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**VERSATILE PILOT SCALE DISTILLATION/EXTRACTION UNIT
TO PROCESS MEDICINAL AND AROMATIC PLANTS**

Prepared by

UNIDO Secretariat

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Foreword

One of the major constraints in the growth of a plant-derived pharmaceutical and essential oils industry, and this is particularly the case in developing countries, is the lack of suitable pilot-scale processing facilities and expertise in chemical processing technology.

Product processing methods achieved at the laboratory level have to be scaled up to ensure process suitability as well as techno-economic feasibility. Furthermore, where clinical trials have to be conducted prior to introduction to the health care system or necessary tests for use in the cosmetic industry, the material for testing has to be processed in reliable and reproducing fashion to generate it in adequate quantity as well.

Processing of plant material varies significantly from one case to another and therefore a pilot plant built for one situation may be quite unsuitable for another case. While discussing the issue of industrial utilization of medicinal plants - process technology and product standardization, the Third Consultation on the Pharmaceutical Industry held in Madrid, Spain, from 5 to 9 October 1987 recommended that UNIDO should further develop the concept and design of a polyvalent versatile pilot plant for process technology for use in developing countries.

In implementation of the recommendation of the Consultation meeting work was undertaken by UNIDO in preparation of preliminary design options for the pilot plant which could enable carrying out a variety of operations. As a result of extensive discussions with UNIDO experts, a versatile polyvalent pilot scale distillation and extraction unit to process medicinal and aromatic plants has been developed with practical modular engineering design complete with itemized scale construction drawings, representations of exploded sections and bills of quantities of the materials of construction involved.

UNIDO believes that this document will be a valuable reference guide for establishment, in particular in developing countries, and will find wide usefulness in local fabrication of polyvalent pilot plants for processing of medicinal and aromatic plants and help in economic production ultimately resulting in technology development, improved drug delivery and health programmes.

The substantive contribution in design ideas was provided by the following UNIDO experts: J.M. Chiocci, C.L. Tikoo, M.B. Narasimha, C.K. Atal, A. Escaut. The working drawings and detailed specifications have been prepared by the UNIDO expert H. Hogekamp. UNIDO wishes to acknowledge the contribution and co-operation afforded by experts and Tournaire, Grasse (France) which enabled it to accomplish the task of compilation of the designs under reference.

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1

VERSATILE PILOT SCALE DISTILLATION/EXTRACTION UNIT
TO PROCESS
MEDICINAL AND AROMATIC PLANTS

1. Introduction

The following describes a pilot scale polyfunctional processing unit of uncomplicated construction to carry out various unit operations in extracting flavour, aroma and medicinal constituents from plant material. The pilot plant has been designed as a frame mounted package unit for easy installation, operation and maintenance.

The required upstream and downstream processing steps as washing, size reduction, drying and packaging much depend on the nature of the raw material processed. For this reason, no general recommendation can be given regarding the components required. These must be considered independently for each case of application and are recommended to be procured externally like the items mentioned below. Reference is made on pages 21 and 22 regarding appropriate grinding and drying equipment.

The description of the distillation/extraction unit is accompanied by a full set of construction drawings for the main components, excluding pumps and filter, and by a bill of quantities of the materials of construction involved. The circulating pump, the vacuum pump, the miscella filters and preferably the condensers should be procured from specialized manufacturers stating the details and parameters of application.

The operations that may be carried out with the plant either in successive steps or simultaneously are

- steam distillation and separation of essential oils,
- fractional distillation of essential oils,
- percolation in a solvent at ambient temperature,
- hot-solvent extraction by the Soxhlet method,
- extraction by repeated leaching with hot or cold solvent,
- filtration of the miscella,
- vacuum concentration of the miscella,
- rectified solvent distillation and recovery.

The unit features the following main components:
(refer to arrangement drawings 9004-01/03)

1. Extraction Vessel
2. Condenser 1
3. Phase Separator (Decanter)
4. Florentine Flask
5. Solvent Circulating Pump
6. Miscella Filter
7. Evaporator
8. Packed Column
9. Condenser 2
10. Cooler
11. Receiving Vessel
12. Solvent Tank
13. Vacuum Pump
14. Supporting Frame

The loading capacity of the extraction vessel is 50 to 100 kilograms per charge, depending on the apparent density of the raw material to be processed. The plant may be scaled up or down by a factor between 1/2 and 2 by generally enlarging or reducing the vessels pro rata. However, before taking this step all other possibilities of increasing the capacity, e. g. adding further modules or vessels for intermediate storage of miscella, should have been checked thoroughly.

The plant has been designed for organic solvent extraction and for these solvents the capacities of extractor, condensers and evaporator have been adjusted. The plant may be used for aqueous extraction but it should be noted that in this case the capacity of the evaporator will be sharply reduced because of the much higher heat of vaporization/condensation of water compared with the commonly used organic solvents (ratio 1:4 to 1:5). In a commercial operation entirely different types of evaporators (of the tube or plate type with forced circulation) would be used and for this reason upscaling forbids itself in this case.

2. Details of Construction

By the nature of the raw materials to be processed as well as the ultimate use of the products it is understood that the entire equipment, including valves and fittings is to be constructed of stainless steel, material no. 1.4571, except for the supporting frame, the vacuum pump and the pipework for steam, condensate, cooling water and vacuum.

2.1 Extraction Vessel

(refer to drawings 9004-10, 9004-11, 9004-12, 9004-01)

The extraction Vessel is of upright cylindrical shape with a dished bottom (Klopper-type) and a collar with a finely machined face at the top. Fixed directly below the collar there are 8 double gussets hinging the clamps that will tighten the lid to the main body (detail X). Approximately the lower half of the body is encased in a stainless steel shell serving as a steam jacket. The jacket is to be insulated with a layer of minimum 50 mm thickness.

In the bottom portion of the main body a ring is provided (detail Y) to support the raw material basket.

The lid consists of a convex disk welded onto a flange finely machined with a trapezoidal recess to accommodate a PTFE-coated sealing ring.

Attachments to the extraction vessel starting at the bottom are:

- 1 Ball valve DN 40, flanged to the bottom opening N5;
- 1 Drainage pipe DN 40, flanged to the ball valve, with T-piece DN 25 for lateral connection;
- 1 Condensate outlet pipe DN 15 with 90° elbow and flange N4;
- 1 Steam injection pipe DN 15 (flange N3) leading directly into the bottom portion of the main body; the pipe is provided with approximately 20 steam holes of 2 mm diameter, evenly distributed over the length of the pipe and facing the bottom of the vessel. The steam holes must face the bottom of the vessel in order to ensure complete draining of the solvent;
- 1 Steam pipe DN 15 (flange N2) to supply the jacket;
- 2 Supporting brackets in stainless steel, welded to the outer shell of the steam jacket;

- 2 Tubes with sight glass assembly for lighting and inspection of the contents during processing; the sight glass assembly should preferably be procured from a specialist manufacturer;
- 1 Vapour outlet tube DN 50 (flange N1); connected to the tube is an elbow piece with pressure gauge and safety valve.
- 8 Clamps to take tilting bolts (detail X), fixed directly below the collar;
- 2 Gussets hinging the lid to the main body;
- 1 Hoop to open the lid;
- 1 Socket (S1) with elbow piece welded to the center of the lid forming the inlet for the circulating solvent.

The elbow piece is connected to a flexible pressure hose with an inner PTFE lining and bayonet joints for easy removal when opening the lid

To spread the solvent over the surface of the material, a cross-tube with holes of 2mm diameter spaced at 20 mm over the length of the tubes is screwed to the socket from inside the lid. Alternatively a 'shower head' type of device may be used.

Accessories to the extraction vessel:

- 1 Set of perforated disks to accommodate the plant material to be extracted.

This equipment component is designed to fit easily inside the extraction vessel so that the plant material to be processed is separated into successive layers of about 150 mm depth, thus avoiding compaction. The set is constructed as follows:

- Six round disks of 2 mm thickness, perforated over the entire surface with holes of 3 mm diameter.
- Each disk is fitted with four spacer rods, 150 mm high, joined at the top by a flat ring of 25 x 5 mm cross section.
- For handling purposes, the disks all have a 22 mm hole in the center to accommodate a central shaft of 20 mm diameter, fitted with a cross piece at the bottom and with a ring at the top for the use of a hook.

- 1 Perforated basket for processing of very light material, e.g. lemon grass.

The basket is made of a perforated sheet metal cylinder of 1.5 mm thickness, perforated with 3 mm holes, closed at the base by a round perforated disk welded to the cylindrical part. The bottom disk is made of 2 mm sheet. At the top the basket is reinforced by a flat iron hoop of 30 x 6 mm cross section fitted with two lifting rings for attachment to a beam able to carry a load of 100 kg.

OPERATING AND TESTING CONDITIONS

- a) Interior of extraction vessel:

Operating pressure: 2.5 bar

Testing pressure: 5 bar

Operating temperature: 138 °C

- b) In the steam jacket:

Operating pressure: 3 bar

Testing pressure: 6 bar

Operating temperature: 140 °C

2.2 Condenser 1

(refer to drawings 9004-20 and 9004-21)

The condenser is of the multi-tube single-pass type. The cooling water circulates outside the tubes deflected by a number of baffle plates whereas condensation occurs inside the tubes. The condenser is fitted with a slight tilt at an angle of 3° from the horizontal. The cooling water enters the shell from the bottom and leaves the shell at the top. The product vapours enter the head compartment at the elevated end from the bottom and the condensate runs by gravity flow through the tubes to the head compartment at the lower end.

Note: The construction of a condenser, especially the welding of the relatively thin tubes into the head plates requires welding experience and great skill if this work is to be performed manually. For this reason even in industrialized countries condensers in most cases are bought from specialized producers.

It is, therefore, recommended that the condenser is procured from a reputable manufacturer!

Attachments to the condenser are:

- 1 Vapour tube DN 50 fitted with flange N8;
- 1 Condensate discharge tube DN 20 fitted with flange N13
- 1 Cooling water inlet tube DN 25 fitted with flange N14;
- 1 Cooling water discharge tube DN 25 fitted with flange N15;
- 2 Sockets 1/2" (S4/S5) fitted in the center of the dished ends to hold dial thermometers;
- 8 Baffle plates inside the shell and evenly spaced over the length of the tubes;
- 1 Air venting tube DN 25 with two 90° elbows welded together to form a semicircle.

OPERATING AND TESTING CONDITIONS

a) In the shell outside the tubes:

Operating pressure: 3 bar

Testing pressure: 4.5 bar

Operating temperature: 100 °C

b) Inside the tubes:

Operating pressure: 0.2 bar

Testing pressure: 1 bar

Operating temperature: 100 °C

2.3 Phase Separator (Decanter)

(refer to drawing 9004-35)

The decanter is designed to separate water from solvent when, after extraction, the solvent is removed from the spent raw material, in part or in full, by purging with steam.

If, as is generally the case, the solvent is lighter than water and non-miscible, the decanter works automatically. However, if the solvent is water-miscible, the decanter is not of much use but neither poses any problem. In case a heavier-than-water non-miscible solvent is used, close monitoring is required and small quantities must be drawn off periodically.

The decanter essentially consists of two parts, the head piece and the glass receptacle.

Welded into the head piece are the various connections for the mixture inlet and the outlets:

- 1 Mixture inlet tube DN 20 reaching down into the glass receptacle closed at the tip by a slotted disk serving as an anti-splash nozzle;
- 1 Heavy liquid outlet tube DN 20 with rise pipe reaching down into the glass receptacle;
- 1 Light liquid outlet tube DN 20, positioned 30 mm higher than the heavy liquid overflow.

The glass receptacle is to be provided with a bottom outlet of 20 mm diameter and flanges of the sizes DN 200 and DN 20. The flanges, PTFE-coated Klingerite joints as well as a ball valve for the bottom outlet should be procured together with the glass receptacle.

2.4 Florentine Flask

(refer to drawing 9004-16)

The florentine flask is used in the steam distillation process to separate the essential oils from the steam condensate. The bottom part of the flask may either be constructed from copper sheet or from stainless steel sheet with a flange at the top to which the cylindrical glass sleeve is fitted in the way shown on sketch 9004-16. It is, however, also customary to use flasks entirely made of glass. Preferably commercially available standard flasks should be procured.

2.5 Solvent Circulating Pump

The solvent circulating pump must be able to overcome a certain pressure build-up in the miscella filters which depends on the amount of fine particles present in the raw material. The pump should be procured from a reputed pump manufacturer and should meet the following specification:

Parts in contact with medium: stainless steel 1.4571

Seal: PTFE-coated, contact surface: tungsten carbide

Maximum throughput: 2000 l/h

Maximum working pressure: 2 bar

Maximum delivery: 25 m water column

Electric motor: 3-phase, flameproof

2.6 Miscella Filter

The miscella filter preferably consists of two units of a commercial basket filter installed in parallel in the delivery line of the circulation pump. Each filter should have an area of approximately 0.1 m². Both filters should be connected by 3-way ball valves in the supply and in the delivery line for easy changeover if one filter gets blocked. The miscella filters should equally be procured from a reputed manufacturer since excellent performance is indispensable in order to obtain a satisfactory product free from insoluble plant material.

2.7 Evaporator

(refer to drawing 9004-30)

The evaporator essentially consists of a hemispherical heating surface with a cylindrical dome covered by a dished boiler end. The heating surface is hemispherical for easy and thorough cleaning between batches and different raw materials. The bottom outlet flange has to be finely machined and precisely welded into the bottom according to the details given in the drawing. All the inner welding seams are to be ground and the interior wall of the evaporator is to be polished to a finish of grain 300 in order to minimize sticking and to guarantee complete draining of the very valuable product from the reboiler.

The hemispherical bottom has an outer casing serving as a steam jacket. The jacket is to be insulated with a layer of 50 mm thickness minimum.

Attachments to the evaporator starting at the bottom are:

1 Ball valve DN 20 with PTFE seal connected directly to the bottom flange N20;

- 1 Drainage pipe DN 20, flanged to the ball valve;
- 1 Condensate outlet pipe DN 15 with 90° elbow and flange N19;
- 1 Tube LN 15 (flange N18) to supply steam to the jacket;
- 2 Supporting brackets in stainless steel, welded onto the cylindrical wall of the evaporator;
- 2 Sight glass assemblies DN 80 (flanges N17,N22), designed to withstand the vacuum and test pressure and for this reason to be procured from a specialized manufacturer;
- 1 Miscella inlet tube DN 20 fitted with flange N26;
- 1 Solvent recirculation tube DN 20 fitted with flange N26A;
- 2 Sockets 1/2" (S7,S8) for vacuum gauge and thermometer respectively.
The thermometer must reach into the outlet opening in order to guarantee temperature indication even of small quantities of product;
- 1 Socket 1/2" (S6) for the vacuum relief valve;
- 1 Vacuum relief ball valve 1/2", connected directly to the socket;
- 1 Inspection port DN 200 (flange N21), machined for an O-ring similar to the groove in the extractor lid (vide drawing 9004-11);
- 1 Top opening DN 200 with flange N16 to connect with the packed column;
- 1 Tube DN 25 with flange N23 to fit the purging lance.

As an accessory to the evaporator a purging lance is provided by which steam or air may be injected under vacuum into the concentrated product in order to drive off remaining solvent. The tip of the lance must reach down to the bottom opening.

The lance is made of a 14 mm tube with a 1 mm nozzle at the tip.

OPERATING AND TESTING CONDITIONS

- a) Interior of the evaporator:

Minimum operating pressure: vacuum

Maximum operating pressure: 1 bar

Testing pressure: 2 bar

Temperature: 130 °C

b) In the steam jacket:

Operating pressure: 3 bar

Testing pressure: 6 bar

Temperature: 142 °C

2.8 Packed Column

(refer to drawing 9004-31)

Through the packed column the vapours generated in the evaporator are carried to condenser 2. In the case of rectified solvent distillation or fractionation of essential oils it is used as a reflux column.

The column mainly consists of the following elements:

- A cylindrical body fitted at both ends with machined flanges N27, N28; alternatively loose flanges and collars may be used.
At the bottom end the column is provided with a fixed grid to hold the packing. The grid in form of a 120° cone is made of 2 mm sheet pierced with 50 holes of 20 mm diameter evenly distributed over the surface of the sheet with one hole in the center.
- A column head composed of a cylindrical glass sleeve DN 200, length 300 mm, together with machined flanges N32, N33, tightening rods and PTFE-coated seals.
- A cap DN 200, with convex base and outlet DN 80 fitted with an elbow piece 90° and flange N31. The cap is further provided with a socket 1/2" (S9) to hold a thermometer and with a reflux tube DN 20 consisting of an elbow piece with a straight pipe end reaching down into the glass sleeve and located in the center of the cylinder.
- A reflux divider of commercial design operated by a timer actuated solenoid valve, pipe connections in DN 20;

OPERATING AND TESTING CONDITIONS

Identical to those of the evaporator.

2.9 Condenser 2

(refer to drawings 9004-25 and 9004-26)

The condenser is of the multi-tube single-pass type. The cooling water passes through the tubes whereas vapour condensation occurs in the shell. The condenser is fitted with a slight tilt at an angle of 3° from the horizontal. The cooling water enters the condenser from the bottom of the lower head compartment and flows through the tubes to the head compartment at the upper end. The condensate leaves the shell at the lower end whereas at the top of the upper end the vacuum line is connected.

Note: The construction of a condenser, especially the welding of the relatively thin tubes into the head plates requires welding experience and great skill if this work is to be performed manually. For this reason even in industrialized countries condensers in most cases are bought from specialized producers.

For this reason it is recommended that also this condenser is procured from a reputable manufacturer!

Attachments to the condenser are:

- 1 Vapour tube DN 80 fitted with flange N34;
- 1 Condensate discharge tube DN 20 fitted with flange N39;
- 1 Cooling water inlet tube DN 25 fitted with flange N40;
- 1 Cooling water discharge tube DN 25 fitted with flange N41;
- 2 Sockets $1/2''$ (S10/S11) fitted in the center of the dished ends to hold dial thermometers;
- 2 Baffle and tube supporting plates inside the shell;
- 1 Vacuum tube DN 25 fitted with flange N42.

OPERATING AND TESTING CONDITIONS

a) In the shell outside the tubes:

Minimum pressure: vacuum

Maximum pressure: 1 bar

Testing pressure: 2 bar

Temperature: 100 °C

b) Inside the tubes:

Maximum operating pressure: 3 bar

Testing pressure: 4.5 bar

Operating temperature: 100 °C

2.10 Cooler

(refer to drawing 9004-34, 9004-01)

The condensate leaves the condenser at a temperature near the boiling point. This requires an additional cooler in order to bring the temperature of the condensate down to near ambient before it enters the receiver or, in case of a solvent, is fed back into the solvent storage tank.

The cooler is composed of two concentric tubes DN 40 and DN 60, the product flowing through the inner tube and the cooling water between the two tubes.

The inner tube is fitted with 12 semi-circular deflectors made of sheet metal with 3 mm holes. The deflectors are fitted on a central stem in an opposed order so that the liquid flows along the inner wall from one deflector to the next.

The unit is closed at the ends by DN 20 tubes fitted with flanges N43, N44. The outer tube has at the ends two sockets S12, S 13 serving as inlet and outlet for the cooling water.

In order to ascertain proper functioning the unit must be assembled and installed strictly vertically.

OPERATING AND TESTING CONDITIONS

Minimum pressure: vacuum

Maximum pressure: 1 bar

Testing pressure: 2 bar

Temperature: 100 °C

2.11 Receiving Vessels

(refer to drawings 9004-36, 9004-01)

When evaporator, packed column and condenser 2 are operated as a fractionating unit the separate fractions may be collected in the two receivers. The receivers consist of vertical cylindrical vessels provided with an oblong inspection glass which serves as a level indicator.

Attached to the vessels are

- 1 Bottom outlet tube DN 15 fitted with flange N52;
- 1 Inlet tube DN 15 with 90° elbow and a straight section fitted to flange N50. The tube should reach approx. 40 mm down into the vessel;
- 1 Vacuum tube DN 15 fitted with flange N51;
- 1 Socket 1/2" (S14) to hold the vacuum relief valve;
- 1 Ball valve 1/2" mounted on socket S14;
- 1 Ball valve DN 15 flanged on to flange N 52;
- 1 Inspection assembly 70 x 400 mm.

OPERATING AND TESTING CONDITIONS

Minimum pressure: vacuum

Maximum pressure: 1 bar

Testing pressure: 2 bar

Temperature: 100 °C

2.12 Solvent Tank

(refer to drawing 9004-40)

The solvent tank serves as a storage vessel and as the receiver for the solvent redistilled via evaporator and condenser under vacuum or under atmospheric pressure.

The solvent tank is a horizontal cylindrical vessel with convex ends. It is equipped with the following attachments:

- 1 Drain tube DN 20 fitted with flange N57;

- 1 Ball valve DN 20 joined to flange N57;
- 1 Discharge tube DN 20 fitted with flange N54;
- 1 Man hole DN 250 with finely machined flange N55 and lid. Lid to be machined with groove for 6 mm PTFE-coated O-ring (vide drawing 9004-11);
- 2 Tubes DN 15 with flanges N58, N59 at the front for the level gauge assembly;
- 1 Solvent inlet tube DN 20 fitted with flange N53;
- 1 Vacuum tube DN 20 fitted with flange N54

OPERATING AND TESTING CONDITIONS

Minimum pressure: vacuum

Maximum pressure: 1 bar

Testing pressure: 2 bar

Temperature: 100 °C

2.13 Vacuum Pump

The vacuum pump is of the liquid-ring-type. It is to be equipped with a small water tank of 10 - 15 l capacity as a sealing water reservoir.

The pump must meet the following specifications:

at a sealing water temperature of 15 °C

Capacity at absolute pressure of 100 torr: 35 m³/h

Capacity at absolute pressure of 40 torr: 15 m³/h

The pump is to be procured together with the sealing water tank, a non-return valve DN 25 and a flame proof electric motor. The power required is approximately 1.5 kW.

2.14 Supporting Frame

(refer to drawing 9004-03)

The supporting frame is essentially constructed of mild steel IPB-beams and channel iron. The entire frame should preferably be galvanized or else have rust protective coating applied. The individual beams should be bolted together using 4 bolts M16 for each junction and patent

washers against loosening. Where channel iron is used the junctions should be reinforced by gusset plates as shown on the drawing.

In order to have access to the elevated parts of the plant as for instance the reflux divider, stairs or a ladder should be provided in accordance with the safety regulations of the country.

2.15 Internal Connections

The sizes of the process pipes between the individual components are determined by the nominal diameters of the flanges given in the drawings and the bill of quantities. All process piping should be executed in stainless steel.

For the dimensions of flanges and pipes refer to the DIN standards given in the drawings and the bill of quantities respectively. If other than DIN standards are to be used the nominal diameters should be close to the ones given in this document.

Bolts and nuts should be in stainless steel and of the size given by the standards for the flanges. As jointing material generally PTFE-coated Klingerite should be used.

The following pipe dimensions are used:

- a) Process pipes (all stainless steel as in Bill of Quantities):
 - aa) Link between extraction vessel and condenser 1: DN 50;
 - ab) Link between packed column and condenser 2: DN 80;
 - ac) Product line from branching point below cooler to receivers: DN 15
 - ad) Product line from condenser 1 to Florentine flask and decanter;
 - ae) Drain pipe underneath extractor: DN 40;
 - af) All other product lines: DN 20.

- b) Steam and condensate pipes (to meet requirements of local boiler inspectorate):
 - ba) Main steam line DN 20;
 - bb) Steam connections to consumers, condensate discharge pipes: DN 15.

c) Vacuum lines (standard galvanized pipe):

ca) From condenser 2 to vacuum pump: DN 25;

cb) From solvent tank to vacuum pump: DN 20;

cd) Connections to receivers: DN 15.

d) Cooling water lines (standard galvanized pipe): DN 25

3. Instrumentation and Safety Equipment

a) on the extraction vessel

- in the vapour line before the pressure control valve

1 pressure gauge, range 0 - 4 bar,

1 safety valve calibrated at 2.3 bar

on the jacket

- 1 pressure gauge, range 0 - 3 bar

- 1 steam trap with by-pass line

b) on condenser 1

- in both the head compartments

1 dial thermometer each, range: 0 - 150 °C

1 sight glass in the product discharge line

c) in the miscella circulating line

- between circulating pump and entrance to the filters

1 pressure gauge, range 0 - 4 bar

- between filters and entrance into extractor

1 thermometer, range 0 - 150 °C

1 sight glass

- d) on condenser 2
- in both the head compartments
 - 1 dial thermometer each, range: 0 - 100 °C
- e) on the evaporator
- 1 thermometer with immersible stem, range 0 - 100 °C
 - 1 vacuum/pressure gauge, range -1 to +1 bar
- on the jacket
- 1 pressure gauge, range 0 - 3 bar
 - 1 steam trap with by-pass line
- f) on the top of the column
- 1 dial thermometer, range: 0 - 100 °C
- in the reflux line
- 1 reflux divider actuated by a time controlled solenoid valve
- g) in the vacuum line
- 1 safety valve calibrated at 0.8 bar
- NOTE: Blow-off from the safety valve must lead into a direction where it cannot harm any person!
- h) in the cooling water supply line
- 1 commercial impurity filter
- i) in the main steam supply line
- 1 safety valve calibrated at 2.8 bar
 - 1 steam trap

4. Construction and Assembly of the Components

4.1 Manufacturing Instructions

Construction of the components should be attempted only by firms who already have experience in the manufacture of stainless steel equipment and who are conversant with the appropriate welding methods. It is recommended that the TIG welding process with internal argon gas scavenging is applied. This work can only be performed by highly skilled welders and under supervision of an experienced engineer.

It is indispensable that appropriate welding rods are used as recommended by the supplier of the sheet and pipe material respectively.

It is recommended that the construction work, if performed in a developing country, be supervised by an experienced expatriate engineer.

As already mentioned above especially the construction of the condensers should only be attempted if adequate welding know-how and skilled welders are available. If this is lacking, too much construction material will be wasted before an acceptable result is achieved.

For the construction of the vessels and the condensers it has been foreseen to use pre-fabrikated dished ends which greatly facilitates welding and guarantees perfect seams.

As a general rule inlet and outlet tubes, sockets and sight glass flanges on the vessels should be welded from both sides inside and outside. With special accuracy the flange for the bottom outlet of the evaporator has to be machined and welded and the seam to be ground and polished before the bottom is welded to the cylindrical part.

Throughout this document the pipe ends attached to the vessels are fitted with welded flanges DIN 2576. This, however, is not essential, also loose flanges DIN 2642 and pipe ends with welded on dished flanges may be used. Great care must be exercised to weld the flanges at right angles or else it may be difficult to get the plant vacuum tight.

The finished components must be subjected to the pressure tests described above for the individual items before being mounted on the steel frame.

NOTE: Notwithstanding the remarks made above regarding the testing conditions of the individual apparatus all conditions must comply with the regulations of the country where the plant is to be operated.

4.2 Erecting Instructions

Assembling commences with mounting the steel frame followed by placing the two main components, the extractor and the evaporator. The following order of erection should then be maintained:

- arrange in position but do not yet fix circulation pump, miscella filters, solvent tank and receivers; in case dimensions different from the ones given in the drawings are used make sure there is enough working space between the components;
 - connect packed column to evaporator; take care that the column is mounted accurately in a vertical position;
 - connect sight glass DN 200 and cap (without vapour pipe DN 80) to head of column;
 - fix the two receivers to the frame;
 - assemble the 3-way valve, cross piece of product pipe, sight glass and cooler; make sure that cooler is mounted accurately in vertical position;
 - before continuing erection ascertain the height required (instructions of supplier) by the reflux divider to be fitted between condenser, column head and cooler;
 - mount condenser 2 on supporting leg;
 - complete connection of vapour line between column head and condenser by cutting the DN 80 tube to size, welding on the flange and the cap of the column head;
 - complete connections between condenser, reflux divider and cooler according to instructions of supplier;
 - place condenser 1 in position on supporting leg;
 - complete vapour line between extractor and condenser inlet; make sure that pressure control valve V1 is put in a position easily accessible by hand when standing on the ground floor;
- NOTE: Safety valve and pressure gauge must be fitted between extractor outlet and valve V1; blow-off from the safety valve must lead into a direction where it cannot harm any person.
- fix the decanter to a bracket and place the florentine flask on a stand mounted on the frame;

- complete connections between condenser product outlet, decanter and Florentine flask;
- complete connection of miscella circuit between extractor, circulating pump, filters and back to top of extractor;
- complete remaining process piping between filters and evaporator, cooler and solvent tank, cooler and evaporator, solvent tank and circulating pump;
- fit drain valves.

For process pipe connections PTFE-coated gaskets and O-rings should be used throughout.

NOTE: Under normal circumstances the plant would not be constructed and for the first time assembled at the place where it is to be operated. For this reason no utility pipework should be carried out before the plant has been erected at its ultimate location, except temporary pipework for testing purposes.

Also the packing material should be charged only after erection of the column at the final location unless test runs are planned at the manufacturer's premises. Consult the supplier for correct charging of the packing material, if of patented make, especially with regard to an even distribution of the reflux over the cross section area of the column.

For easy transportation the individual beams of the frame as well as the apparatus and the supporting frame should be bolted together rather than welded.

The electrical switching cabinet must be either hermetically sealed (explosion proof) or else must be installed in a room separate from the plant, where there is no possibility of the ambient air getting loaded with inflammable vapours.

Where climatic conditions permit the plant should be installed 'open-air', however under a roof providing protection from rain and direct sun radiation.

5. Notes on Ancillary Equipment

5.1 Size Reduction

In processing of medicinal and aromatic plants for the extraction of valuable constituents virtually the whole range of plant material is used, comprising flours, fruit, seed, leaves, stems, bark, wood and rhizomes, in a fresh as well as dried state. Knowing the variation in physical properties of the various fractions of a plant, especially in hardness and tensile strength, it is evident that no single machine could be proposed which would give optimum results in all possible cases of application.

Frequently appropriate size reduction can only be achieved in two steps using machines of different grinding principles, notably where volatile or heat sensitive constituents are involved. The most commonly used grinders are

- cutting mill
- roller mill
- hammer mill

The cutting mill normally is used for pre-grinding of soft to semi-hard as well as fibrous and cellulose containing material. The roller mill is often used for seed and where only coarse breaking is required. The hammer mill e.g. may be applied in further reduction of products pre-ground in a cutting mill or in cases where heating or high volatility are of no concern. Before purchasing a grinder it is in any case advisable to have grinding trials conducted by an experienced manufacturer of grinding equipment paying special attention to heat damage and loss of volatile matter.

5.2 Drying

With the organic solvent evaporating the product is collecting in the evaporator usually in form of a viscous syrup. In the case of essential oils or oleoresins the product is traded as such and no further treatment is required.

In case the plant should also be used for aqueous extraction of medicinal plants the products will similarly be of syrupy consistency. The products cannot be marketed in this form but must be transferred into the dry state. This requires an additional drying step. Three types of dryers are most commonly used:

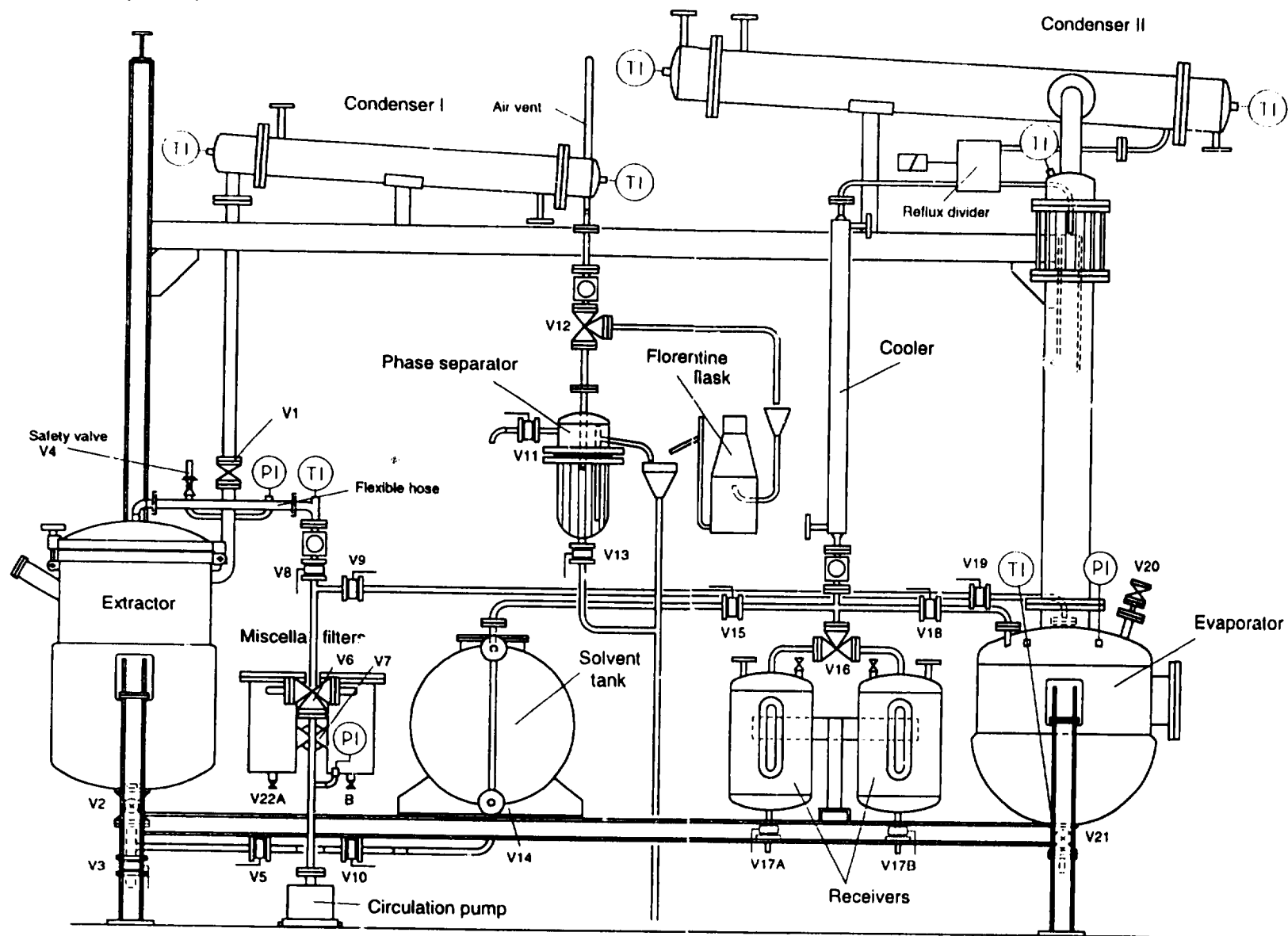
- tray drying cabinet at atmospheric pressure or vacuum;
- spray dryer at atmospheric pressure.

- drum (roller) dryer at atmospheric or reduced pressure;

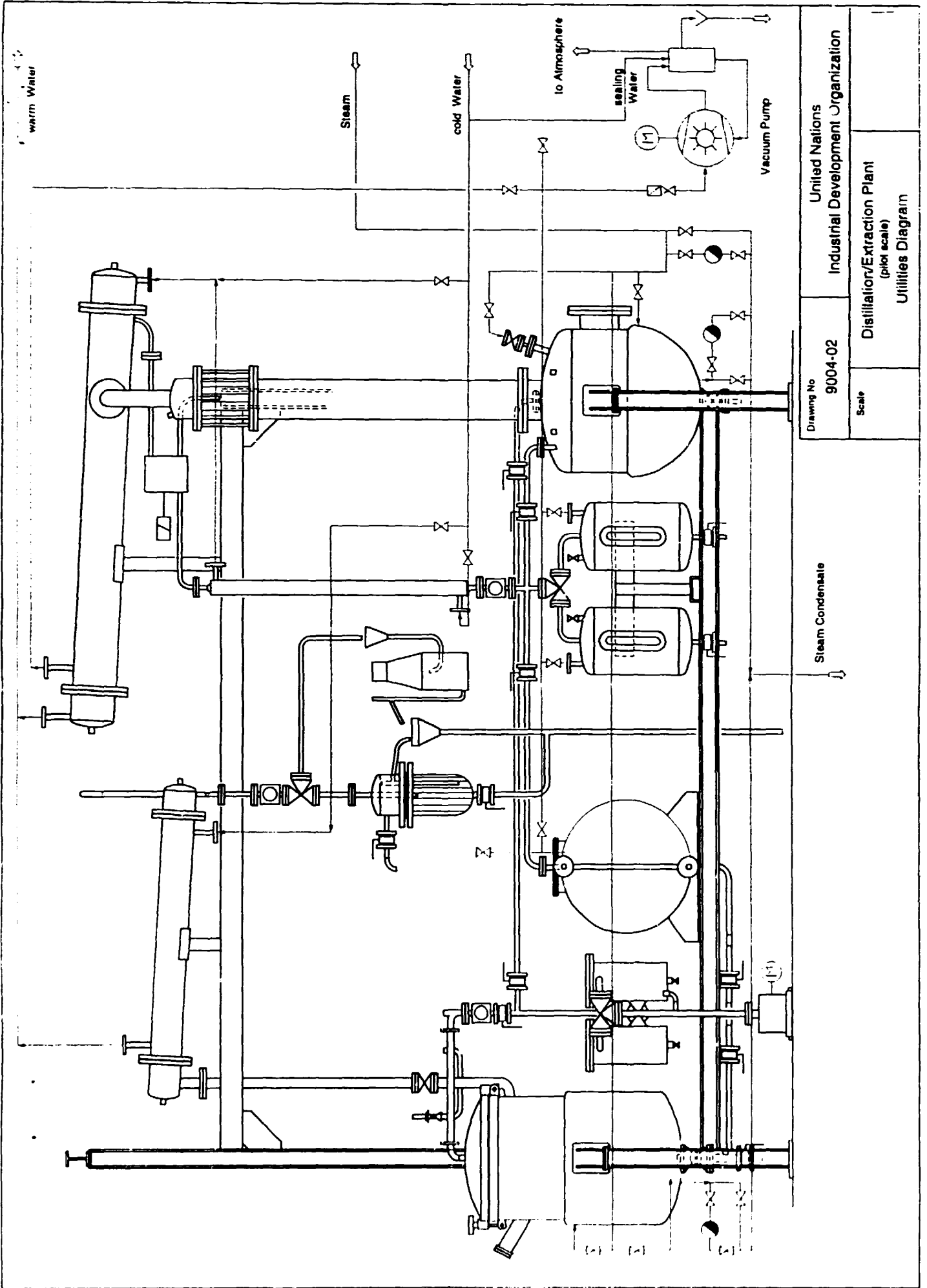
The choice of the dryer is determined by the properties of the product, mainly viscosity, hygroscopicity and heat sensitivity. To match the in case of aqueous extraction reduced capacity (vide page 2) of the extraction unit normally a laboratory drying unit of about 1 - 2 kg/h capacity of dry product would be sufficient. Dryers of this capacity are available from various suppliers of laboratory equipment, with the exception of drum dryers which usually are of 10 - 20 kg/h capacity.

Here, too, it is advisable to have samples of the products tested with prospective suppliers in order to be able to select the appropriate dryer. Not only residual moisture is an important criterium but equally appearance, density or bulk weight, possible heat damage, and dissolvability.

UNIDO Distillation/Extraction Plant			List of Drawings	
No.	Title	No.	Remarks	
1	Arrangement and Instrumentation	9004 - 01		
2	Utilities Diagram	9004 - 02		
3	Supporting Structure	9004 - 03		
4	Extraction Vessel 250 l	9004 - 10		
5	Extraction Vessel Detail X1	9004 - 11		
6	Extraction Vessel Detail X2	9004 - 12		
7	Plate Inserts	9004 - 13		
8	Florentine Flask	9004 - 16		
9	Condenser 1	9004 - 20		
10	Condenser 1 Head Plate	9004 - 21		
11	Condenser 2	9004 - 25		
12	Condenser 2 Head Plate	9004 - 26		
13	Evaporator	9004 - 30		
14	Packed Column	9004 - 31		
15	Cooler	9004 - 34		
16	Phase Separator	9004 - 35		
17	Receiver	9004 - 36		
18	Solvent Tank	9004 - 40		
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	Date	Name	Order No.	
Compiled				
Checked				
Revised				



Drawing No. 9004-01		United Nations Industrial Development Organization	
Scale	Distillation/Extraction Plant (pilot scale)		
Arrangement & Instrumentation			



Steam Condensate

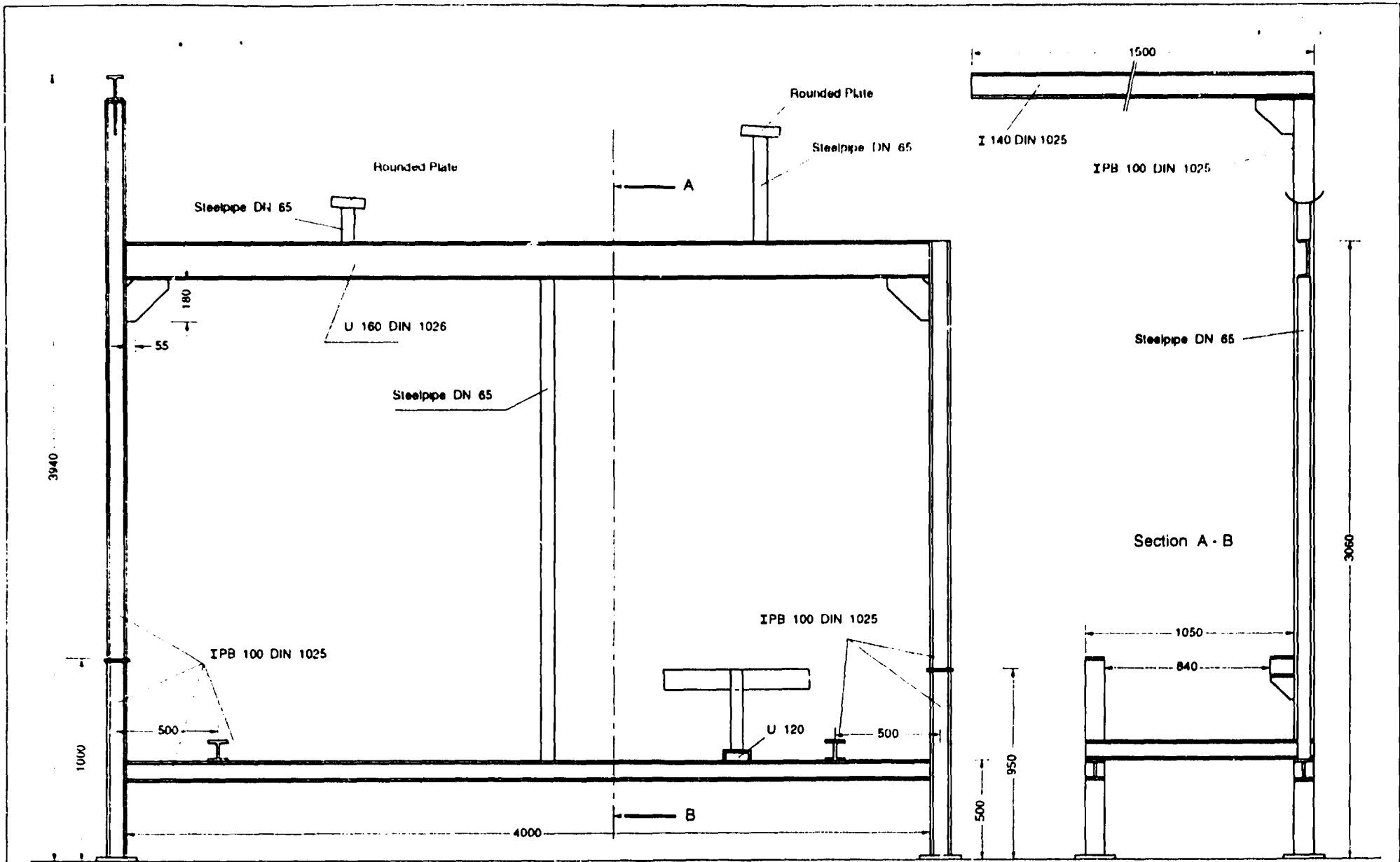
Drawing No
9004-02

United Nations
Industrial Development Organization

Distillation/Extraction Plant
(pilot scale)

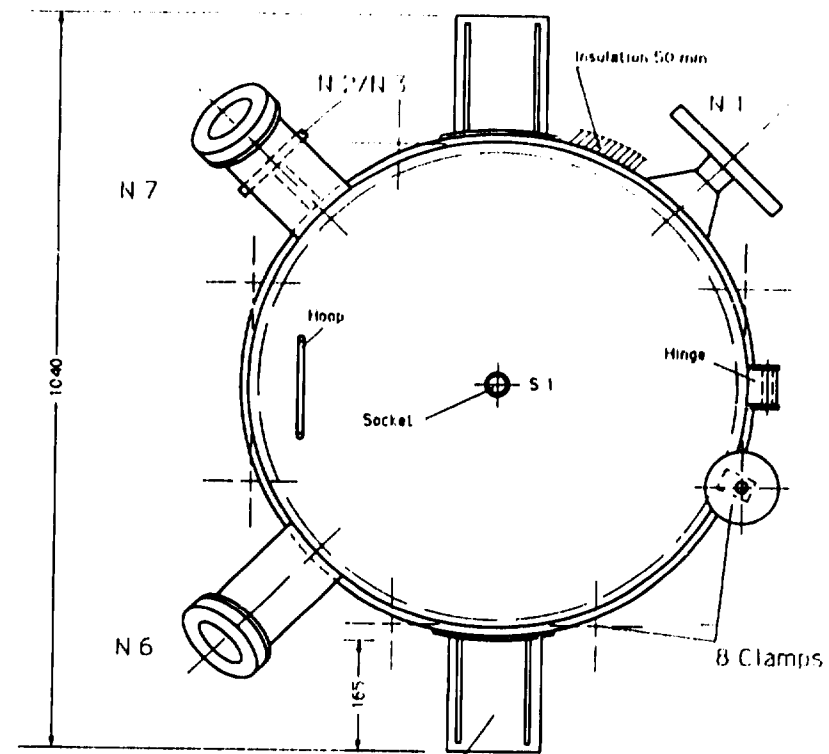
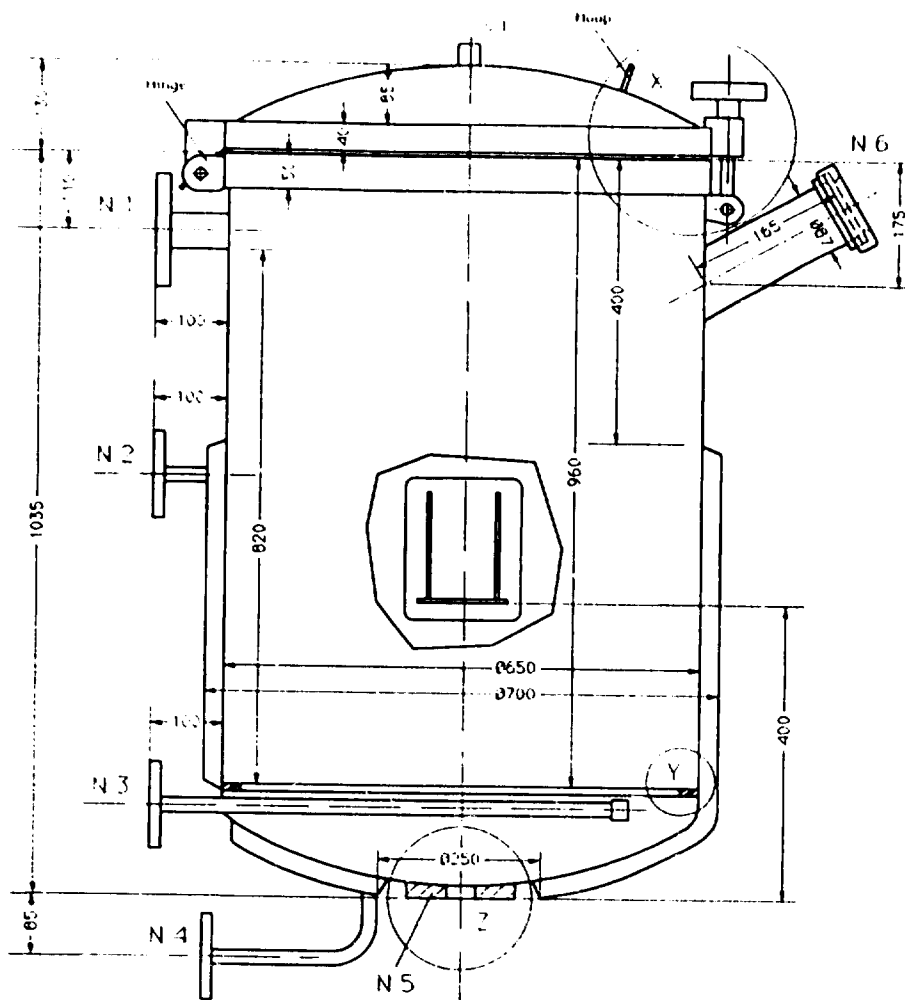
Scale

Utilities Diagram

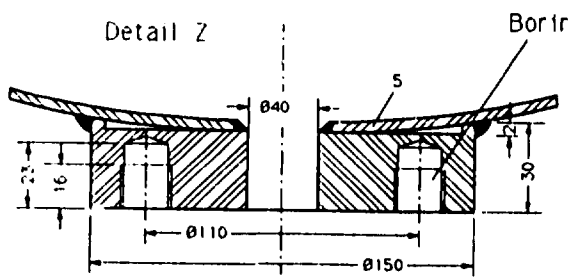
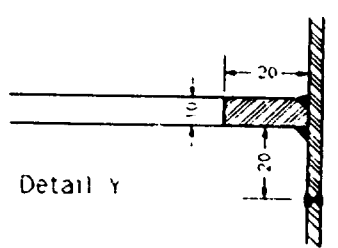


Drawing No 9004-03		United Nations Industrial Development Organization	
Scale	Distillation/Extraction Plant (pilot scale)		
	Supporting Structure		

Position of pipe connections according to top view only!

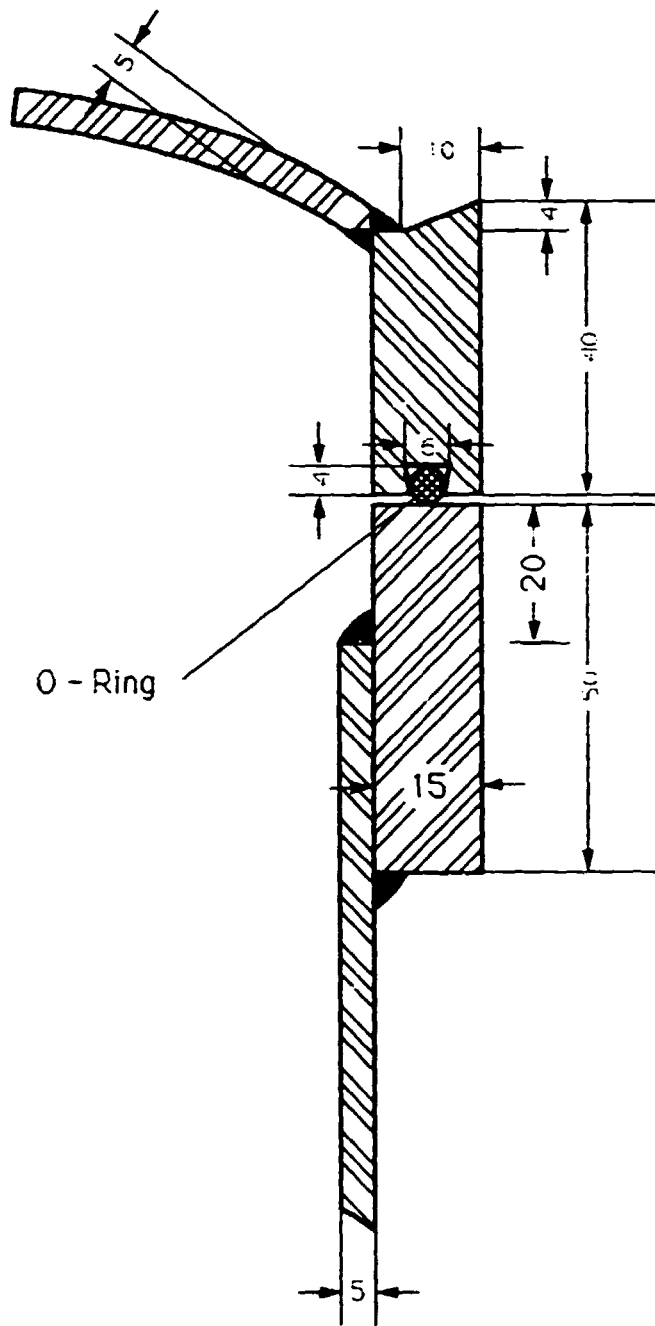


2 Brackets DIN 28083 - A2D



Flange	DN	PH	DN	Application
N 1	50	10	2576	Vapour
N 2	15	10	2576	Steam
N 3	15	10	2576	Steam
N 4	15	10	2576	Condensate
N 5	40	10	28117	Drain
N 6	80	10	11851	Sight glass
N 7	80	10	11851	Light glass
S 1	3/4"	10	2986	Solvent recirculation

Drawing No. 9004-10		United Nations Industrial Development Organization	
Scale	Distillation/Extraction Plant (pilot scale)		
	Extraction Vessel 250 l		



Drawing No.

9004-11

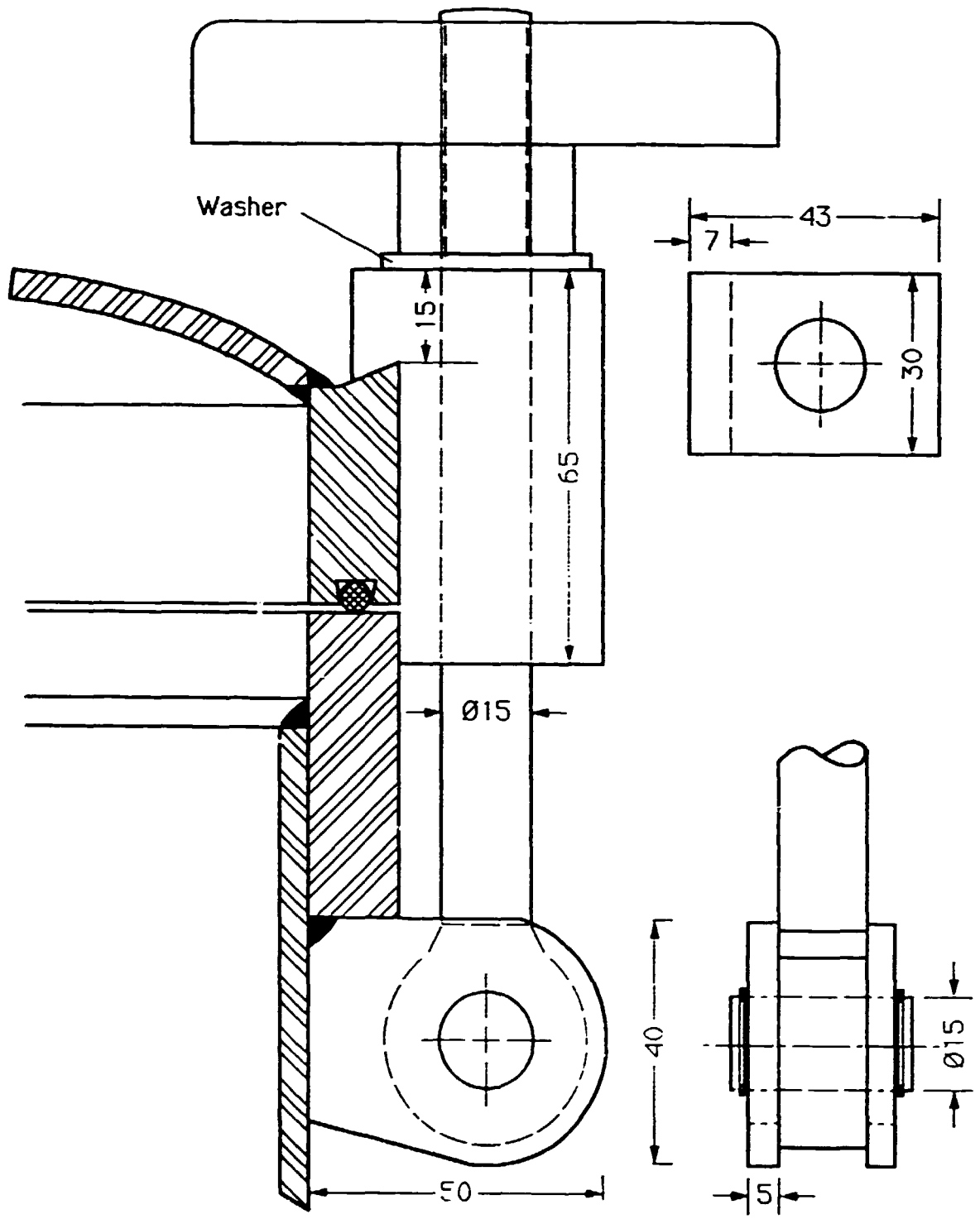
United Nations

Industrial Development Organization

Scale

Distillation/Extraction Plant
(pilot scale)

Extractor Detail X1



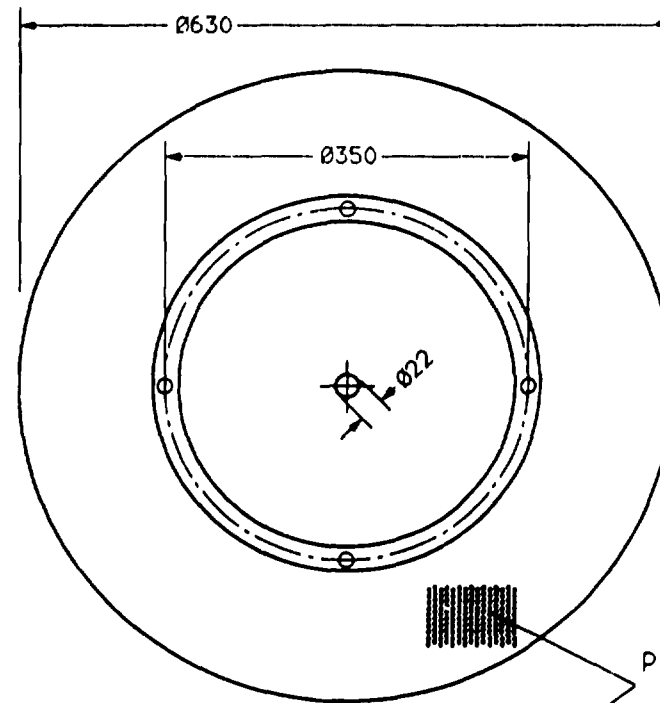
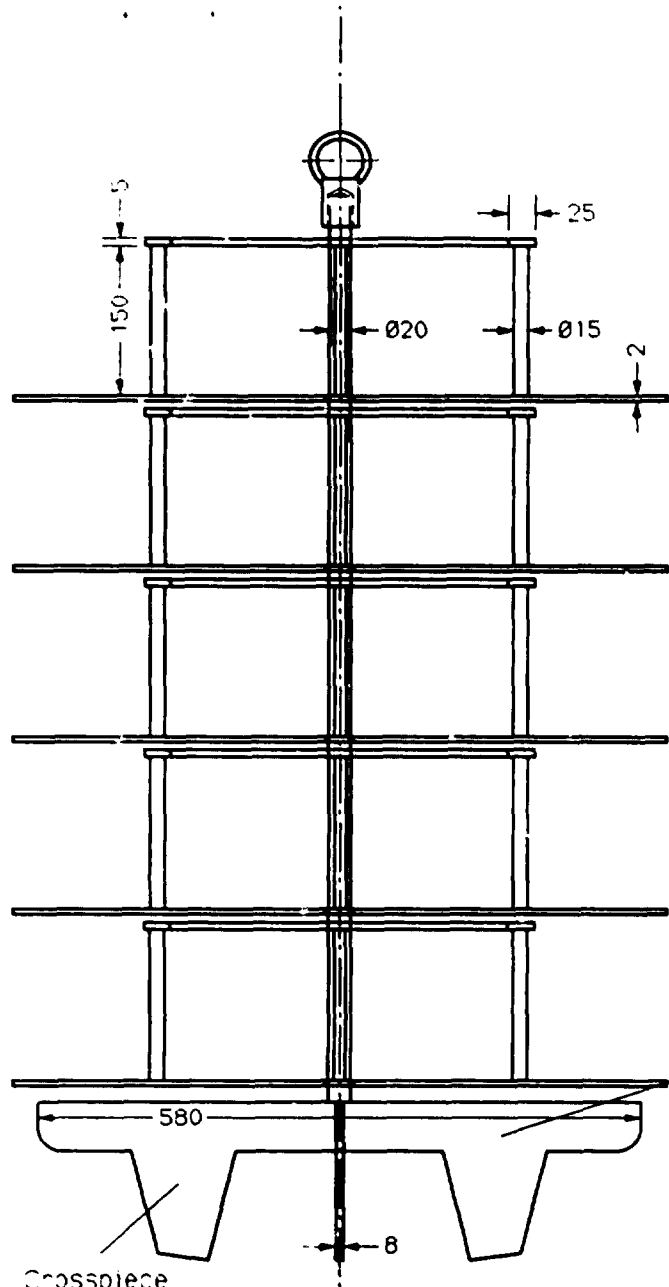
Drawing No.

9004-12

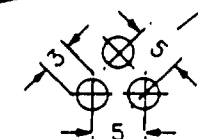
United Nations
Industrial Development Organization

Scale

Distillation/Extraction Plant
(pilot scale)
Extractor Detail X2



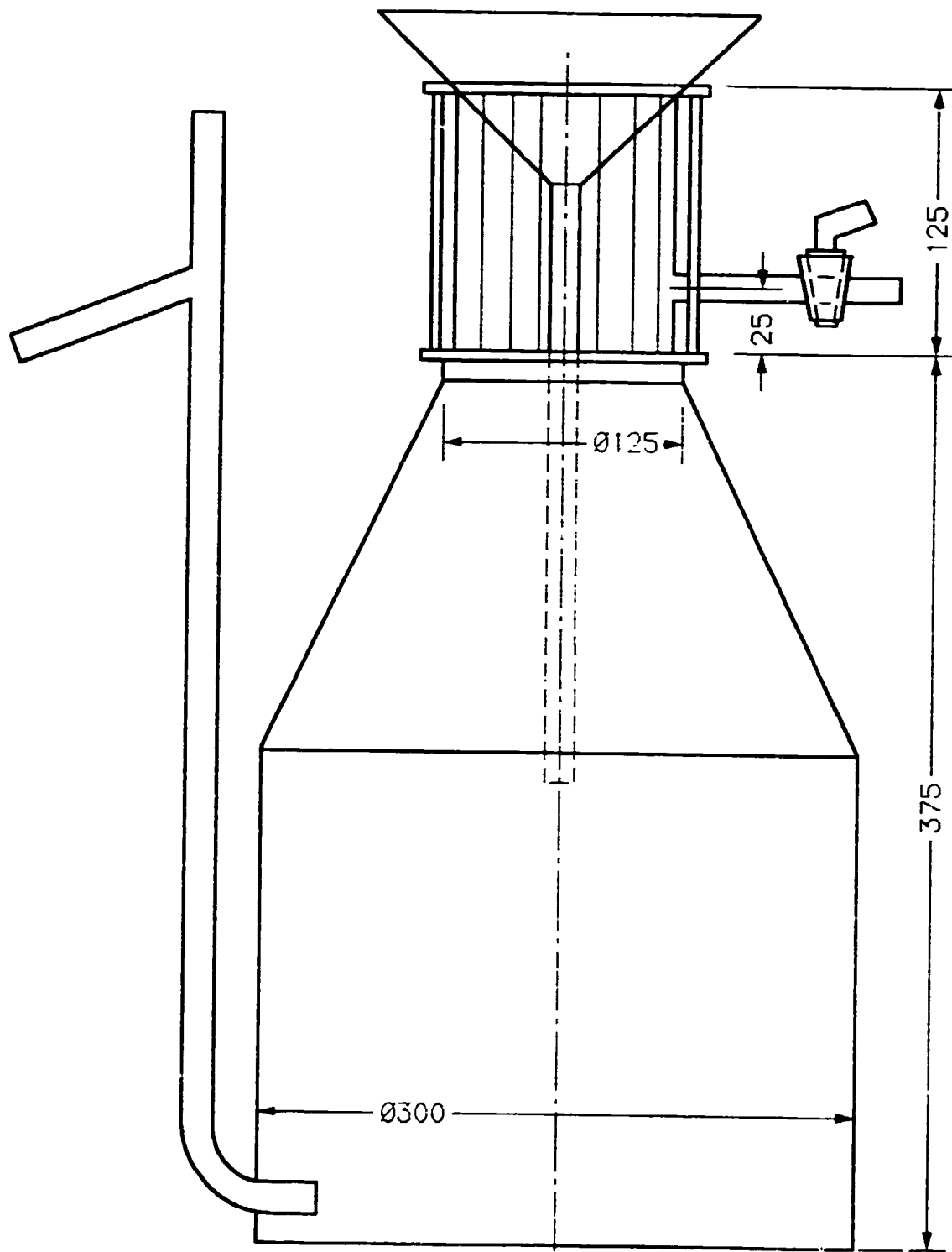
Pattern of perforation



Dimensions depending on shape of dished bottom

Crosspiece

Drawing No. 9004-13		United Nations Industrial Development Organization	
Scale	Distillation/Extraction Plant (pilot scale)		
	Plate Inserts for Extractor		



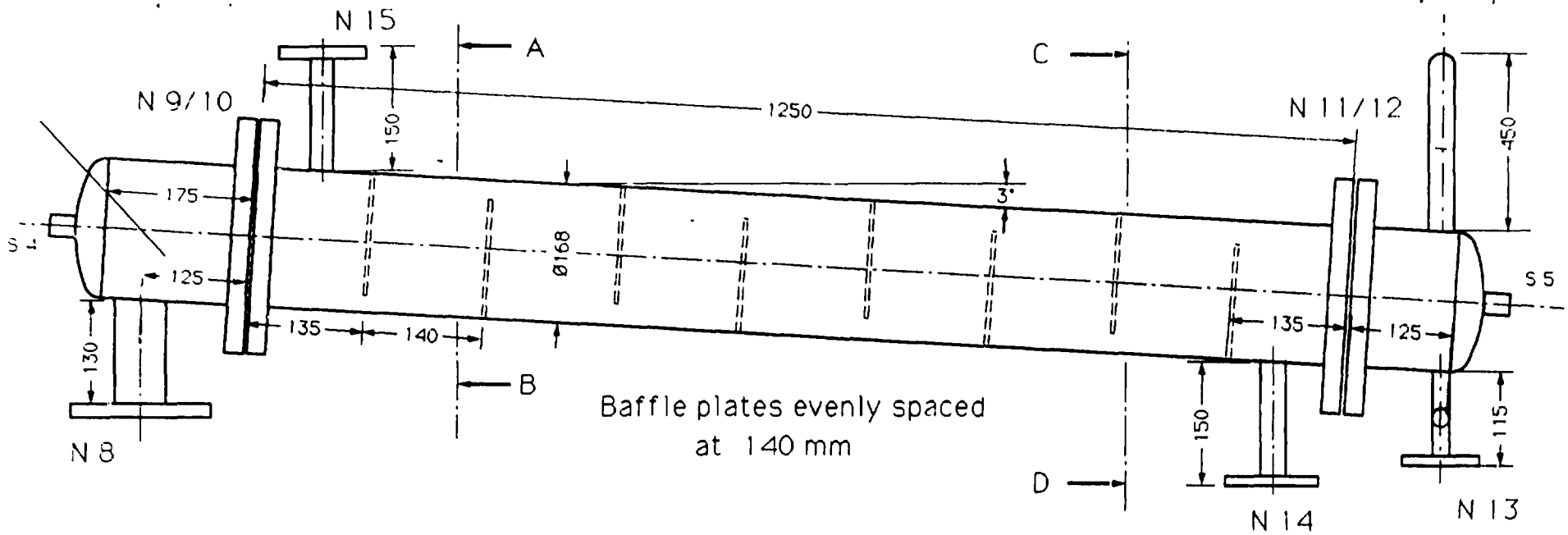
Drawing No.

9004-16

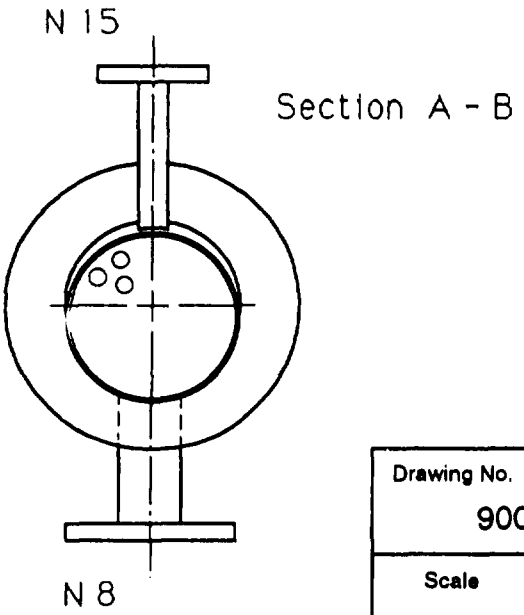
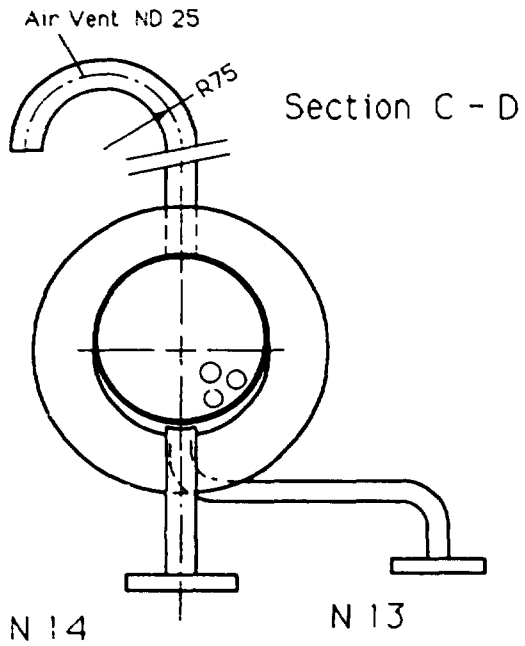
United Nations
Industrial Development Organization

Scale

Distillation/Extraction Plant
(pilot scale)
Florentine Flask

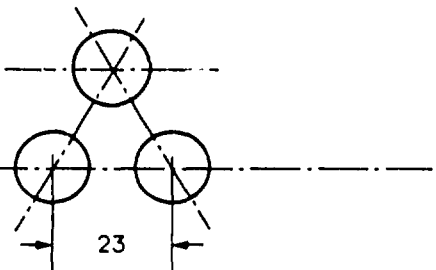
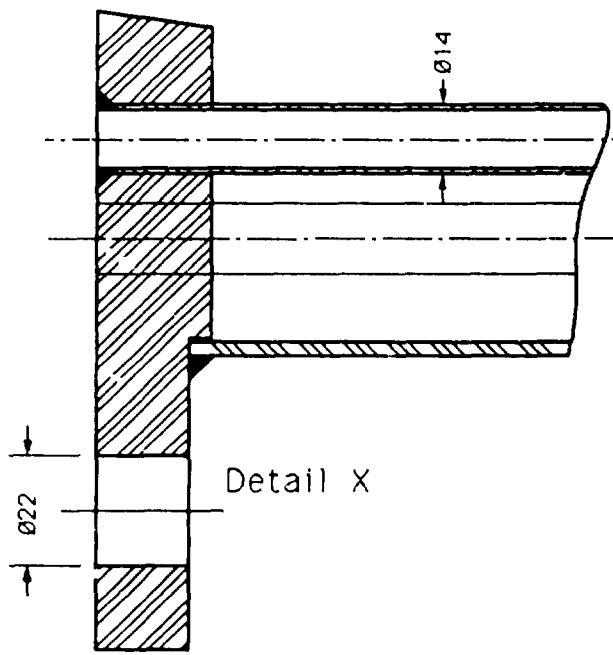
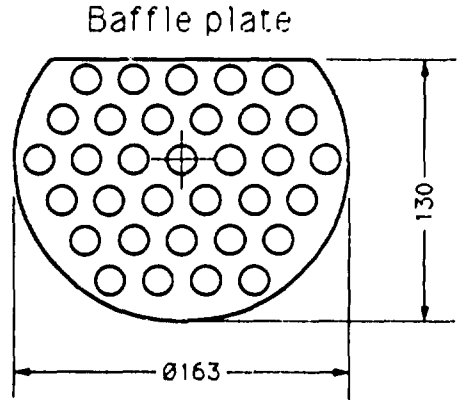
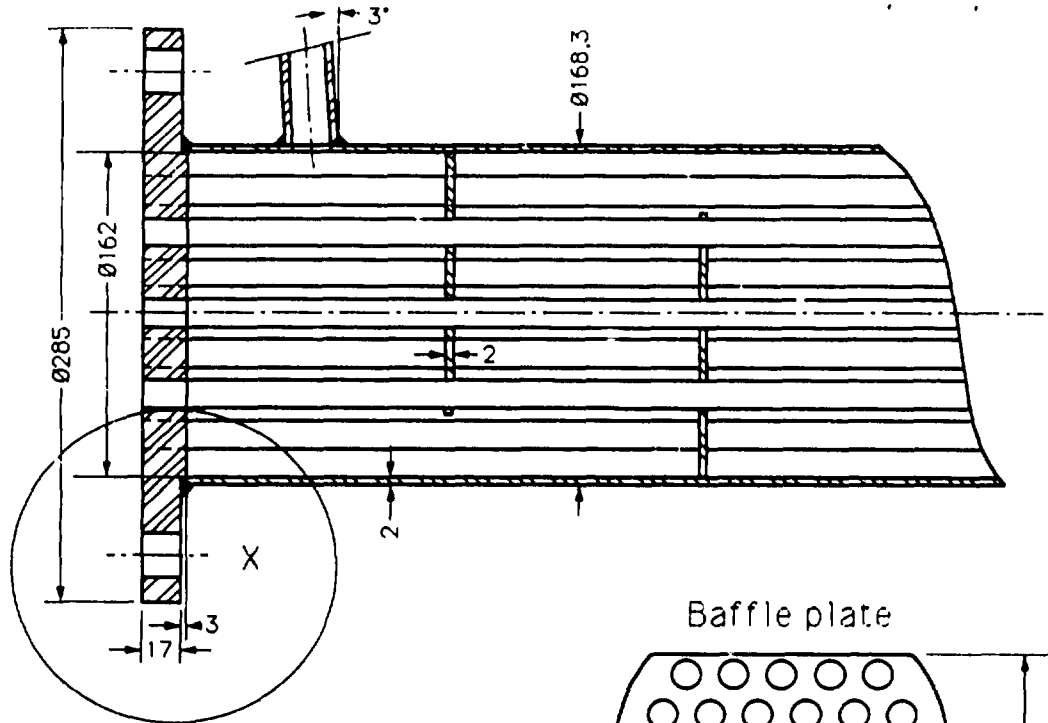
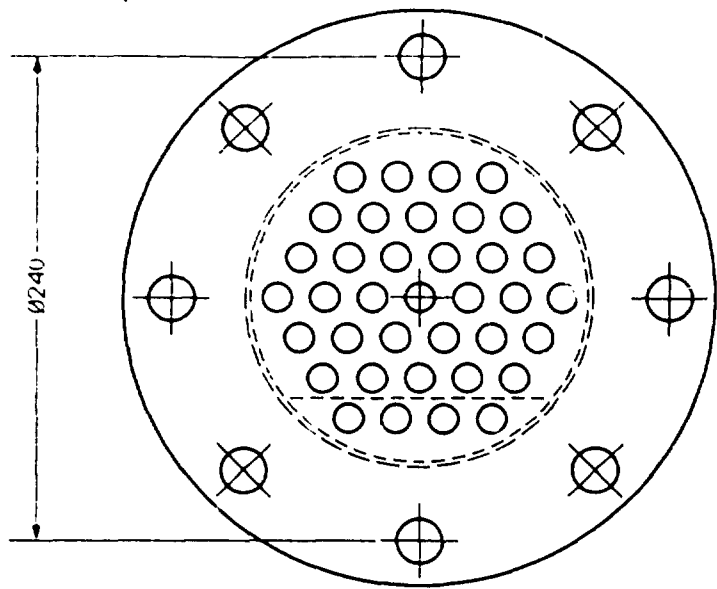


Baffle plates evenly spaced at 140 mm

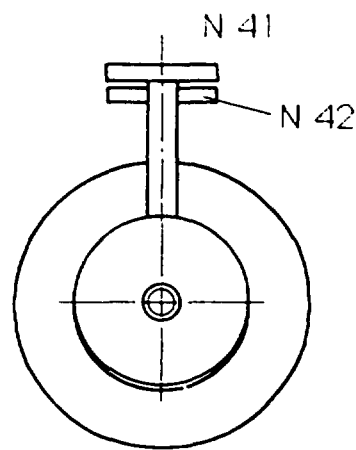
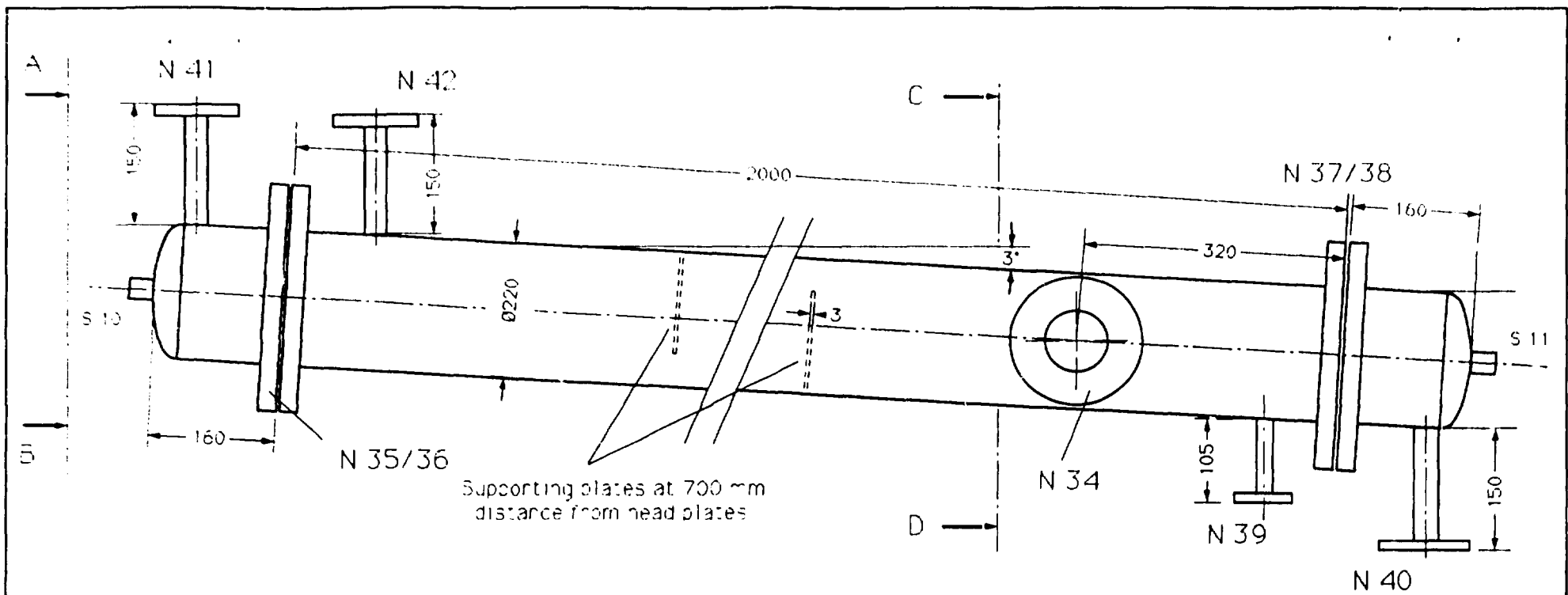


Flange	DN	PN	DIN	Application
N 8	50	10	2576	Vapour
N 9/10	150	10	2576	Head plate & counter flange
N 11/12	150	10	2576	Head plate & counter flange
N 13	5	10	2576	Product discharge
N 14	25	10	2576	Cooling water supply
N 15	25	10	2576	Cooling water discharge
S 4	1/2"	10	2986	Temperature indication
S 5	1/2"	10	2986	Temperature indication

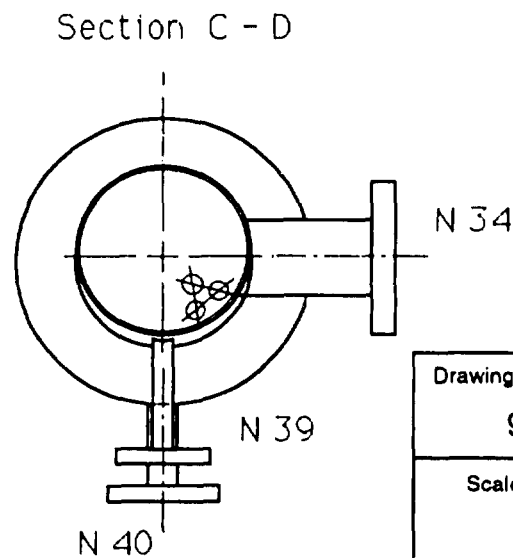
Drawing No. 9004-20		United Nations Industrial Development Organization	
Scale	Distillation/Extraction Plant (pilot scale) Condenser 1		



Drawing No. 9004-21		United Nations Industrial Development Organization	
Scale	Distillation/Extraction Plant (pilot scale)		Condenser 1 Head Plate

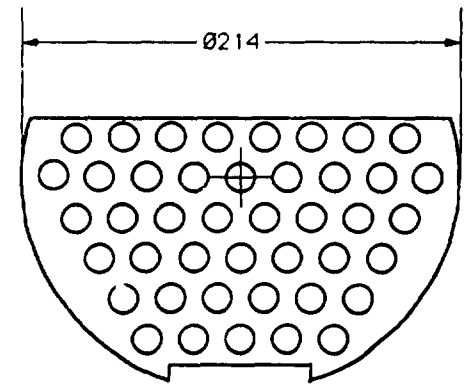
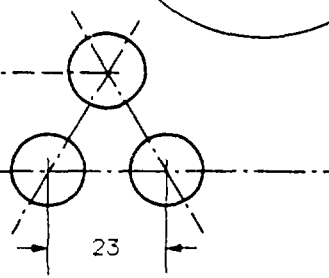
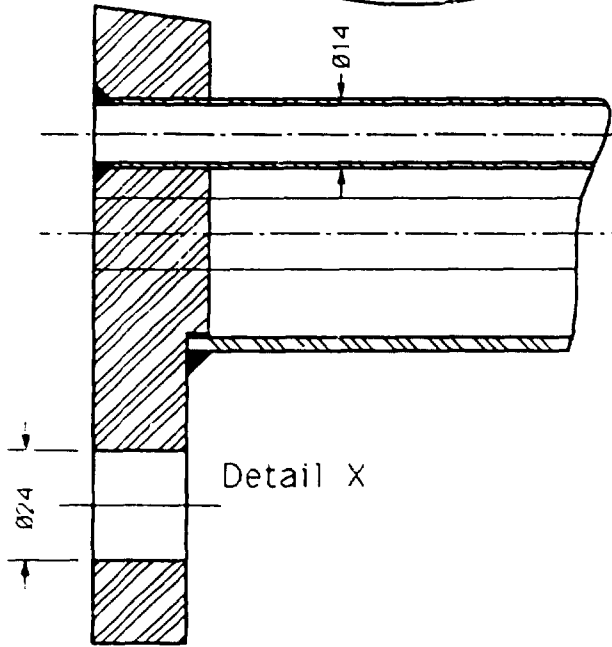
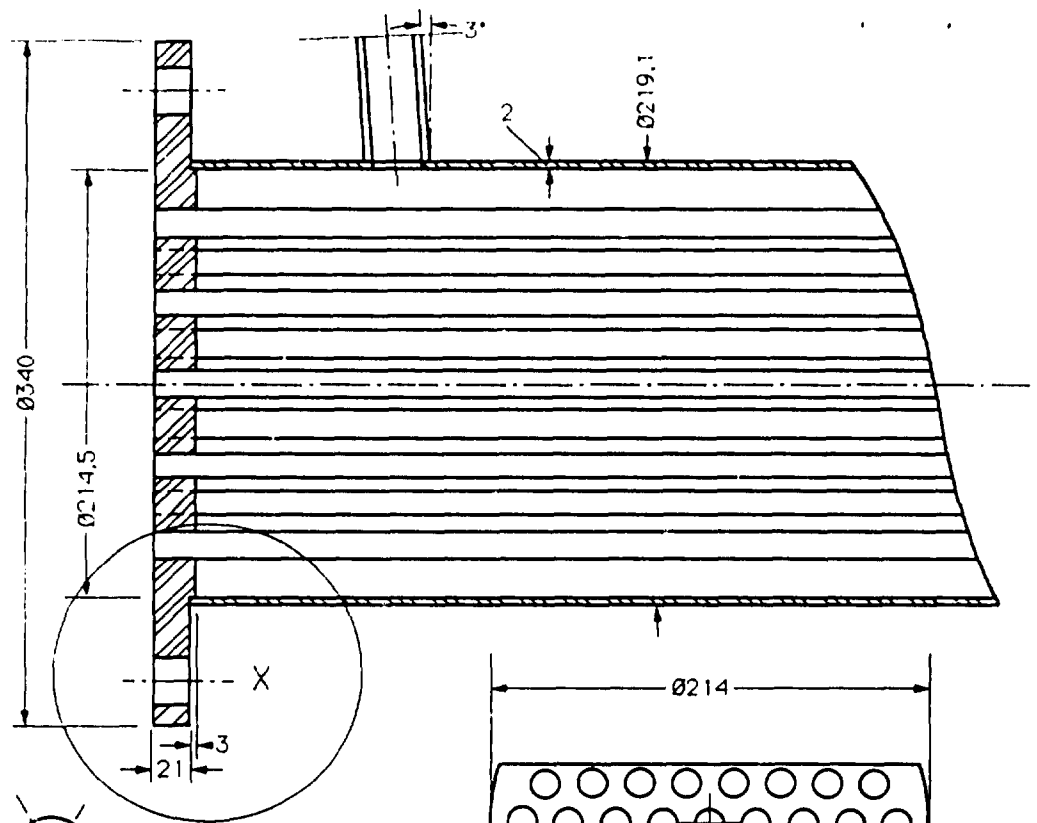
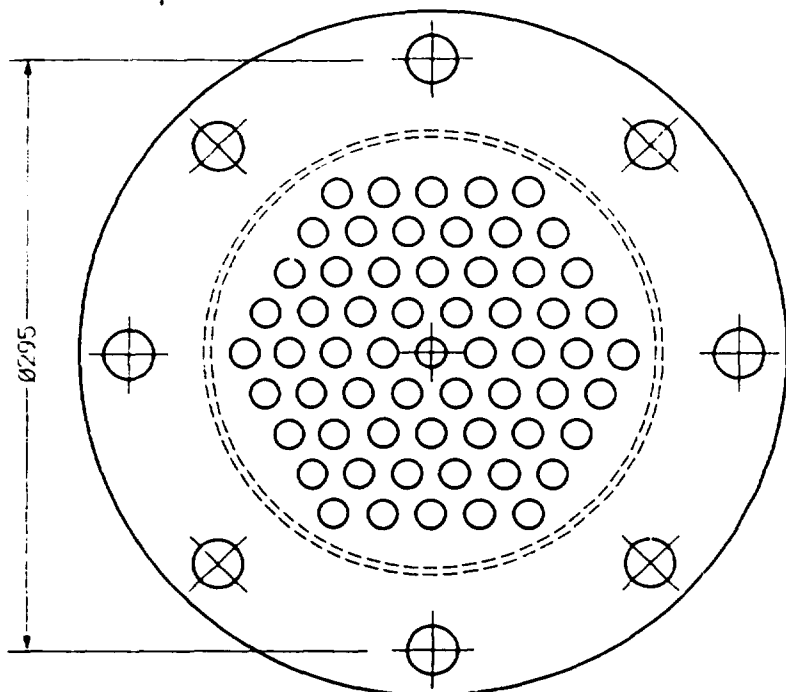


View A - B



Flange	DN	PN	DIN	Application
N 34	80	10	2576	Vapour
N 35/36	200	10	2576	Head plate & counter flange
N 37/38	200	10	2576	Head plate & counter flange
N 39	20	10	2576	Product outlet
N 40	25	10	2576	Cooling water inlet
N 41	25	10	2576	Cooling water outlet
N 42	25	10	2576	Vacuum pipe
S 10	1/2"	10	2986	Thermometer
S 11	1/2"	10	2986	Thermometer

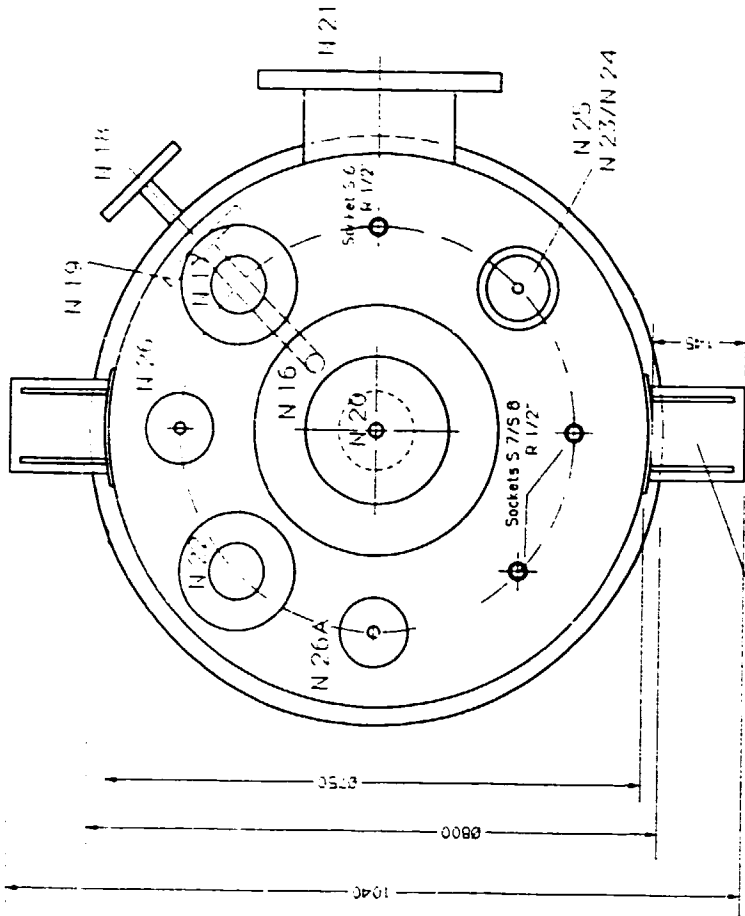
Drawing No. 9004-25		United Nations Industrial Development Organization	
Scale	Distillation/Extraction Plant (pilot scale) Condenser 2		



Tube supporting plate

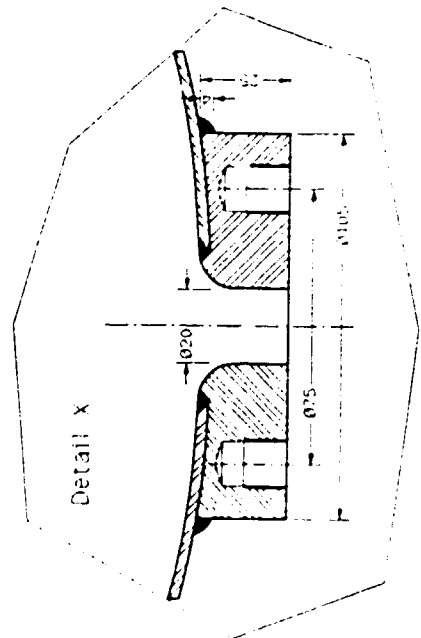
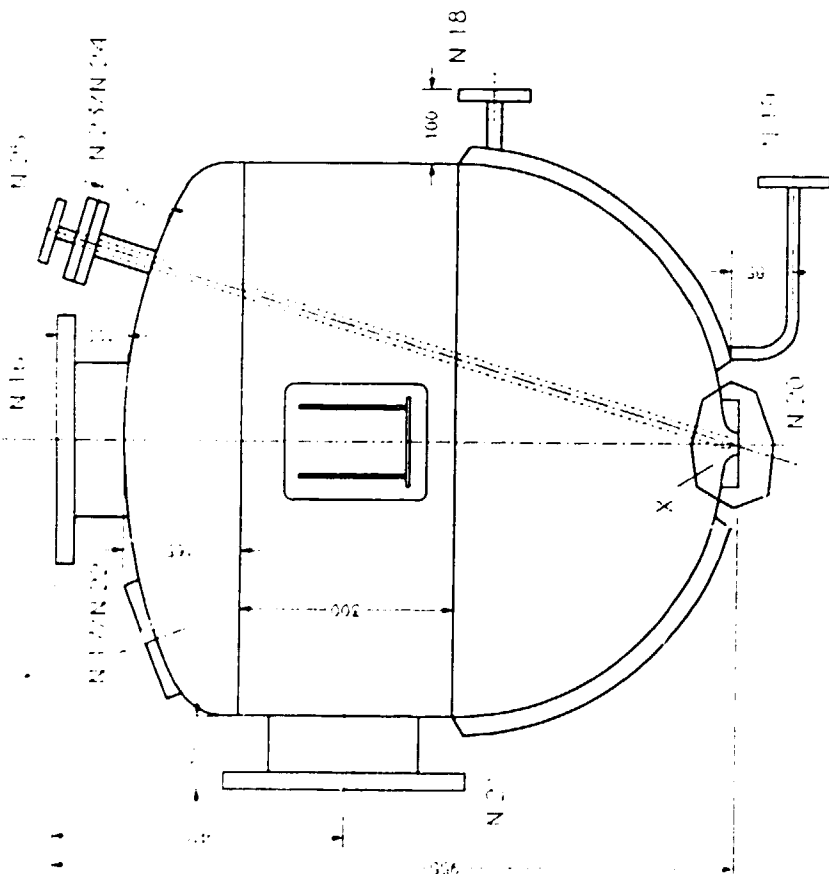
Drawing No. 9004-26		United Nations Industrial Development Organization	
Scale	Distillation/Extraction Plant (pilot scale)		
	Condenser 2 Head Plate		

Position of pipe connections according to top view only



Flange	DN	PN	DIN	Application
N 16	200	10	2576	Solvent vapour
N 17	80	10	28120	Sight glass
N 18	15	10	2576	Steam
N 19	15	10	2576	Steam condensate
N 20	200	10	28117	Product outlet
N 21	200	10	2576	Inspection port
N 22	80	10	28120	Inspection light
N 23	25	10	2576	Purging lance assembly
N 24	25	10	2576	
N 25	15	10	2576	
N 26	20	10	2576	Extract inlet
N 26A	20	10	2576	Solvent recirculation
S 6	1/2	10	2986	Vacuum relief
S 7	1/2	10	2986	Vacuum gauge
S 8	1/2	10	2986	Thermometer

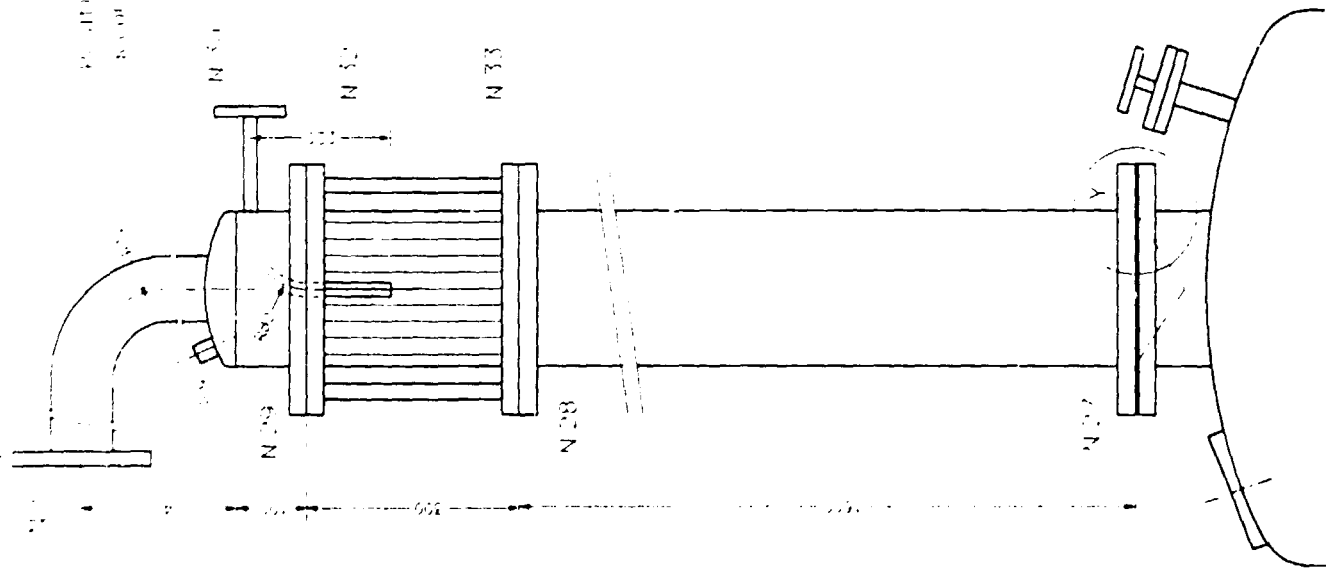
2 Brackets
DIN 28083 - A2D - 1 4571



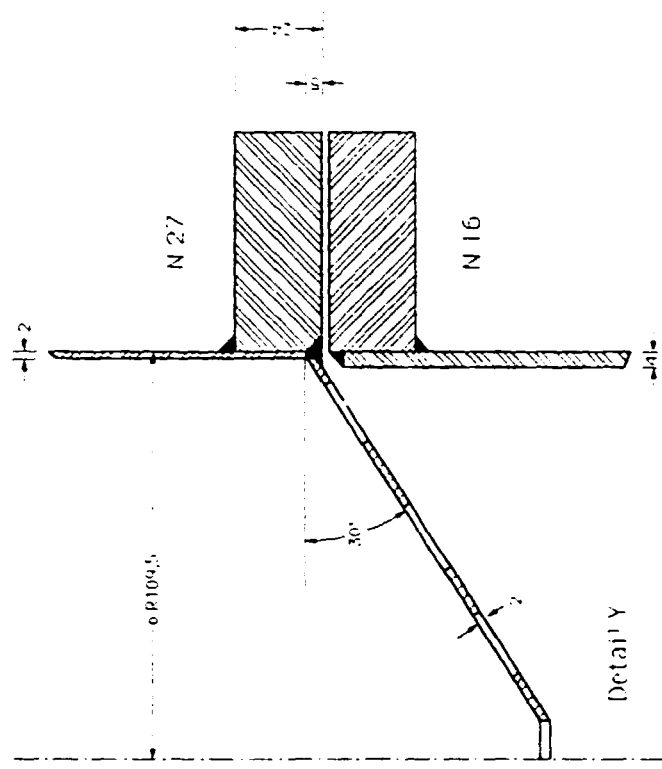
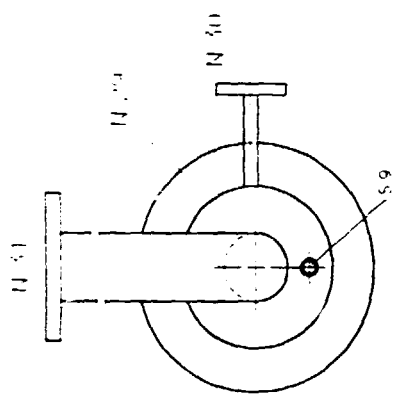
Drawing No
9004-30
Scale

United Nations
Industrial Development Organization
Distillation/Extraction Plant
(pilot scale)
Evaporator

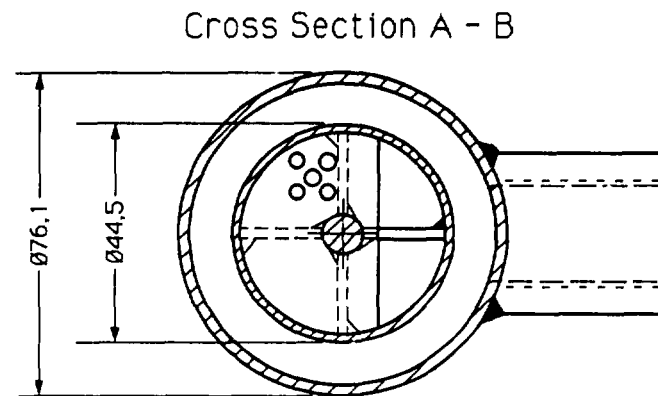
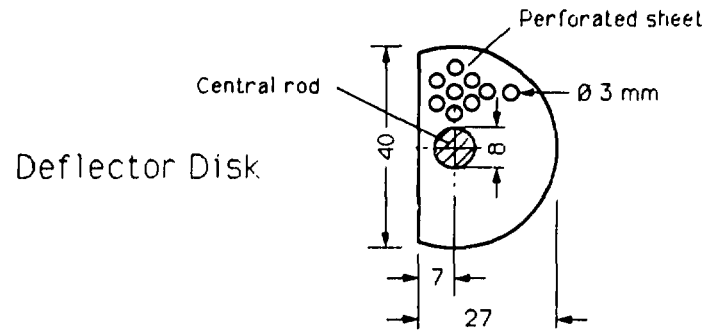
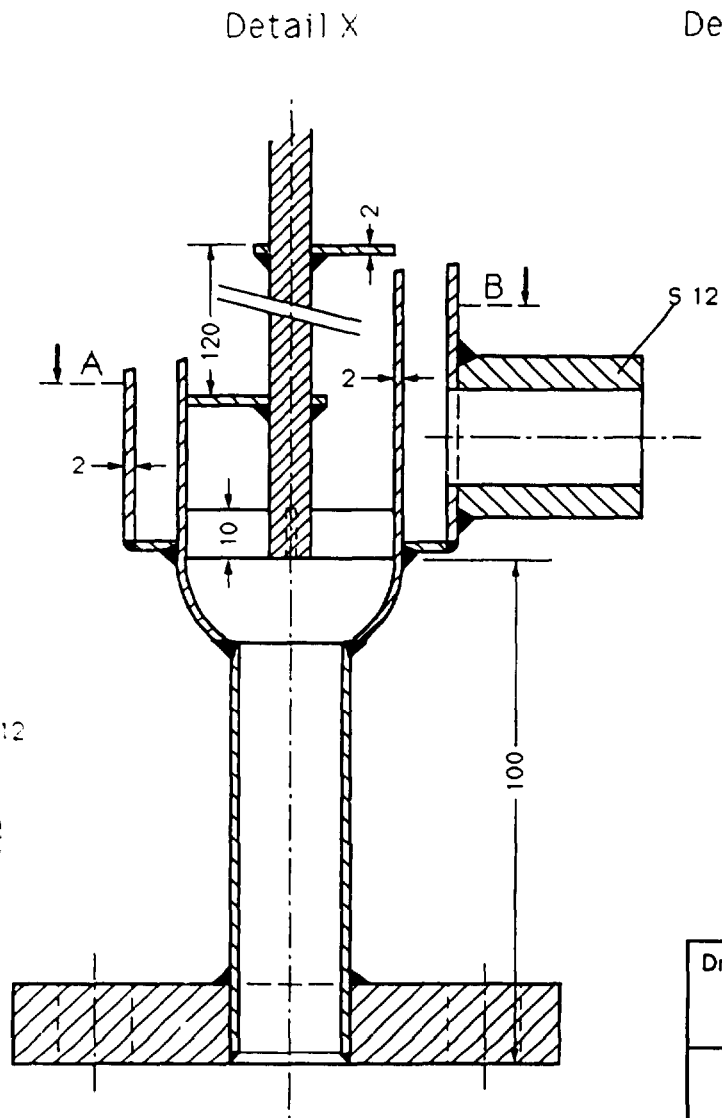
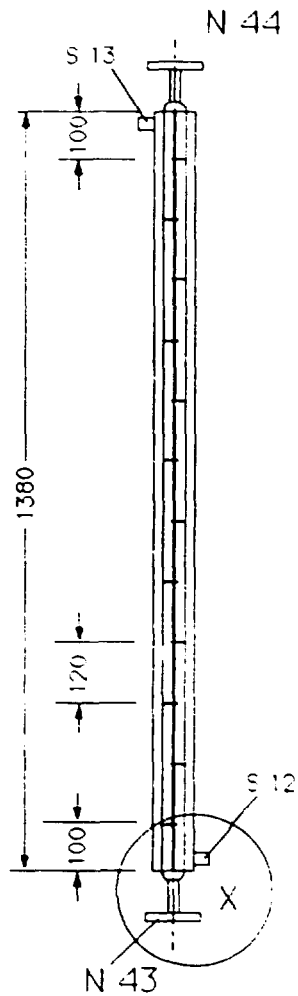
Detail of pipe connection
is omitted to top view only



Range	DN	PN	DN	Application
N 27	200	10	2 1/2"/6	Bottom end of column
N 29	200	10	2 1/2"/6	Top end of column
N 30	200	10	2 1/2"/6	Cap
N 31	80	10	2 1/2"/6	Reflow pipe
N 32	200	10	2 1/2"/6	Vapour outlet
N 33	200	10	2 1/2"/6	Sight glass
N 35	200	10	2 1/2"/6	Sight glass
N 36	172	10	2 1/2"/6	Thermometer

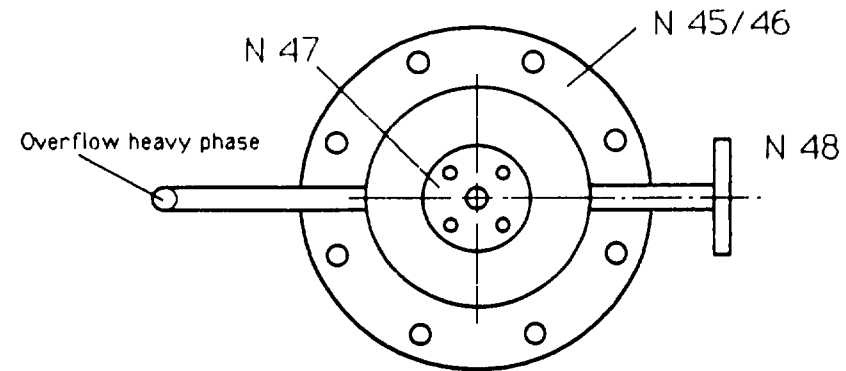
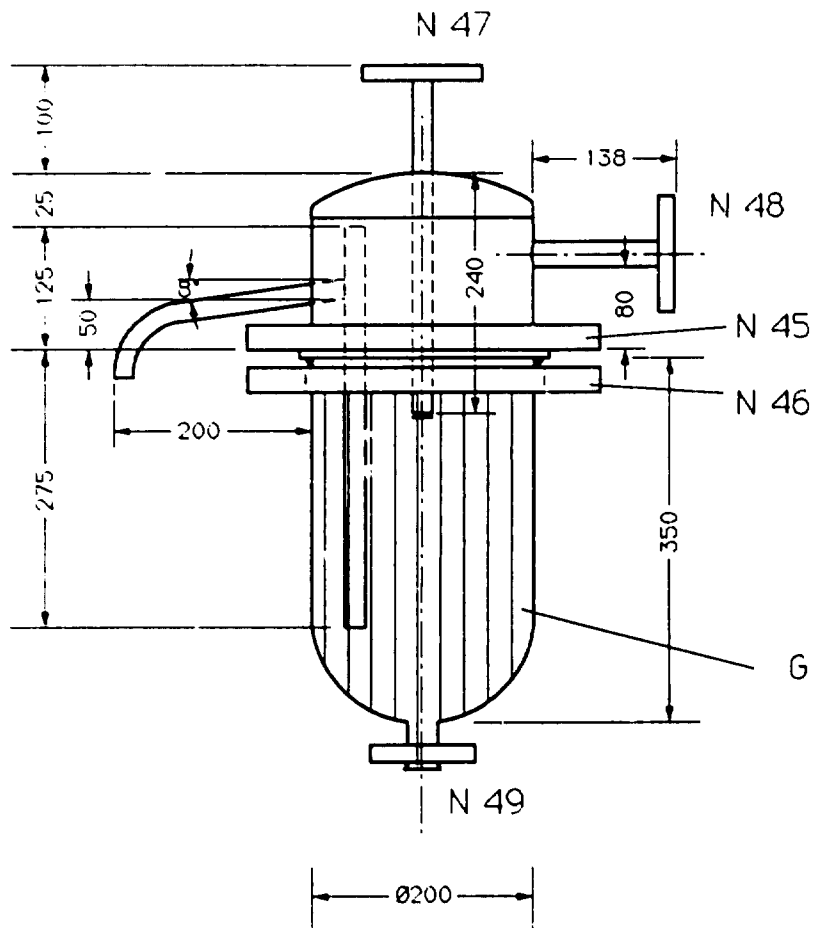


Drawing No 9004-31	United Nations Industrial Development Organization	
	Scale	Distillation/Extraction Plant (pilot scale) Packed Column



Flange	DN	PN	DIN	Application
N 43	20	10	2576	Bottom end of cooler
N 44	20	10	2576	Top end of cooler
S 12	3/4"	10	2986	Cooling water in
S 13	3/4"	10	2986	Cooling water out

Drawing No. 9004-34		United Nations Industrial Development Organization	
Scale	Distillation/Extraction Plant (pilot scale) Cooler		



N 46 (Flange to be machined according to instructions of supplier of glass receptacle)

Glass receptacle

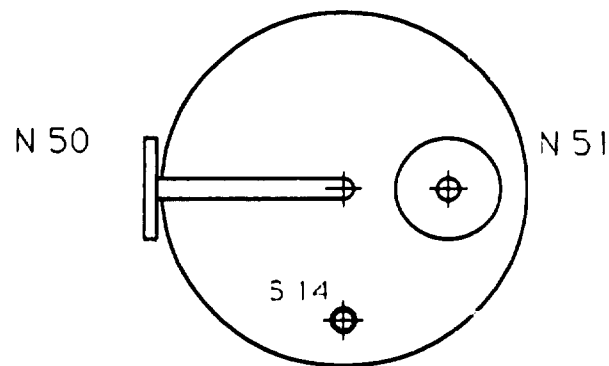
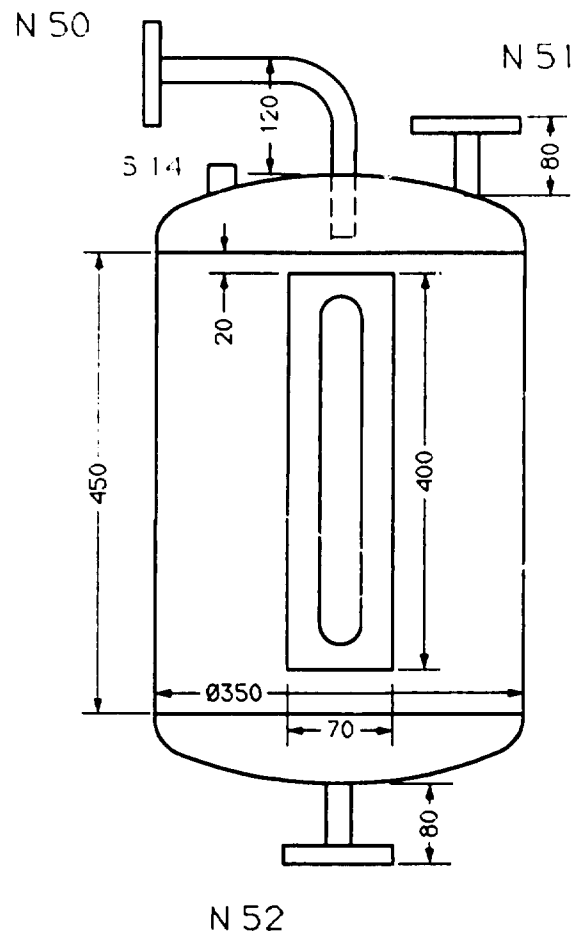
Flange	DN	PN	DIN	Application
N 45	200		2576	Cap
N 46	200		2642	Glass receptacle
N 47	20		2576	Mixture inlet
N 48	20		2576	Overflow light phase
N 49	20		2642	Drain
	20			Overflow heavy phase

Drawing No.
9004-35

United Nations
Industrial Development Organization

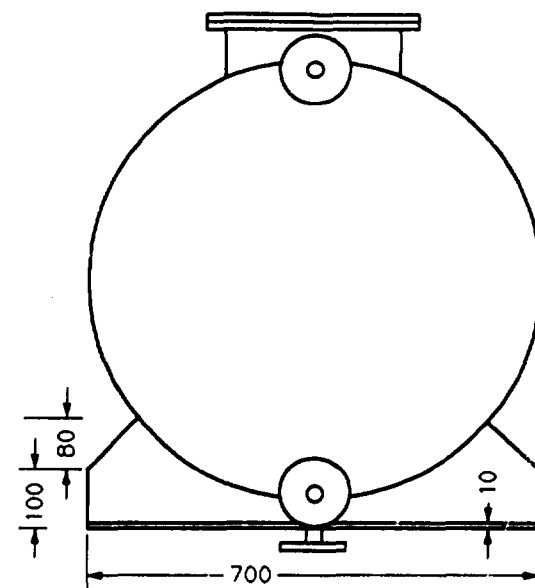
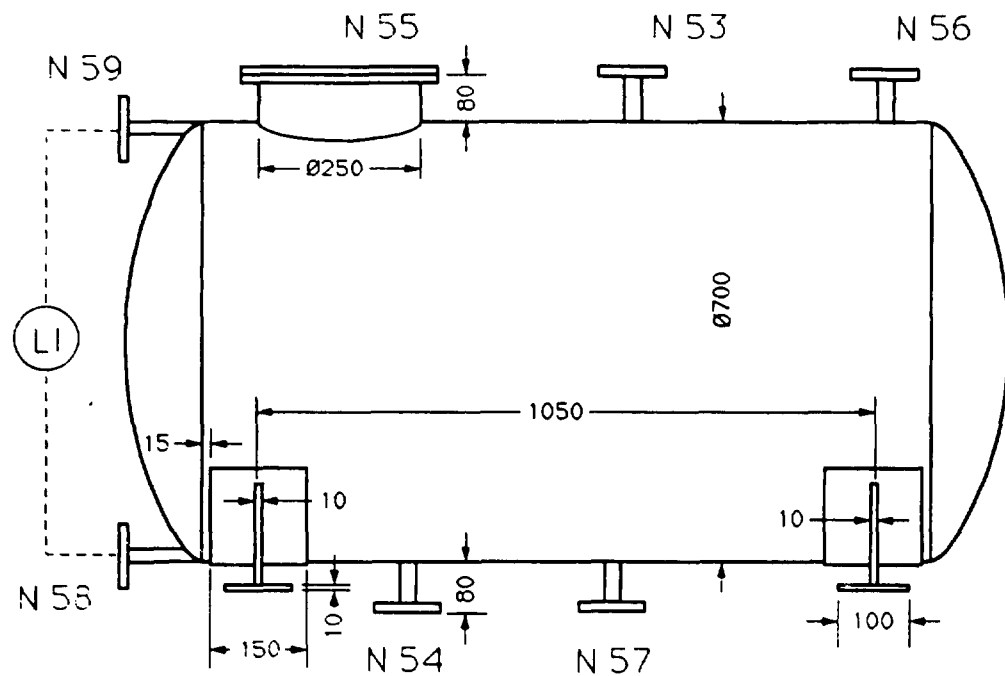
Scale

Distillation/Extraction Plant
(pilot scale)
Phase Separator



Flange	DN	PN	DIN	Application
N 50	15	10	2576	Product supply
N 51	15	10	2576	Vacuum
N 52	15	10	2576	Product outlet
S 14	1/2"	10	2986	Vacuum relief

Drawing No. 9004-36		United Nations Industrial Development Organization	
Scale	Distillation/Extraction Plant (pilot scale) Receiver		



Flange	DN	PN	DIN	Application
N 53	20	10	2576	Solvent recirculation
N 54	20	10	2576	Solvent discharge
N 55	250	10	2576	Man hole
N 56	20	10	2576	Vacuum
N 57	20	10	2576	Drain
N 58	15	10	2576	Level gauge
N 59	15	10	2576	Level gauge

Drawing No. 9004-40		United Nations Industrial Development Organization	
Scale	Distillation/Extraction Plant (pilot scale) Solvent Tank		

**UNIDO
Distillation/Extraction Plant**

Bill of Quantities

No.	Item	Qty.	Unit	Dimensions	Remarks
Material of construction: Stainless steel 1.4571, unless otherwise stated					
1	Pipe	16	m	DN 20/26,9	
2	Pipe	5	m	DN 15/21,3	
3	Pipe	2	m	DN 50/60,3	
4	Pipe	1	m	DN 40/48,3	
5	Elbow	20	pce.	DN 20/26,9 90°	
6	Elbow	10	pce.	DN 15/21,3 90°	
7	Elbow	1	pce.	DN 50/60,3 90°	
8	Butterfly valve	8	pce.	DN 20	
9	Butterfly valve	3	pce.	DN 15	
10	Ball valve	1	pce.	DN 40	
11	Ball valve	1	pce.	DN 20	
12	Ball valve	2	pce.	DN 15	
13	Slide valve	1	pce.	DN 50	
14	Cock	4	pce.	1/2" threaded	
15	3-way ball valve	2	pce.	DN 15	
16	3-way ball valve	3	pce.	DN 20	
17	Sight glass	1	pce.	DN 15	
18	Sight glass	2	pce.	DN 20	
19	Safety valve	1	pce.	3/4" threaded	
20	Pressure gauge	2	pce.	1/2" range: 0 - 10 bar	
21	Vacuum gauge	1	pce.	1/2" range: -1 - +3 bar	
22	Thermometer	5	pce.	1/2" x 100, range 0 - 200 °C	
23	Thermometer	1	pce.	1/2" x 60, range 0 - 200 °C	
24	Thermometer	1	pce.	1/2", 0 - 200 °C to reach bottom of evaporator	
25	Flange, loose	36	pce.	DN 20/26,9 DIN 2642	
26	Flange, loose	16	pce.	DN 15/21,3 DIN 2642	
27	Flange, loose	4	pce.	DN 50/60,3 DIN 2642	
28	Butterfly valve	1	pce.	DN 40	
29	Flange, loose	3	pce.	DN 40/48,3 DIN 2642	
	Date	Name	Process Piping, Fittings & Instrumentation		Drawing No. 9004-01
Compiled					
Checked					
Revised					