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

21548

Distr.
RESTRICTED

ISED/R.69/Rev.1
28 May 1996

UNITED NATIONS
INDUSTRIAL DEVELOPMENT ORGANIZATION

ORIGINAL: ENGLISH

CONSULTANCY SERVICES FOR ENGINEERING DESIGN OF A PILOT PLANT FOR THE PRODUCTION OF IODIZED OIL

SF/THA/95/001/11-51

THAILAND

Technical report: Findings, work performed and recommendations*

Prepared for the Government of Thailand
by the United Nations Industrial Development Organization

Based on the work of N.B. Marla,
chemical technologist

Backstopping Officer: T. De Silva
Chemical Industries Branch

* This document has not been edited.

V.96-83883

TABLE OF CONTENTS

		Page No.
ABSTRACT		i
I.	**INTRODUCTION**	1
	Iodized oil, BP, USP	2
II.	**ACTIVITIES**	2
	Major constraints	2
	Process	3
	Site visit to the proposed location of pilot plant	4
	Design of pilot plant	4
III.	**CONCLUSIONS**	5
IV.	**RECOMMENDATIONS**	6
Acknowledgements		7

ANNEXES

Annex 1	-	Job Description	8
Annex 2	-	Design drawings of a pilot plant for the production of iodized oil	9-29
Annex 3	-	Specifications of standard equipment	30-32
Annex 4	-	A letter from the NPD for inclusion of silica gel column	33-34
Annex 5	-	A note on safety aspects of solvent extraction plants	35-37
Annex 6	-	List of persons contacted	38

ABSTRACT

The expert on mission to the project SF/THA/95/001 - Consultancy Services for Engineering Design of a Pilot Plant for the Production of Iodized Oil, was briefed by the Backstopping Officer at Vienna, during 27-28 November 1995, who gave him the benefit of advice and general guidelines in the implementation of the project.

It has been reported that "the Government of Thailand aims to reduce the number of people with iodine deficiency disorders (IDD) to just 5% of the current 15 million people suffering from IDD by December 1996". Hence the project to produce iodized oil assumes national importance for the health care of a large segment of the population of Thailand. Towards this end Research and Development Institute of Government Pharmaceutical Organization, developed a process to iodize a vegetable oil on a laboratory scale and entered into an agreement with UNIDO under a self financed project to provide the GPO an Engineering Design of a Pilot Plant for the Production of Iodized Oil.

The UNIDO expert on mission to the project fielded from 9 January 1995 to 13 February 1996, held discussion with the National Project Director (NPD) and the counterpart scientists who participated in the demonstration of the process at GPO. On careful evaluation of the process, the UNIDO consultant and NPD jointly came to the conclusion that the process suffers from various constraints/drawbacks and may not merit scale-up to the pilot plant stage, due to its inherent potential for causing environmental pollution and very long batch time and due to the formation of severe emulsions with wash water.

At the suggestion of NPD, that the production of iodized oil was of utmost importance for the health care of a large segment of Thai citizens suffering from IDD, the UNIDO consultant assisted the NPD and counterpart scientists in planning and successfully adapting and updating a published process (see "Activities" for reference) for iodization of soyabean oil to the desired level. Refining of iodized and esterified oil fatty acids, dark in colour on laboratory scale was under progress when the consultant left the mission site at the end of his mission.

It may be mentioned here, that the demonstration of the oil process and its evaluation, adaptation and modification of a published method took two weeks of mission time.

As a result of the application of the later process for iodization of soyabean oil, the number of modules of the pilot plant was considerably reduced, consequently reducing capital investment and lowering the cost of production of the product.

The combined efforts of the UNIDO consultant, NPD and the counterpart scientists in the adaptation and modification of a known process, made the safe and economic production of iodized oil possible.

The consultant discussed with the NPD the modules required for the pilot plant equipment and designed a pilot plant and worked out the specifications of the standard equipment to be purchased.

I. INTRODUCTION

It has been reported that an estimated 15 million people in Thailand are suffering from varying degrees of iodine deficiency disorders (IDD).

Lack of iodine causes slow mental and physical growth. Iodine deficiency can cause poor eye-hand coordination, partial paralysis, deaf-mutism, dwarfism, facial and physical deformity and brain damage.

Thailand aims to reduce the number of people with iodine deficiency to just five percent of the current estimated 15 million people suffering from IDD by next December, according to the Director General of the Health Department of the Government of Thailand.

Since the iodized oil project is of national importance for the health care of a large segment of the population of Thailand, Research and Development Institute, a Constituent of Government Pharmaceutical Organization (GPO), a wholly owned Government of Thailand Unit, developed a process to iodize a vegetable oil on a laboratory scale.

GPO sought and entered an agreement with UNIDO under a self financed project SF/THA/95/001 for providing consultancy services for engineering designs for a pilot plant for the production of iodized oil. Under this agreement, the consultant fielded by UNIDO from 9 January 1995 to 13 February 1996, was mandated to carry out the duties as given in the Job Description (Annex I).

The process developed at GPO for iodization of vegetable oil in brief was that hydriodic acid of 57% concentration was added dropwise to a mixture of red phosphorous and iodine in a flask fitted with a magnetic stirrer. The resulting HI gas dried over red phosphorous reacted with soyabean oil placed in a series of three erlenmeyer flasks, immersed in an ice bath (to absorb the heat of reaction), till the colour of soyabean oil changed to dark brown. Free iodine from the reactants was removed by washing 4 to 5 times with sodium metabisulphite solution. Excess sodium metabisulphite and hydrogen iodine ware removed by washing with warm water, until the pH of the solution reached about 6-7, entrapped water was then removed by extraction with absolute ethanol, until the solution became transparent. The traces of ethanol were removed under high vacuum.

The series of washing operations were repeated, if the colour of the resultant iodized oil was dark brown.

The light brown coloured iodized oil was esterified with sodium ethoxide under stirring. The esterified solution was neutralised with dilute hydrochloric acid. Traces of iodine, liberated during esterification process, excess acid, ethanol and salt were removed by repeated washing operations.

It was reported that the iodized oil obtained by this process matched with that of iodized oil procured from China.

The extra pharmacopoeia (1982-1994), of the Royal Pharmaceutical Society, U.K. volume 80 states that "Iodine is an essential trace element in the human diet, it is necessary for the formation of thyroid hormones. It is used for the prophylaxis and treatment of iodine deficiency disorders, such as endemic goitre, in areas where the diet is deficient in iodine. It may be administered as potassium or sodium iodide or as iodized oil".

"The chief alternative to supplementation with iodinated salt is iodized oil, usually given by intramuscular injection; it is useful where salt consumption is unreliable or inadequate or where immediate action is necessary to correct severe iodine deficiency".

The most commonly used type of iodized oil is poppy seed oil containing about 38% w/w of iodine. Single intramuscular dose of 1 ml. can provide adequate protection from iodine deficiency for at least 3 years, while a dose of 2 ml. provides adequate coverage for 3½ to 5 years.

Iodized oil, BP, USP

"A sterile iodine addition product of ethyl esters of fatty acids obtained from poppy seed oil. The B.P. injection specified 37% to 39% w/w, combined iodine. USP injection specify 35.2 to 38.9% of combined iodine. The injection is a straw coloured yellow or amber coloured oily liquid, which has not more than a slight alliaceous odour, practically insoluble in water. soluble in acetone, chloroform, ether, and petroleum spirit. The B.P. injection is sterilised by filtration. Store in an atmosphere of carbondioxide or nitrogen, protect from light".

II. ACTIVITIES

The consultant on mission fielded by UNIDO, arrived on 10 January 1996, at the site, Research and Development Institute (R & D Inst.) of GPO, Bangkok, held detailed discussions with the Director, the counterpart scientists and advisors of the project and reviewed the process that has been developed at the Institute. It was mutually agreed that the process would be demonstrated in the laboratory for evaluation and assessment, before the consultant sets about the designing of the pilot plant.

The consultant, at the end of the demonstration of a couple of batches, spread over a few days, came to the conclusion that the process developed at GPO, produces iodized oil of quality equivalent to that produced in China. However, it suffered from constraints/draw backs given below and may not merit scale-up to the pilot plant level because of the inherent potential for causing environmental pollution and formation of severe emulsions of reactants with wash water, which defied several methods for separation. As a result a batch may make 8-10 days for completion.

- a large amount of iodines was used.
- sublimed iodine deposited inside the reactor vapour lines and completely covered the red phosphorous used as drying agent.
- fairly a large amount of iodine also dissolved in the oil.
- to eliminate dissolved iodine from the oil, fairly a large amount of 8% sodium metabisulfite was used, two to three times, followed by washing with water 4-5 times, until washings were neutral.
- entrained water was removed by treating it with absolute ethanol.
- entrained ethanol was removed under high vacuum using a oil displacement vacuum pump.

Major constraints

Environment pollution.

- Every time the reaction was opened for recharging with a fresh lot of iodine (three times during one batch), iodine vapours escaped into the atmosphere.
- Formation of severe emulsions, every time when sodium metabisulphite solution was added and much worse when washed with water.
- Emulsion does not break up and defied several methods in its separation.

The Institute's Director who is also the NPD who actively followed the progress of the demonstration expressed her concurrence with the opinion of the consultant who however, suggested that since some sections of Thai citizens, estimated to be around 15 mission were suffering from iodine deficiency diseases and the production of iodized oil was of utmost national importance for the health

care of its people and cannot be postponed, hence an alternate process may be developed/adopted, suitable for scale-up.

The consultant, assisted the NPD and the scientists of the Institute in planning and successfully adapting and updating a published process "Iodocyclohexane" by Stone H, and Schahter H, Org. Synth. Coll. Vol. 4, 1963 PP 543-544, for the iodization of soyabean oil.

The adapted and modified process worked satisfactorily in the laboratory, yielding iodized oil of required degree of iodine content. However, the colour of iodized oil produced varied from amber to dark brown.

Process

The adapted, revised and updated process in brief was that 95% orthophosphoric acid was prepared by the reaction of 85% phosphoric acid with phosphoric anhydride. Potassium iodide was added to the cooled solution, followed by the addition of a requisite amount of refined soyabean oil. The mixture was stirred and heated to the desired temperature and for the appropriate period of time. It was allowed to cool and then extracted with n-hexane, the extract separated and washed with water, decolorised with aqueous sodium thiosulphate solution, washed with saturated sodium chloride solution and dried with anhydrous sodium sulphate. Hexane was evaporated on a steam bath.

The iodized oil thus obtained was esterified with sodium ethoxide and neutralised and dilute hydrochloric acid. The separated oily layer was decolorized with sodium thiosulphate solution and separated from the aqueous layer, washed with water till pH was 7. Separated oily layer was dried over anhydrous sodium sulphate and filtered.

The iodized fatty acids mixture thus obtained was of amber to dark brown in colour, combined iodine content varied from 35-40%.

Experimentation in the purification of the dark coloured iodized fatty acid, dissolved in n-hexane, over silica gel was being done at the time of departure of the consultant from Bangkok. The NPD (vide a fax message of 19 February 1996 - Annex 4) desired to include an ion exchange column for this purification. The same has been incorporated in the proposed pilot plant.

It may be mentioned that the demonstration of the old process, study of its results, adaptation and updating of a published method took two weeks of the mission time. However, it was an important and a very necessary step as otherwise production of iodized oil would have been both problematic and expensive.

As a result of adaptation and modification of a known process for iodization of soyabean oil, the number of modules of pilot plant could be considerably reduced, consequently leading to a considerable reduction in not only capital investment but also running costs of the pilot plant and in overall reduced cost of production of iodized oil.

At the end of this stage, the consultant in consultation with the NPD (Director, Research and Development Institute of Government Pharmaceutical Organization) regarding the modules of pilot plant equipment decided to provide the design and specifications of the following equipment:

1. Glass lined reactor of 250 litre volume with stirrer, variable speed drive (10-100 rpm), flame proof starter, shell and tube condenser, receiver, water ring vacuum pump with flame proof motor and flame proof starter.

2. Raw material storage tanks of AISI 316/AISI 304.

3. Centrifugal pumps.

4. Super centrifuge (10,000 - 15,000 rpm) of 200-500 litres per hour capacity, to be used for the separation of mild emulsions. This unit to be provided with an additional clarification bowl for the separation of fine suspended solids.

5. An additional conical separator for separation of oily and aqueous layers.

6. Rotameters to be included to measure the quantity of n-hexane, vegetable oil etc.

7. To include a silica gel filled column with accessories like overhead tank, receiver and feed pump for refining iodized fatty acids (final product).

8. Interconnecting process piping and service piping, i.e. water, steam, power connections.

9. Carbon steel support structure.

Site visit to the proposed location of pilot plant

The consultant accompanied by counter part scientists visited the proposed location of the iodized oil pilot plant at the Rangsit District located at about 50 km from Bangkok. The Chemical Plant and the premises are owned and managed by G.P.O. Adequate covered space was available to house the pilot plant. However, some old disused plant and equipment were lying all over the premises.

Effluents treatment plant was being constructed at a nearby site. The Engineer-in-Charge assured the consultant, that the effluent plant can treat both acidic and alkaline effluents and was of sufficiently large capacity to take care of the acidic effluents of the proposed iodized oil pilot plant.

A large water chilling plant to cool water from ambient temperature to about 9° C. was reported to be under construction for serving heat exchangers of the chemical plant. The Engineer-in-Charge conveyed that he could spare an adequate amount of chilled water to the pilot plant.

The details of power available at plant site according to the Engineer-in-Charge were:

220 volts, 50 Hz 1 phase
380 volts, 50 Hz 3 phase

The consultant noticed that the electrical wiring at the plant site was flame proof, time at his disposal was not enough to check-up the condition of wiring and switches at site. GPO authorities could make necessary arrangements to check-up and inform UNIDO.

Design of Pilot Plant

A pilot plant has been designed based on adapted, modified and updated process, to make iodized fatty esters using soyabean oil as the raw material. Depending upon the amount of chemicals and solvent used, the pilot plant would have the capacity to process about 30-50 litres of oil per batch.

The pilot plant consists of two glass-lined reactors of about 250 litres capacity. Each one is connected to a shell and tube condenser, receiver and a water ring vacuum pump.

The reactors, pumps, storage tanks, separators, super centrifuge, silica gel column, breather etc. are inter-connected (Annex 2) through a manifold of pipes and fittings in such a way that one of the two reactors could be put to use, to generate hydrogen iodide gas and to react with soyabean oil in situ to iodize the same. Simultaneously when one of the reactors is under iodization process, the other reactor could be used for esterification of iodized oil obtained from the previous batch. The rest of the equipment shown are supplementary/complimentary to these main reactors. Silica gel column is necessary for removing the colour of iodized and esterified fatty acids from dark brown to light yellow.

A safety measure has been provided in the form of a breather to trap the vapours of n-hexane a highly inflammable solvent and HI gas. The outlet from the breather to the atmosphere is the only point of vent, stripped of n-hexane vapour and HI gas to the entire pilot plant, located at a safe point above the pilot plant building.

The design drawings of the following equipment and specifications of standard equipment are enclosed (Annexes 2 and 3).

The design of the proposed pilot plant is both versatile and adequate to undertake upgradation of phosphoric acid from 85% to 95%, iodization of soyabean oil, partial bleaching and washing operations, preparation of requisite amount of sodium ethoxide, followed by esterification of iodized oil and series of neutralization, washing and drying operations.

The iodized, esterified oil could be further purified using the silica gel column to yield a straw yellow to light brown coloured product.

The pilot plant is so designed that when not in use for making iodized oils/fatty acid, it could be used for carrying out reactions, extractions, distillations and other evaporation operations.

III. CONCLUSIONS

It is concluded that the adapted, modified and updated process for iodization of soyabean oil, is both practical and superior to that process developed earlier at GPO.

That the process developed earlier at GPO has the inherent potential for causing environmental pollution and consumed large amounts of iodine and some amount of red phosphorous, an explosive material.

That the process takes about 8-10 days for completion of one batch due to the formation of severe emulsions during processing.

Hence it was concluded that the earlier process does not merit scale-up to pilot plant level.

The following criteria were taken into consideration for the choice of the process (adapted, modified and updated).

- Minimized environmental pollution.
- Safety to lives and property.
- Low cost of raw materials to reduce the cost of product.
- Import substitution.

The process proposed in itself is a safe and an economical method for the production of iodized oil, a vital health care product necessary in the prevention and effective treatment of iodine deficiency disorders.

IV. RECOMMENDATIONS

Refined soyabean oil has been used in all the laboratory experiments. To cut down the cost of production of iodized oil, commercial oil (unrefined) may be tried in the laboratory, before being used in pilot plant production.

Laboratory research should be continued at the R&D institute of GPO to optimize the use of chemicals and process time:

- for iodization of soyabean oil
- esterification
- refining of iodized and esterified fatty acids.

Optimization could lead to the increased capacity and overall economy in the production of iodized oil.

A single supplier may be chosen for the supply of the entire pilot plant, i.e. fabricated items of plant and equipment, standard items, interconnecting pipes and fittings, support carbon steel structures.

Pilot plant should be inspected in an installed condition at the premises of the suppliers, before despatch to the plant site in Bangkok/Rangsit, Thailand.

UNIDO has extensive and global experience in the procurement, installation and commissioning a wide range of pilot plants. It is recommended that the GPO use UNIDO services to set-up the pilot plant on turn-key basis.

The supply of the pilot plant should include the following spare/extra parts:

- two lengths of stainless steel pipes of each size and quality.
- welding electrodes suitable for welding AISI 304 and AISI 316 pipes/tubes and sheets.
- Two spare valves of each size used in the pilot plant.
- four pairs of flanges of each size used.
- Teflon gaskets 6 numbers of each size used.
- bolts and nuts with spring washers 20 numbers of each size used.

As considerable amounts of n-hexane, a highly inflammable solvent would be used in the pilot plant, when commissioned for the manufacture of iodized oil, it is imperative that the premises housing the pilot plant should be equipped with appropriate fire fighting equipment, preferably foam type and a fire proof storage area.

A note on safety aspects of solvent extraction plants is appended (Annex 5). These should be strictly followed during implementation of the project and operation of pilot plant.

Acknowledgements

The consultant acknowledges with thanks the advise and help extended by Mr. Tuley De Silva, Special Technical Adviser, Chemical Industries Branch, UNIDO, Vienna, Mr. Ari Huhtala, UNIDO Country Director, Bangkok, for briefing and follow up of the process, Ms. M. Hinte, Programme Officer, for the constant help and assistance during the consultant's stay in Bangkok, Dr. K. Kraisintu, Director, R & D Institute, GPO, Bangkok, for advise and the benefit of regular discussions and for whole heartedly placing the needed laboratory facility as well as two young scientific staff members of RDI for the demonstration of the earlier process and in the adaptation and successful modification of a published process for iodization of soyabean oil.

He also acknowledges with thanks the young counterpart scientists Mr. W. Sastrawathit and Ms. O. Srimun for full time assistance in the updated process development and Dr. (Ms.) Mayuree and Ms. T. Jaipetch for extending appropriate advice and help during demonstration and updated process development. Last but not least his thanks are due to Ms. P. Lakgrod for drawing the pilot plant equipment.

T. de Silva
5.10.95

Annex 1

UNIDO

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

JOB DESCRIPTION
SF/THA/95/001/11-51

Post Title: Chemical Technologist

Duration: 1.0 m/m

Date Required: ASAP

Duty Station: Bangkok, Thailand

Purpose of Project: Consultancy services for engineering design of a pilot plant for the production of iodized oil

Duties: The consultant in collaboration with the management and counterpart staff of Government Pharmaceutical Organization (GPO) of Thailand will carry out the following:

- Discuss and evaluate the requirements of the organization for a pilot plant for the production of iodized oil. The stages will consist of: generation of hydrogen iodide, drying of HI iodization, removal of iodine and water removal of traces of ethanol, esterification of iodized oil and purification of the iodized ester.

- Design the plant components required and prepare detailed engineering drawings with complete technical specifications, with the assitance of a draftsman .

- Prepare a list of accessory equipment (with detailed technical specifications) required for the commissioning of the plant.

- Provide details of services needed and lay out for the installation of the pilot plant.

F nally, the expert will furnish a report including the engineering drawing. and outlining his recommendations to both UNIDO and the Government Pharmaceutical Organization of Thailand.

Qualifications: A graduate in Chemical/Mechanical Engineering/Chemical Technology with over 10 years experience in engineering design of pilot scale chemical plants. Supervision and experience in fabrication of chemical reactors will be useful.

Language: English

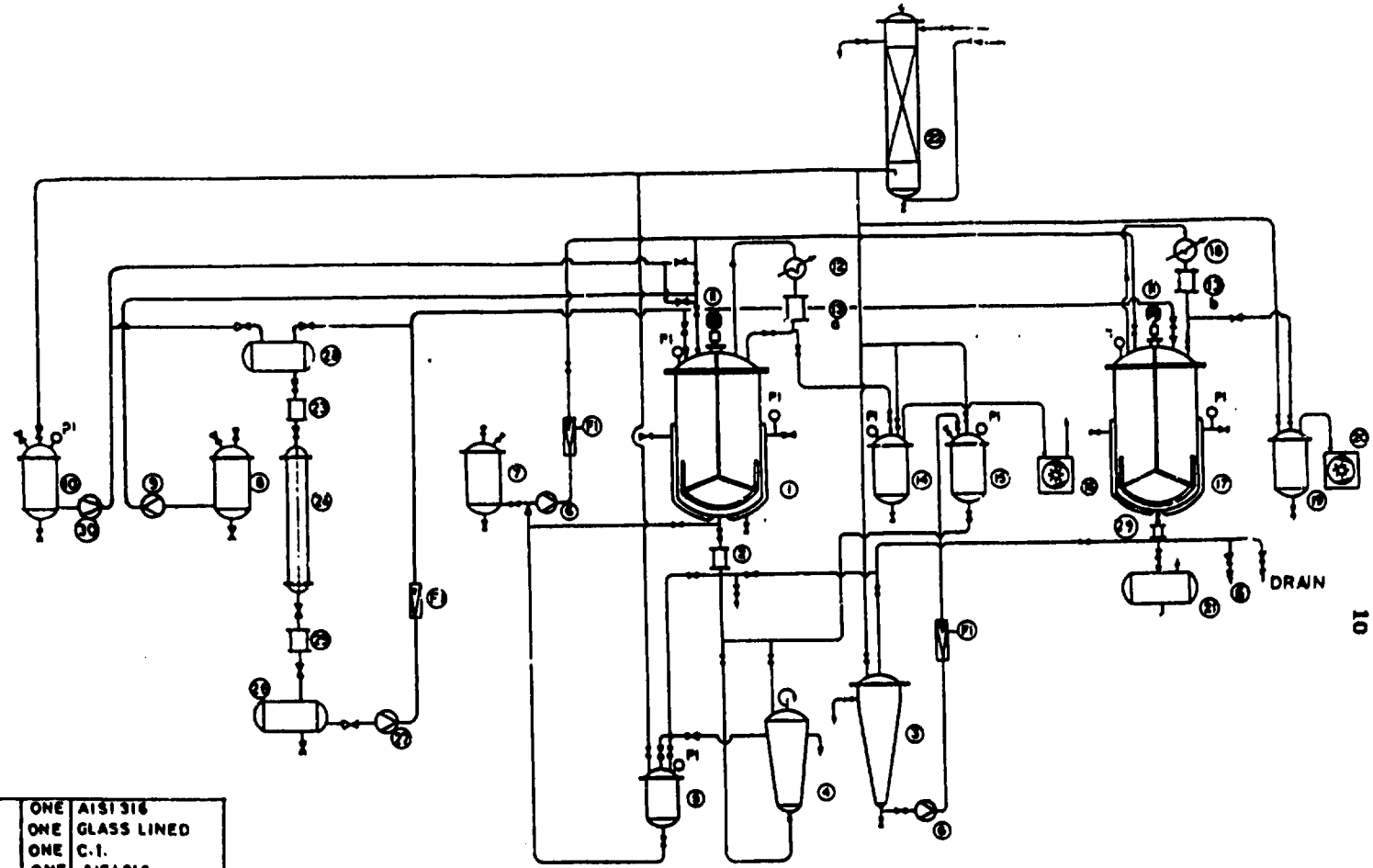
APPLICATIONS AND QUALIFICATIONS REGARDING THIS JOB DESCRIPTION
SHOULD BE SENT TO:
Project Personnel and Fellowship Service,
Operational Support Division,
UNIDO,
Vienna International Centre, P.O. Box 300, A1400 Vienna, Austria

DESIGN DRAWINGS

OF A

PILOT PLANT FOR THE PRODUCTION OF

IODIZED OIL



No	ITEM	REQD	MATL OF CONSTN.
18	HEAT EXCHANGER	ONE	AISI 316
17	REACTOR II	ONE	GLASS LINED
16	WATER RING VACUUM PUMP	ONE	C.I.
15	OVERHEAD TANK	ONE	AISI 316
14	RECEIVER	ONE	AISI 304
13	VISEUR (a & b)	ONE	GLASS
12	HEAT EXCHANGER	ONE	AISI 316
11	MOTOR & REDUCTION GEAR BOX	ONE	FLAME PROOF
10	HEXANE TANK	ONE	AISI 304
9	PUMP	ONE	AISI 304
8	OIL TANK	ONE	AISI 304
7	PHOSPHORIC ACID TANK	NOT	NEEDED
6	PUMP	ONE	AISI 316
5	MISCELLA TANK	ONE	AISI 316
4	SUPER CENTRIFUGE	ONE	AISI 316
3	SEPARATOR	ONE	AISI 304
2	VISEUR	ONE	GLASS
1	REACTOR I	ONE	GLASS LINED

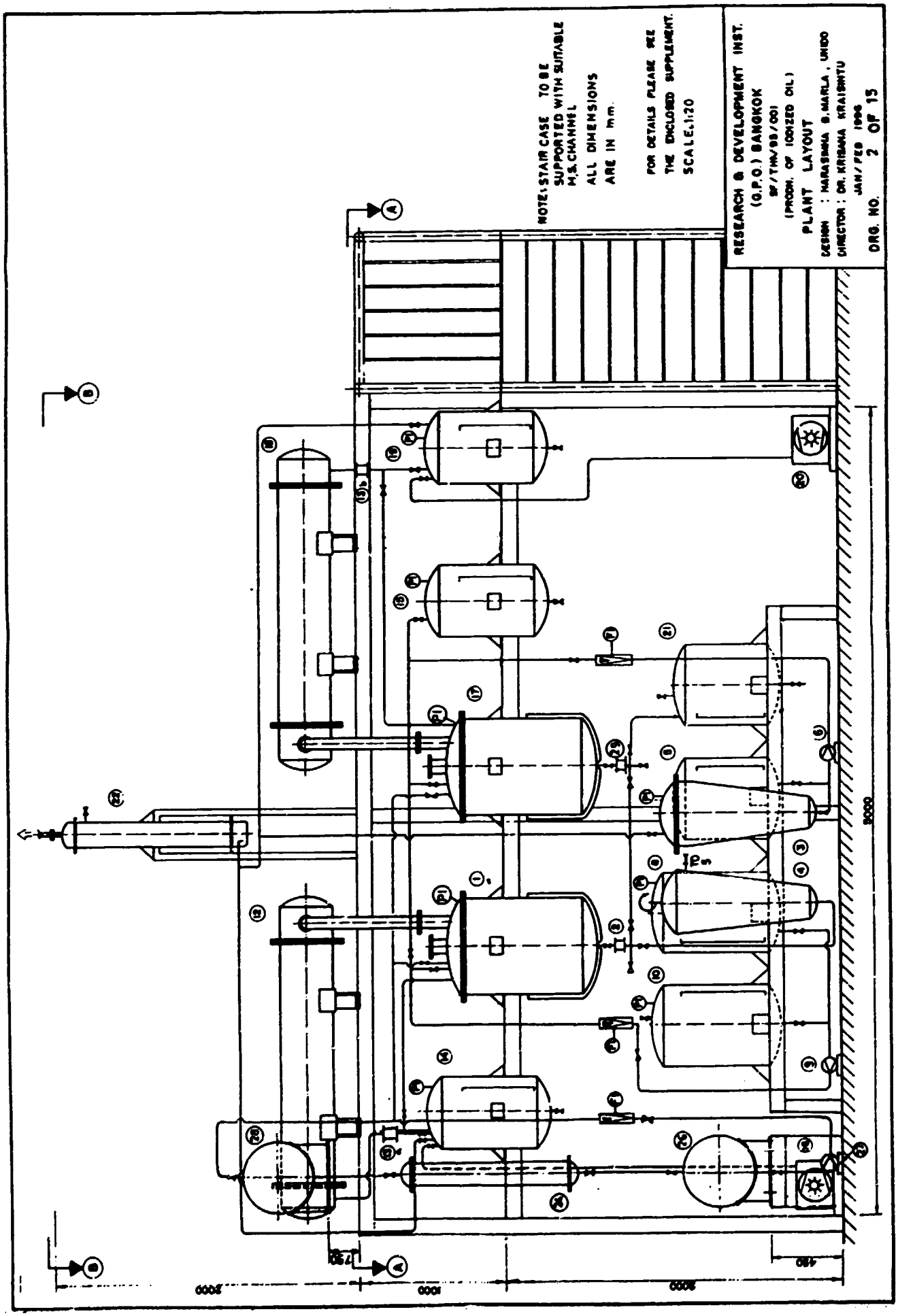
No	ITEM	REQD	MATL OF CONSTN.
30	PUMP	ONE	AISI 304
29	VISEUR	ONE	GLASS
28	OVER HEAD TANK	ONE	AISI 316
27	PUMP	ONE	AISI 316
26	REFINED MISCELLA TANK	ONE	AISI 316
25	VISEUR	ONE	GLASS
24	SILICA GEL COLUMN	ONE	AISI 316
23	VISEUR	ONE	GLASS
22	BREATHER	ONE	AISI 304
21	PRODUCT RECEIVER	ONE	AISI 316
20	WATER RING VACUUM PUMP	ONE	C.I.
19	RECEIVER	ONE	AISI 304

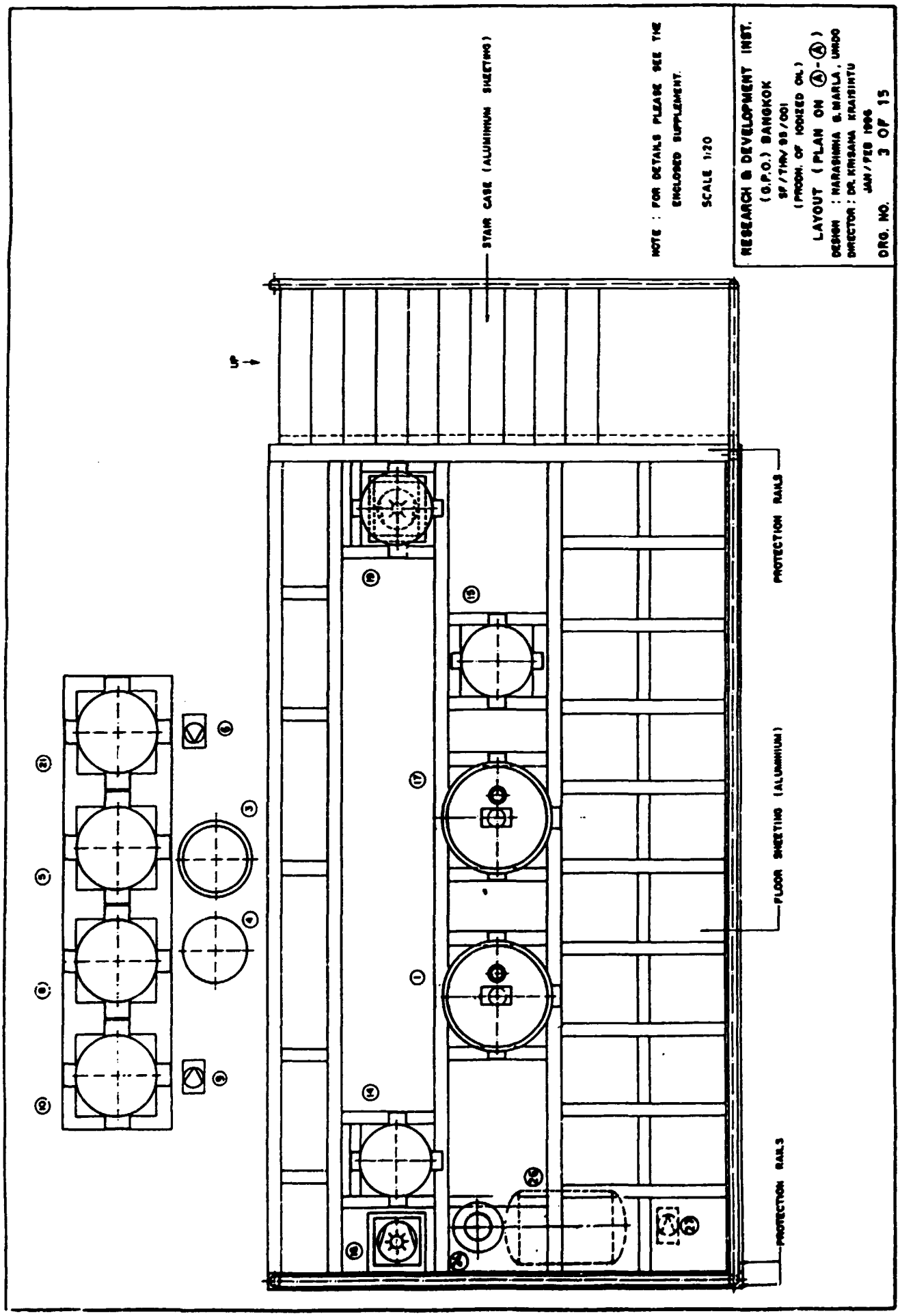
RESEARCH & DEVELOPMENT INST.
(G.P.O) BANGKOK
SP/ THI/ 95/001
(PRODN. OF IODIZED OIL)

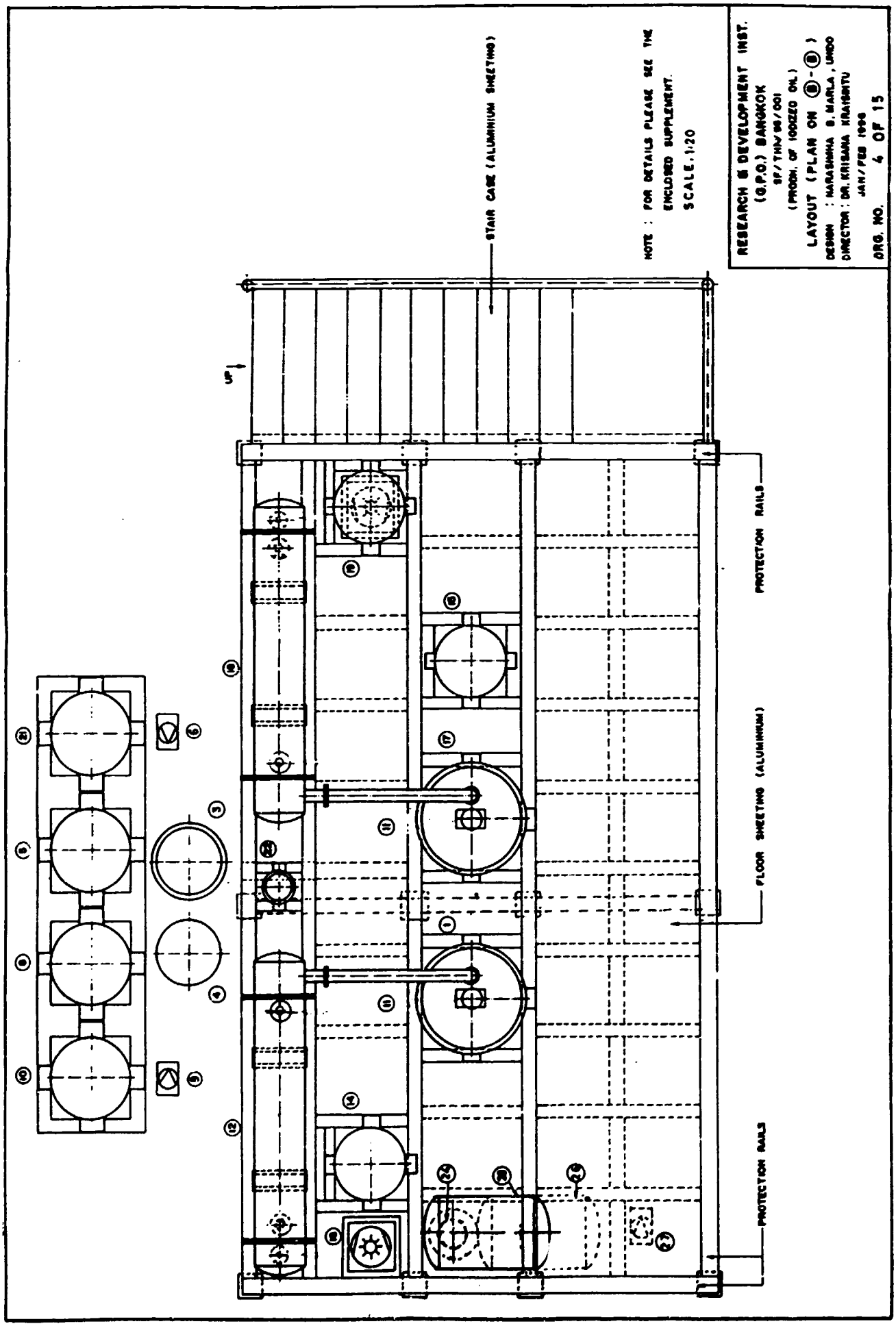
FLOW DIAGRAM

DESIGN: NARASIMHA. B. MARLA, UNIDO
DIRECTOR: KRISANA KRAISINTU
JAN/ FEB 1996

DRG. No. 1 OF 15

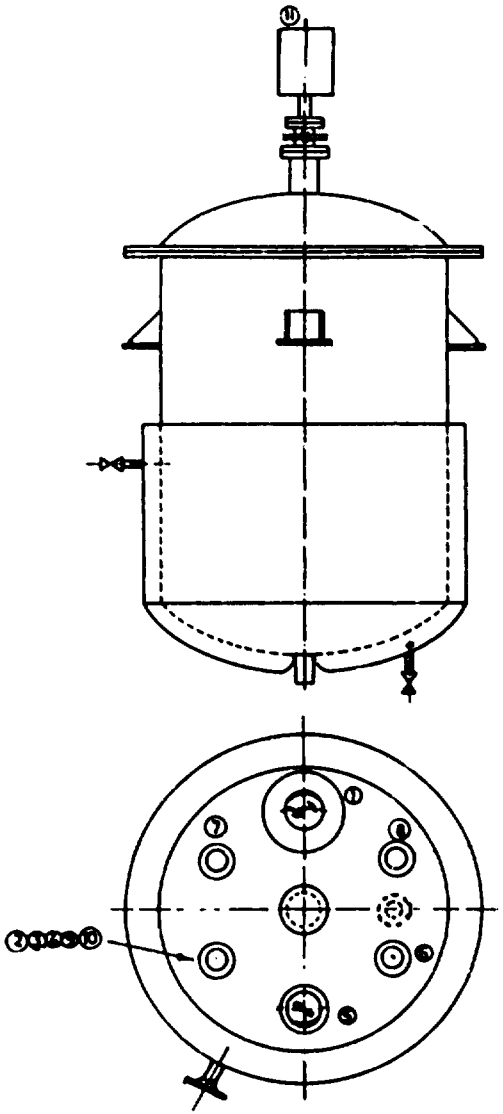

NOTE: STAIR CASE TO BE SUPPORTED WITH SUITABLE H.S. CHANNEL
ALL DIMENSIONS ARE IN mm.
FOR DETAILS PLEASE SEE THE ENCLOSED SUPPLEMENT.
SCALE 1:20
RESEARCH & DEVELOPMENT INST. (G.P.O.) BANGKOK
SF/THA/88/001
(PRODN. OF IODIZED OIL)
PLANT LAYOUT
DESIGN : NARASIMHA B.MARLA, UNIDO
DIRECTOR : DR. KRISANA KRAISINTU
JAN/FEB 1996
DRG. NO. 2 OF 15
9000
3000
1000
3000
750
450

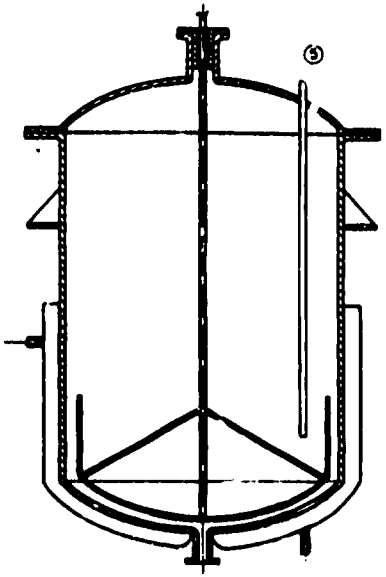

RESEARCH & DEVELOPMENT INST.
(G.P.O.) BANGKOK
SF/THV 98/001
(PROGM. OF IODIZED OIL)
LAYOUT (PLAN ON Ⓐ-Ⓐ)
DESIGN : NARASIMHA S.MARLA, UNIDO
DIRECTOR : DR. KRISANA KRAISINTU
JAN/FEB 1996
DRG. NO. 3 OF 15
NOTE : FOR DETAILS PLEASE SEE THE ENCLOSED SUPPLEMENT.
SCALE 1:20
STAIR CASE (ALUMINIUM SHEETING)
PROTECTION RAILS
FLOOR SHEETING (ALUMINIUM)
PROTECTION RAILS

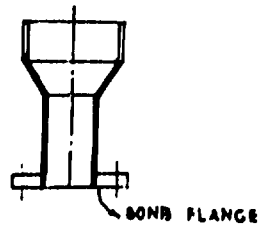

STAIR CASE (ALUMINIUM SHEETING)
NOTE : FOR DETAILS PLEASE SEE THE ENCLOSED SUPPLEMENT.
SCALE. 1:20
RESEARCH & DEVELOPMENT INST. (G.P.O.) BANGKOK
SF/THA/88/001
(PROGN. OF 100SIZED ONL)
LAYOUT (PLAN ON Ⓑ-Ⓔ)
DESIGN : NARASIMHA B. MARLA, UNIDO
DIRECTOR : DR. KRISANA KRAISRITU
JAN/FEB 1990
DRG. NO. 4 OF 15
PROTECTION RAILS
FLOOR SHEETING (ALUMINIUM)
PROTECTION RAILS

SUPPLEMENT TO DRG Nos. 2 OF 15, 3 OF 15, & 4 OF 15, PLANT LAYOUT

1. REACTOR 1
2. VISEUR
3. SEPARATOR
4. SUPERCENTRIFUGE
5. MISCELLA TANK
6. PUMP
7. -
8. OIL TANK
9. PUMP
10. HEXANE TANK
11. MOTOR AND REDUCTION GEAR BOX
12. HEAT EXCHANGER
13. VISEUR (a and b)
14. RECEIVER
15. OVERHEAD TANK
16. WATER RING VACUUM PUMP
17. REACTOR II
18. HEAT EXCHANGER
19. RECEIVER
20. WATER RING VACUUM PUMP
21. PRODUCT RECEIVER
22. BREATHER
23. VISEUR
24. SILICA GEL COLUMN
25. VISEUR
26. REFINED MISCELLA TANK
27. PUMP
28. OVER HEAD TANK
29. VISEUR







ALL CONTACT PARTS GLASS LINED
ALL NOZZLES ARE TO BE FITTED G.I. VALVES

NOTE - IMPORTANT

THIS DRG. IS FOR GENERAL GUIDELINES ONLY
STANDARD GLASSLINED REACTORS OF ABOUT 250 L VOLUME (EACH) - TWO NOS. ARE NEEDED.
CONNECTIONS ON TOP AND BOTTOM AS PER ENCLOSED

DUTY: REACTIONS INVOLVING PHOSPHORIC ACID 85% CONC. CRYSTAL POTASSIUM IODIDE HYDROGEN IODIDE GAS SODIUM METAL TO REACT WITH C_2H_5OH, n-Hexane, dil HYDROCHLORIC ACID
TEMP 70°C - 100°C
ALL GASKETS - TEFLON

RESEARCH & DEVELOPMENT INST.
(G.P.O.) BANGKOK
SF / THA / 85 / 001
(PRODN. OF IODIZED OIL)
REACTOR I & II
DESIGN : NARASIMHA S.MARLA, UNIDO
DIRECTOR : DR. KRISANA KRAISINTU
JAN / FEB 1986
DRG. NO. 5 OF 15 (ITEM - 1, 17)

SUPPLEMENT TO DRG.NO.5 OF 15 REACTOR

CAPACITY ABOUT	:	250 L VOLUME
MAT.OF CONSTN.	:	ALL CONTACT PARTS. INCLUDING STIRRER Etc, FLANGES, VALVES, GLASS-LINED.
STIRRER	:	ANCHOR TYPE, VARIABLE SPEED
SUITABLE	:	ADJUSTABLE FROM 10 TO 100 R.P.M.
MOTOR & STARTER	:	FLAME PROOF
POWER AVAILABLE	:	220V $50H_z$, 1 PHASE
AT SITE	:	380V $50H_z$, 3 PHASE
JACKETED	:	FOR STEAM HEATING CUM WATER COOLING
MAX. TEMP	:	120°c
THERMOSTATIC	:	$\pm$ 0.5°c
CONTROL		
INSULATION	:	50mm thick GLASS-WOOL TO COVER ENTIRE REACTOR & BOTTOM DISH, CYLINDRICAL PORTION TOP DISH, VAPOUR OUTLET LINE UPTO CONDENSER AND COVER IT WITH 2mm AISI 304 SHEET. FINISH GRIND POLISHED TO GRIT 180
THERMOWELL WITH THERMO COUPLE	:	SUSPENDED FROM TOP UPTO LOWER PORTION OF THE REACTOR. TO BE FITTED WITH DIGITAL INDICATING THERMOMETER

CONNECTIONS ON TOP (Ret.DRG.3 of 12)

MANHOLE	:	CIRCULAR/OVAL MANHOLE 150 NB WITH INSET SIGHTGLASS FITTED WITH FLAME PROOF LIGHT FLAME PROOF SWITCH. (1).
NOZZLE	:	FOR THERMO WELL WITH DIGITAL INDICATING THERMOMETER 0-150°c (RANGE) (6)

NOZZLE : 80 NB FOR LIGHT GLASS (5)

NOZZLE : FITTED WITH VACUUM MEASURING INSTRUMENT, DIGITAL INDICATING TYPE.(7)

FLANGED GLASS LINED NOZZLE 50 NB

FITTED WITH 50 NB GLASS LINED VALVE, AND A HOPPER (AISI 316) 150 NB (TOP) 150 Ht. FOR CHARGING CRYSTALLINE POWDERS, ORGANIC AND INORGANIC ACIDS (2,3,4,9 & 10)

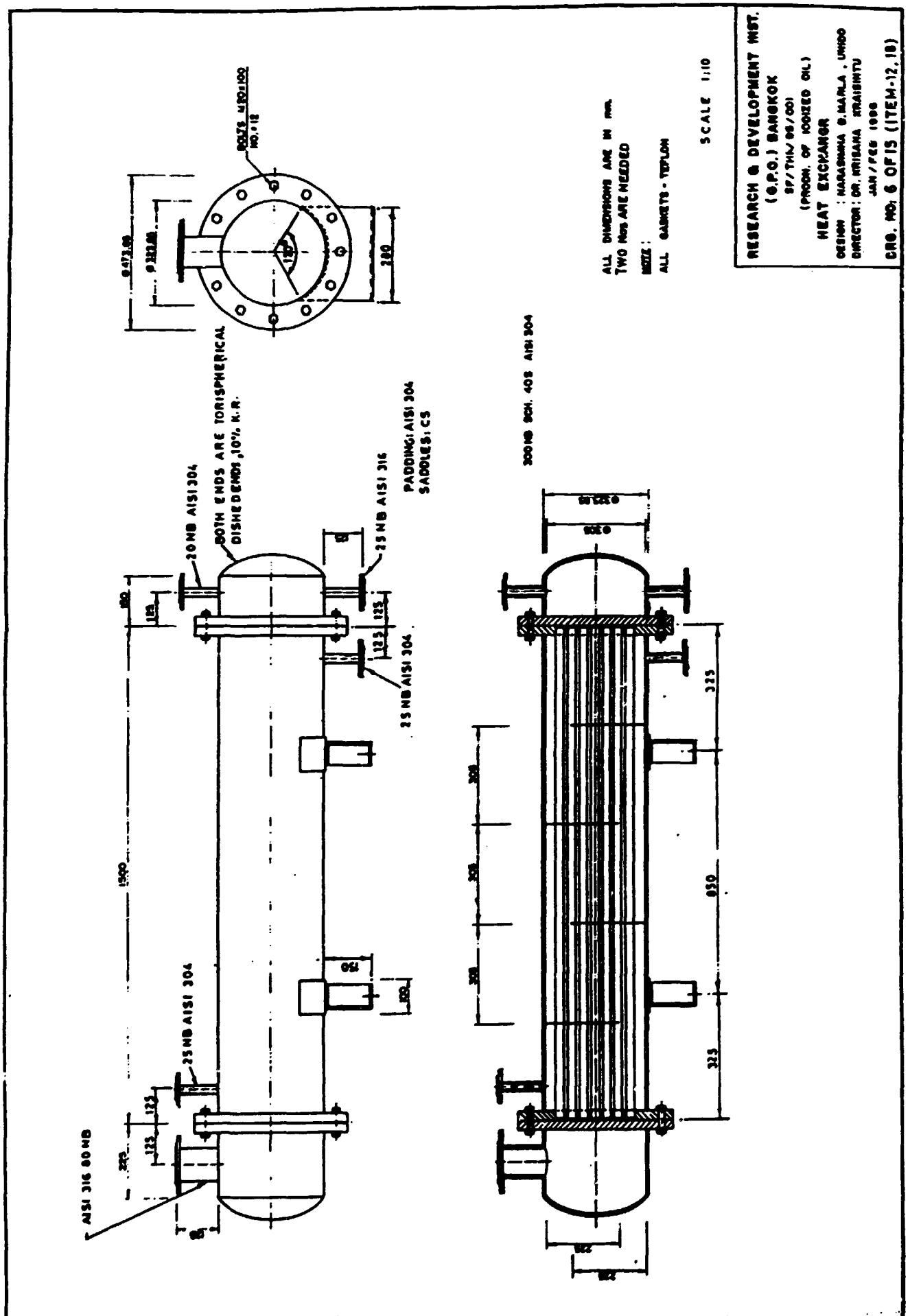
FLANGED GLASS-LINED

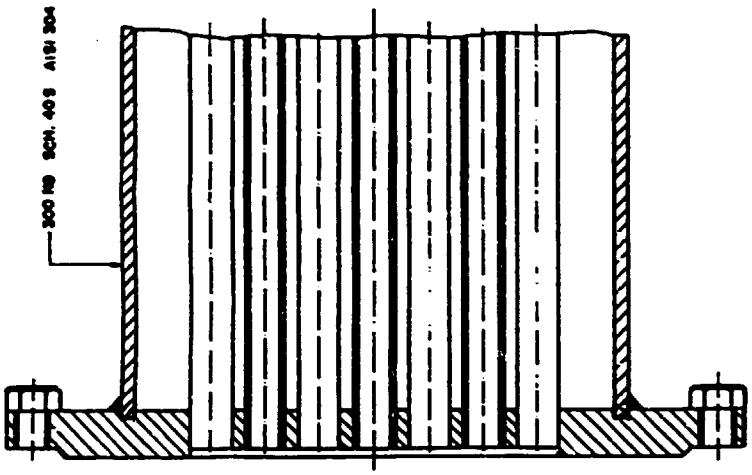
NOZZLE : 80 NB : FOR VAPOUR OUTLET. (8)

BOTTOM CONNECTIONS : FLANGED BOTTOM OUTLET 50 ϕ FLUSH WITH BOTTOM, FITTED WITH Y-TYPE GLASS LINED VALUE.

ON JACKET : TEE-NOZZLE FOR STEAM INLET WITH 25 NB VALVE.

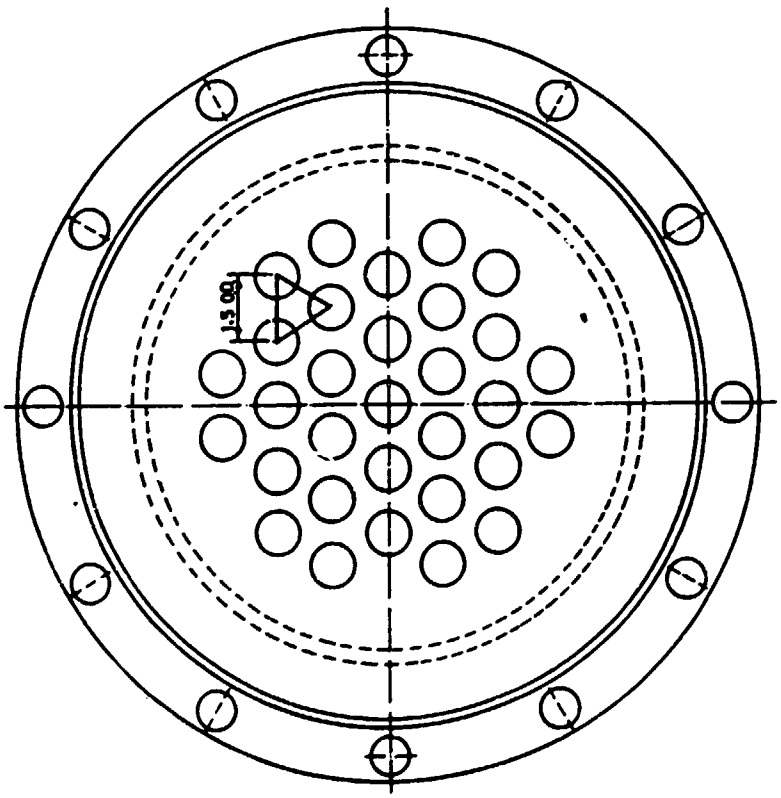
NOZZLE 15 NB WITH VENT COCK, PRESSURE GAUGE, SAFETY VALVE. CONDENSATE OUTLET WITH STEAM TRAP, WITH 'TEE' CONNECTION FOR WATER INLET.


RESEARCH & DEVELOPMENT INST.
(O.P.O.) BANGKOK
BP/THA/85/001
(PROGR. OF IODIZED OIL)
HEAT EXCHANGER
DESIGN : NARASIMHA B.MARLA , UNIDO
DIRECTOR : DR. KRISANA KRAISINTU
JAN/FEB 1986
DRG. NO. 6 OF 15 (ITEM-12,18)
ALL DIMENSIONS ARE IN mm.
TWO Nos ARE NEEDED
NOTE :
ALL GASKETS - TEFLON
SCALE 1:10
BOLTS M20x100
NO.12
20 NB AISI 304
BOTH ENDS ARE TORISPHERICAL
DISHED ENDS ,10% K.R.
25 NB AISI 316
PADDING: AISI 304
SADDLES: CS
25 NB AISI 304
25 NB AISI 304
AISI 316 80 NB
300 NB SCH. 40S AISI 304
1500
850
325
325



NOTE :
ALL GASKETS - TEFLON
TWO Nos ARE NEEDED

SCALE : 1:3



RESEARCH & DEVELOPMENT INST.
(R.P.O.) BANGKOK
SF/THA/93/001
(PROGR. OF ROUZED OIL)
DETAILS OF TUBEPLATE
DESIGN : NARASHIMA S.MARLA, UNIDO
DIRECTOR : DR. KRISANA KRAISINTU
JAN/FEB 1996
DRG. NO.7 OF 15 (ITEM-12, 18)

SUPPLEMENT TO DRG.Nos.6 OF 15, 7 OF 15
HEAT EXCHANGER

SHELL AND TUBE-TYPE

- SHELL AND TUBE WITH CROSS FLOW BAFFLING.
- COOLING WATER PASSES THROUGH SHELL SIDE.

SHELL : 300 NB. SCH 40 S, AISI 304 PIPE
WALL THICKNESS 9.52
LENGTH 1500

TUBES : 20 NB, SCH 40S, AISI 316 TUBES
NUMBER OF TUBES 31
TUBE LENGTH 1500
TRIANGULAR PITCH 1.5 x TUBE OD

TUBE SHEET: THICKNESS : 20 (AISI 316)
FLANGE : 25

BAFFLES: SEGMENTAL BAFFLES
HEIGHT: 75% OF SHELL ID
THICKNESS: 6, AISI 316
SPACING: 305

NOTE: WHERE EVER POSSIBLE, BAFFLES TO BE WELDED TO INSIDE OF SHELL

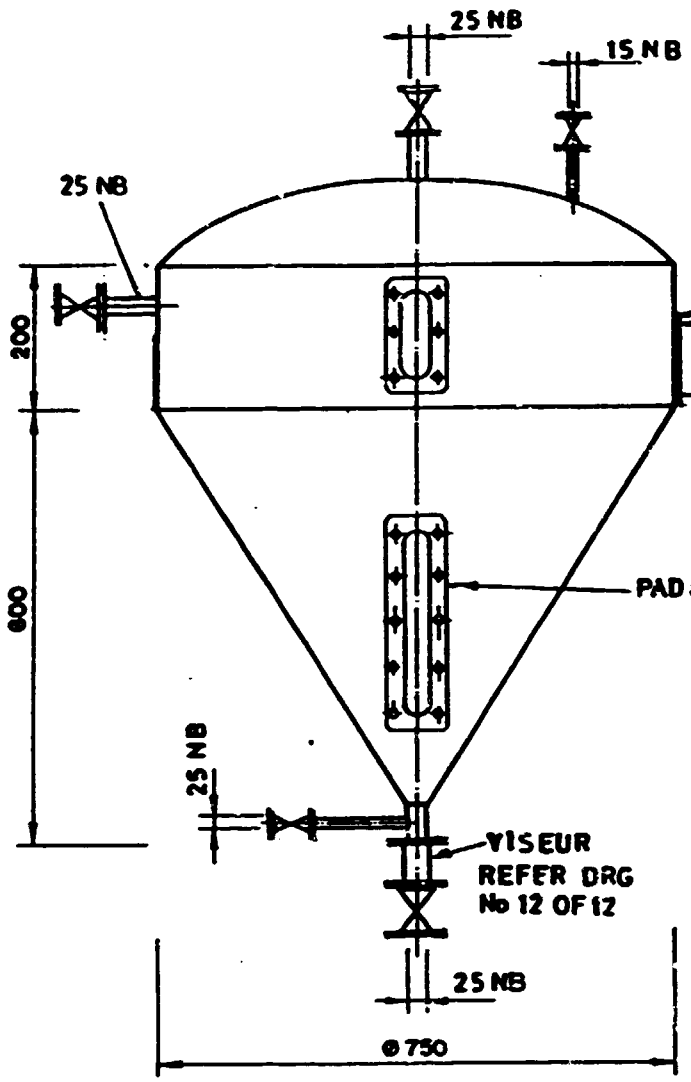
BONNETS :

VAPOUR INLET SIDE

300 NB, SCH. 40S PIPE, AISI 316 LENGTH:225, DISHED END WELDED ON ONE SIDE, THE OTHER END WELDED TO TO A FLANGE (AISI 316) WITH RAISED FACE AND CIRCULAR GROOVES. FLANGED NOZZLE 80 NB SCH 40S AISI 316 WITH RAISED FACE AND CIRCULAR GROOVES. WELDED FOR VOPOUR INLET.

CONDENSATE OUTLET SIDE: 300 NB SCH 40S AISI 316 PIPE SEGMENT 150 LENGTH. SAME AS VAPOUR INLET SIDE EXCEPT CONDENSATE OUTLET. IT SHOULD BE FLANGED NOZZLE 25 NB SCH 10S AISI 316

NOTE: FINISH WHEREVER POSSIBLE, OUT SIDE SURFACES TO BE GROUND POLISHED TO GRIT 180

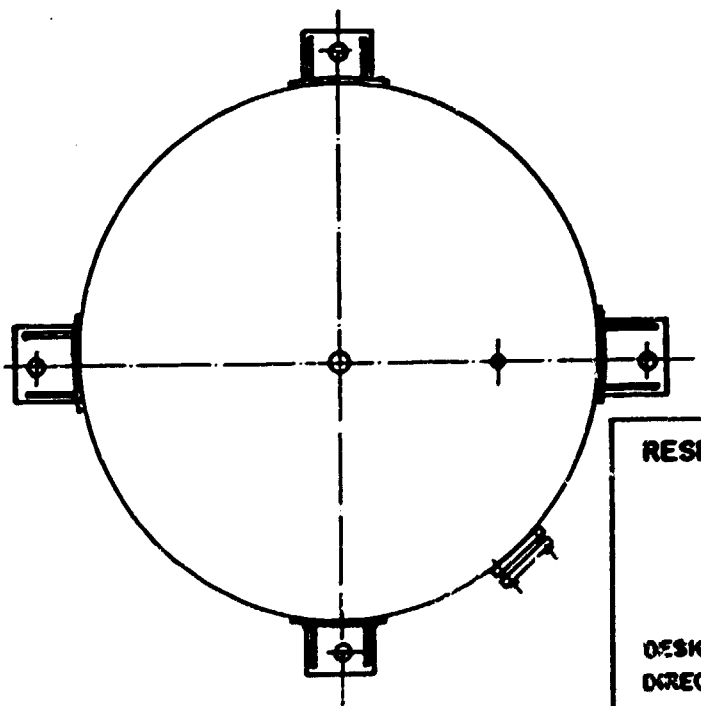


THICKNESS : 5

VALVES : BALL VALVES

MAT. CONSTRUCTION AISI 316

ALL DIMENSIONS ARE IN mm



SCALE 1:10

RESEARCH & DEVELOPMENT INST.
(G.P.O.) BANGKOK

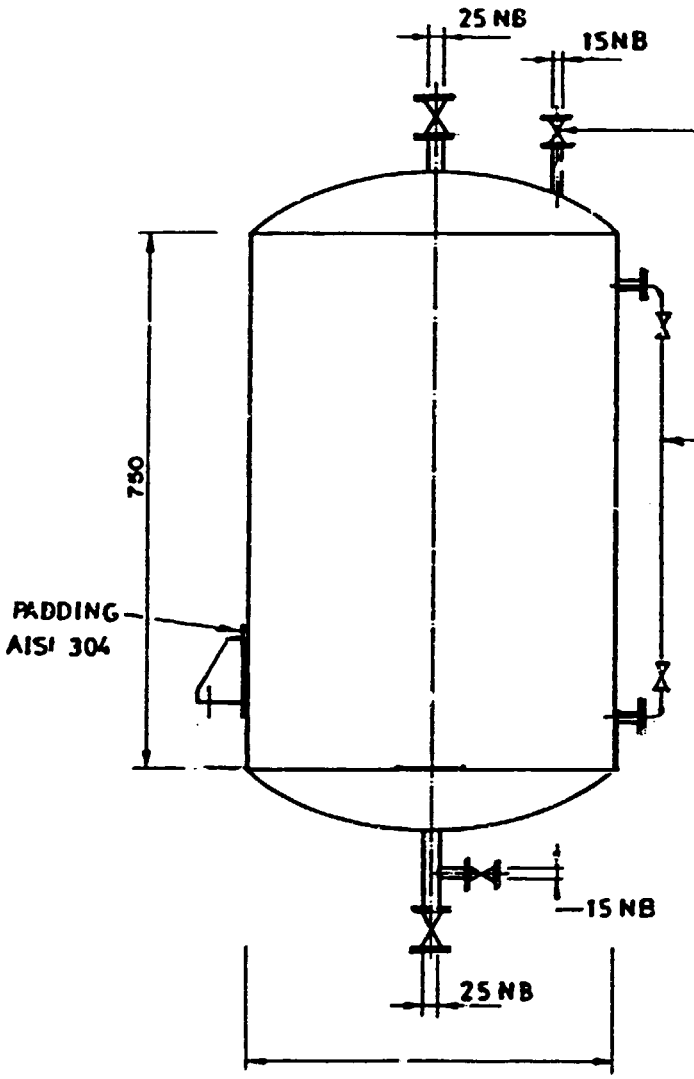
SF / THA / 95 / 001
(PRODN. OF IODIZED OIL)

SEPARATOR

DESIGN : NARASIMHA B. MARLA , UNIDO
DIRECTOR : DR. KRISANA KRAISINTU

JAN / FEB 1996

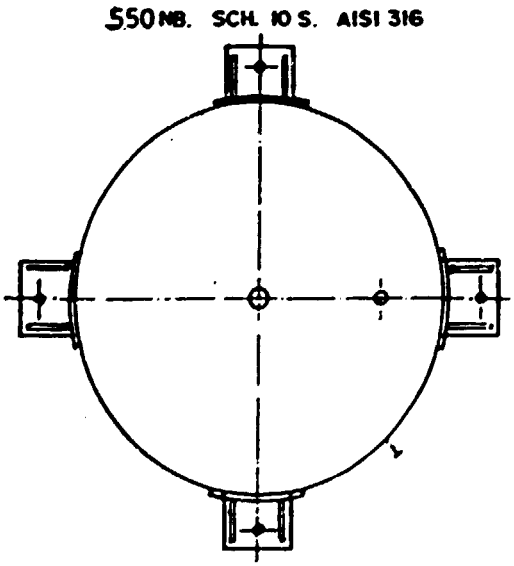
DRG. NO. 8 OF 15 (ITEM-3)



ALL DIMENSIONS ARE IN mm

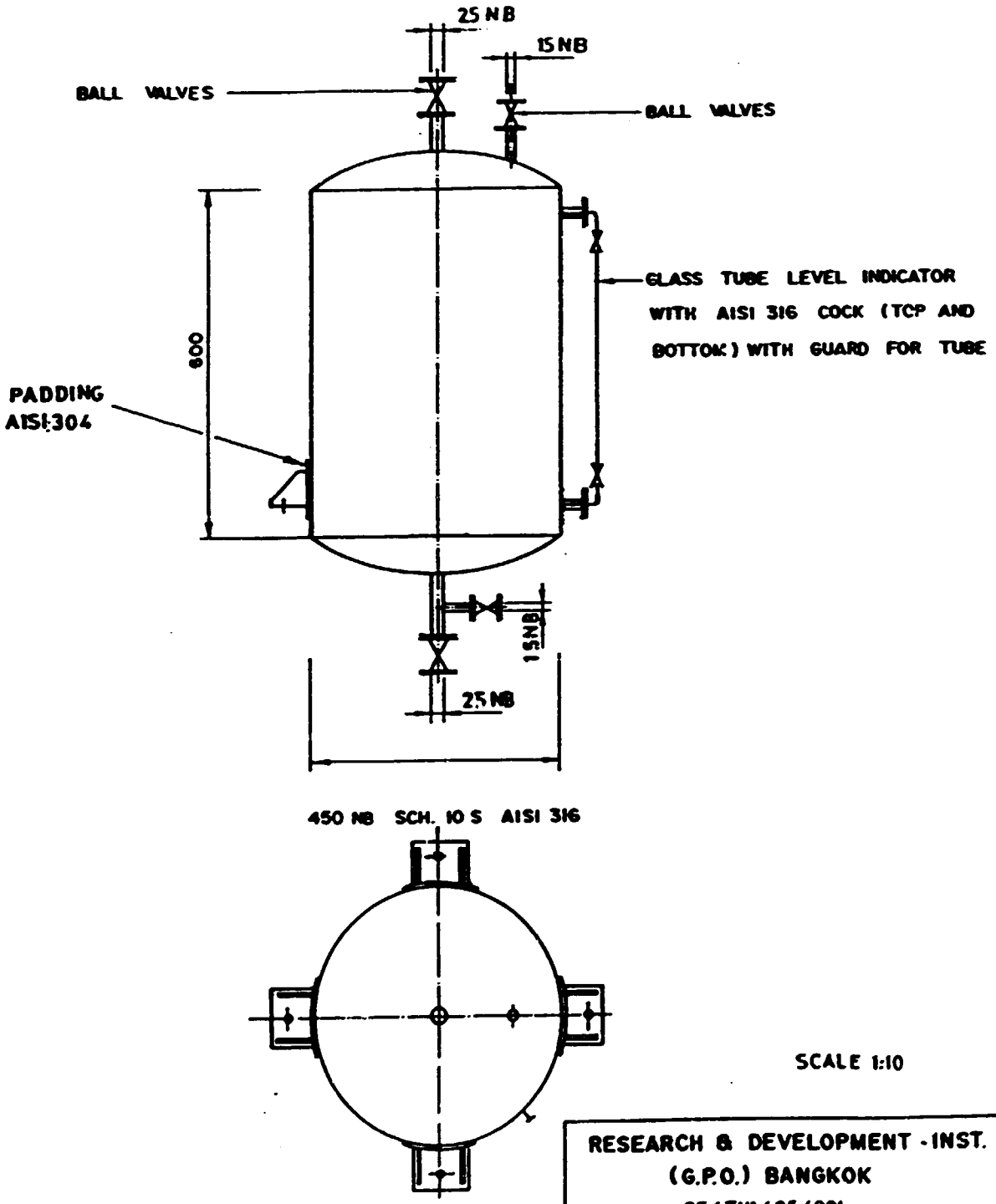
ALL BALL VALVES: AISI 316

ALL GASKETS: TEFLON



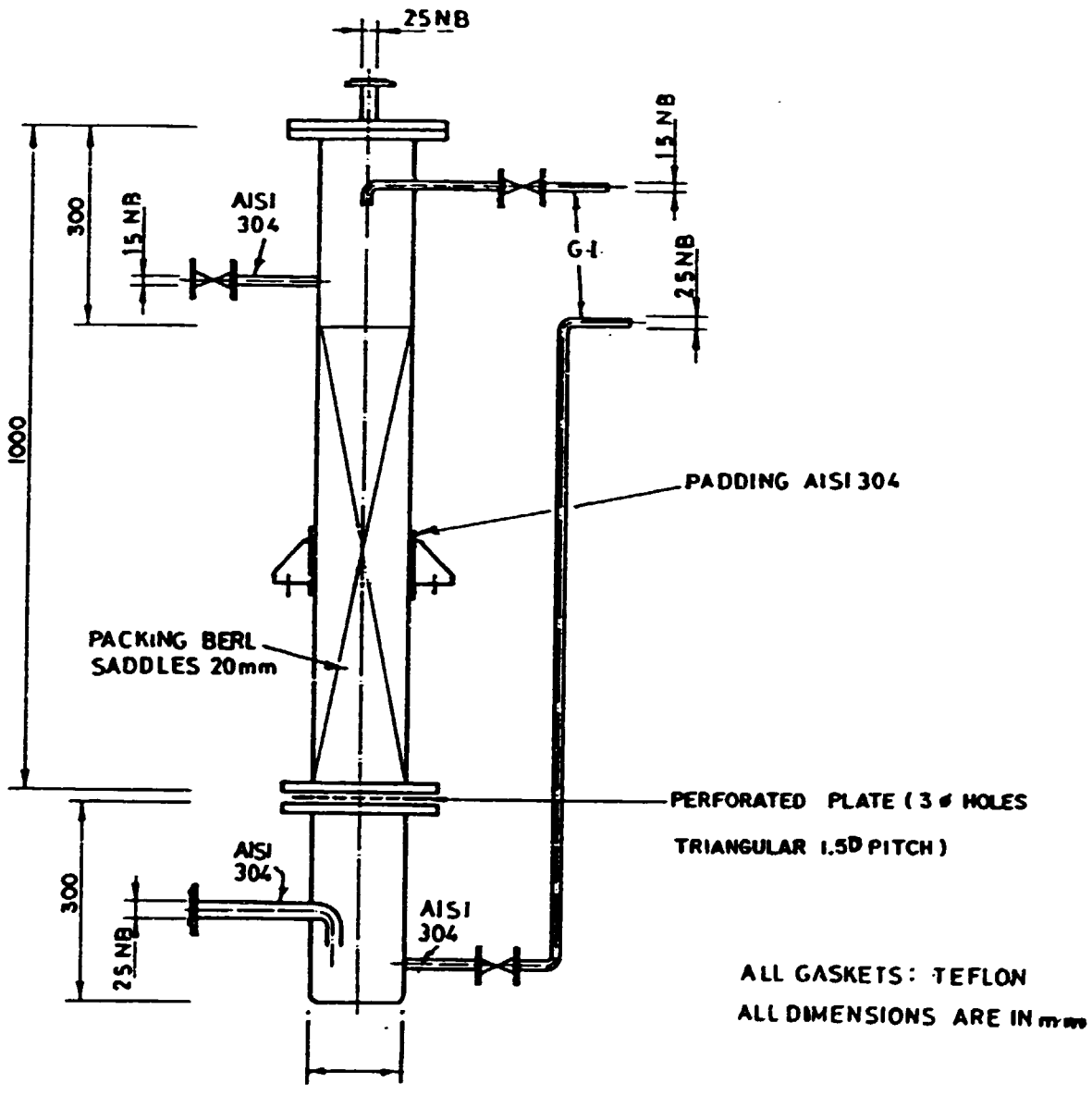
SCALE 1:10

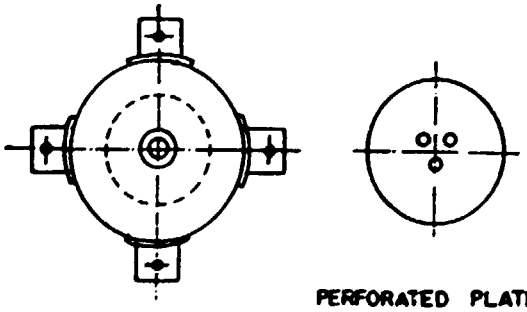
RESEARCH & DEVELOPMENT. INST.
(G.P.O.) BANGKOK
SF/THA/95/001
(PRODN. OF IODIZED OIL)
PROCESS TANKS
DESIGN : NARASIMHA B. MARLA, UNIDO
DIRECTOR: DR. SISANA KRAISINTU
JAN/FEB 1996
DRG. NO. 9 OF 15 (ITEM-5,10,15)



SCALE 1:10

RESEARCH & DEVELOPMENT - INST.
(G.P.O.) BANGKOK
SF / THA / 95 / 001
(PROON. OF IODIZED OIL)
PROCESS TANK
DESIGN : NARASIMHA B. MARLA , UNIDO
DIRECTOR : DR. KRISANA KRAISINTU
JAN / Feb 1996
DRG. NO. 10 OF 15 (ITEM-8)





PERFORATED PLATE

SCALE: 1:10

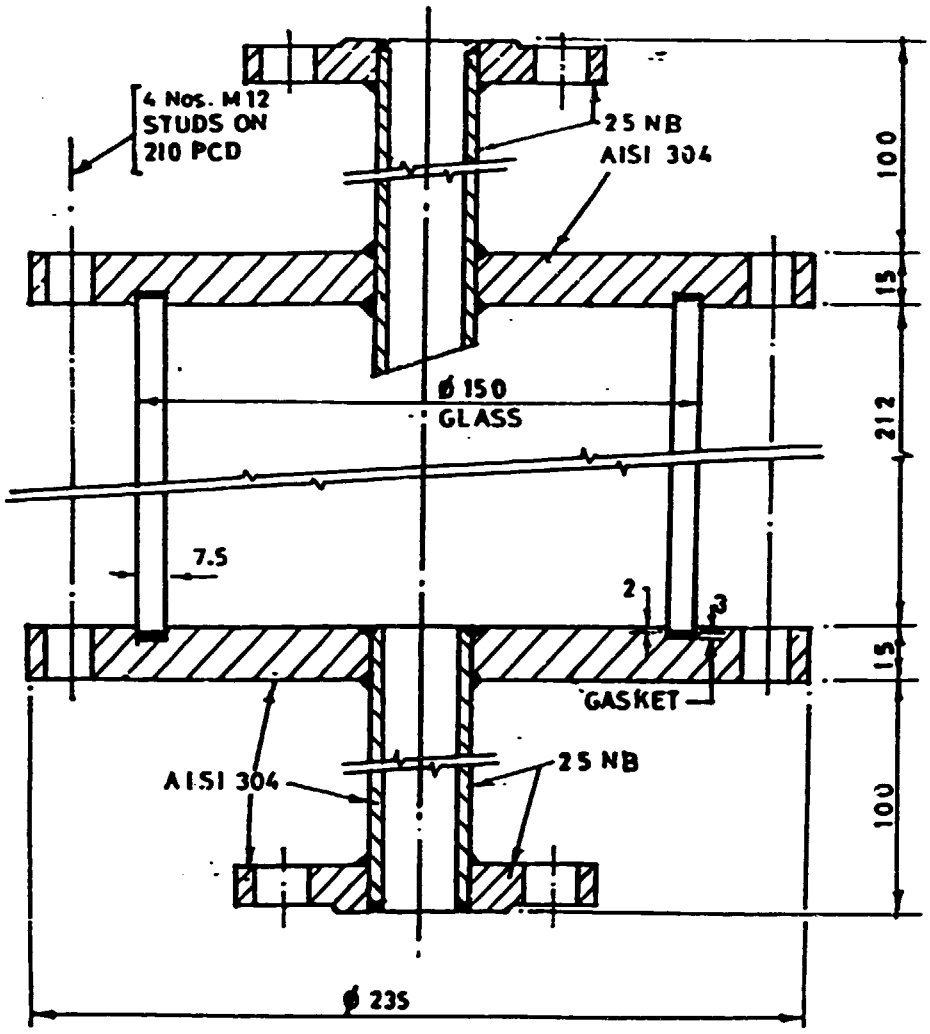
RESEARCH & DEVELOPMENT INST.
(G.P.O.) BANGKOK

SF/ THN/ 95/ 001
(PRODN. OF IODIZED OIL)

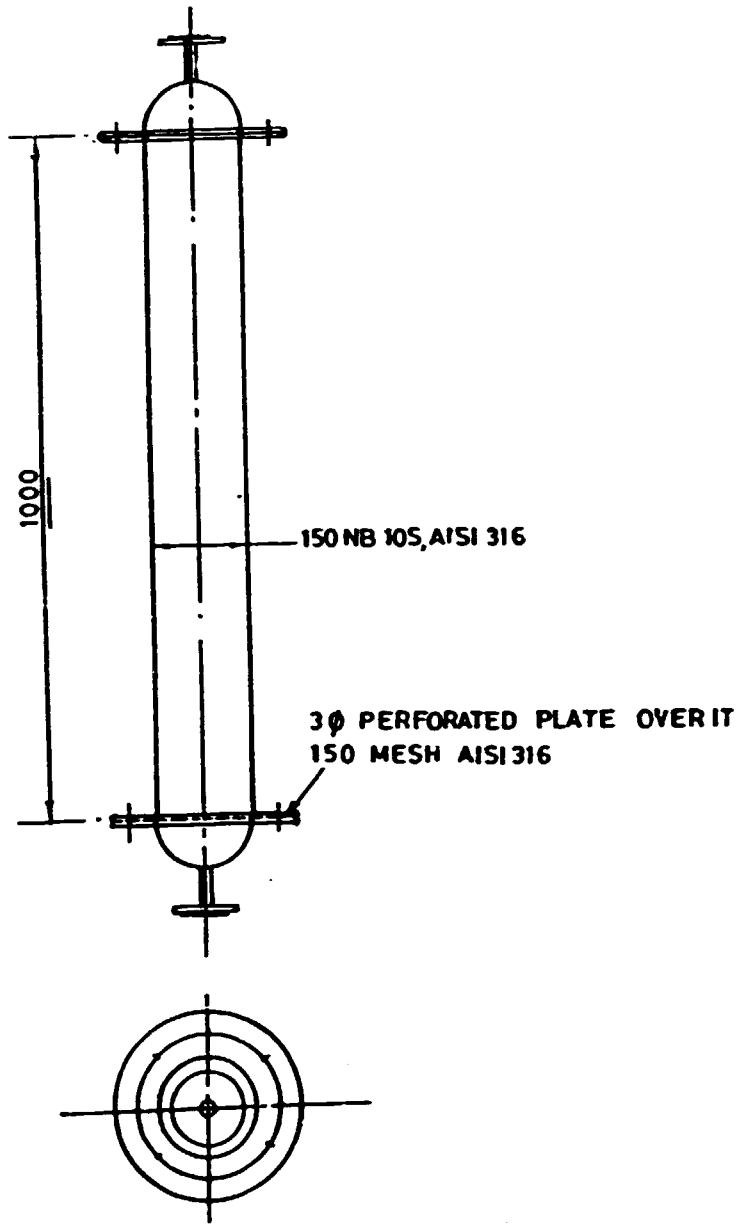
BREATHER

DISIGN : NARASIMHA B. MARLA, UNIDO
DIRECTOR : DR. KRISANA KRAISINTU
JAN / FEB 1996

DRG. NO. 11 OF 15 (ITEM-22)

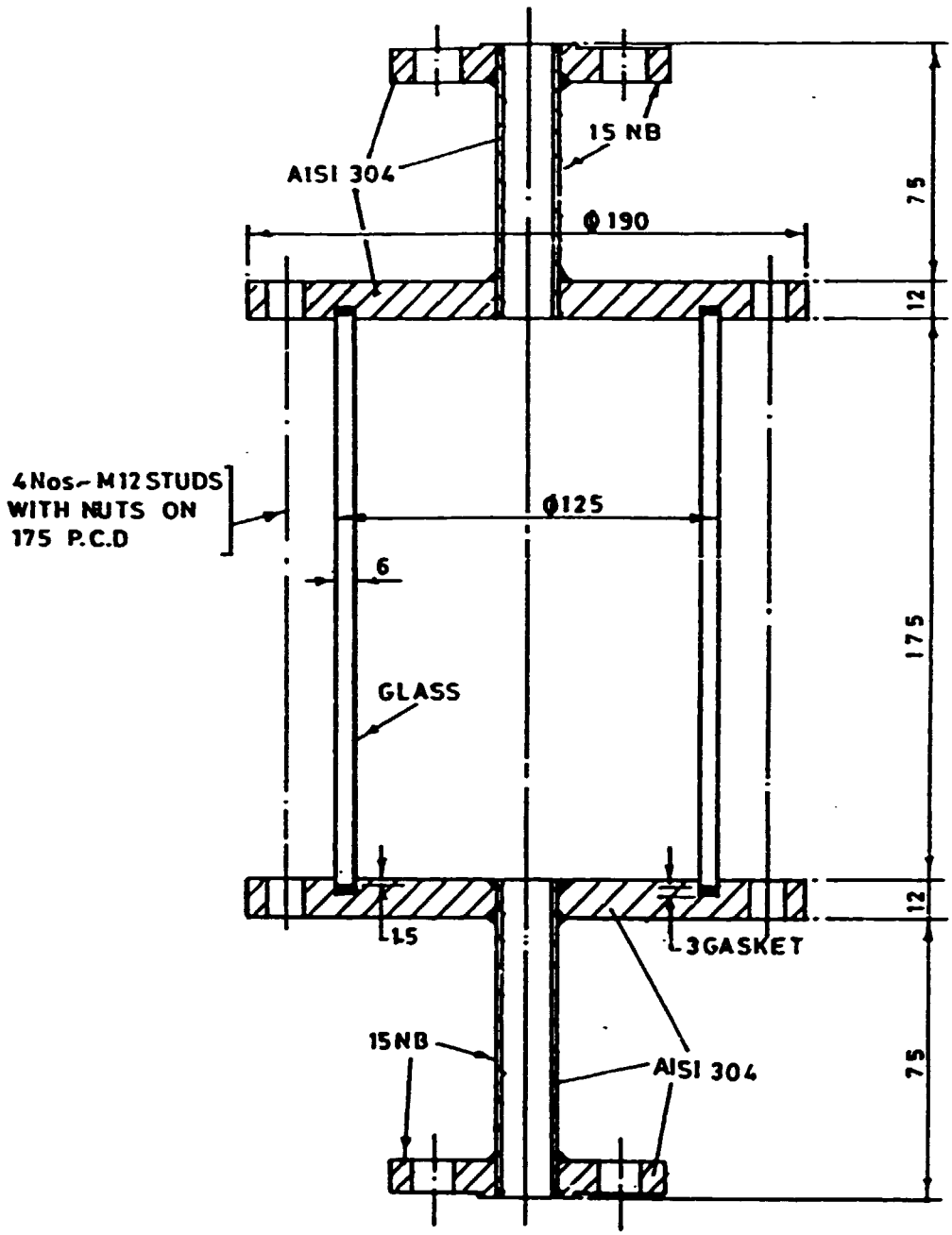


RESEARCH & DEVELOPMENT INST.
(G.P.O.) BANGKOK
SF/THA/95/001
PRODN OF IODIZED OIL
VISEUR
DESIGN: NARASIMHA B.MARLA, UNIDO
DIRECTOR: DR.KRISANA KRAISINTU
JAN/FEB 1996
DRG. NO. 12 OF 15 (ITEM 2,13ab,29)



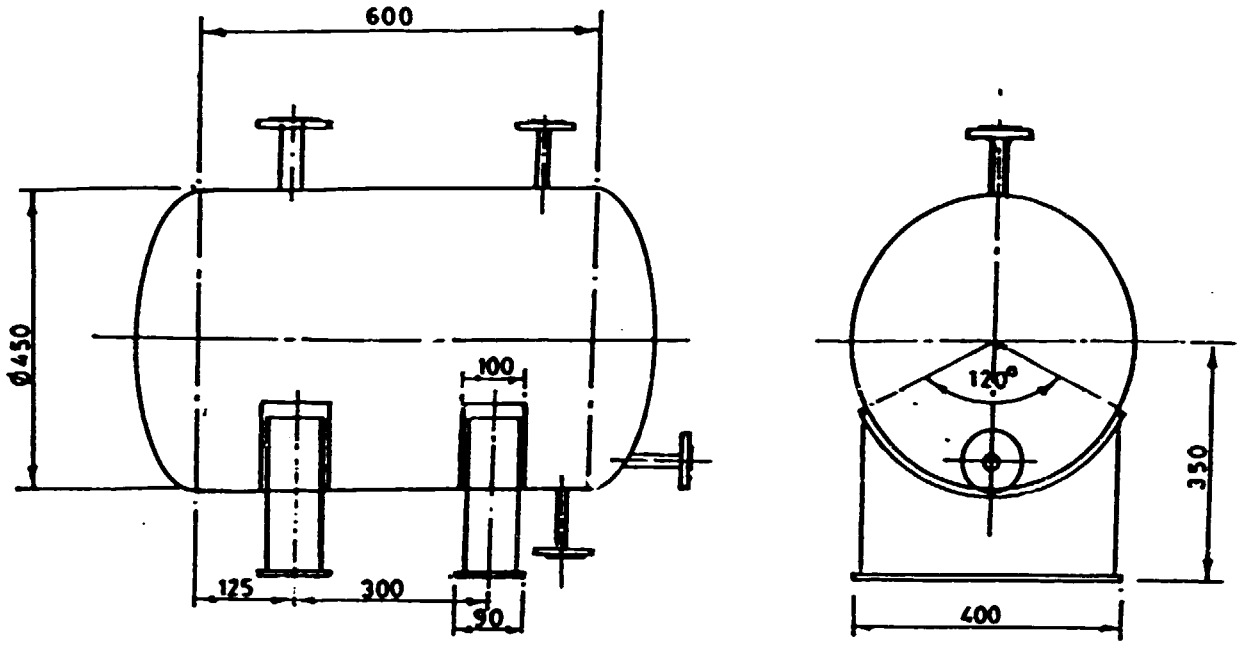
RESEARCH & DEVELOPMENT INST.
(G.P.O.) BANGKOK
SF/THA/95/001
(PRODN. OF IODIZED OIL)

SILICA GEL COLUMN
DESIGN: NARASIMHA B. MARLA, UNIDO.
DIRECTOR: DR. KRISANA KRAISINTU
JAN/FEB 1996
DRG. No. 13 OF 15 (ITEM 24)



NOTE:-
1. ALL DIMENSIONS ARE IN mm
2. FLANGES ANSI 16.5 & AISI.316
3. GASKET: TEFLON

RESEARCH & DEVELOPMENT INST.
(G.P.O) BANGKOK.
SF/THY/95/001
(PRODN OF IODIZED OIL)
VISEUR
DESIGN: NARASIMHA B MARLA
UNIDO.
DIRECTOR: DR.KRISANA KRAISINTU
JAN/FEB 1996
DRG. NO: 14 OF 15 (ITEM. 23,25)



MATL. OF CONSTN: AISI 316
(ALL CONTACT PARTS)
PADDING: AISI 304
SADDLE: C.S.

RESEARCH & DEVELOPMENT INST.
(G.P.O) BANGKOK
SF/THA/95/001
(PRODN OF IODIZED OIL)
PROCESS TANK
DESIGN: NARASIMHA B.MARLA, UNIDO
DIRECTOR: DR. KRISANA KRAISINTU
JAN/FEB 1996
DRG No. 15 OF 15 (ITEM 21, 26, 28)

SPECIFICATIONS OF STANDARD EQUIPMENT

1. Water-ring vacuum pumps required number	:	Two
Service	:	To create vacuum in iodization reactor
Fluid to be handled	:	n-hexane vapour, iodine vapour and a small amcunt of hydrogen iodide vapour
Capacity	:	About 1000 lpm (Suction capacity 60 M^3/hr)
Operating temperature	:	Ambient (30/35°c)
Total vacuum	:	50 mm Hg. absolute
Material of construction Casing & impellor)	:	Cast iron (close grained)
Gaskat	:	C A F
Nozzles Suction and discharge	:	Flanged RF (Raised Face)
Drive (Directly coupled)	:	Electric Motor (TEFC), Flame Proof with Flame-Proof starter.
Power available at site	:	380 V, 50 H_z, 3 Phase 200 V, 50 H_z, 1 Phase
H.P.	:	Vendor to specify

Scope of supply shall include vacuum pump, flame proof motor directly coupled, flame proof starter, base frame, non return valve, vacuum gauge (digital indicating type), valves, Vendor shall indicate the flow rate of water required for the liquid ring of the vacuum pump.

2. SUPER CENTRIFUGE		
Required number	:	One
Service	:	To separate iodized vegetable oil and water emulsion (mild form) additionally to be fitted with a clarification bowl to clarify iodized oil containing less than 1% solids.

Fluid to be handled	:	Iodized vegetable oil, water emulsion containing, phosphoric acid, hydroiodic acid (dilute solutions) n-hexane, iodine in solution.
Capacity	:	About 200 to 500 l ph (or smallest available unit needed for a pilot plant)
Operating temperature	:	Ambient (30/35°c)
Material of construction	:	All contact parts AISI 316
Gaskets	:	Teflon
Nozzles	:	Flanged RF (Raised Face)
Drive	:	Electric Motor (TEFC/Flame proof) V-belt drive, variable speed (final r.p.m. between 10,000 to 15,000) flame proof starter
Power available at site	:	380 V, 50 H_z, 3 Phase/200 V, 50 H_z, 1 Phase
H.P.	:	Vendor to specify

Scope of supply should include super centrifuge with flame-proof motor, flame proof starter (final r.p.m. to vary from 10,000 min. 15,000 max.), all contact parts AISI 316, inlet and out valves (ball valves).

3. CENTRIFUGAL PUMPS

Required number	:	Four
Material of	:	AISI 304 -- Three AISI 316 -- One
Type	:	Mono Block closed impellor
Service	:	To pump (1) n-hexane (2) Vegetable oil, and (3) iodized vegetable oil containing n-hexane, phosphoric acid, dissolved iodine.
Capacity	:	About 500 lphr.
Head	:	Positive suction delvery head 10 M
Opeating temperature	:	Ambient (30/35°c)

Gaskets	:	Teflon
Nozzles	:	Flanged RF (Raised Face)
Drive	:	Electric motor (TEFC) flame-proof, flame-proof starter, directly coupled to pump.
H.P.	:	Vendor to specify
Power available at site	:	380 V, 50 H_z, 3 Phase / 220 V, 50 H_z, 1 Phase.

Scope of supply shall include centifugal close impellor type mono block pumps (directly coupled to flame-proof motors), flame proof starters, base frame, non-return valves flanged ball valves, tee connection with flanged ball valves in the delivery lines for by pass into the feed tanks. Shall also include rotameters in the delivery lines.

FAX Transmission

From	Dr. Krisana Kraisintu	Research & Development Institute.
		Organization
Fax No	(662) 2462134	
To	Mr. M B. Narasimha	
Fax No	00-91-40-7740582	
Date	19 February, 1996	
No.of page	(including this one) 2	

Message :

Dear Mr. Narasimha,

I hope you had a pleasant journey home and have fully recovered from tiring work in Thailand.

I wish to express my sincere thank and appreciation to you for your assistance, understanding and patience with us and most of all your sense of humour during our hard work here at the institute.

Regarding the purification experiment with silica gel, Vatit and Onsiri have done the followings :

1. Oil used in the experiment was from the combination of previous experiments (dark brown colour in all of them).

2. With column chromatography, the following data was obtained

Silica gel - No. art 7734

- Bulk density 0.47 g/ml

Weight of silica gel 50 g

Column diameter 30 cm

Column height	23	cm
Volumn of Oil	25	ml
Volume of hexane used	175	ml
Ratio of silica gel (g) : oil (ml)	[illegible]	
hexane (ml) : oil (ml)	7	1
Esterified oil used	25	ml
Purified esterified oil obtained	10	ml

From the results above, I can conclude that the yield of the light yellow oil is still low and a lot of hexane was needed. However, it was more promising than our previous experiments, so we'll try again to optimise both the reaction and purification. My conclusion now is that the column is still required. I, therefore, would appreciate your consideration into this matter.

Thank you again for your kind cooperation

With best regards.

K. Kraisintu

Dr. Krisana Kraisintu

A note on safety aspects of solvent extraction plants

Normal hexane is generally used in the extraction operations at all the stages of process/product development viz., bench scale, pilot plant and commercially, since it has properties most suitable for extraction purposes. However, the high inflammability of n-hexane makes it necessary that the design and operation of solvent extraction plants should aim at elimination of fire hazard.

Inflammability indicates the rate at which a material undergoes combustion. Combustion is a chemical reaction by which the oxygen combines with the elements of the combustible materials forming mostly carbon-dioxide. The reaction is highly exothermic and in the case of highly inflammable materials the heat of combustion is very high so much so, that once the reaction is set in by application of external heat, the heat of combustion itself will provide enough heat for further ignition and the heat of reaction is released as fire. The heat of combustion of n-hexane is 21,000 Btu/lb. and the reaction start at an ignition temperature of 260 °C.

Hence for a solvent to catch fire, two conditions are to be simultaneously fulfilled. These are:

- there should be a correct amount of air
- there should be enough heat to bring the temperature to 260 °C to start the combustion reaction.

Absence of any one of these conditions renders the solvent extraction safe. The design of extraction plants should aim at the elimination of both of these conditions required for starting the combustion of the solvent to ensure perfect safety.

The mixture of air and hexane may not always be flammable unless proportionate amount of oxygen and hexane vapour is present in the mixture. The flammable limits for n-hexane is 1.2% by volume minimum and 6.9% by volume maximum in air, in other words any mixture containing less then 1.2% and more then 6.9% hexane vapour is considered safe.

The sources of ignition in an extraction plant can be of two types:

- mechanical
- electrical.

In the extraction plant area metal to metal impact should be avoided.

All the electrical equipment such as starters, cables, light fittings, motors, etc., required in the extraction plant are necessarily to be flame proof type equipment, any spark occurring is confined inside a perfectly air tight casing, so that it is impossible for the spark to come into contact with the outside atmosphere.

An electrical spark can be set off, due to static electricity generated in the equipment, due to friction between various parts of the extraction plant. To avoid such a contingency it is necessary to ensure the electrical continuity between any two points in an extraction plant and earth the entire plant, so that statically generated electricity is transferred to the earth as and when produced. Lightning arresters grounded to the earth are also installed at suitable points so that in case of lightning in the solvent extraction area it is immediately grounded.

All heating operations in the extraction plant are carried out by steam heating only to ensure that the temperature in the plant at any point does not reach the ignition temperature of 260°C. Temperature of steam at a pressure of 150 p.s.i.g is only 185 °C.

Under abnormal conditions, solvent can find way out of the equipment and likely to create hazardous situation. To avoid such abnormal conditions the following general safety desigr features are to be incorporated.

Solvent vapours from distillation as well as desolventisation sections are lead to a system of condensers and then the uncondensed gases are lead through a "breather" or an oil absorber or through a chilled condenser, a cold trap and then vented into the atmosphere. Under normal operation conditions the gases vented out contains traces of solvent vapours much below the flammable limits. But in the case of failure or inadeqacy of cooling water in the condensing system the gas will consist mainly of solvent vapours, hexane vapour being three times heavier than air would settle in the extraction area creating a very hazardous atmosphere. It therefore becomes necessary that the person in charge of the plant must be alerted in advance of such a situation occurring so that he can take immediate preventive action. For this purpose a vent thermostat is installed in the vent line. The function of this thermostat is to actuate an electrical contact as soon as the temperature of the vent reaches 45 $^{\circ}$ C, an alarm is set off thus warning of the impending danger. For further precaution, this electrical contact is installed in sequence with an electrically operated steam value installed in the main steamline. As soon as this contact is activated the main steam valve is automatically closed, thus cutting off the supply of steam, so that no further evaporation of solvent occurs in the extraction plant.

In case of power failure, the main steam valve is automatically shut off thus cutting off the steam supply.

Live steam is used for final stripping of the oil and this is condensed along with the solvent vapours and is seperated from the solvent in a separator.

The vent of exhaust gases of the extraction plant must be located at a minimum height of 7 meters, so that traces of solvent vapour present in the gas gets diluted with atmospheric air below the flammable limits and does not settle down near the floor.

Solvent extraction plant area should be well ventilated so that concentration of vapours due to any leaks in the equipment does not take place.

LIST OF PERSONS CONTACTED.

1.	Dr. Noppadol Somboon	:	General Manager, G.P.O. Bangkok
2.	Dr. Krisana Kraisintu	:	Director, R&D Institute, GPO (NPD)
3.	Mr. Ari Huhtala	:	U.C.D., Bangkok
4.	Ms. Mario Hinte	:	Programme Officer, Office of UCD Bangkok.
5.	Mr. Wathit Sastrawathit	:	R&D Institute, G.P.O.
6.	Ms. Onsiri Srikun	:	R&D Institute, G.P.O.
7.	Dr. Mayuree	:	Adviser to the Project
8.	Ms. Thaworn Jaipetch	:	Adviser to the Project
9.	Dr. Prakongsiri Booncong	:	R&D Institute, G.P.O.
10.	Ms. Achara	:	R&D Institute, G.P.O.
11.	Mr. Jullajak Uthairungsri	:	Material Handling Engg., G.P.O.