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STRENGTHENING OF ESSENTIAL OIL INDUSTRY IN KOREA

DP/DRK/88/001

DEMOCRATIC PEOPLE'S REPUBLIC OF KOREA

Technical report: Installation and commissioning of equipment for distillation and extraction*

Prepared for the Government of the Democratic People's Republic of Korea
by the United Nations Industrial Development Organization,
acting as executing agency for the United Nations Development Programme

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Vienna

* This document has not been edited.

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ABSTRACT

Title of project:	Strengthening the Essential Oil Industry in Korea
Number of project:	DP/DRK/88/001/A/01/37
Number of mission:	DP/DRK/88/001/11-01 DP/DRK/88/001/11-53
Duty station:	Pyongyang DRK
Duration:	47 days
Post title:	Supervision Expert and Chemical Technologist

The UNIDO consultants fulfilled their tasks at the Pyongyang Essential Oil Research Centre (PEORC) during their missions in end of May/June 1992.

The special tasks were,

- to carry out the supervision of installation and mechanical testing of bench scale equipment delivered from Germany in accordance with the contract between UNIDO and the contractor agro-consult dresden (ACD), UNIDO-purchase-No. 15-0-0344 M, as well as

- to put this equipment into operation and to train the PEORC staff in operating the bench scale equipment.

These tasks have been performed with positive results and a corresponding protocol was signed by the partners.
The National Project Director (NPD) confirmed the fulfilment of the consultant's obligations on the end of their work in Pyongyang.

I. INTRODUCTION

This report is given by Mr. Böhme, planning engineer at the "Ingenieurbetrieb Anlagenbau Leipzig GmbH", in O-7010 Leipzig, Germany and by Mr. Langner, project manager, at the "Linde-KCA-Dresden GmbH", in O-8012 Dresden, Germany.

Both experts employed with and working on behalf of agro-consult dresden GmbH have put into operation and tested successfully the pilot plant for essential oils in PDR-Korea.

The report gives a survey on the activities which were done at PEORC

- to carry out the supervision of installation and mechanical testing of bench scale equipment delivered from Germany in accordance with the contract between UNIDO and the contractor agro-consult dresden (ACD), UNIDO-purchase-No. 15-0-0344 M,

- to put this equipment into operation,

- to train the PEORC staff in operating of the bench scale equipment

The mission started in Pyongyang on May 25, 1992, and was finished on July 01, 1992 (including traveltime) without debriefing stop in Vienna. The last one will take place after August/September 1992.

This part report No. 4 continues and finishes the reporting about the design, the planning of technological main and auxiliary units, supplying, erection and commissioning of bench scale equipment. See also - reports from Mr. Langner from December 1989, January 1991 and September 1991.

All the objectives planned were attained and a respective protocol was signed.

II. TECHNICAL REPORT ABOUT BENCH SCALE EQUIPMENT

A. Description
General

The bench scale equipment consists of

- main system No. 1, vacuum falling-film evaporation,
- main system No. 2, discontinuous vacuum rectification as well as the auxiliary systems
- No. 1, vacuum generation 1 and 2,
- No. 2, cold generation,
- No. 3, steam generation.

The vacuum falling-film evaporation serves for removing and recycling of solvent n-hexane under saving the product conditions (short stay time, low temperature) from extracts of solvent extraction of essential oils from plant raw material mainly from flowers such as Rosa rugosa and other.

The discontinuous vacuum rectification has got the technological task raw essential oils produced by steam distillilation also under saving the oil conditions (vacuum, low boiling temperature) to clean and to fractionate.

The mentioned auxiliary systems serve to provide the main systems with vacuum, coldness and steam.

The main systems are accomplished as compact units completely premounted at the manufacture's workshop in Germany and in this form transported by ship to Korea.

The a. m. equipment and systems are in detail explained and specified in the project documentation from February 1991. Here will be given only a short description.

Layout of equipment systems

For illustration-see annexes No. 5 and 6, figures No. 1-18 and installation plan, draw-No. 51/02493(0) a, b.

The equipment systems described in this report are installed on the premises of the PEORC institute, Pyongyang, DPRK. They are accommodated in two (2) roofed buildings, i.e. in the machine room and equipment hall in which the temperature does not fall below +5 °C.

The machine room is an existing building which is adjacent to the northern wall of an available laboratory building and was rebuilt to meet the new requirements.

The machine room is dimensioned as follows:

layout:	3.6 m x 11.5 m
height:	abt. 3.5 m

The machine room houses the electric distribution V1, the compact-type chiller/refrigerating unit 1N1 and the automatic steam generator 2N1.

The new equipment hall (layout: 5 m x 11.5 m, height: abt. 12 m) is located at a distance of 1 m from the northern wall of the machine room. This hall houses the vacuum falling-film evaporation and discontinuous vacuum rectification units, the vacuum generation units No. 1 & No. 2.

The filling stations for the falling-film evaporation and discontinuous rectification units are located outside the equipment hall, on the northern side.

The access roads are on the western side of the buildings. Mounting girders are provided for to set up the framings.

Main system No. 1, Vacuum Falling-Film Evaporation

This system is shown in annex No. 6, PID, drawing-No. 51/02723a(0).

It is arranged in a light-weight steel framing as compact unit with the main sizes:

- layout: 1,2 m x 3,1 m
- height: appr. 8,8 m

All parts of the compact unit are completely made from stainless steel (X8CrNiTi18.10).

The unit consits of

- 6 pieces vessels 1V2-1V7 with a volume from 26 l to 260 l,
- 2 pieces heat exchangers (1C1, 1H1),
- 1 piece evaporator (1E1),
- 1 piece demister (1V1),
- all pipes and valves,
- instrumentation and control systems with lokal indication of technological parameters values,
- cold and hot insulation of equipment and pipes,
- light steel structure with ladders and platforms for observation and operating of the system,
- lightening system.

The compact unit ist working under following main technological parameters:

- Flow rate of raw material:
  57 kg/h of n-hexane with a wax concentration of 1.5 g/l
- evaporated and condensed quantity of n-hexane: 50 kg/h
- absolute pressure: 13.32 kPa (100 mm Hg)
- Temperature of cold water at the inlet: -15 °C
- Temperature of hot water at the inlet: 60 - 70 °C

The raw product coming from the extraction stage is made available in a barrel located in the fillingstation outside the equipment hall. Due to the underpressure of P_{abs} = 13.32 kPa available in the evaporator system, the raw product flows form the barrel into the tank 1V2.

A flash evaporation can take place in the tank 1V2 in case of ambient temperatures exceeding the boiling temperature of hexane under vacuum.

The hexane vapours thus produced are directly transferred to the top of the condenser 1C1 (max. 4 to 5 kg/h).

The level in the tank 1V2 is automatically controlled through control loop LIC 1700.

The product flows to the evaporator 1E1 by way of free gravity.

The main part of the evaparator is a vertical tube (diameter 50 mm, length 2,5 m) with jacket tube (diameter 70 mm).

Inspection glasses arranged in the bottom part of the evaporator and in the evaporator head of the it. 1E1 serve to control the evaporator function and the quantity fed by sight whilst manual regulation is effected by a regulating valve. The latter ist arranged at the level +/- 0 m.

The evaporation surface is heated up through hot water which flows through the jacket of evaporator.

In the evaporator, the hexane vapours are led in countercurrent flow to the wetted-wall film and flow subsequently via the precipitation tank 1V1 to the condenser 1C1 where the vapours are mixed together with the respective portion originating from the it. 1V2. The vapour flow is distributed over the condenser tubes to be converted into the liquid phase. The condensate and the non-condensed portions as well are flowing to the coil cooler 1H1. The condenser 1C1 has been designed as vertical tubular heat exchanger of 125 mm dia. and 1200 mm length. The inner tubes are 16 x 1 mm dimensioned. Cooling is effected through cold water.

The coil cooler 1H1 serves to condense the residual hexane vapours. The condensate originating from the it. 1C1 is cooled down from an inlet temperature of 14 °C to an outlet temperature of -10 °C. Both the condensate and the non-condensed portions are flowing together in the pipe coil. The heat to be dissipated is transferred to the cold water.

The condensate plus the non-condensed portions are separated in the buffer tank 1V4. The condensate gets into the tank 1V5. The latter can be drained via bottom valve during system operation once the feed valve an the inert gas line valve been closed and the vent valve has been opened. The condensate produced in the meantime remains in the tank 1V4 and is then transferred to the it. 1V5 once the operating condition has been set on the latter. In order to avoid reevaporation of hexane the it. 1V5 is cooled in its bottom section by way of cold water.

The tank 1V6 serves as separator vessel to separate condensate particle from the inert gas flow. It is furnished with a cooling jacket to hamper reevaporation.

The in the evaporator 1E1 non-evaporated portion of 7 kg/h is accumulated in the tank 1V3 and can directly be withdrawn either after batch end once the system has been appropriately vented or via tank 1V7 at existing operating pressure. The tank 1V7 is arranged beneath the it. 1V3 to allow flowing of the product to the it. 1V7. The tank contents can be taken off via the tap once the feed and vacuum valve has been closed and the vent valve located on the it. 1V7 has been opened.

Both products - the liquid n-hexane and the concentrated bottom product of the evaporator - are discharged in proper vessels located at the filling station outside of the equipment hall.

Main system No.2,
Discontinuous Vacuum Rectification

Refer to dwg. No. 51/02723a(0), annex No. 6.

This system is also arranged in a lightweight steel framing as completely premounted compact unit with the same sizes like the main system No. 1. All parts of the unit which are contacted with product are also completly made from stainless steel (X8CrNiTi18.10).

The unit consists of

- 1 piece column system (2K1) with
  . destillation still with heating jacket, total volume 180 l
  . column of
    .. inside diameter: 150 mm
    .. number of packing layers: 4
    .. height of one packinglayer: 1 m
    .. kind of packings: Pall-rings 15x15 mm
  . top condenser, F = 1 m^2
- 1 piece coil cooler (2H1)
- 1 piece condenser (2C1)
- 3 pieces vessel (2V1, 2V2)
- all pipes and valves,
- instrumentation and control systems with local indication of technological parameters,
- cold and hot insulation of equipment and pipes,
- light steel structure with ladders and platforms for observation and operating of the unit,
- lightening system.

The compact unit is working under following main technological parameters:

- capacity of raw essential oil: 100 l per distillation cycle
- absolute pressure at column top: 6,7 mbar
- pressure drop at the column: 0 - 40 mbar
- max. temperature of essential oil in the still: 175 °C

- max. steam pressure in still jacket:	9 bar abs.
- inlet temperature of cooling water:	25 °C
- inlet temperature cold water:	-15 °C
- reflux ratio:	changable and adjustable in a wide range

The technology is following:

The feed mixture is filled into the jacket-heated receiver appurtenant to column 2K1 and is heated up through indirect steam heating up to the boiling point. The product vapours occurring are passing the packing layer by Pall rings and are separated in countercurrent flow by rectification with liquid top product, the lower-boiling component being enriched towards column top. Here, the vapours are able to condense through a directly mounted condenser. The discharged top product gets into a reflux distribution (2 solenoid valves) where a certain ratio of reflux to top product can be set according to the available separation conditions which finally defines quality and quantity specifications of the product.

The product gets into the cooler 2H1, is cooled down to abt. 40 °C by way of cooling water and subsequently transferred to the vessels 2V1.1 and/or 2V1.2. An inspection glass arranged beneath the coil cooler 2H1 assures appropriate flow control. Samples can be taken from the tanks 2V1.1 and/or 2V1.2 for the purpose of quality control. The filling level of the tanks can be controlled by built-in inspection glasses. Some non-condensed vapours are withdrawn via column top and fully condensed through cold water (abt. -15 °C forerum temperature) in the condenser 2C1. Thus, the product vapours cannot get into the rotary slide-valve pump that are used for vacuum generation. The tank 2V2 has been arranged immediately ahead of the pump in order to precipitate residual liquid.

The condensate stemming from the heat exchanger 2C1 is collected in an expanded DN 100 tube and added to the product once quality control has been effected.

In case of batch change, the steam is directly fed to the jacket-heated receiver via a separate nozzle while the unit is connected with the atmosphere.

The steam flows up to top condenser, is condensed and so the column and still are suitably cleaned.

The condensate is collected in the still and draining into proper vessels.

Considering total reflux at the very beginning the top product quantity is increased step by step through a changing ratio of reflux to top product. The condensate is collected in the tanks 2V1.1 and/or 2V1.2 and drained into barrels.

Auxiliary systems
Auxiliary systems No. 1,
Vacuum gereration No. 1 and 2

Refer to drawing-No. 51/02724(0), 51/02723(0), annex No. 6

In accordance with the required system pressures for every main system a rotary slide-valve pump (capacity: 60 Nm^3/h) has been provided for functioning even in the medium-high vacuum range and ensuring a constant suction speed at extreme outdoor conditions.

However, this pump type is very susceptible to aggressive and corrosive gases that may enrich the luboil, reducing the efficiency and finally in pump failure.

That is why cooling traps and separators and a fresh oil metering device have been provided for. The so-called "fresh-oil consumption" has to be determined by test.

Auxiliary system No. 2, Refrigeration

See drawings annex 5 and 10

A glycol-water mixture (40 wt-% of $C_2H_4-(OH)_2$/60 wt-% of H_2O) is uses as cold water.

A complete chiller (refrigerating) unit is used for refrigeration (capacity: 12 kW at -15 °C cold water temperature).
It contains a 500 l capacity insulated cold-water basin with one intern circulating pump for circulation of cold water throgh the evaporator of cold agent the heat fed to the cold water during falling-film evaporation and discontinuous rectification and to dissipate this heat to the atmosphere via an air-cooled condenser.

The compact-type chiller unit accommodates the circulating pumps 1P1.1 and 1P1.2.

The circulating pump 1P1.1 delivers the cold water to the main system No. 1 and the pump 1P1.2 to the main system No. 2.

The forerun temperature of the cold water is -15 °C whereas the reflux temperature amounts to -9 °C.

Non-return valves mounted in the pipes of the circulating pumps avoid overflowing of the cold-water basin through system drainage in case of standstill position of the circulating pumps.

Venting and holding of the basin's liquid level is effected through vessels via a funnel-shaped inlet nozzle.

Auxiliary system No. 3, steam generation

Refer to drawing-No.: 51/02725a(1), annex 6 and principal flow diagramm in annex 10

An automatically operating electric steam generator 2N1, type "EDT 27" has been provided for to heat up the jacket-heated receiver 2K1 with water steam. Featuring a power input of max. 30 kW, a total of abt. 35 kg/h of steam with a temperature of max. 175 °C can be made available. Thereby, the boiler output is automatically controlled within the limits of the selected power stage, dependent on the required quantity. This also applies to the water level control, i.e. automatic cutting-off is effected at lowest and highest water level and the pump arranged in the electric steam producer is automatically switched off when the feed water tank is lacking of water. The same refers to general power failure.

B. Assembling and mechanical testing of bench table equipment

Conditions found when arriving at the Pyongyang site

On May 28, 1992, the first working day, a site inspection was carried out to check the delivered two pilot plants for completeness and their outer appearance, and to find out the progress of construction to be planned and performed according to the civil and heating and ventilation tasks handed over by ACD to PEORC in February 1991.

Participants of this inspection were the director of the institute, Dr. Choi Dueng Gwang, and his deputy, Li Myong Ho, as well as the authors of this report.

The following had been found:

- The apparatus hall to accomodate the vacuum falling-film evaporators, the vacuum rectification, the vacuum generation and the adsorption plant (PEORC` s scope of delivery) was newly built and the compact units for the vacuum falling-film evaporation prefabricated in Germany already set up.

- In the machine room, an exisiting building, where the refrigerating unit 1N1, the steam generator 2 N 1 and the main electric distribution were to be installed, the floor had been finished, and the door and the windows rebuilt according to the tasks given.

- In addition to the two compact units mentioned above the refrigerating unit, box No. 4/10, the steam generator, box No. 1/10 and the vacuum pumps, box No. 2/10 were unpacked and stored in the machine room.

  After a visual check shipping damage had been found with the two compact units and the steam generator. The delivery was complete.

- The other packages for ethylene glycol, drum No. 3/10, electrical material, boxes No. 5a/10 and 5b/10, the steel structure, piping and insulation material, boxes No. 8-10/10 were intact and were unpacked. Everything was complete and undamaged.

Remaining construction work

Parallel to pipe laying, and partially prior to that work, the following construction work was still to be carried out:

- wall openings for pipes in the apparatus hall and machinery house as well as for fresh and exhaust air for the refrigerating unit in the machine room and the wall fans in the apparatus hall.

- Fixing of pipe holders at the northern wall of the apparatus hall.

- Completion of the filling stations at the external northern wall of the apparatus hall.

- Installation of the wall fans in the apparatus hall (PEORC's scope of delivery). The fans had not been installed before the authors of this report left the site.

- Completion of the vacuum pump foundations.

Equipment installation and pipe laying

The task was to set up the units prefabricated in Germany and connect them with each other by pipes according to their functions. All equipment and piping elements were delivered from Germany to the People`s Republic of Korea. The equipment and piping were mounted between June 6 and 25, 1992.

PEORC being repsonsible for the installation at the site, had to provide the necessary machinery, tools, devices and auxiliary materials.

Despite the fact that this was a new type of task for the Institute, its limited material potential and certain initial difficulties, the work had been done with a high commitment. All workers came from the Institute and partially carried out such kind of work, like pipe insulation (insulation for cold and heat conservation) and pipe laying, for the first time.

That's why the pipe laying and welding were carried with an increasing severity beginning with

- pressureless carbons steel lines followed by
- pressure lines of carbon steel
- Cr-Ni-steel lines for refrigerant and
- Cr-Ni-steel lines for vacuum generation.

One problem was that no inert gas could be provided to weld the Cr-Ni-lines so that the lines had to be welded with heavily covered Cr-Ni-steel electrodes under normal atmosphere. This resulted in a lower welding seam quality. Since these lines do not carry corrosive media corrosion can be excluded.

Piping insulation was partially carried out parallel to the pipe installation. The insulation was completed after the pressure test had been carried out.

The pressure tests were performed at maximum operating pressure obtained by the installed pumps as no booster pumps were available.

The vacuum lines were tested under vacuum.

All systems of the test plant had been tested after having been installed in Germany.

After having done the most essential construction work in the machine room the refrigerating unit 1N1 and the electric steam generator 2N1 were arranged at the place designed for. For the two systems no foundations were needed because they have their own feet.

Still before the supervisor had arrived the complete vacuum falling-film evaporator plant and the vacuum rectification had been placed by PEORC on the foundations in the apparatus hall (see above).

The vacuum pump 1P2 (system 1) and the vacuum pump 2P1 (system 2) were to be screwed on the foundation by simply using plastic dowels.

The problems mentioned above were solved by the high commitment and the rapid acquisition of skills by the Korean staff and the comprehensive assistance by the institute management and representatives of the GBCIO. The task was fulfilled.

Installation of the electrical equipment

All electrical equipment and installation material from the power supply connecting point in the machine room to the individual consumer was supplied by the seller of the plant. The installation was carried out by qualified personnel of PEORC. The electrical installation took place at the same time as the other erection and assembly work between June 4 and 25, 1992.

The feeder cables of the machines and apparatuses have been laid in cable troughs and protective tubes according to the project.

While carrying out this work the transformer station was switched over from delta connection to star connection and (PEORC's scope of delivery and services) the main energy distribution connected in the machine room. The electrical specialists also adjusted the measuring and control instruments of the vacuum falling-film evaporators and the vacuum rectification.

The tasks have been fulfilled.

Mechanical functional tests

The contract between ACD and the UNIDO was to be considered fulfilled when all systems of the pilot plants have been proved to be serviceable.

The systems were put into operation as soon as these were ready and their serviceability tested.

The mechanical functional tests were carried out between June 19 and 25, 1992.

The successful completion of the mechanical tests has been confirmed by the "Protocol about finishing of assembling and mechanical function certification of the bench scale equipment" which you can find enclosed to this report.

-Cold water recooler, item 1N1

After the pipes of the cold water system had been laid the pumps 1P1.1 and 1P1.2 of the external circuits were put into operation for checking the tightness of the system at maximum operating pressure (the valves of the reflux lines were closed).

This pressure test was carried out with cooling water at a temperature of 20°C. After this test the refrigerating unit was connected and the cooling water chilled to 1 oC. After having finished the insulation of the entire refrigeration cycle,the system was completely emptied and then filled with the refrigerant, a mixture of 40 % ethylene glycol and 60 % water. Then, after the
relevant electrical work had been carried out, the external circulation and the refrigerating unit were again started, the refrigerant cooled to a set value of -15 °C and the refrigeration cycle operated at this temperature.

The mechanical functioning of the plants was proved.

- Steam generator, item 2N2

Prior to starting the steam generator repairs were necessary.

The transport and the long storage time of about 11 months caused damage of the electrical system and the water pump which, however, could be repaired at the site. Then the testing began. Drinking water was used for steam generation.

There was a normal operation of the automatic water supply to the steam generator, the automatic connection and disconnection of the relevant heating steps and the holding of the steam pressure at the given value of the steam generator.

The shut-off valve of the elutriation line of the steam generator appeared to have an increased leakage so that a ball valve was planned to be additionally installed. Up until the departure of the author of this report the ball valve had not yet been installed. (At that time the necessary Cr-Ni steel welding electrodes of the buyer were not yet available.)

The testing of the steam and condensate system was successfully completed after a several-hours-operation.

- Vacuum pumps 1P2, 2P1 and the vacuum systems of the falling-film evaporators and the rectification

Finally the vacuum pumps were installed and connected to the electrical and piping network.

After pipe laying and initial filling of the vacuum pumps with vacuum oil (supplied by PEORC) the vacuum pump 1P2 was started, and, after successful operation, the vacuum system of the falling-film evaporation evacuated. The necessary vacuum of 13.5 kPa was reached after approx. 4 minutes. A supply of external air at the pump suction nozzle allowed to keep the vacuum stable with the pump running. A mechanical fault of the magnetic valve upstream from the vacuum pump 1P2 destroyed the coil (see Annex 4, item 3).

After the coil had been repaired the system was again ready for operation.

Following the start-up of the vacuum pump 2P1 the vacuum of the rectification was generated. Initial leakages of flanged connections in the system were eliminated by a tightening of the bolts.

The electronic vacuum measuring devices of the falling-film evaporation and the rectification were adjusted during the test run.

The testing of the vacuum pumps and the vacuum systems of the falling-film evaporation and the rectification could be successfully completed.

- Warm and cooling water systems

The warm-water connection lines (PEORC's scope of delivery) to the falling-film evaporation and cooling water lines to the rectification were laid parallel to the other assembly work.

After completion the systems were operated under maximum operating conditions.

The tightness and serviceability of the systems were proved.

- Electrical installation

The distribution board for electrical energy and the other electrical equipment
operated during the test run of the machines and apparatuses. This electrical system ran according to the project and ensured the smooth operation of the electrical drives and control systems.

C. Thermical testings and first operation of bench scale equipment with original products

Thermical testing of discontinuous vacuum rectification and steam generation

This testing was carried out on June 27 in accordance with the testing programme, point 1.3, contained in annex 7.
The steam generator worked with full power 30 kW.

A water quantity of 28 l/h was evaporated in the column still and condensed in the top condenser under normal pressure. The maximum steam productivity of the steam generator is 35 kg/h. So 80 % of heating energy can be used for the distillation process. This is a sufficient high efficiency for such systems and demonstrates the good insulation quality of it.

First operation of discontinuous vacuum rectification with essential oil

The first operation of a.m. system was carried out on June 28 with 50 litres Thuja oil (Thuja orientalis L.) in accordance with the operating instruction, variant 2, contained in annex 8.

The aim of the first operation was to demonstrate the principal function ability of the unit with essential oil and to purify the oil.

Stable working conditions were attained after about 2 hours from the beginning of the heating process.

Befor stabilisation of the process the vacuum pump 2P1 was several times automatically switched off due to too high current reception of it drive motor. The lubrication oil was discoloured to dark brown. After changing of the oil the pump worked normally.

After stabilisation of the process following parameter values were observed for example at 14.30:

- Temperature head, TI 2201: 74 °C
- Temperature still, TI 2200 110 °C
- Absolute pressure head, PI 2501: 5,0 mbar
- Absolute pressure before vacuum pump, PI 2504: 6,0 mbar
- Pressure drop, PDI 2500: 39,0 - 39,5 mbar
- Reflux ratio, KC 2800: 0,5 (return) to 1,0 (destillat)
- Steam pressure, PI 2530: 4,5 bar overpress.

- Temperature of cooling water
  inlet, PI 2221: 24 °C
  outlet,PI 2220: 27 °C

- Power of steam generator: 18 kW
- Cold water temperature, inlet: -16,3 °C
- Distilled off quantity of essential oil compounents under a.m. conditions: 2 l per 5 minutes equal 24 l/h

After finishing of the distillation process following quantities of substances were obtained:

- water: 0,5 l
- residue: about 3 l
- cleaned essential oil: about 46,5 l

The cleaning of the system was perfomed by directly fed steam into the distillation still in accordance with the operating instruction in annex 8.

The results of the first operation of the compact unit vacuum rectification together with auxiliary systems can be commended as follows:

- All set values of technological parameters in the system necessary for its normal function such as absolute pressure, pressure drop, head and still temperatures, reflux ratio were obtained and kept stable or changed at the required level.
- The distillation productivity of the unit is sufficient high in order to distill a normal batch of 100 l essential oil in a justifiable time (8 - 10 hours).
- The heating system of the column by steam produced in a separate steam generator functioned without any problems. After setting of a defined power value at the steam generator in accordance with the pressure drop in the column the boiling temperature of oil in the still and the steam pressure in the still jacket are slowly increasing white distillation process without any additional control actions.
- The vacuum pump is a sensitive place of the unit. This demands a careful operating in the first distillation stage especially when the system is conveyed to stable conditions (increasing of vacuum while boiling of oil in the still) and a continuous feeding of fresh lubrication oil or regular changing of the lubrication oil.

First operation of vacuum falling-film evaporation

The first operation of a.m. system was carried out on June 26 with 100 l n-hexane in accordance with the operating instruction contained in annex 9.

The aim of the first operation was to demonstrate the principal function ability of the unit. The unit has been tested without the adsorption stage before the vacuum pump 1P2 because it was not ready yet (scope of PEORC).

Hot water from the central heating system of the town (temperature 60 - 70 °C) has been used for heating of the evaporator.

The unit worked about 2 hours at the pressure (PI 1501) 133 mbar (100 mm Hg) and approximately 70 - 80 l n-hexane was evaporated.

The results of the first operation of the unit can be commended as follows:

- The principal function ability of the unit was demonstrated.
- All set values of technological parameters in the system such as absolute pressure, cold water temperature (-15 °C) for condensation of n-hexane vapors, flow rate at the inlet of evaporator were obtained and kept stable at the required level.
- The sight glasses on the top and the outlet of the evaporator allow very good observe and control its normal function.
- There were not any problems with the vacuum pump 1P2. N-hexane vapors were not solved in observable quantity in the lubrication oil because a lot of air was sucked into the pump to get the relativly high pressure 133 mbar (100 mm Hg).

D. Training of PEORC staff in operating of equipment

The training of PEORC staff by the consultants in operating of equipment was carried out while the assembling, testing and first operation of units.

Training in operating of auxiliary equipment

The consultants visited in Germany before the beginning of their mission the factory for electric steam generators "Dampfautomat Leipzig" in the town Leipzig and a factory in the town Halle which is using a complete refrigarating unit type Etscheid. They got there the necessary practical know-ledge and experience in operating and nursing of this aggregates.

The representatives of the manufacturers handed over some additional spare parts which were taken to Pyongyang. Detailed operating instructions were compiled and prepared in Pyongyang for all 3 types of auxiliary equipment and given with additional drawings (Prinzipal flow diagrams for steam generator and refrigerating unit, see annex 10) to PEORC staff. The staff was trained by theoretical explanations and practical handling at every item of equipment on the base of handed over instructions.

Training in operating of main units

The PEORC staff knows very well the technology and the design of the main unit No. 1 vacuum falling-film evaporator. A similiar normal pressure falling-film evaporator is used for removing of solvent n-hexanes from extracts of Rosa rugosa oil on the fields of PEORC at the east cost of Korea (see reports of Mr. Thomann and Mr. Langner from 1991). Therefore the training was focused on the new aspects of evaporation that is working under vacuum and low temperature for condensation and cooling. A short operating instruction for main unit No. 1 was elaborated at home and will be handed over with this report (see annex 9).

The technology and design of the main unit No. 2 batch vacuum rectification is new for PEORC staff.

The chemical process engineer Mr. Langner elaborated under using of the "UNIDO practical manual on the essential oil industry", No. 2, fractionation and production of isolates a detailed temporary operation instruction for this unit and handed over it to the PEORC staff. The final version of this instruction is included in this report (see annex 8).

The PEORC staff was intensivly trained in practical handling of the unit on June 26 and 27, 1992.

III. FINDINGS AND RECOMMENDATIONS

1. When exporting to developing countries the plants should be prefabricated as far as possible, and cutting and welding and similar work should be reduced to a minimum.

   This particularly applies to plants where special requirements are to be met concerning assembly. These are, for example, Cr-Ni steel plants and plants subject to thermal stress as well as plants operating under pressure where special welding techniques and test methods are needed.

2. For the seller and buyer it would be helpful, if prior to the installation of a plant, a competent representative of the seller who is well acquainted with the detailed project, would go to the site (the buyer's country) about two months before commencing this work in order to discuss open questions of the buyer regarding the detailed project and take the necessary building measures at the prospective site. It is necessary to get a survey on the material and technical potential of the buyer in order to be able to optimally prepare for the special conditions and inform the buyer, if he is going to carry out the assembly, what kind of devices, machinery and other aids will be required.

3. PEORC is recommended to develop for every kind of essential oil a special destillation technology with concrete values for main process parameters.

   The available in PEORC gas chramotograph should be used for this in close co-operation between technologists of distill-ation and analytists.

4. PEORC is recommended to pay a special attention to further qualification of it's members in operation of the delivered not simple units and to the observing safety of rules mentioned in the project documentation.

Annex 1

T.De Silva/jbg
29 October 1991

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

JOB DESCRIPTION
DP/DRK/88/001/11-53

Post Title: Supervision expert / Engineer

Duration: 1.5 m/m

Date Required: January 1992

Duty Station: Pyongyang

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

Duties: The expert will work in collaboration with the National Project Director and will have particular responsibility for the supervision and direction of the staff from the implementing agency for the erection and commissioning of pilot scale equipment delivered from FR Germany

The expert will prepare a full report on his work and recommendations.

Qualifications: A plant engineer with at least 10 years experience for supervision, erection and construction of plant projects.

Language: English/Russian

Background Information: As part of a new initiative, the Government of DPR Korea has recently established the Pyongyang Essential Oil Research Centre (P.E.O.R.C) to act as a central body to supervise the production of essential oils and related products, principally from rose, lilac and lily of the valley, to serve a growing domestic market for scented toiletries as well as export. The Government is receiving assistance from UNDP through UNIDO project designed to facilitate the development of P.E.O.R.C. The project will enhance rural development and provide raw materials for local industry (including that with high added value such as cosmetics) in a hitherto somewhat neglected sector of the economy.

Ultimately, by producing material for export, will contribute to foreign exchange earnings.

Applications and communications regarding this Job Description should be sent to:

Project Personnel Recruitment Section, Industrial Operations Division
UNIDO, VIENNA INTERNATIONAL CENTRE, P.O. Box 300, Vienna, Austria

atj

T.De Silva/jbg
24 February 1992

NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

JOB DESCRIPTION
DP/DRK/88/001/11-01

Post Title: Chemical Technologist

Duration: 1.5 m/m

Date Required: As soon as possible

Duty Station: Pyongyang

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

Duties: The expert will work under the supervision of the National Project Coordinator and will have particular responsibility for assisting in the installation and commissioning of distillation and extraction equipment, training local staff in their initial use and development of processing parameters using them.

..../..

Qualifications **A chemical technologist with a thorough grasp of the up-to-date technology of distillation and extraction of essential oils, including field distillation.**

Language English

Background information As part of a new initiative, the Government of DPR Korea has recently established the Pyongyang Essential Oil Research Centre (P.E.O.R.C) to act as a central body to supervise the production of essential oils and related products, principally from rose, lilac and lily of the valley, to serve a growing domestic market for scented toiletries as well as export. The Government is receiving assistance from UNDP through UNIDO project designed to facilitate the development of P.E.O.R.C. The project will enhance rural development and provide raw materials for local industry (including that with high added value such as comestics) in a hitherto somewhat neglected sector of the economy.
Ultimately, by producing material for export, will contribute to foreign exchange earnings.

Annex 2

List of partners

Mr. Karcher	-	Representative of UNDP in Pyongyang
Mr. Li Song Ho	-	National project officer in UNDP-office Pyongyang
Mr. Han	-	Director General in the General bureau for cooperation with international organizations (GBCIO)
Mr. Ko Ju Chol	-	leading member in GBCIO
Mr. Choi Dung Gwang	-	Director of the PEORC, National Project Director
Mr. Li Myong Ho	-	Deputy director of PEORC
Mr. Li Chan Il	-	interpreter
Mr. Boehme	-	UNIDO-expert, supervisor
Mr. Langner	-	UNIDO-expert, consultant

Several members of PEORC

Annex 3

Time schedule of activities

24/05 - 26/05/1992:
flight Leipzig - Frankfurt/Main - Beijing (staying one night) - Pyongyang

27/05 - 29/05/1992:
working in Pyongyang

30/05 - 01/06/1992:
flight Pyongyang - Beijing - Zuerich (staying one night) - Leipzig

Planned:
08/09 - 11/09/1992:
Debriefing Vienna including travel time for Mr. Langner

Annex 4

DPR of Korea, Pyonyang.
26. 06. 1992.

Protocol about finishing of assembling and mechanical function certification of the bench scale equipment.

1. Subject.

- Project: DP/DRK/88/001, Essential Oils.
- UNIDO purchase number: 15-0-0344M
- Establishment of a pilot plant for essential oils.

2. Participants.

Mr. Choi - National project director, Director of PEORC ; DPRK.
Mr. Li - Deputy director of PEORC, DPRK.
Mr. Boehme - UNIDO-expert, supervisor, representative of contractor acd.
Mr. Langner - UNIDO-expert, Consultant.
Several members of PEORC.

3. Performance and results.

After finishing of assembling following item of equipment were tested from 19/06/92 to 25/06/92;
- Cold water recooler, pos. 1N1.
- Steam generator, pos. 2N1.
- Distribution board for electrical energy, pos. V1.
- Vacuum pumps,pos. 1P1, 2P1.
- Compact units No1 (Vacuum film evaprator) and No2 (Batch vacuum rectification) for vacuum and vacuum measurement.
- Pipes for cold water and steam for tightness.

All a.m. equipment was tested with positive result with one exception of magnetic valve for the vacuum pump 1P1. The spool was destroyed due to the mechanical defect in the valve. The contractor acd declare his readyness to deliver a new spool if repairing onsite no possible.
The other systems (steam,warm and cooling water) have worked also without problems.
With the hereby certified mechanical function of the equipment the scope of services of the contractor in accordance with clause 2,07d has to be considered as finished and equipment warranty according clause 2.08 of the contract enters into effect.

Contractor
ACD Dresden
Supervisor Boehme

Implementing agency
PEORC Pyongyang
Director Choi

Executing agency
UNIDO Vienna
Consultant Langner.

Photos

Annex 5

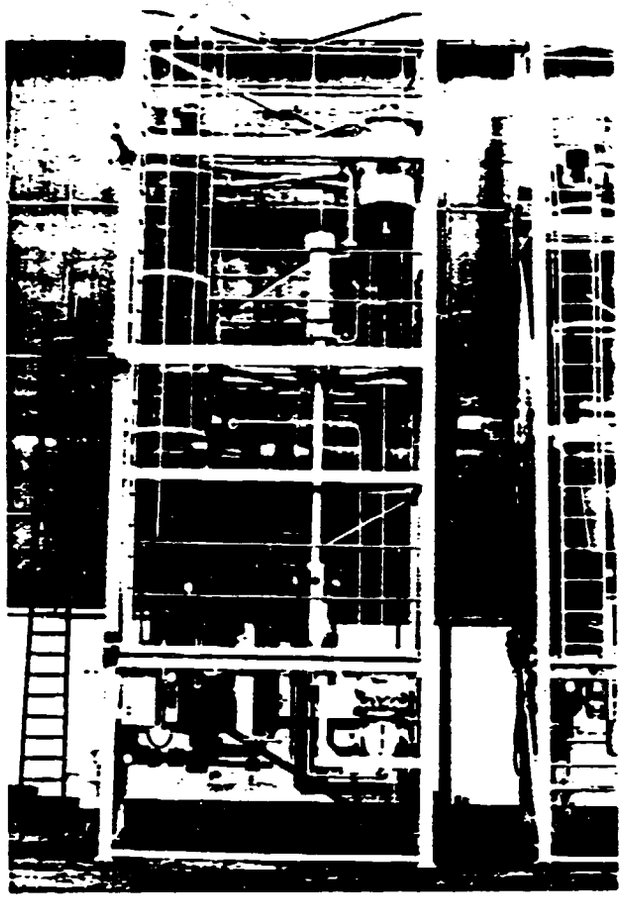


Fig. 1

Compact unit No. 1 while assembling in Germany

Fig. 2 General view of equipment hall and machine room in PEORC/Pyongyang

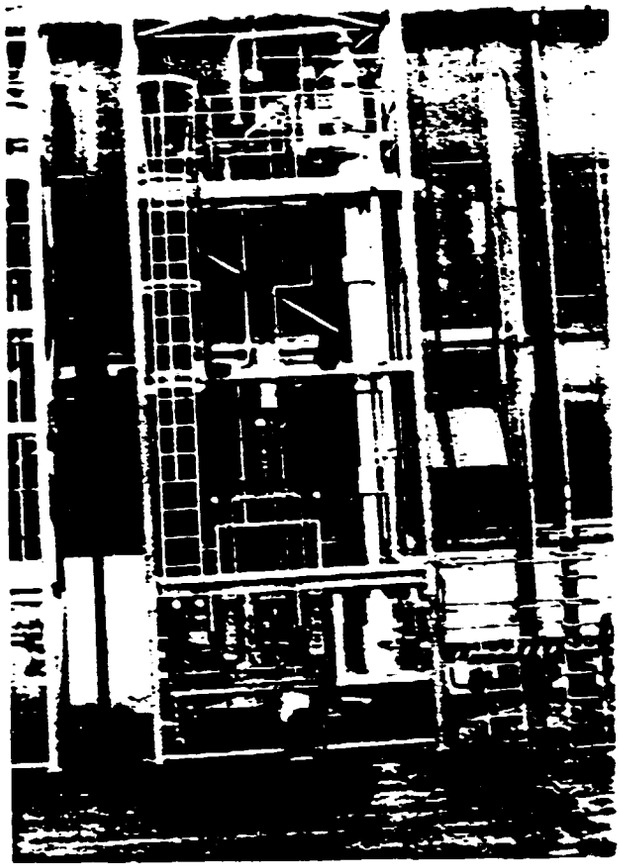


Fig. 3

Compact unit No. 2 while assembling in Germany

Fig. 4 Machine room in the beginning

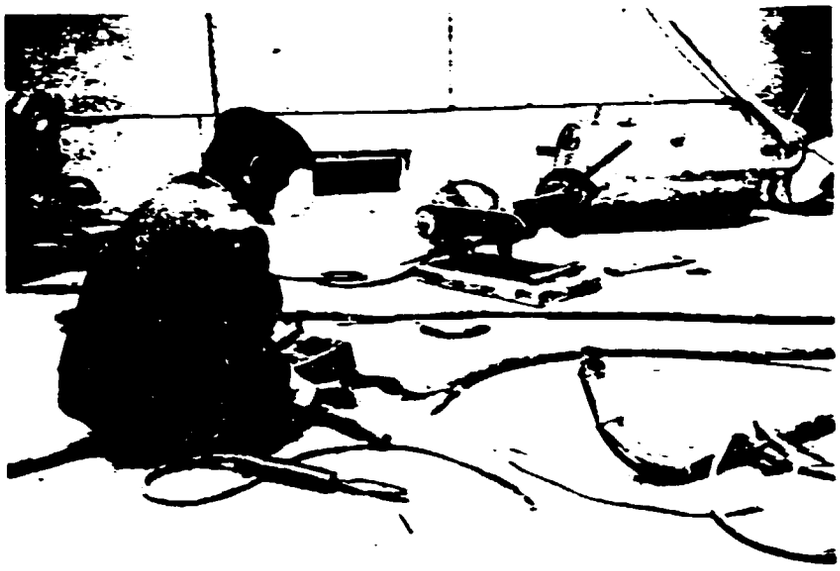


Fig. 5

Welding of pipes

Fig. 6

Cold insulating of pipes

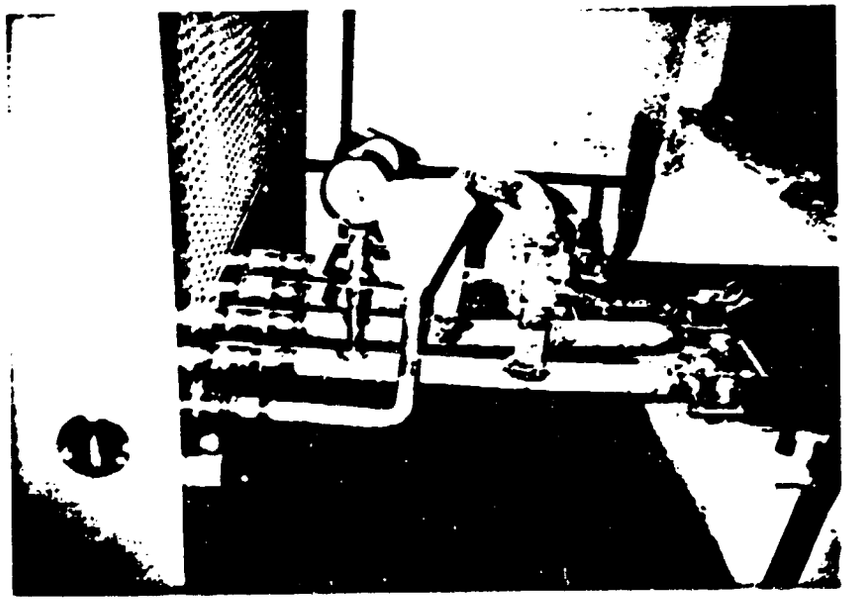
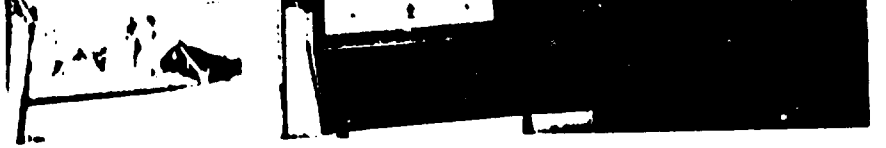


Fig. 7

Pipe connections to the refrige-ration unit 1N1 in machine room



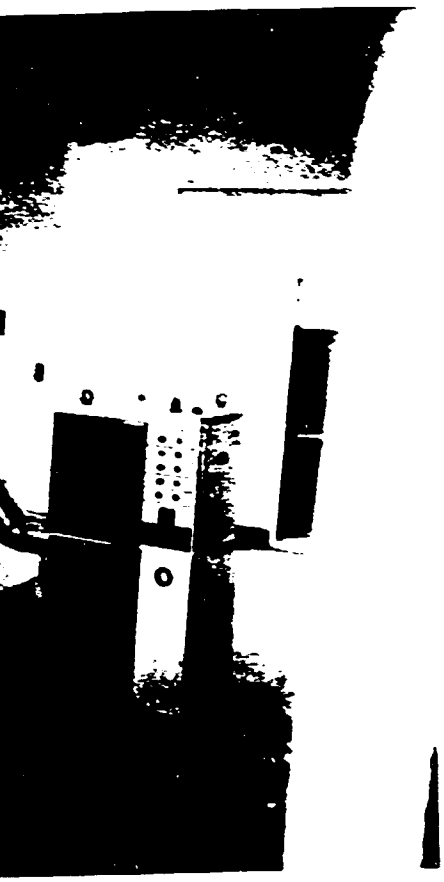


Fig. 8

Machine room
with steam
generator 2N1

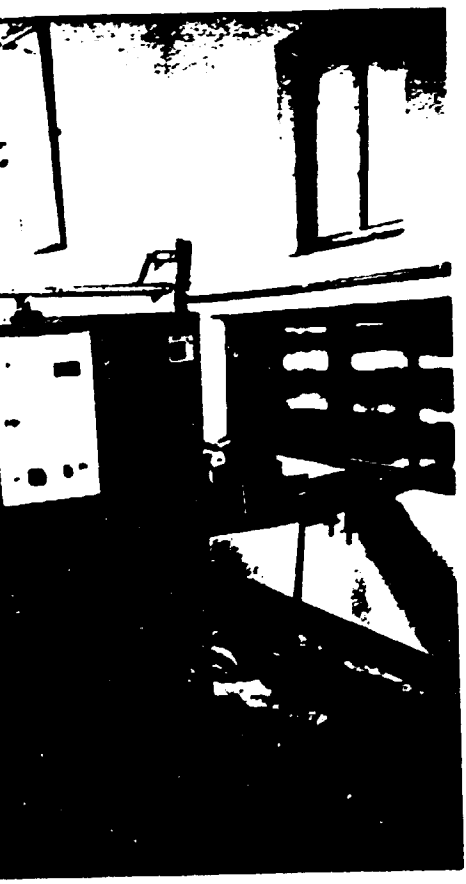


Fig. 9

Machine room
with refrigeration
unit 1N1

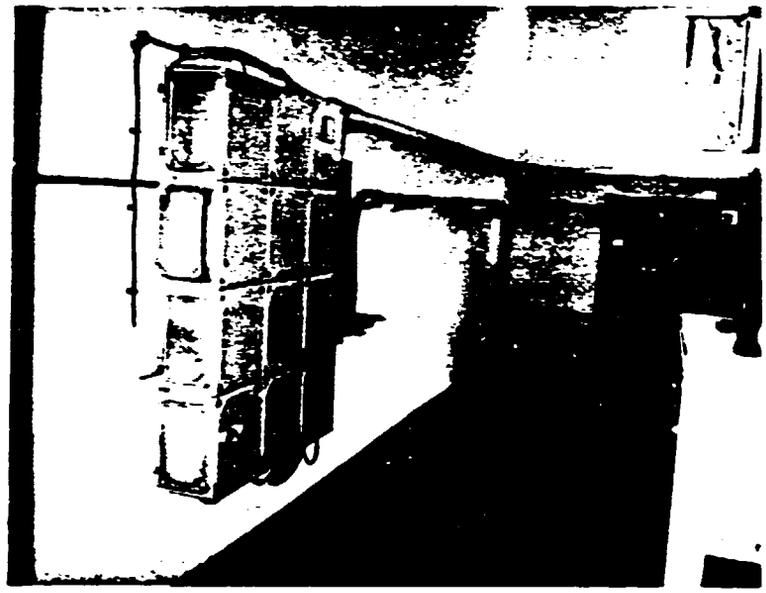


Fig. 10 Machine room with electrical distribution board V1, refrigerating unit and a part of steam generator

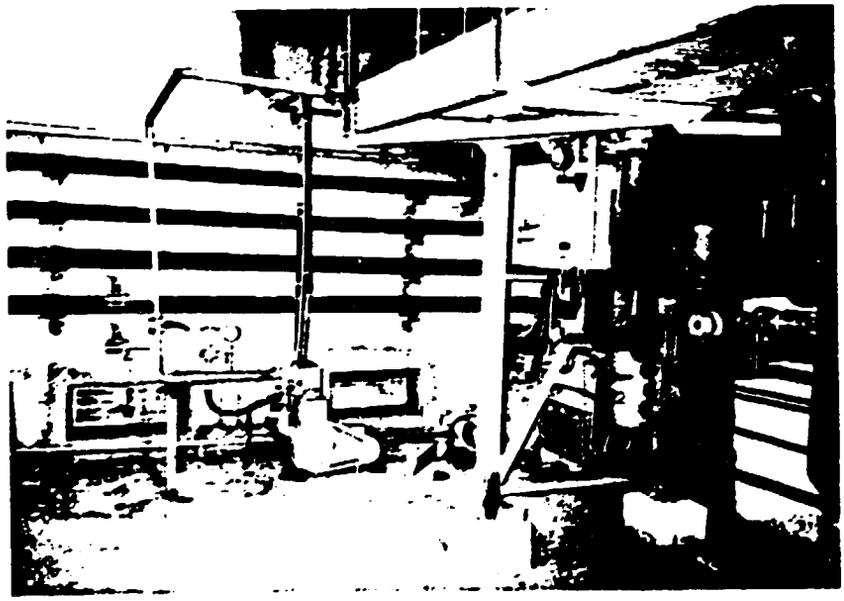


Fig. 11 Equipment hall, lower part of compact unit No. 1 and vacuum pump 1P2

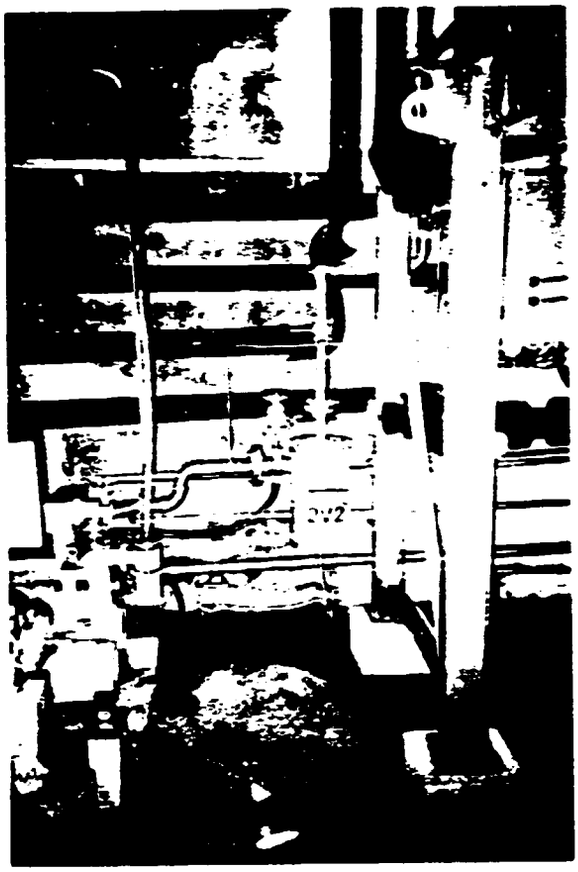


Fig. 12

Equipment hall, vacuum pump 2P1 and separator 2V2

Fig. 13 Lower part of compact unit No. 2 with distillation still, equipment hall

Fig. 14

First start of vacuum pump, equipment hall

Fig. 15

First steam, equipment hall

Fig. 16 First evaporated and condensated n-hexane, filling station

Fig. 17 Discharging of residue after first distillation of essential oil, equipment hall

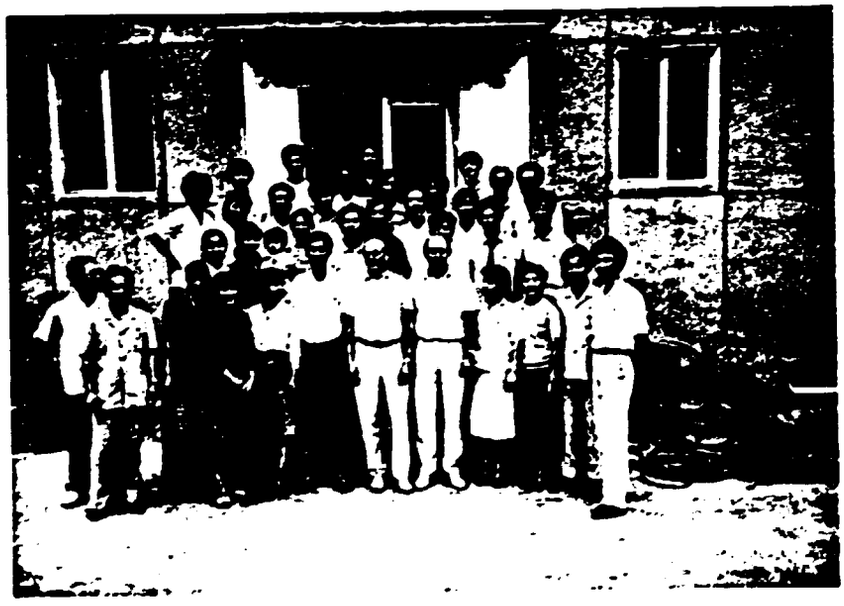


Fig. 18 The team of participants in assembling and commissioning of bench scale equipment

Annex 6

Drawings

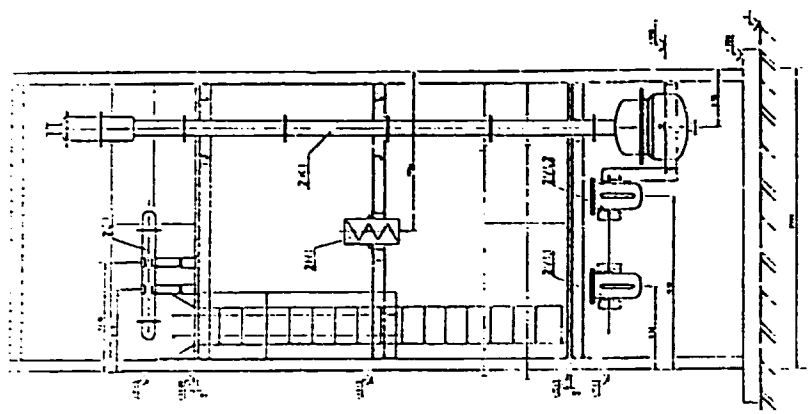
Following drawings are enclosed in reduced scale:

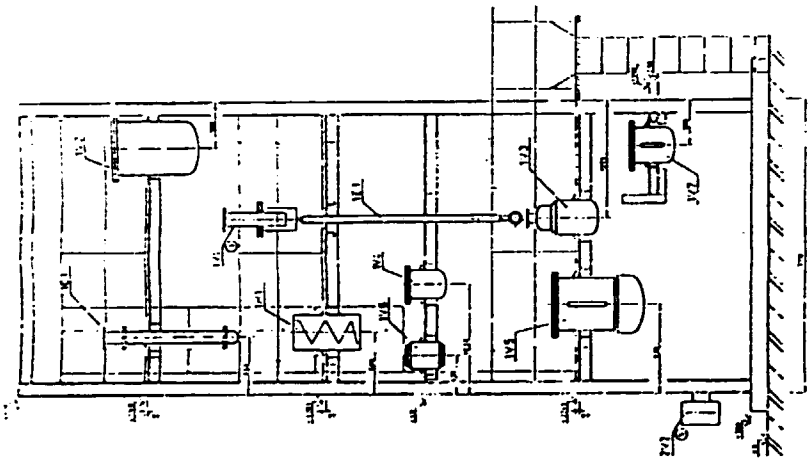
- PID, Vacuum falling-film evaporation,
  drawing-No.: 51/02724 a (0)
  from 27/09/1991

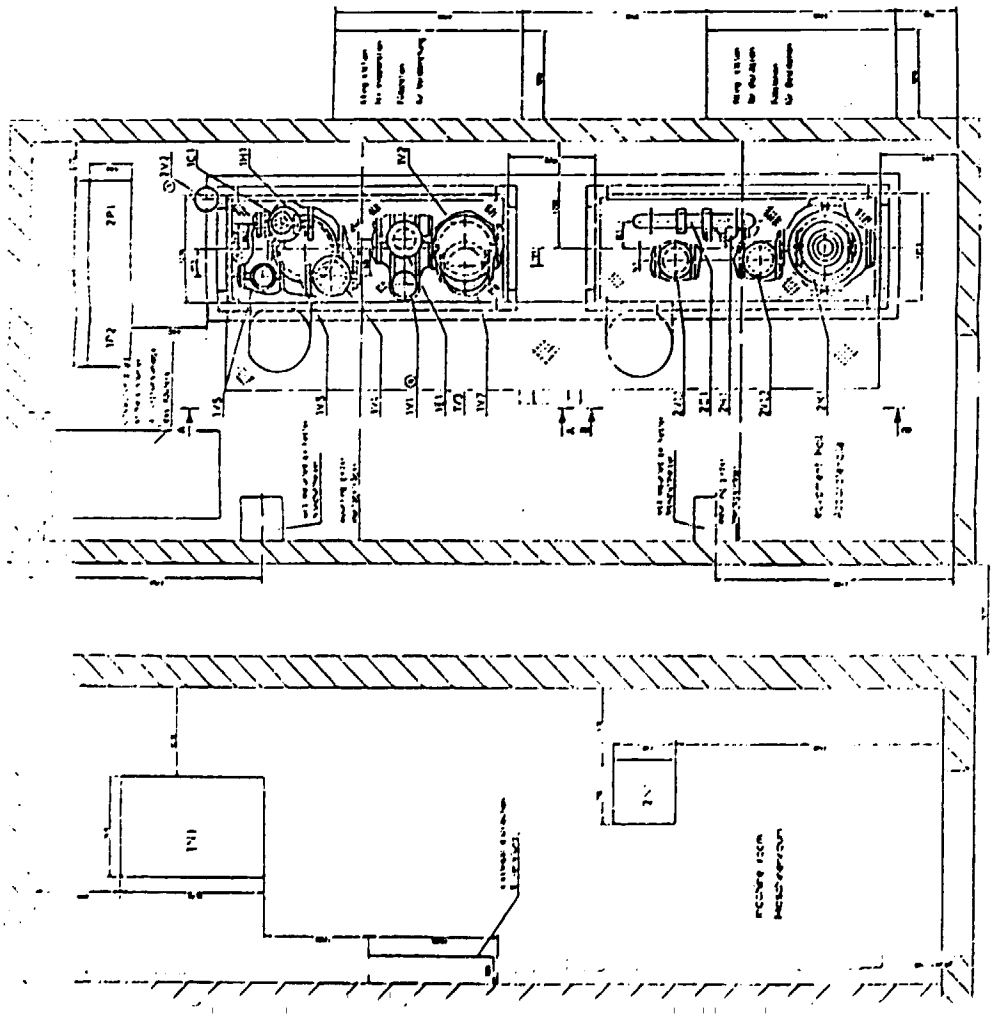
- PID, Discontinuous vacuum rectification,
  drawing-No.: 51/02723 a (0)
  from 27/09/1991

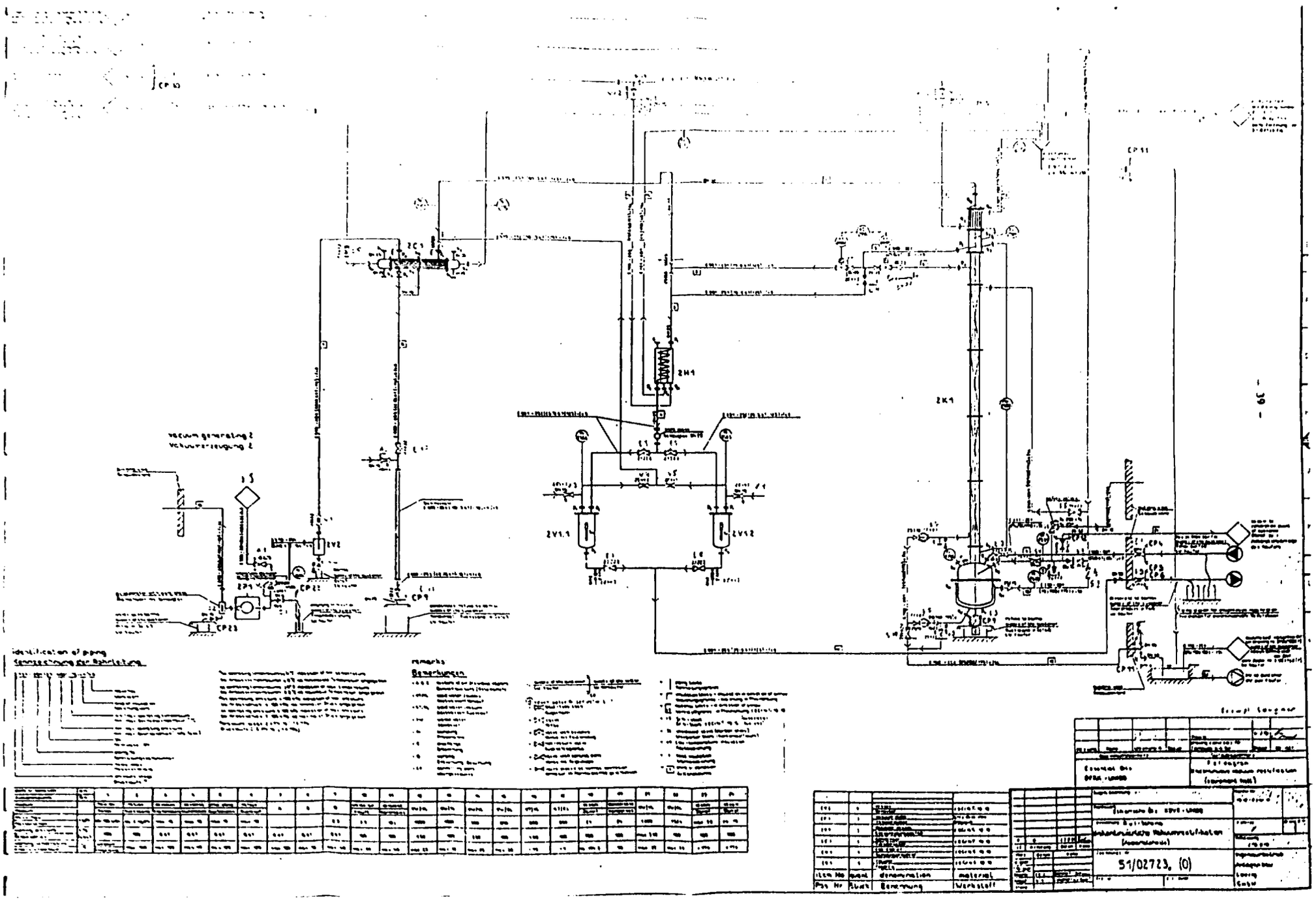
- PID, steam generation and refrigeration
  (machine room),
  drawing-No.: 51/02725 a (0)
  from 27/09/1991

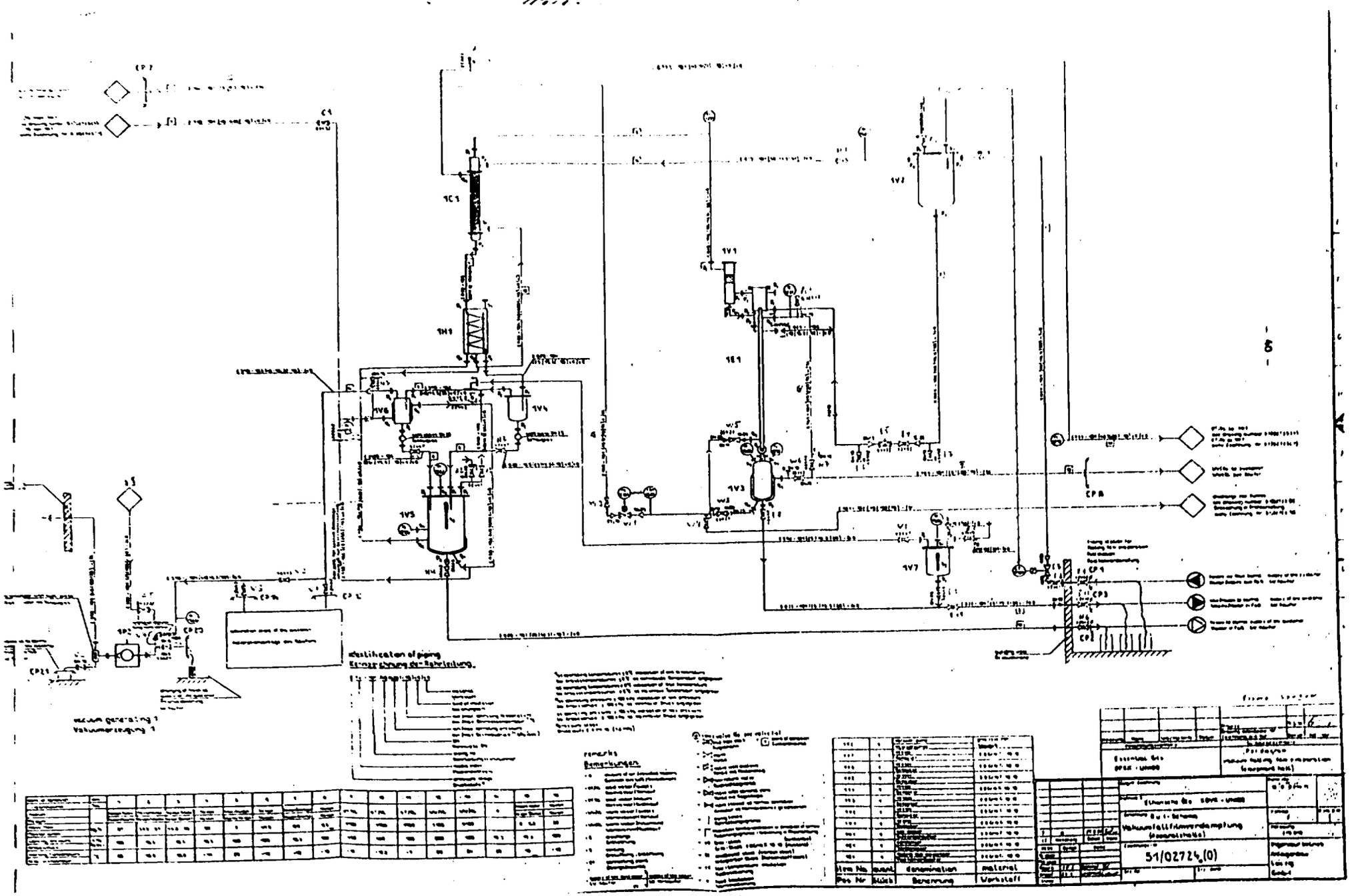
- Installation plan,
  drawing-No.: 51/02493 a, b (0)
  from 27/09/1991







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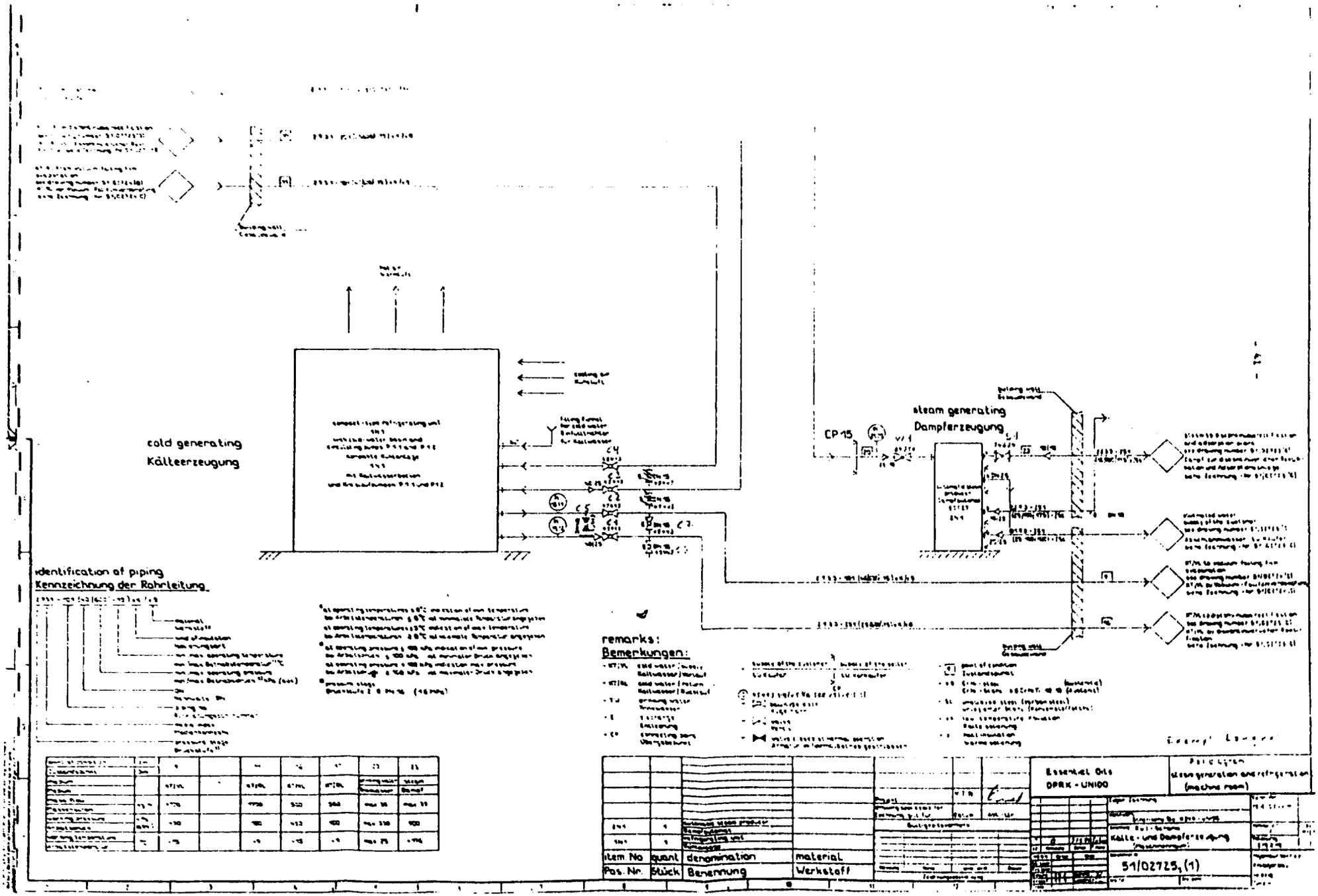

cold generating
Kälteerzeugung
steam generating
Dampferzeugung
CP 15
identification of piping
Kennzeichnung der Rohrleitung
remarks:
Bemerkungen:
Essential Oils
DPRK - UNIDO
steam generation and refrigeration
(machine room)
Kälte- und Dampferzeugung
51/02725,(1)
item No
Pos. Nr.
quant
Stück
denomination
Benennung
material
Werkstoff

Programme for mechanical and thermical testings of equipment

See also at PID in annex 6.

1 Testing of heating system for compact unit No. 2 with steam generator, pos. 2N1

1.1 Steam generator, pos. 2N1

- Checking of all installations (pipes, electrical system) according to right performance/excecuting.
- Put into operation the steam generator acc. to operating instructions of the supplier "Dampfautomat Leipzig". Steam valve DV is closed.
- Steam generator works without delivery of steam to the consumer for 30 minutes.
- Checking of all components of steam generator acc. normal function.

1.2 Steam generator 2N1 together with column 2K1

- Steam generator is working acc. point 1.1.
- Give slowly steam to the jacket of distillation still. Pressure of steam at PI 2530 must be $\leq$ 1 at.
- After stabilisation of steam pressure PI 2530 on 1 at increase slowly step by step the pressure to maximum 10 at.
- Work 1 hour at this pressure.
- Open slowly the steam valve for heating of the coil at the column 2K1.
- Work 1 hour with parallel heating of distillation still and coil on the column at the pressure 10 at.
- Check the system for normal function.

1.3 Steam generator 2N1 and column 2K1 with evaporation of water in the distillation still

- Check all systems of the column, especially KC 2800 (reflux devider), PDI 2500 (pressure drop).
- Give cooling water to top condenser, 2H1.

- Start the vacuum pump 2P1, set a small vacuum, suck into the distillation still 100 l pure water.
- Close the vacuum valve before 2P1, connect the system with the atmosphere.
- Beginn slowly to heat the still with steam, reflux devider KC 2800 is switched for full return to the column.
- After stabilisation of process work 2 hours, check the system and note the values of all parameters (PI 2530, TI 220, PDI 2500, TI 2201, TI 2220).
- Switch reflux devider KC 2800 to full discharging of condensate, collect 90 l water condensate.
- Switch off the steam to the distillation still, after cooling for same hours discharge the waste water from the still.

2 Testing of vacuum pumps 1P2, 2P1

2.1 Testing of pumps without system

- The valves to the systems are closed. The pumps are ready for working (filled with oil etc.). The pressure measurements PI 1503, PI 2504 are switched on. The valves for air sucking into the pumps are closed.
- Switch on the pumps.[1)] Record the pressure values on PI 1503, PI 2504.
- Open slowly the valves for air sucking into the pumps. Set the working values PI 1503 (100 mm Hg) and PI 2504 (5 mm Hg) of pressure.
- Switch off the pumps, test the automatical valves in the gas outlets of pumps.

2.2 Testing of pumps with systems

- The valves for air sucking into the pumps are closed. The systems of compact units No. 1 and 2 are closed.
- Open the valves between pumps and system. Switch on the pumps. Record every 5 minutes the values of PI 1501, PI 1502, PI 1503, PI 2501; PI 2502, PI 2503, P 2504.
- Close the valves between pumps and the systems. Switch off the pumps. Record every 1 hour the values of pressure above mentioned.

1) acc. to the operating instructions of manufacturer

3 Testing of cold water recooler 1N1

3.1 Cold water recooler 1N1 without systems

- Open the recirculation valves from the pumps P 1.1, P 1.2 back to the water tank.
- Close the valves C1 and C2 between recooler and systems.
- Fill in the mixture ethylenglykol/water 40:60 % into the cold water tank.
- Start the recooler[1]; check that the temperature of cold water is set at -15 °C; after achieving of temperature -10 °C check all components for right function.

3.2 Cold water recooler 1N1 with systems

- The recooler is working accordingly to point 3.1.
- Open slowly the valves C1, C2 and close in the same manner the recirculation valves from the pumps P 1.1, P 1.2 back to the water tank, fill the systems with cold water. Let out the air from closed spaces in the systems (1H1, 1C1, 2C1). Check all systems for tightness.
- While circulation of cold water between recooler and systems the temperatur is decreasing to -15 °C. Work some hours under these conditions, check the cold water circuit for cold places (forming of ice). If such places are available remove them by improvement of insulation.

1) in accordance with manufactures operating instructions

Operating instruction for batch vacuum rectification

See annex 6, piping and instrumentation diagramm, drawings-No. 51/02723 a (0).

I. Checking

1. Check if any residue, water, organic liquids etc. are in the rectification unit. Remove if any.
2. Check all joints, connections (flanges), screws if tight; valves, if no blocked.
3. Check all water connections for leaks.
4. Check all electrical and control connections.
5. Check vacuum:
   - Switch on mains cable at the distribution board V1.
   - Connect vacuum control system.
   - Switch on the vacuum pump 2P1; valve A1 for air sucking in is closed.
   - Observe vacuum true value with unit empty, clean and dry at PI 2501, PI 2502, PI 2503, PI 2504 after 5 min. according to pump capacity.
   - Pressure should drop to a minimum of ≤ 3 mm Hg (≈ 4 mbar). If higher, check all connections (under vacuum) for possible leaks and tighten if necessary.
   - If the rectification is working normally, switch off the vacuum pump and slowly open aeration valve A3 or A4 over the vessels 2V 1.1 or 2V 1.2 until the pressure stabiliser to around 760 mm Hg. Now the unit is ready for work.

II. Working with essential oils

1. Follow checking and cleaning instructions until the unit is ready for work. All the elements of the unit switched off and the heating system is cold.
2. Close all inlets and outlets of the unit. Open the valve A1 for air sucking into the vacuum pump 2P1. Start the vacuum pump and set by valve A1 pressure PI 2502, PI 2503, at -0,3 bar.
3. Charge the essential oil (100 l) from the barrel at filling station trough the pipe No. 2800-201 by opening of valves E2, E1.
4. After charging switch off the vacuum pump, air the unit, close the valves.

5. Set the unit for distillation:

   - Turn on cold and cooling water to 2C1 and 2H1, check the flow rate of cooling water in the funnel.
   - Switch on the cooling unit 1N1 and set the temperature at -15 °C.
   - Connect unit with vacuum pump and open the air sucking in valve A1.
   - Set reflux K 2800 to full return to the column.

Now the unit is ready for distillation. The **first stage of distillation** can be carried out in 2 variants.

a) Variant 1

6a) Switch on the vacuum pump, slowly decrease the pressure and observe the pressure drop in the column PDI 2500 as well as the vacuum true value indicators PI 2502, PI 2503.
If boiling starts there will be a pressure drop in the column (PDI 2500).

7a) If no boiling starts at -0,87 bar (100 mm Hg) set the pressure -0,935 bar (50 mm Hg).

8a) If boiling starts at -0,87 bar decrease the vacuum step wise to -0,935 bar (50 mm Hg) and stay at this point until the boiling slows down or stops. Sometimes water appears at this moment or boiling continues at this pressure. In such cases open the reflux distill of the water and the lower boiling fraction. Usually at conditions described above, very low boiling products go trough the condenser and are trapped in cold trap 2C1 only.

9a) At -0,935 bar (50 mm Hg) start low rate heating in the evaporator (TI 2200 50-80 °C, steam generator 9 kW); valves S5, S3, S10 are closed; S2 is open; valve S9 is so throttled, that no steam only condensat can flow to the waste water pit.
If the boiling starts, it can be observed at the pressure drop measurement PDI 2500, vapour enters the column upto the condenser and comes back via the reflux KC 2800 (closed).

10a) After stabilisation of the temperature both in the evaporator TI 2200 and the column head TI 2201, set the reflux ration at 10:1 (1 part distillate, 10 parts return to the column) and start collection of the distillate.

11a) When boiling slows down (pressure drop PDI 2500 decreases) and/or head temperature decreases, set the vacuum step wise down (-0,950; -0,970; -0,990 bar; 8 mbar), controlling each time the unit performance as in point 9a, 10a above.

12a) At the lowest pressure PI 2501, PI 2504, when boiling slows down (look PDI 2500) increase heating rate (Steamgenerator 18 kW) and continue step wise using the same manner as with the vacuum decrease above. Than the second stage of distillation is reached (working under lowest pressure).

b) Variant 2

6b) Connect the system with the atmosphere (values A3, A4, E5, E6 open), switch on the steam generator 2N1 and give steam into the jacket of the distillation still (vaives S5, S3, S10 are closed, S2 open, valve S9 is so throttled that no steam only condesat can flow to the waste water pit).
Observe the temperature of oil in the still TI 2200 and the pressure drop PDI 2500 in the column. The steam pressure in the still jacket must be > 0.

7b) If boiling starts under this conditions (PDI 2500 shows > 0 mbar) stay at this point until the values of head temperature T 2201, pressure drop PDI 2500 and TI 2200 stabilised or the boiling slows down or stops.
In the first case open the reflux KC 2800 and distill off the water or/and the lower boiling fractions.

8b) Switch on the vacuum pump, slowly decrease the pressure PI 2502, PI 2503 and observe PDI 2500, TI 200, PI 2530. When the pressure in still jacket PI 2530 is near to 0 bar and/or the pressure drop PDI 2500 is to high (> 40 mbar) stop the pressure decreasing.

9b) After stabilisation of the temperature both in the evaporator TI 200 and the column head TI 2201, set the reflux ratio KC 2800 at 10:1 (1 part distillate, 10 parts return to the column).

10b) When boiling slows down (PDI 2500 decreases) and/or head temperature decreases, set the vacuum step wise down without decreasing of steam pressure in still jacket PI 2530 lower 0 bar, controlling each time the unit performance as in point 9b above.

11b) At the lowest pressure PI 2501, PI 2504, if boiling slows down increase heating rate (steam generator 18 kW) and continue step wise using the same manner as with the pressure decrease above. Than the second stage of distillation is reached (working under lowest pressure).

Second stage of distillation

13. When the fractionation is stable, the lowest possible vacuum fraction should be collected in the following manner:

    - When the temperatur in the column head TI 2201 is increasing (vacuum stable at set point PI 2501, PI 2504) the reflux ratio KC 2800 should be set at 20:1 to 10:1 (20 or 10 part return to column, 1 part discharged) and fractions equal to around 1 percent of the volume of the oil taken to fractionation should be collected (interfractions).

    - When the vacuum and the temperatur in the column head TI 2201 stabilises at a certain value and boiling in the evaporator is quit (PDI 2500), the reflux can be changed to 5:1 and 1:1 ratio and fraction could be collected until increase in temperature to 1 - 2 °C is observed (TI 2201).
      The collected fraction (isolated product) should be discharged from the vessels 2V 1.1 or 2V 1.2.
      The reflux ratio KC 2800 set back to 20:1 and the procedure continued.

14. Discharge of the fractions from 2V 1.1 or 2V 1.2 should be performed very carefully. It is important that disturbances in the vacuum should be minimal as any vacuum changing will affect the separation quality.

15. Fractionation is continued as long as required according to the oil contents and aim of process. After attaining of 180 °C in the still product (TI 2200) the distillation should be finished, the volume of residue should be 5 - 10 l.

16. Finishing of distillation shall be carried out as follows:

    - Switch off the heating and wait until boiling stops (PDI 2500) and the temperature in the head TI 2201 and evaporator TI 2200 decreases significantly (around 50 °C in the evaporator TI 2200).

    - Switch off the vacuum pump and cut off the pump unit connection (valve V1).

    - Very slowly allow air into the unit until vacuum true value stabilises at around 760 mm Hg, valves A3 or A4.

    - Discharge residue from the still when warm, valve E3 and from the tube under 2C1, valves A2, E10, E11.

    - Clean the column immediatly after this with steam (valves S3, S4) as follows:

      Pay attention (!):

      . Reflux works for full return to the column ($R = \infty$).
      . Top condenser is water cooled.
      . Valves A3, A4, V4, V5, E5, E6 must be open!!!

After 2 hours switch off the steam (close S3, S4), discharge the condensate from the column still, valve E3.

17. All controls should be switched off and the unit cut off from the power. The cooling system should be also switched off (unit 1N1 and cooling water).

18. During the whole process, each action is noted in the records as well as each change in parameters. If the process is running for a long time at the same parameter, notes should be taken every 30 minutes. Fraction should be numbered according to the records for easy identification and weighed. The basic parameters to be noted in the records are:

    - Time
    - Temperature in evaporator TI 2200
    - Temperature in column head TI 2201
    - True vacuum value PI 2501, PI 2504
    - Reflux ratio KC 2800
    - fraction collected number and weight and others

19. Every time while work pay a special attention to the rules of safety mentioned in the project documentation from 27th February 1991, chapters 13 and 14. Especially the general and specific requirements of fire and explosion protection in point 6 of chapter 14 have to be observed strictly.

    It is no permissible to work in the main units with substances which are more dangerous than n-hexane respectively essential oils for example with other solvents such as ethanol, methanol etc.

Operating instruction for vacuum falling-film evaporation

See annex 6, PID, drawing-No.: 51/02724a(0)

I. Checking

Carry out the checking of the unit in same manner as described in annex 8, operating instruction for vacuum rectification.
The required absolute pressure is 133,2 mbar (100 mm Hg).

II. Working with essential oil extract

1. Follow checking instructions until the unit is ready for work.
   All the elements of the unit switched off.
   All inlets and outlets of the unit are closed.

2. Put into operation the refrigerating unit 1N1 with cold water circuit. Open the valves C2, C4, C1.
   Set temperature at the unit and at
   TI1212: -16 °C

3. Switch on the hot water flow to the evaporator jacket 1E1.
   Open the valves W2, W1 (TV 12301, set temperature 70 °C), W5, W7. Valves W3, W6 are closed.

4. Start the vacuum pump 1P2.
   Set pressure by valve A5 at 133,2 mbar (100 mm Hg) PI1501.
   If working without adsorption plant valve V2 is open; V1, V3 are closed. Also open valves H3, H2, V2, H1, E4.

5. Now the unit is ready for receiving of extract from the barrel in the filling station outside the equipment hall via hose. Open the cocks E1, E2. The extract flows due to vacuum in the system into vessel 1V2. After getting of the normal liquid level in this vessel the automatic level control system LC 1700 with solenoid valve LV 17001 will keep the level constant.

6. Beginn to charge the extract (57 kg/h) on the top of the evaporator 1E1 by opening of valve with control cone E5. Check the flow rate of extract and the normal function of evaporator at the sight glasses on the top and at the outlet of evaporator.
   The n-hexane condensat flow can be observed at the sight glasses under the items 1V4 and 1V6.

7. When the vessel 1V5 is full with liquid n-hexane it has to be discharged to the filling station.
Close valves H2, H3; open A2, H4, H6. After discharging close H6, H4, A2; open slowly V2 and than H2, H3.

8. The contents of vessel 1V3 has to be discharged to the filling station via vessel 1V7 in the same manner as described in point 7 above.

9. During the whole process, each action is noted in the records as well as each change in parameters.
If the process is running stable at the same parameter, notes should be taken every 30 minutes. The discharged products should be numbered according to the records for easy indentification and the volume has to be defined.

The basic prarameters to be noted in the records are:

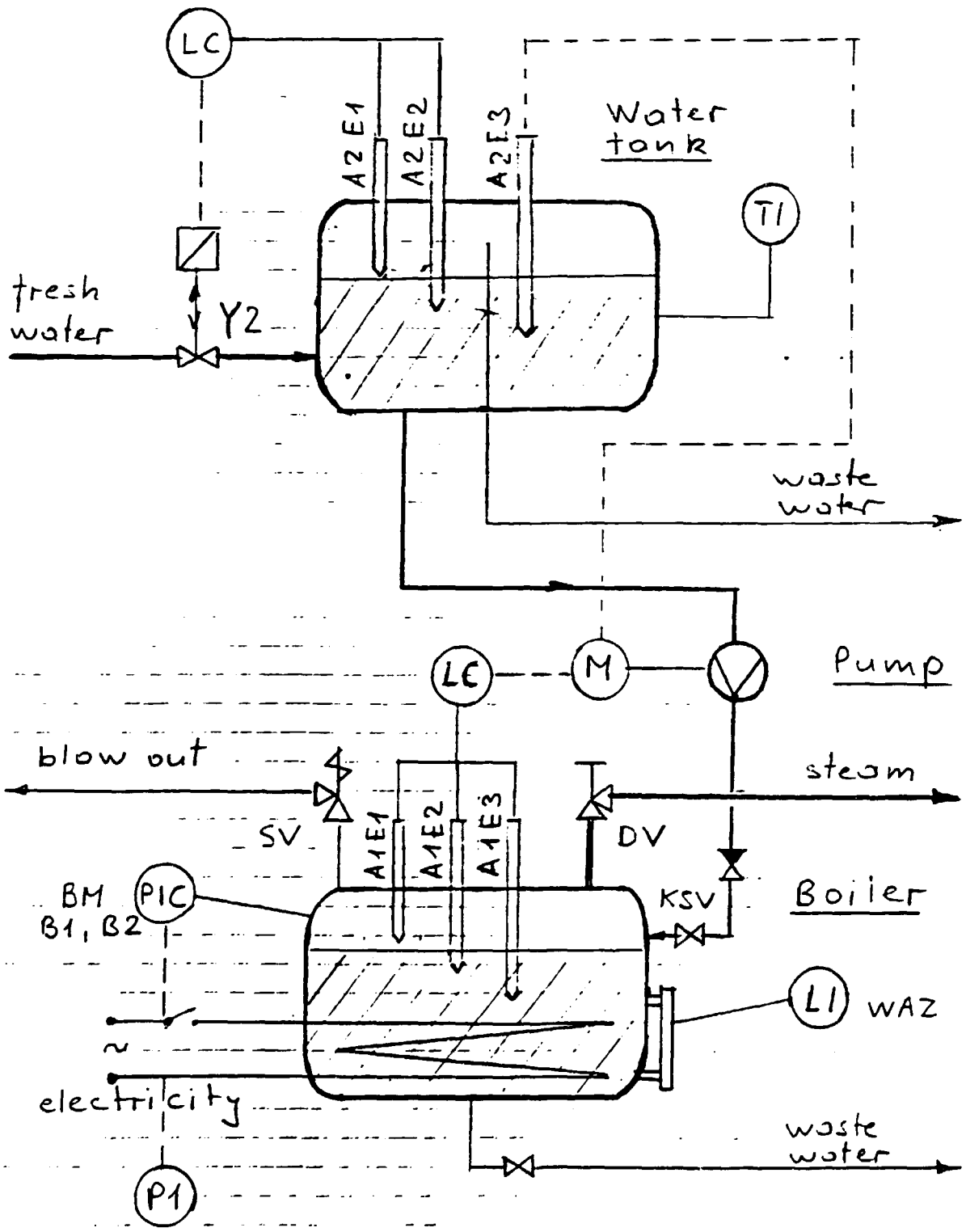
- Time
- Pressure PI 1503, PI 1501, PI 1502, PI 1500, PI 1504
- Hot water temperature TSA 1230, TI 1231
- Flow rate of extract (range of opening of valve E5)
- Cold water temperature TI 1212
- Temperature of n-hexane vapors and condensat TI 1201, TI 1203
- Temperature of residue TI 1200

19. Finishing of process shall be caried out as follows:

- Finish the charging of extract, close valves E1, E2. Empty vessel 1V2.
- Cut off hot and cold water.
- Switch off thes vacuum pump and allow air into the unit.
- Discharge all rest liquids.

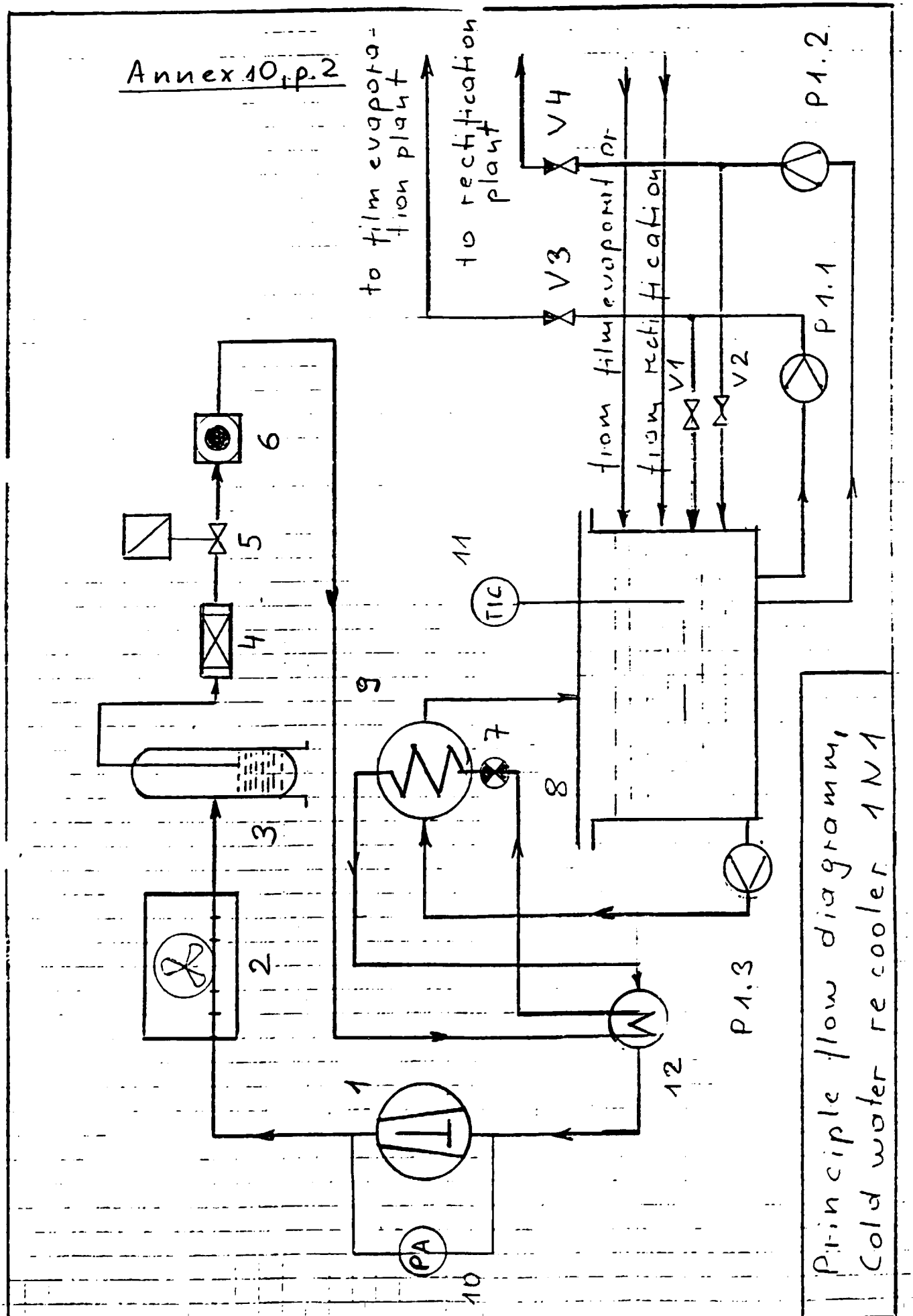
11. Every time while work pay a special attention to the rules of safety mentioned in the project dacumentation from 27th february 1991, chapters 13 and 14.

Annex 10, p.1



Principle flow diagramm
Steam generator, pos. 2 U1

Annex 10, p. 2



Principle flow diagram, Cold water recooler 1 N 1

Cold water recooler 1N1

Designations of positions in the flow diagramm

1 - motorcompressor

2 - condenser with axial ventilators

3 - liquid collector

4 - dryer

5 - solenoid valve

6 - sight glass

7 - thermostatic expansion valve

8 - cold water tank

9 - evaporator

10 - high pressure/low pressure control (pressostat)

11 - thermostat

12 - liquid separator

P 1.1 - Extern circuit pump for cold water to fallfilm-evaporator

P 1.2 - Extern circuit pump for cold water to rectification plant

P 1.3 - Intern circuit pump for cold water

De Silva/jbg
20 Oct. 1992

Backstopping Officer's Technical Comments
based on the work of R. Böhme and S. Langner
DP/DRK/88/001

The report contains an account of the installation and commissioning of the equipment for distillation and detailed operating instructions. The instructions would be very useful in training the operators. Only one trial run has been done to demonstrate the functioning of the equipment. More experiments have to be performed to optimize the parameters for every material to be distilled. The use of analytical data for development of parameters for each oil have still to be imparted by an experienced consultant in the field of essential oils. It is hoped that the ways to maintain the equipment and solve trouble shooting have been imparted.