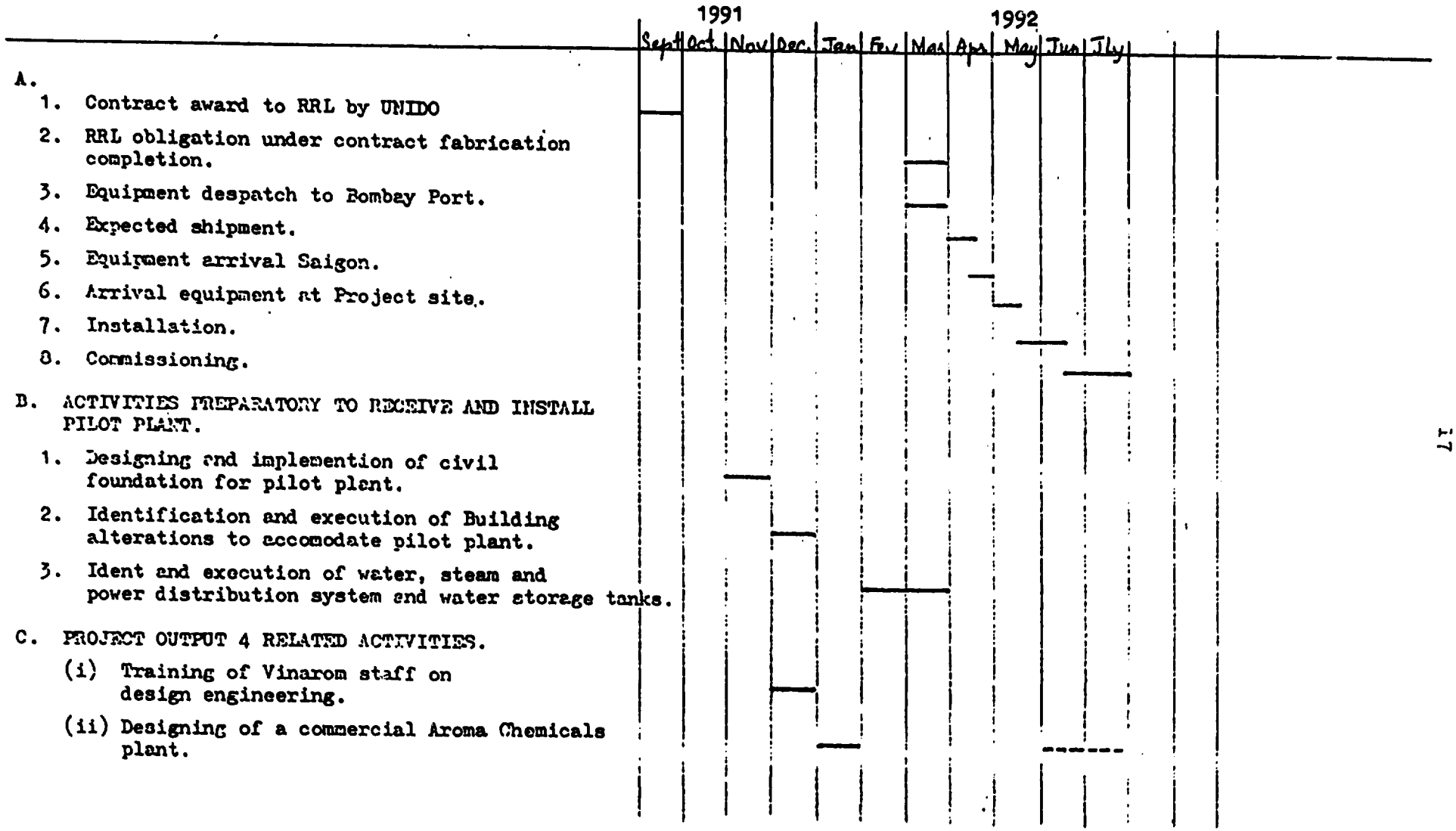
The expert will furnish a complete and fully processed terminal report at the completion of his mission outlining the findings of his mission and his recommendations for follow-up action.

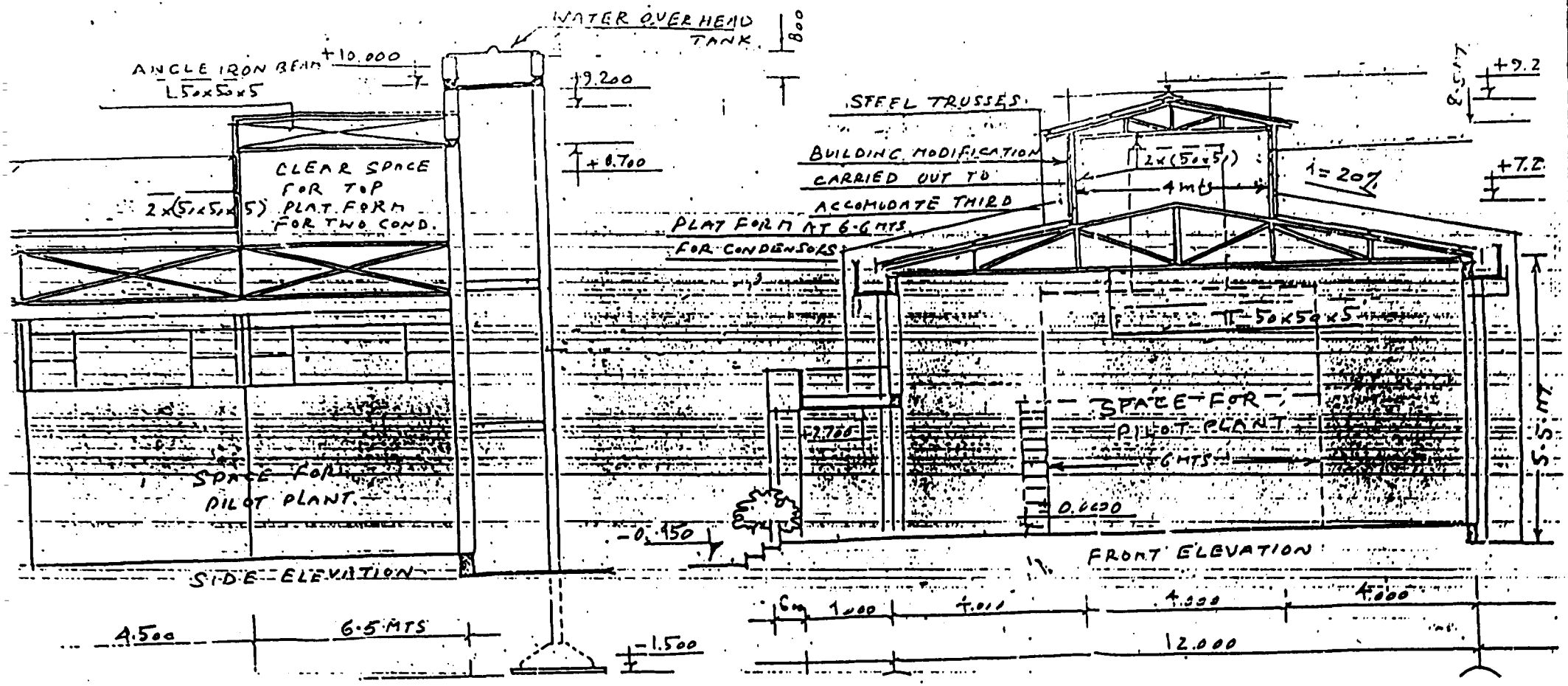
..../..

Annex 2

PROJECT NO. DP/VIE/86/033
AROMA CHEMICALS
ACTIVITY CHART

UP DATED 08 NOV 1991

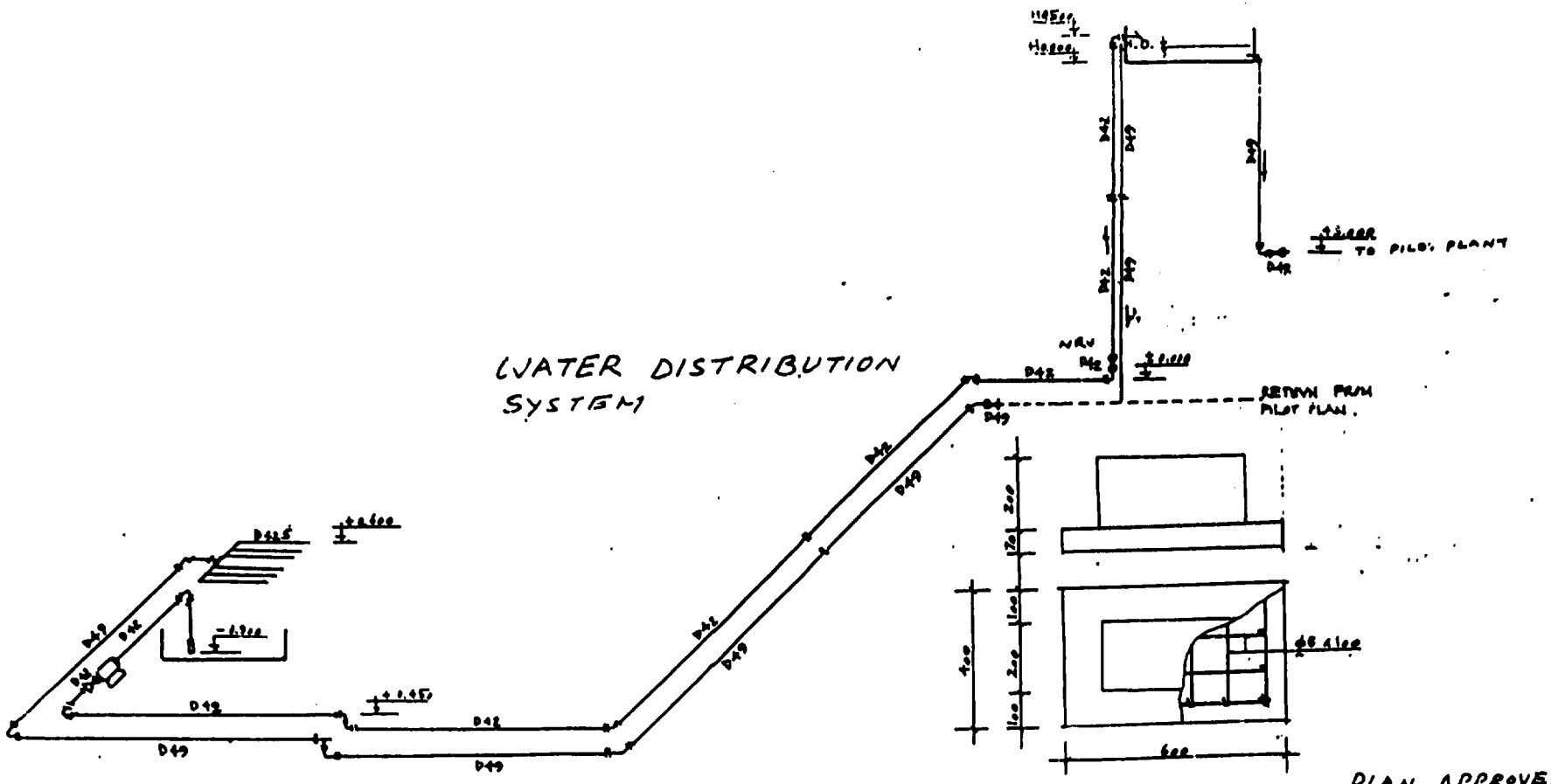




SPACE FOR PILOT PLANT HAS BEEN SHOWN 9 MTS X 6 MTS. BUT THE PLANT SHOULD BE WITHIN 6 MTS X 5 MTS TO LEAVE FREE SPACE FROM SIDES

C.L. TIKOO
PLANT ENGINEER

PROJECT :- DP/VIE/86/033



PLAN APPROVED.

C. L. TIKOO
PLANT ENGINEER
PROJECT DP/VIE/86/033

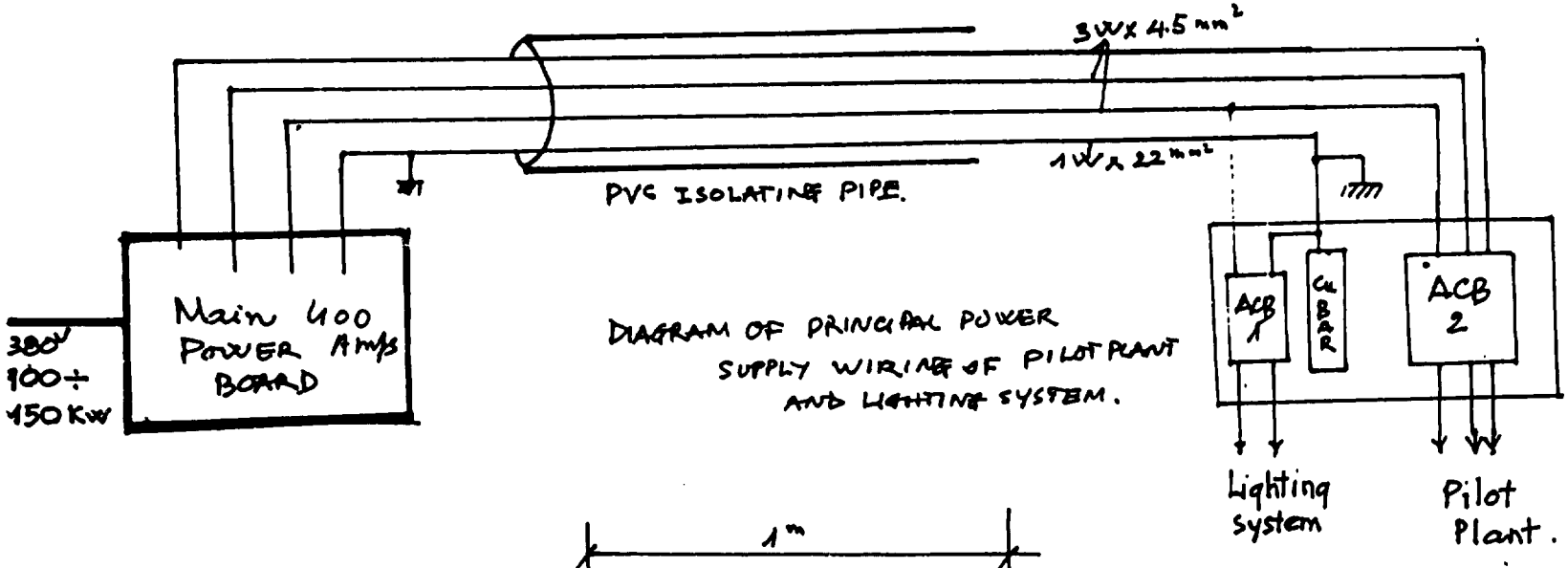
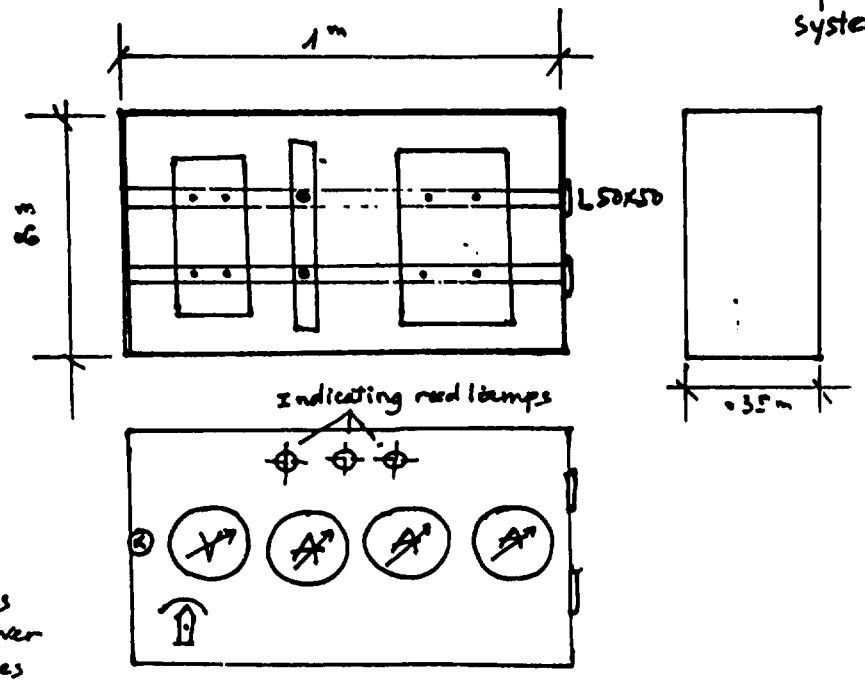


DIAGRAM OF PRINCIPAL POWER SUPPLY WIRING OF PILOT PLANT AND LIGHTING SYSTEM.

Distribution board (wall-mounted)

ACB 1:- 30 Amps

ACB 2:- 200 Amps



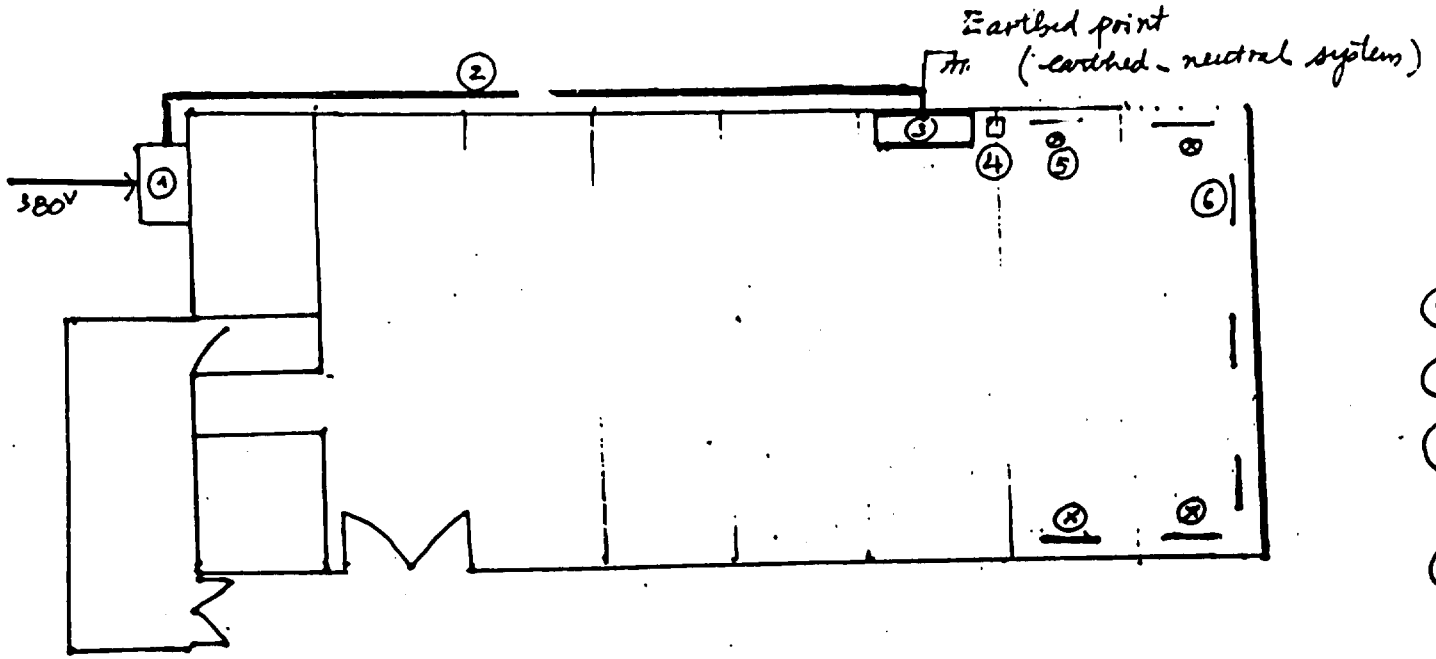
DISTRIBUTION BOARD DIAGRAM

1mW

C.L. TIKU

PLANT ENGINEER

DP/VIE/86/03

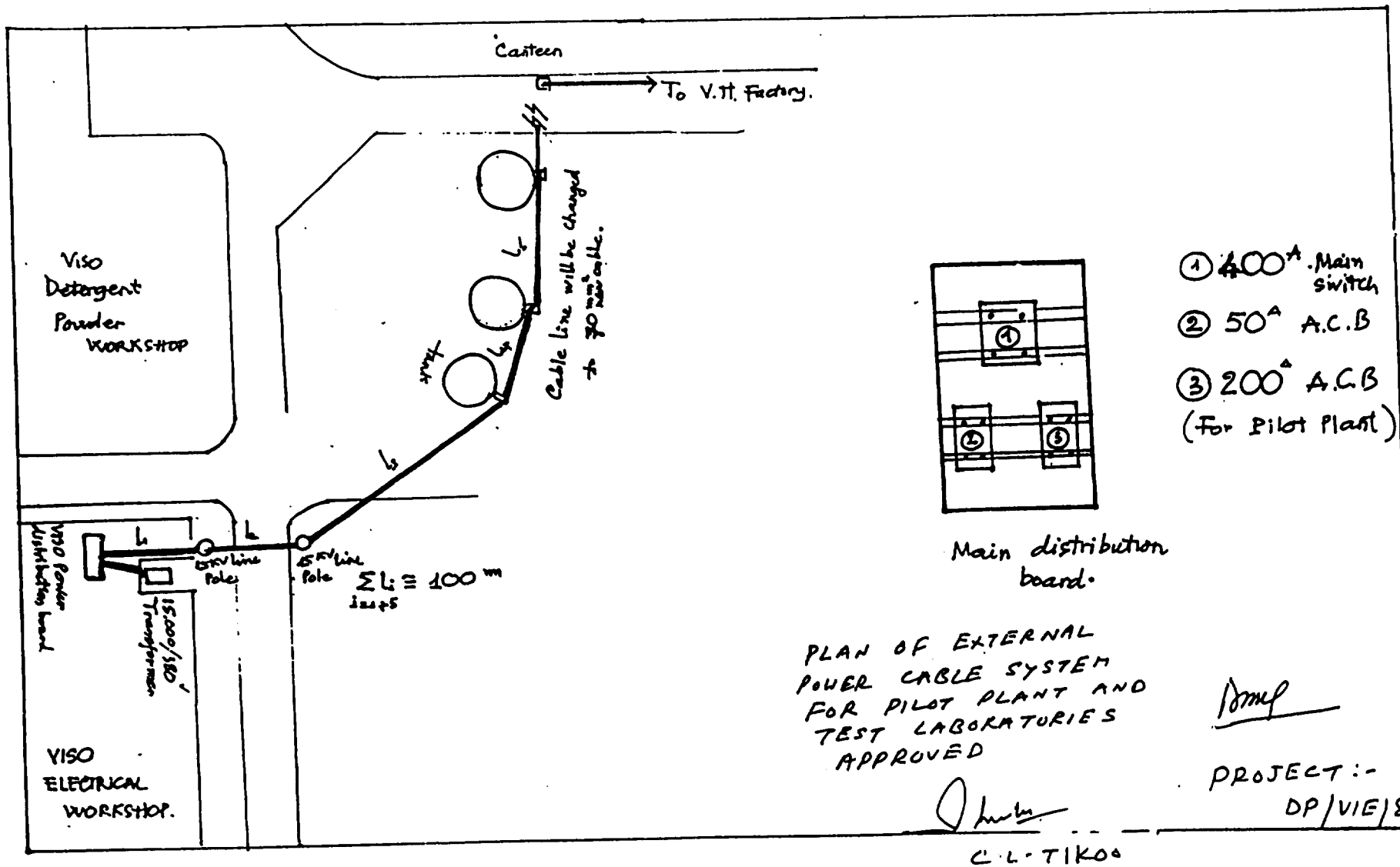


1. Main Power Board
2. Cable line
3. Pilot & Lighting system distribution board
4. Lightswitches and Socket outlet
5. Wall-mounted Fan
6. 1.20m Fluorescent lamp

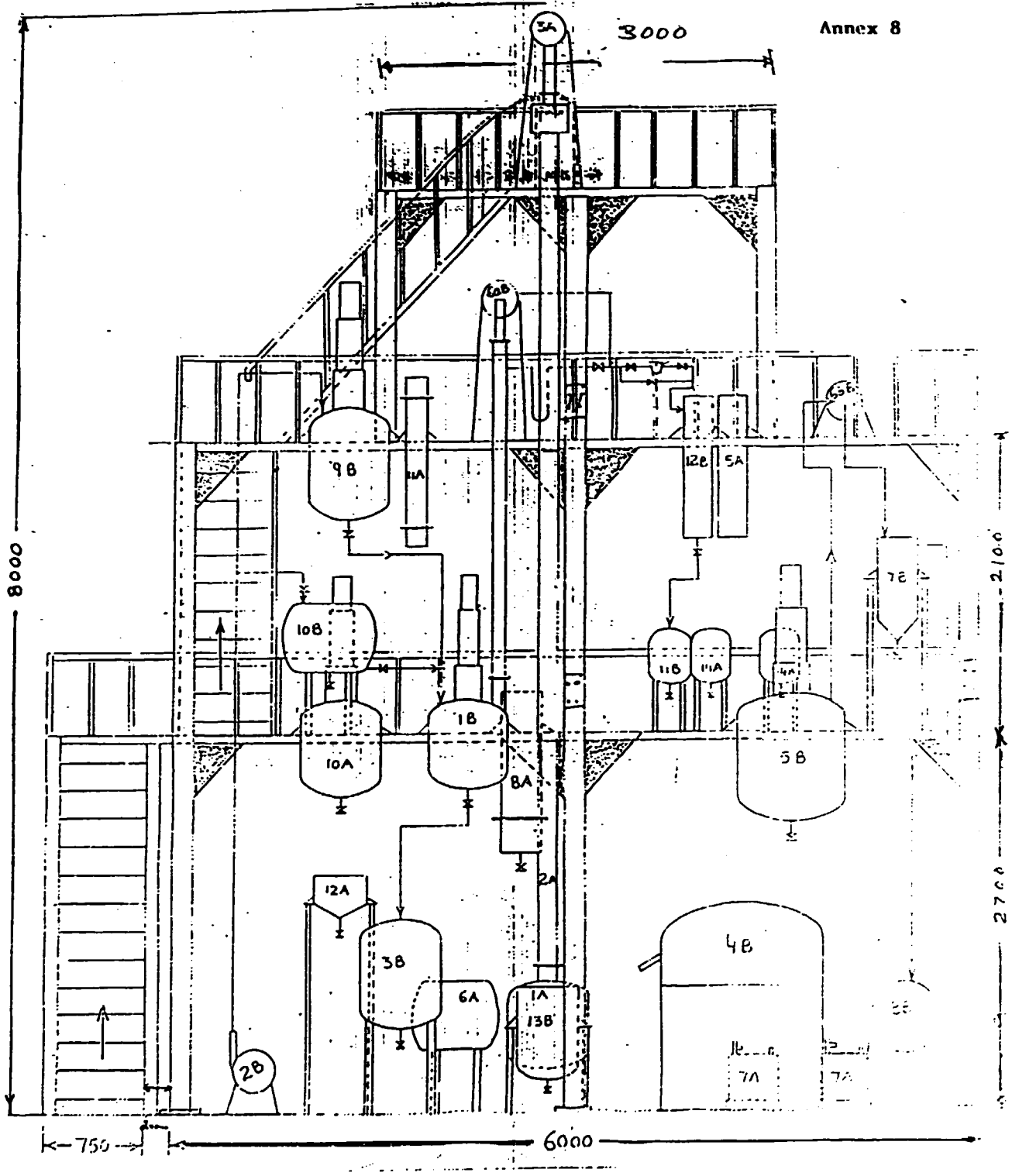
Lighting and wiring system DIAGRAM.

PROJECT :- DP/VIE/86/033

PLAN FOR INTERNAL LIGHTING SYSTEM IN PILOT PLANT APPROVED


Canteen
To V.H. Factory.
Viso Detergent Powder WORKSHOP
Cable line will be changed to 70 mm² cable.
Viso Power distribution board
15.000/380 V Transformer
VISO ELECTRICAL WORKSHOP.
Σ Li ≡ 100 m
i=1 to 5
① 400A Main switch
② 50A A.C.B
③ 200A A.C.B
(For Pilot Plant)
Main distribution board.
PLAN OF EXTERNAL POWER CABLE SYSTEM FOR PILOT PLANT AND TEST LABORATORIES
APPROVED
C.L. TIKOO
PROJECT :-
DP/VIE/86/033

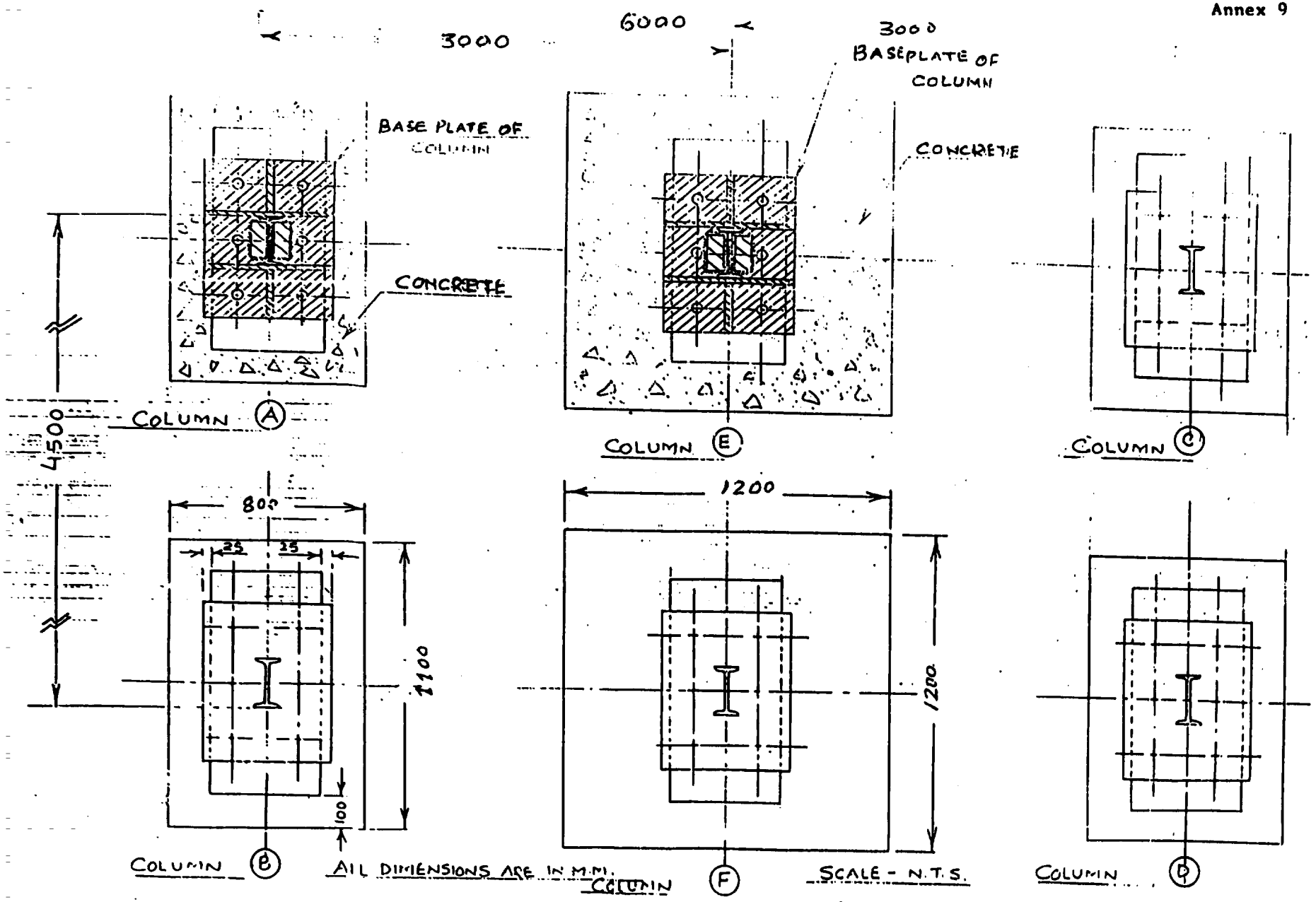
Annex 8



PLANT LAYOUT OF AROMA CHEMICAL PILOT PLANT.

C. L. TIKO
PLANT ENGINEER

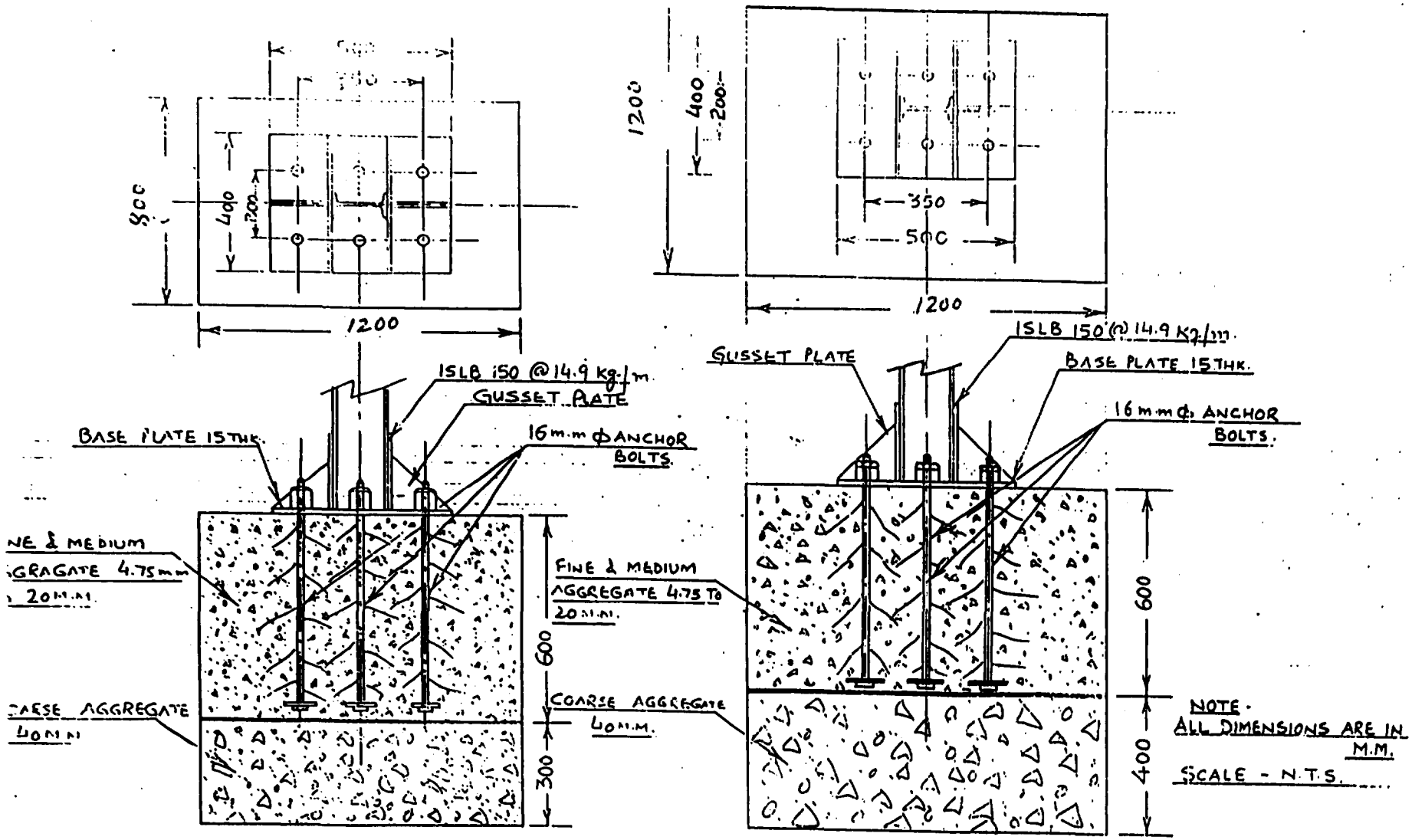
PROJECT :- DP/VIE/86/033



PLAN OF STEEL STRUCTURE FOUNDATION FOR AROMA CHEICAL PILOT PLANT

C L T KO
PLAN-
ENGINEER

PROJECT:
DP/VIE/75/033



FOUNDATION DETAILS OF COLUMNS A, B, C & D

FOUNDATION DETAILS OF COLUMNS E & F

DESIGN OF A FRACTIONATING UNIT FOR ESSENTIAL OILS.

The consultant has designed a fractionating unit to separate out valuable isolates from citronella oil. However this unit can be used for other essential oil also by changing the process parameters.

The unit consists of

1. S.S. reboiler of 500 litre batch capacity.
2. S.S. column packed with multinit packing material.
3. S.S. reflux distributors.
4. S.S. shell and tube condenser.
5. S.S. isolating receiver of 25 litre capacity.
6. S.S. product receiver of 100 litre capacity.
7. S.S. vapour trap.
8. Reflux divider consisting of 25 mm I.D. three way S.S. solenoid valve with timer.
9. Two rotary vacuum pumps.
10. A small chilling plant.

Detailed engineering drawings have been prepared which consists of.

1. Drawings of each component.
2. P.I. diagram.
3. Over all assembly of the unit.
4. Isometric view of the unit.
5. Specifications of the rotary pump.
6. Specifications of the chilling plant.

Basis for the design:

1. Batch capacity 500 litres.
2. Boil up rate 300 litres/hr.
3. Pressure drop 15 mm.
4. Vacuum attained upto 1 mm.
5. Packing used multinit wire mesh.
6. Condenser water inlet 25°C.
7. Condenser water outlet 35°C.
8. Product temp. 30°C.
9. No of theoritical plates 40 at total reflux.
10. Batch cycle - 16 hours.
11. Effective cycle - 12 hours.

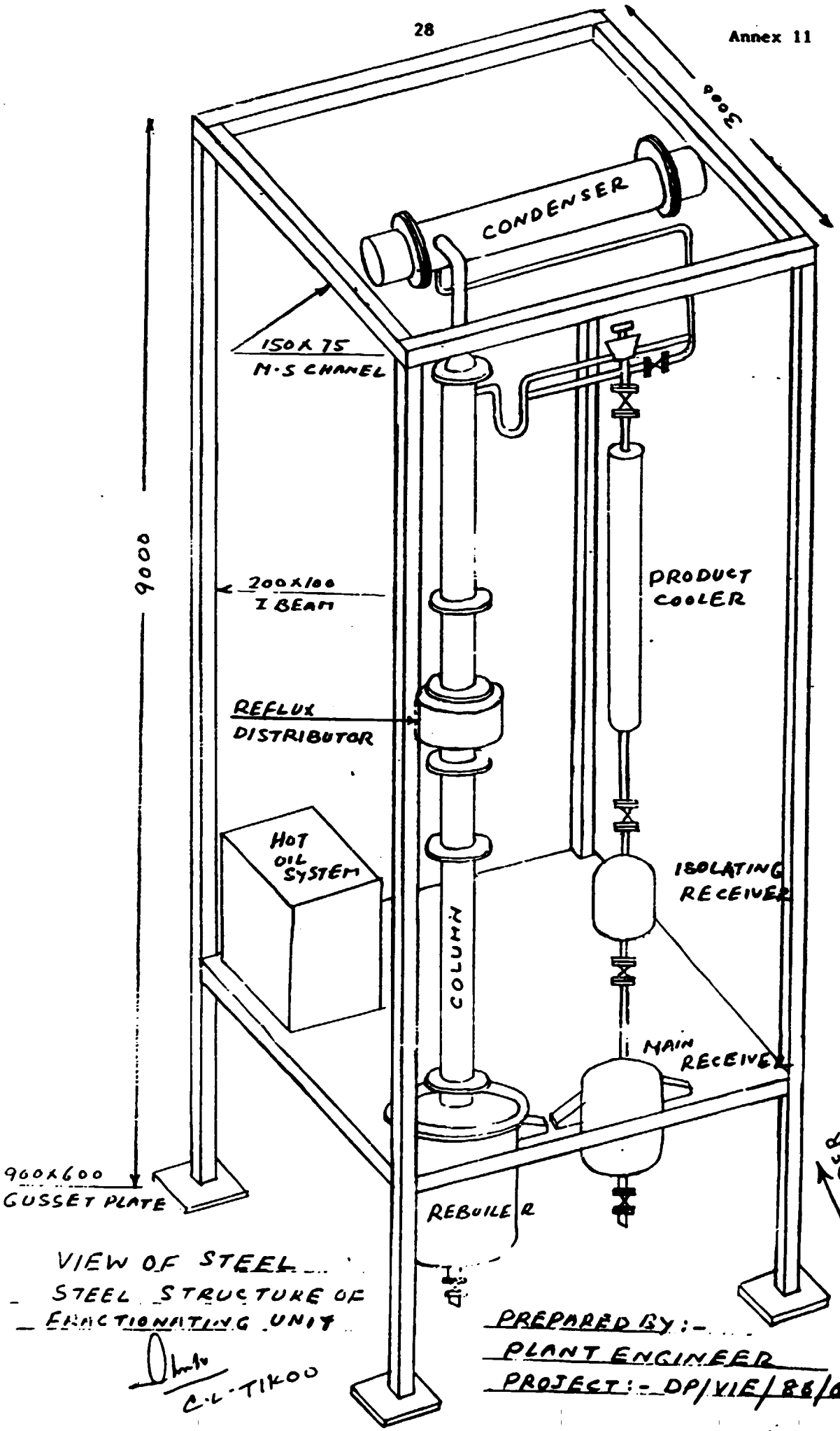
Specifications of chilling plant.

Skid mounted compact chilling unit with air cooled condenser capacity 2 ton refrigeration capacity at -10° to 10°C provided with brine solution storing vessel bait capacity 200 litre connected to a 1 HP centrifugal pump complete with all interconnecting pipe line valves and controls. The unit should have a compact panel control. one set.

Rotary vacuum pump.

Rotary vacuum pump with displacement capacity of 80 cum per hour at N.T.P. to give ultimate vacuum of 1 mm Hg. The pump to be coupled with suitable motor starter and provided with efficient vapour trap non return valve. by pass valve vacuum gauge etc. one set.

Rotary vacuum pump same as above but with displacement cap of 20 cum per hour one set.



VIEW OF STEEL ...
STEEL STRUCTURE OF
FRACTIONATING UNIT

C.L. TIKOO

PREPARED BY :-
PLANT ENGINEER
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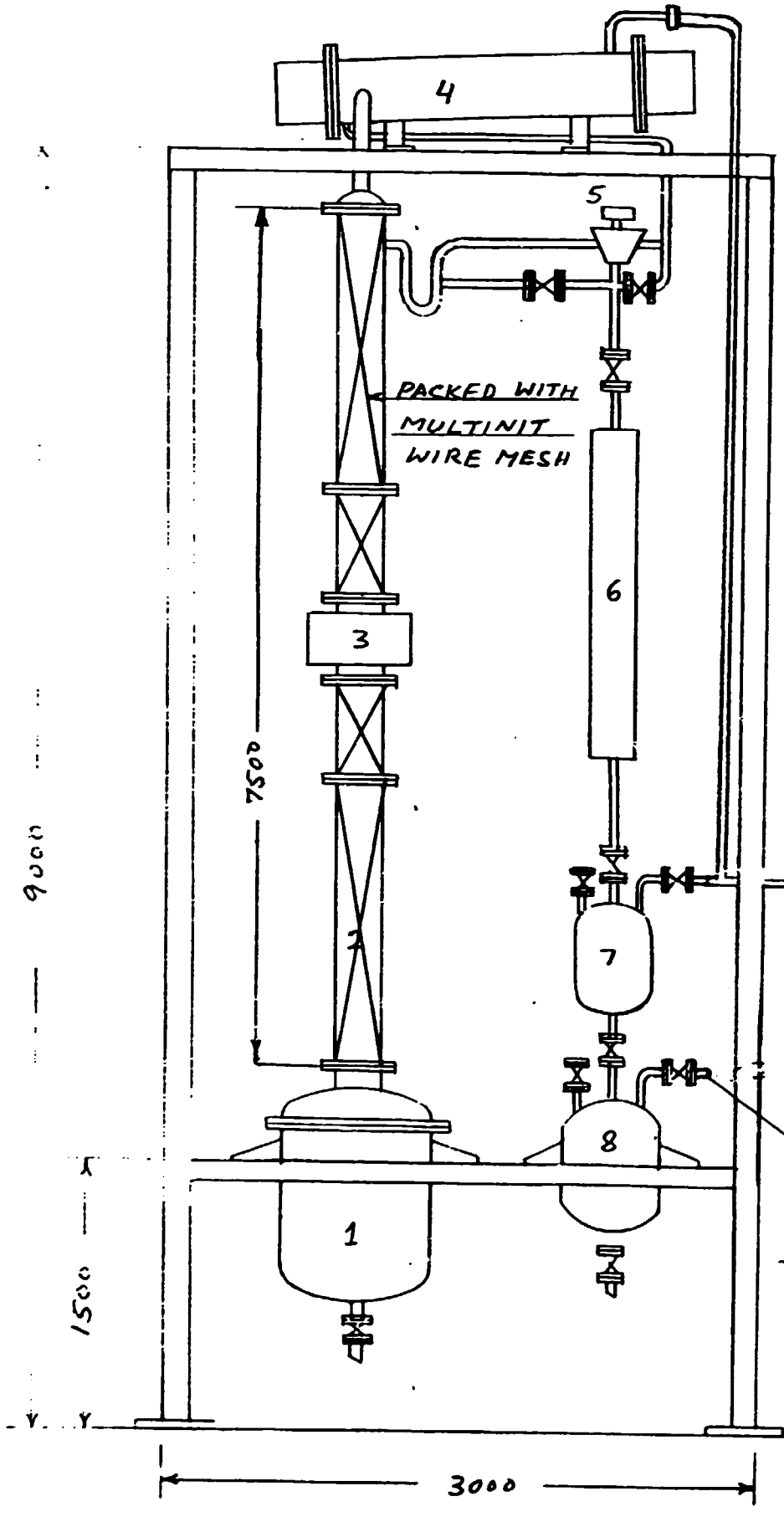
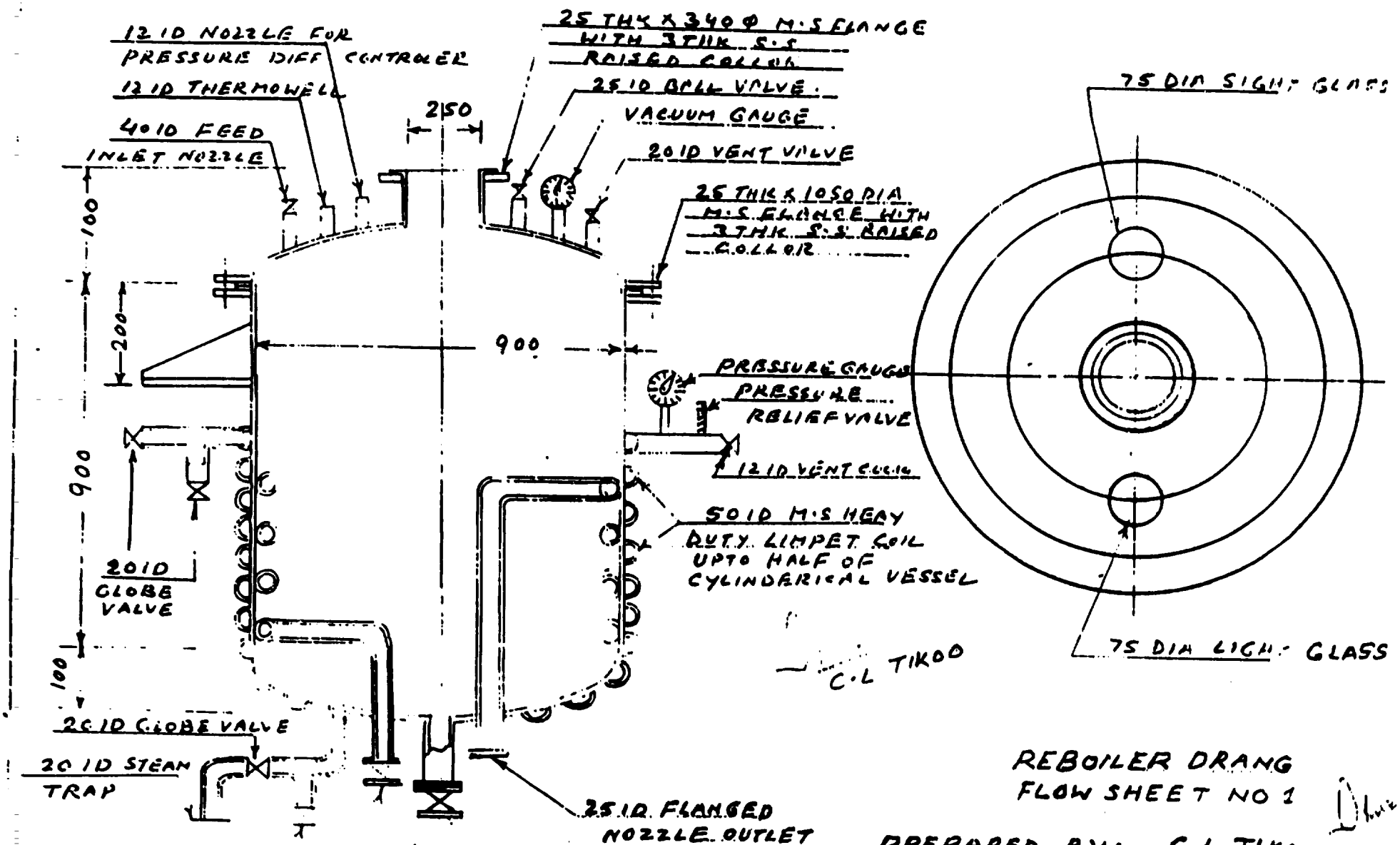


FRACTIONATING
COLUMN ASSEMBLY
AND FLOW SHEET

PREPARED BY :-
PLANT ENGINEER

PROJECT :- DP/VIE/86/



20 ID GLOBE VALVE
25 ID FLANGED TYPE CAST STEEL VALVE

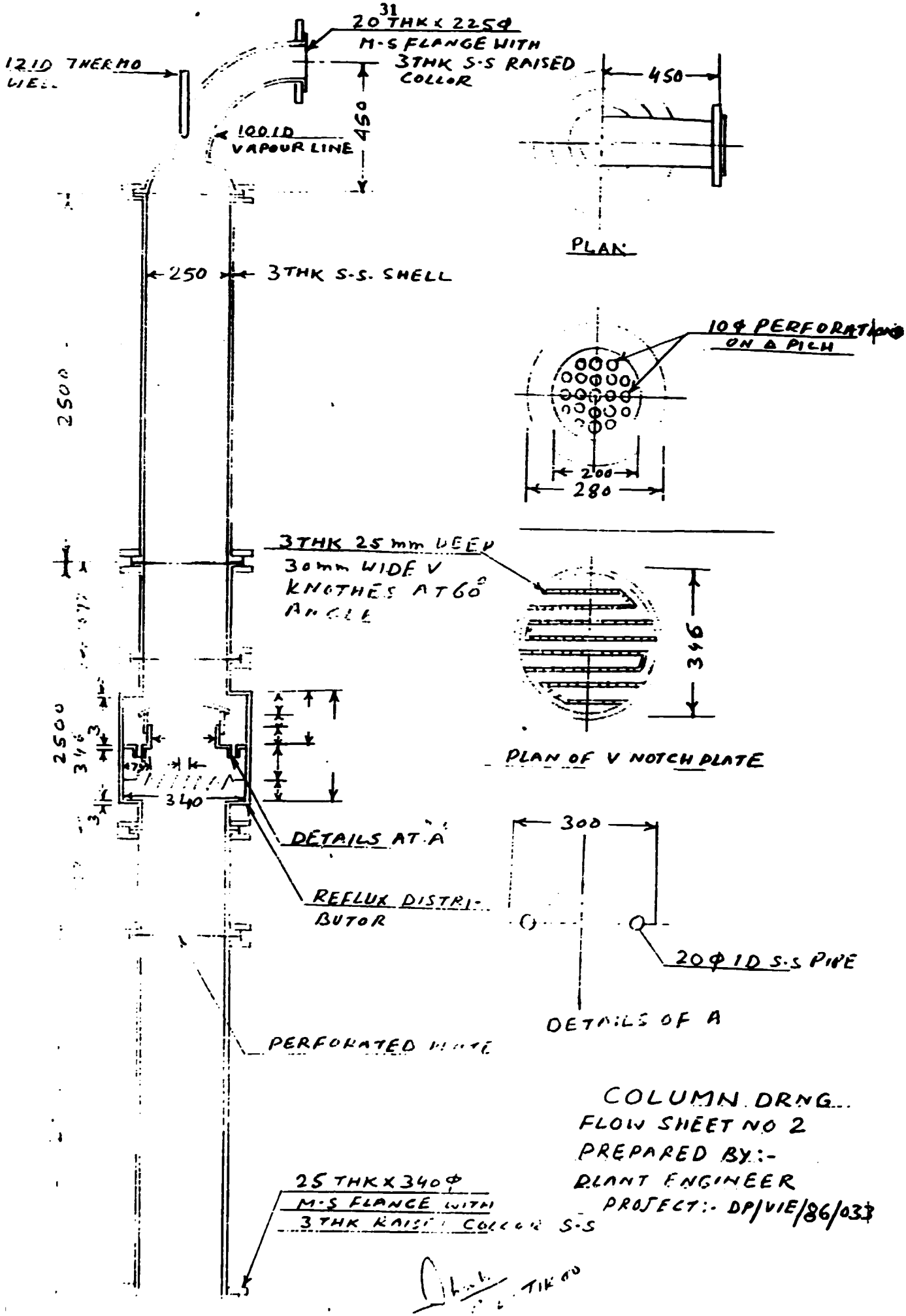
40 ID FLANGED TYPE S.S. BALL VALVE

NOTE:- ALL DIMENSIONS IN MM.

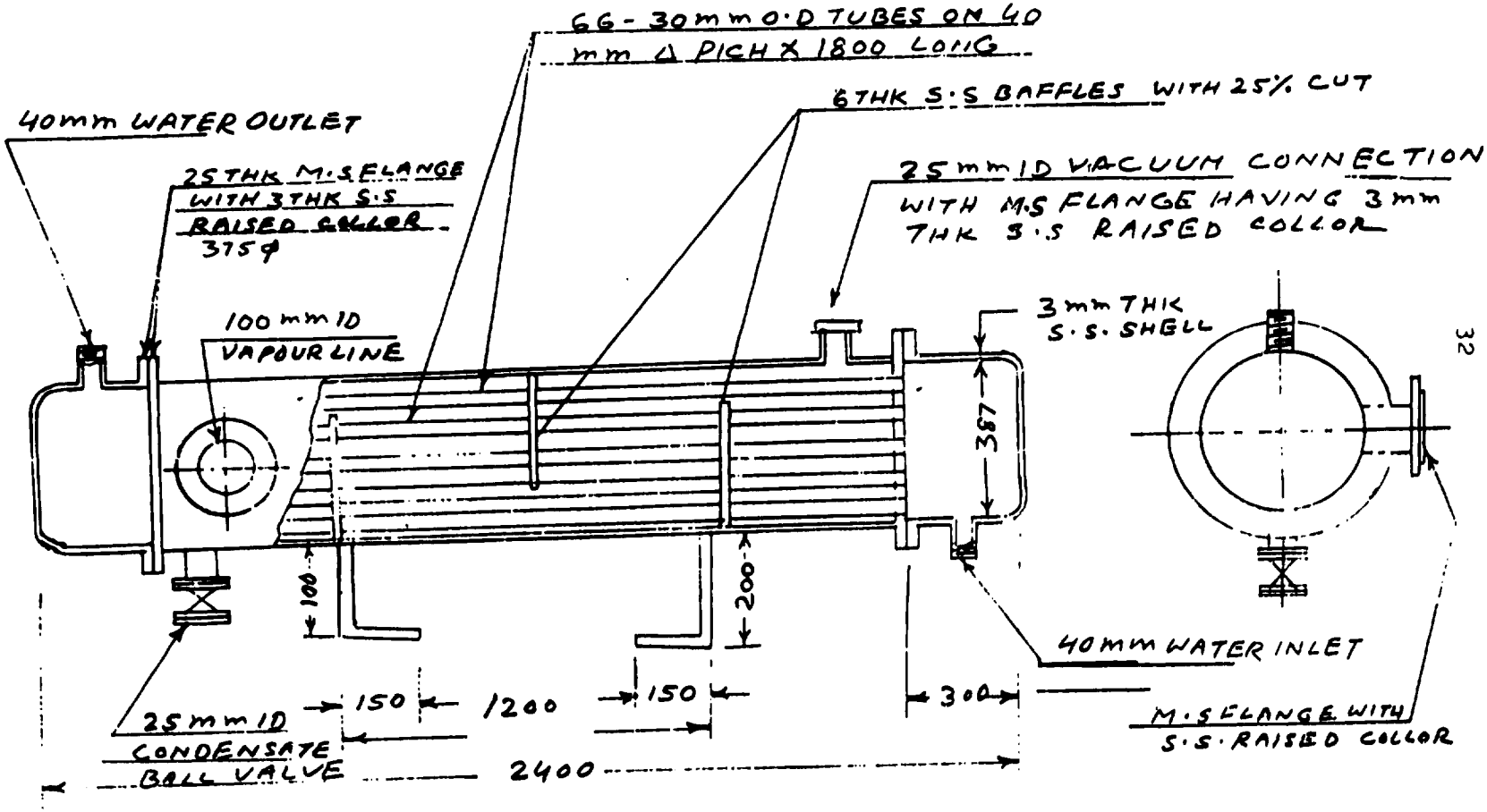
REBOILER DRANG
FLOW SHEET NO 1

PREPARED BY: C.L TIKOO
PLANT ENGINEER

PROJECT :- DP/VIE/86/033



COLUMN DRNG..
FLOW SHEET NO 2
PREPARED BY:-
PLANT ENGINEER
PROJECT:- DP/VIE/86/033



PREPARED BY:-

C.L. TIKOO PLANT ENGINEER

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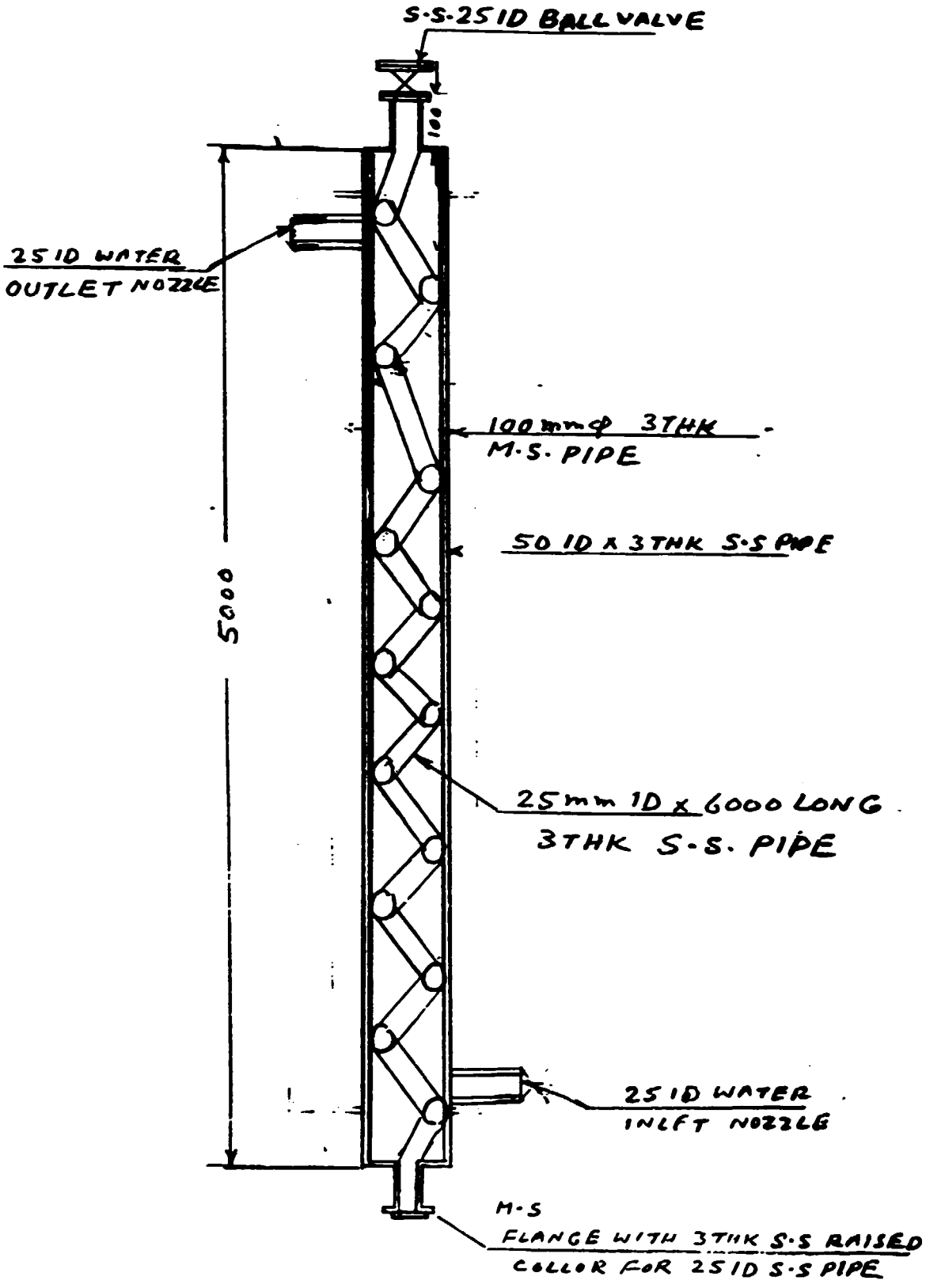
C.L. TIKOO

CONDENSER DRNG.

FLOW SHEET NO 4

NOTE:- ALL DIMENSIONS IN MM



C L TIKOO

PRODUCT COOLER DRNG

FLOWSHEE NO: 6

PREPARED By :-

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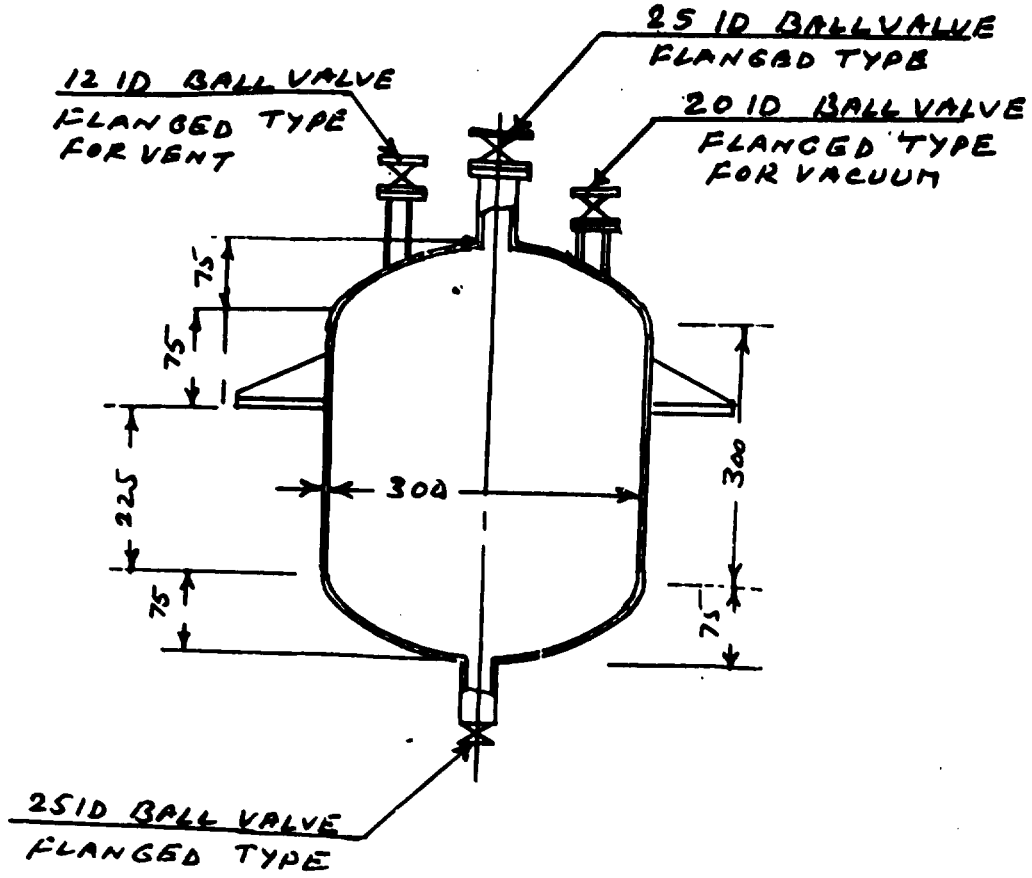
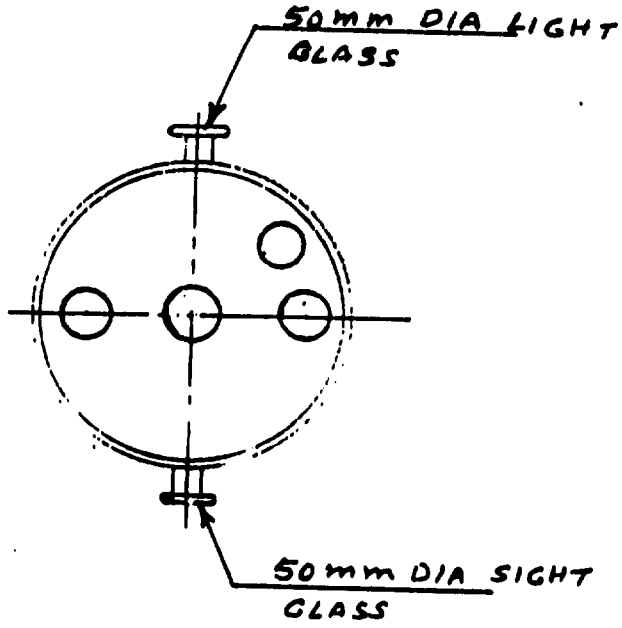




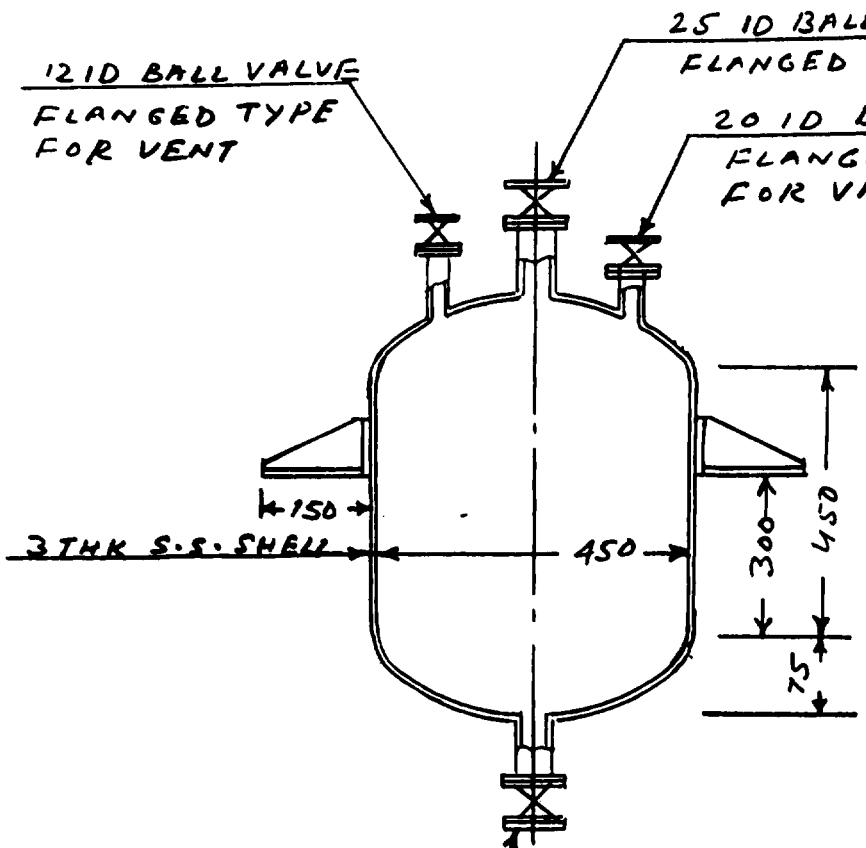
RECEIVER ORNG

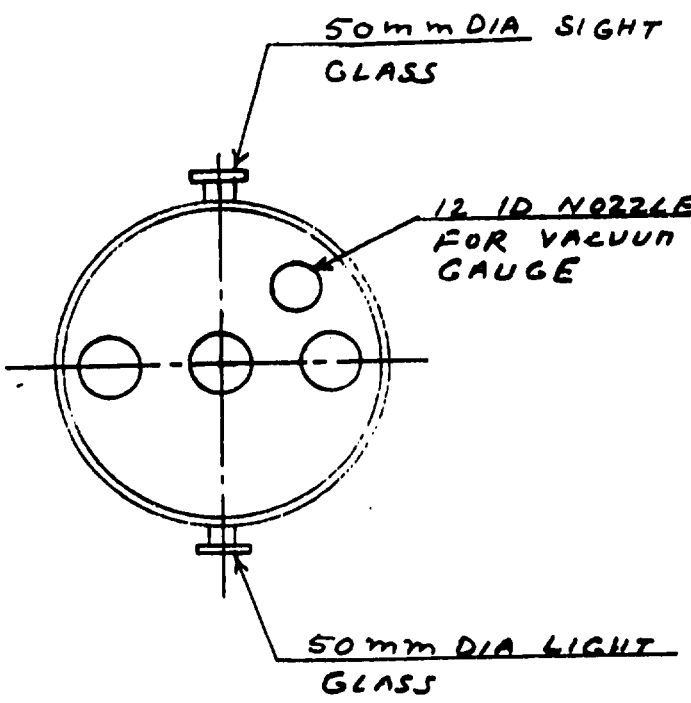
FLOWSHEET NO:- 9

PREPARED BY :-

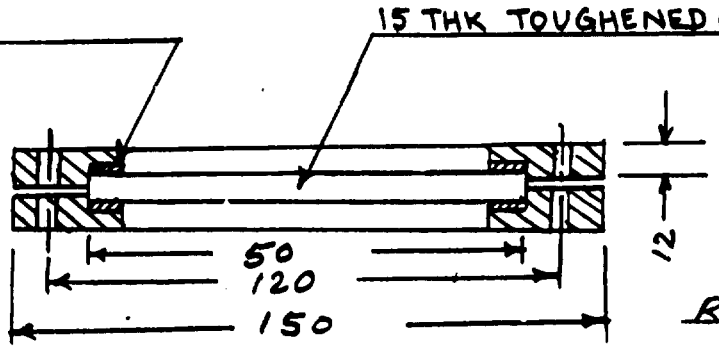
PLANT ENGINEER

PROJECT :- DP/VIE/86/0033





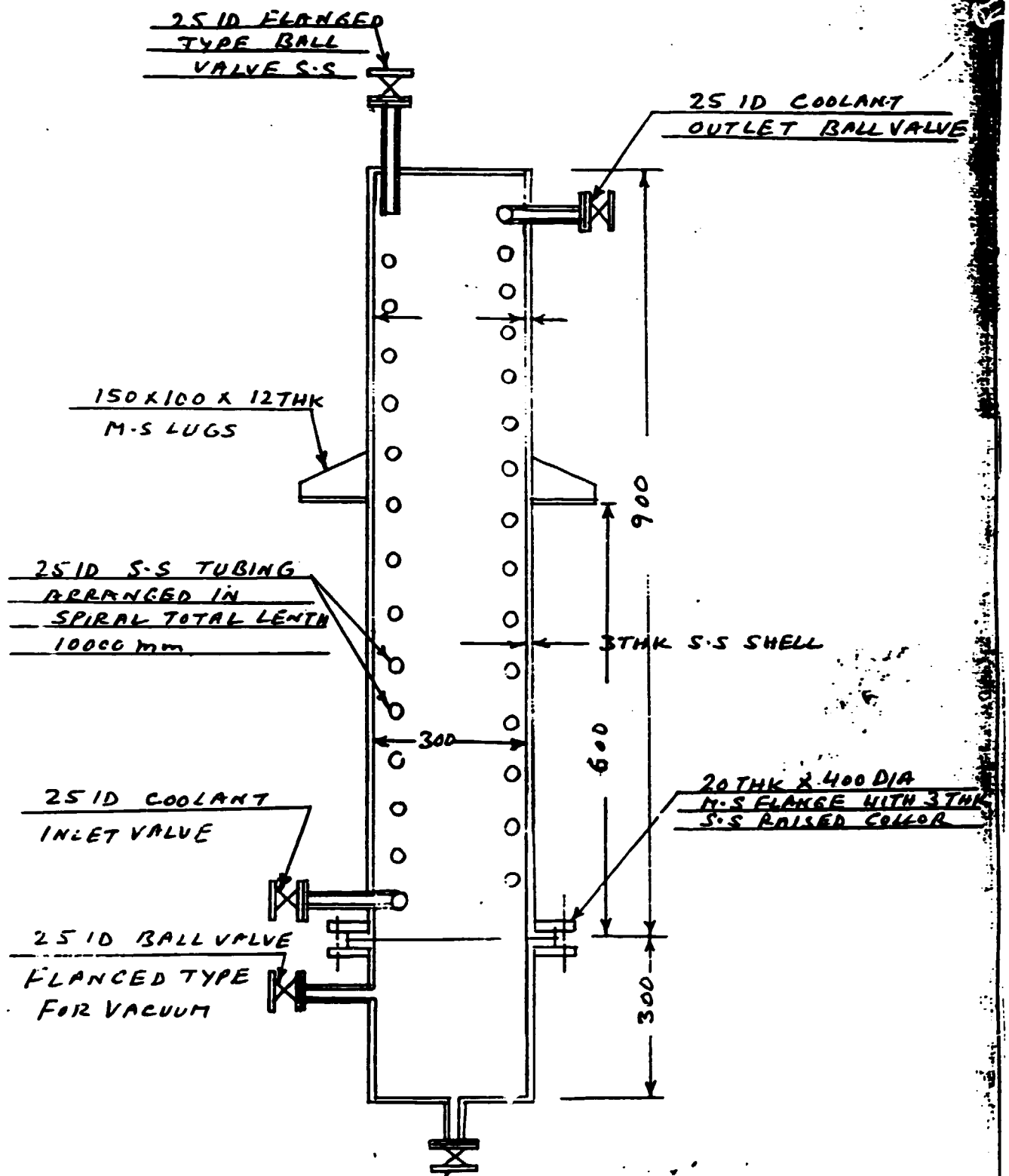
C.L. TILAK



DETAIL OF SIGHT GLASS

PREPARED BY:-
PLANT ENGINEER
PROJECT:- DP/VIE/86/001

RECEIVER DRNG
FLOW SHEET NO 8



VAPOUR TRAP DRNG

FLOWSHEET NO

PREPARED BY :-

PLANT ENGINEER

PROJECT :- DP/VIE/86/033

C.L. TIKOO

Backstopping Officer's Technical Comments
based on the work of Mr. C.L. Tikoo
DP/VIE/86/033/11-55

This report contains the activities of the consultant in detail. He has completed the work assigned except for what was expected of him during installation and commissioning of the pilot plant which unfortunately did not arrive as scheduled. His major task of training the counterpart staff in designing a commercial scale distillation unit has been successfully achieved. It is hoped that the Institute will fabricate this unit when large scale production is to be undertaken.

Design for power and water distillation systems and the pre-installation requirements for the pilot plant have been completed. The work to be undertaken by the supplier and the Institute when installing the pilot plant has been detailed. The Backstopping Officer wishes to stress the timely action needed with regard to the last two recommendations of the expert namely the need to have a stable power supply and a sump for neutralization of corrosive waste. These two items have to be done on a high priority basis before the installation and commissioning of the pilot plant.