

Operation Manual



PETRODIST 100 S

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		NOTE
	Important! - Read carefully before use - Keep for future reference! This operating manual is a component of the system and must be available to the operating and maintenance personnel at all times. The safety instructions contained therein must be observed. In case of a resale of the system, this operating manual must always be supplied. Make sure to read the Data Sheets of the product to be distilled, General Safety Remarks of PILODIST® and of peripheral devices. This distillation-system is only to be operated under current safety-standards for ventilation.	

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1 Introduction

The design of the new computer-controlled distillation system is very user friendly.

The first chapter describes the individual system components and their functions.

In the second chapter (distillation operation) the different operation phases will be explained, examples of application and troubleshooting instructions will be given.

The third chapter (software operation manual) contains an exact description of the system software with all program functions and an illustration of the screen masks.

1.1 Plant Identification (type plate)

	 NOTE The instructions given in this manual are valid only for the plant that type is specified on the title page. The type plate with the type number is located on the control cabinet of the plant. When used for a different type, technical differences can lead to property damage or serious / fatal injuries. Important for all queries is the correct indication: - the designation "PD 100 S", - the type no. "PD 100 S", - the plant no. (Serial / Project Number) Only then is a flawless and fast processing possible.
	 CAUTION In this distillation system sealings are used that can be harmed by certain products deviating from the ASTM-D2892. In this case durability of sealings has to be tested on a case-by-case basis. Special sealings can be ordered directly from PILODIST, feel free to contact us.
	 WARNING Only connect Nitrogen (N₂) to the N₂ port. Do NOT connect pressurized air.

1.2 Short Description of the System

PETRODIST 100 S is a semi-automatic distillation system for fractionation of stabilized hydrocarbon products.

The system was developed for the separation of crude oil and hydrocarbon samples into their different cuts (for example: Naphtha, Kerosene, Diesel) according to ASTM D2892. High level of automatization brings unmatched performance comparing to other semi-automatic systems available on the market.

The system is equipped with a distillation column packed with ProPak packing to ensure a proper separation efficiency between 14 and 18 Theoretical plates. The fully automatic fraction collector is optimized to collect big individual cuts either small different cuts. It is equipped with overfilling protection to exclude any loss of even a big cut.

The whole process is performed semi-automatically through 4 distillation steps (Debutanization, ATM distillation, VAC 100 Torr distillation, VAC 99-2 Torr distillation) with minimum user intervention. A quench coil, in case if connected to a tap water supply or external thermostat, reduces the intermediate cooling phases and in combination with a fast heat-up process the runtimes can be reduced to a unique minimum.

The system basis consists of a control unit and a glass apparatus. Vacuum pump, cold traps and gas trap are integrated into the system.

The new touchscreen control panel is swivel-mounted for the best operation mode and control cabinet is installed on a shelf.

System can be equipped with several safety options (optional). These options are highly recommended to raise the safety level of the system and to be provided either by manufacturer or customer:

1. Automatic fire extinguisher

Automatic fire fighting system with a steel cylinder with 10 kg volume of CO₂. UV sensors for monitoring the system control electronics and pneumatic valve. In case of fire the complete distillation system will be shut-down and the system will be inflated by CO₂.

According to safety regulations for transportation the fire extinguisher system will be shipped emptied (without CO₂) and the steel cylinder needs to be filled at customer's site with CO₂. It is recommended to the end-user to arrange an annual maintenance of the fire extinguisher system by an authorized local company.

The effectivity of the fire extinguishing system needs either a complete sheathing of the system as offered above or it needs to be placed in an on-site available fume hood!

2. Gas Detector

Monitoring of hydrocarbons escaping from the system by means of a gas detector. In case combustible gases concentration is detected (20% LEL, adjustable), alarm will be given. When the alarm is ignored, the distillation will be switched off after a pre- selected time.

3. Hydrogen Sulphide Alarm

Monitoring of hydrogen sulphide escaping from the system. Sensor for detection of hydrogen sulphide with digital display of concentration from 1 to 20 ppm with adjustable alarm limit (standard value - 4-6 ppm). When the alarm is ignored, the distillation will be switched off after a pre- selected time.

1.3 General safety remarks

1.3.1 Information and Explanations

	 DANGER <i>"DANGER" warns of dangerous situations. Avoid these dangerous situations!</i> Failure to do so will result in serious injury or death.
	 WARNING <i>"WARNING" warns of dangerous situations. Avoid these dangerous situations!</i> Otherwise serious injury or death may result.
	 CAUTION <i>"CAUTION" in conjunction with the warning symbol warns of dangerous situations. Avoid these dangerous situations!</i> Otherwise minor injuries may result.
	 NOTE <i>"NOTICE" gives you recommendations for action which do not result in personal injury.</i> However, follow the recommendations for action to prevent problems and damage to property!
	 NOTE <i>Notes in the operating instructions / documentation are marked with a "book" (see external documentation).</i> However, follow the recommendations for action in order to avoid problems and damage to property!

1.3.2 Explanation of the Security Symbols used

	DANGER Hazards due to dangerous electrical voltage, over 1000 Volt alternating voltage with additional voltage indication. Failure to do so will result in serious injury or death. Access only allowed for electrician or electrically instructed person.
	DANGER Burning risk / Hot surfaces Failure to do so will result in serious injury or death. (From 60 ° C, danger: wear suitable protective gloves)
	DANGER EX area, prohibited access for unauthorized persons. Failure to do so will result in serious injury or death. (No access for unauthorized persons!)
	DANGER An area whose access is forbidden to unauthorized persons. Failure to do so will result in serious injury or death. (Danger: unsafe areas for untrained persons)
	NOTE The protective earth connection is indicated at the connection points by one of these symbols. Failure to do so may result in injury, also by replace or confuse these connections during repairs.
	NOTE Eco-label identifies measures of environmental protection (warning against environmental pollution, in the disposal chapter). Otherwise, damage to the environment is the result. Incorrect disposal can lead to major environmental damage.

1.3.3 Built-in Security Systems

The built-in safety devices shall be tested at regular test intervals and with appropriate test methods (see the following table).

Test interval	Test method
s = per shift	
d = daily	
w = weekly	
m = monthly	V = Visual inspection
¼ y = quarterly	F = Function test
½ y = semi-annual	M = measurement
y = yearly	
4 y = every 4 years	

Table 1: test intervals security systems

1 Power supply disconnection (Main Switch)

When the main switch is actuated, the plant is connected to the power supply network or disconnected from the power supply network. The individual switches are located on the units, the main switch is located: at the front of t.

Test	
interval	method
y	F





DANGER

If the main switch is switched off due to the execution of cleaning, maintenance or repair work, unauthorized re-activation is to be ensured.

Failure to do so will result in serious injury or death.

Disregarding the shutdown procedures / securing against reconnection: lethal and / or unexpected movements are possible.

2 Earthing

The earthing of all conductive parts of the plant must be checked regularly.

Test	
Interval	Method
y	V, M

3 Residual current circuit breaker (30 mA)

An RCD (residual current circuit breaker) is to be installed in power supply line separately. It must be checked regularly for proper function.

Test	
Interval	Method
¼ y	F





DANGER

All locked plug-in connections are revision connections and may only be disconnected and plugged in without voltage by trained personnel.

Otherwise, serious personal injury and property damage may occur!

1.3.4 Obligations of the Carrier

		CAUTION
	<i>In the EEA (European Economic Area) the national implementation of the framework Directive (89/391 / EEC) has to be respected and implemented as well as the related individual directives and especially Directive (2009/104 / EC) on the minimum requirements for safety and health protection in the case of the use of work equipment by employees at work, in the valid version, in each case.</i> Otherwise, injuries can result. <i>The operating instructions are written for specialist personnel and the personnel must be suitably trained before working on the machine. In Germany, the Operational Safety Ordinance of June 2015 (BetrSichV) has to be observed (implementation of the above-mentioned guideline in national law).</i>	

The carrier must obtain the **local operating permit** and observe the related requirements.

In addition, he must comply with the local statutory provisions for:

- The safety of personnel (e.g. operating instructions, personal protective equipment (PPE), preventive examinations).
- The safety of the work equipment (protective equipment, work instructions, process risks and maintenance).
- Product procurement (safety data sheets, list of hazardous substances).
- Product disposal (waste disposal law).
- The material disposal (decommissioning, waste disposal law).
- Cleaning (detergents and disposal).
- As well as the current environmental protection requirements.

In addition, the following must be observed:

-
- A permanent hazard assessment of the workstations is to be carried out. The measures must be fixed in operating instructions and the personnel must be instructed accordingly (training of the operating personnel).
- The necessary personal protective equipment (PPE) is to be provided.
- A skin protection plan and a hygiene plan must be drawn up.
- The supervisors have to check compliance with the measures from the operating instructions.
- Work instructions must be drawn up.
- The following applies generally to the staff: no work under the influence of alcohol, drugs or fatigue.
- Safety deficiencies at components of the plant are to be reported by the operator to the supervisor (conductor).
- Protective devices, e.g. suitable hand-held fire extinguishers shall be installed in a prescribed number and size at easily accessible places.
- In case the PILODIST system has been ordered including an integrated fire extinguishing system we highly recommend to study the instructions for operation and maintenance from the

manufacturer (GLORIA), which are enclosed, as well as to consider locally available instructions for use and maintenance of fire extinguishing systems in the laboratory of the enduser company.

- Dangers caused by lightning strikes: the carrier must provide the system with a coarse and medium protection (lightning protection) for the power supply.
- A properly grounded power supply socket and an RCD (30 mA) must be available for the plant (if not built in).
- The illumination of the workstations shall be established by the operator according to the local regulations.
- Periodic checks must be carried out on the occurrence of trip hazards.

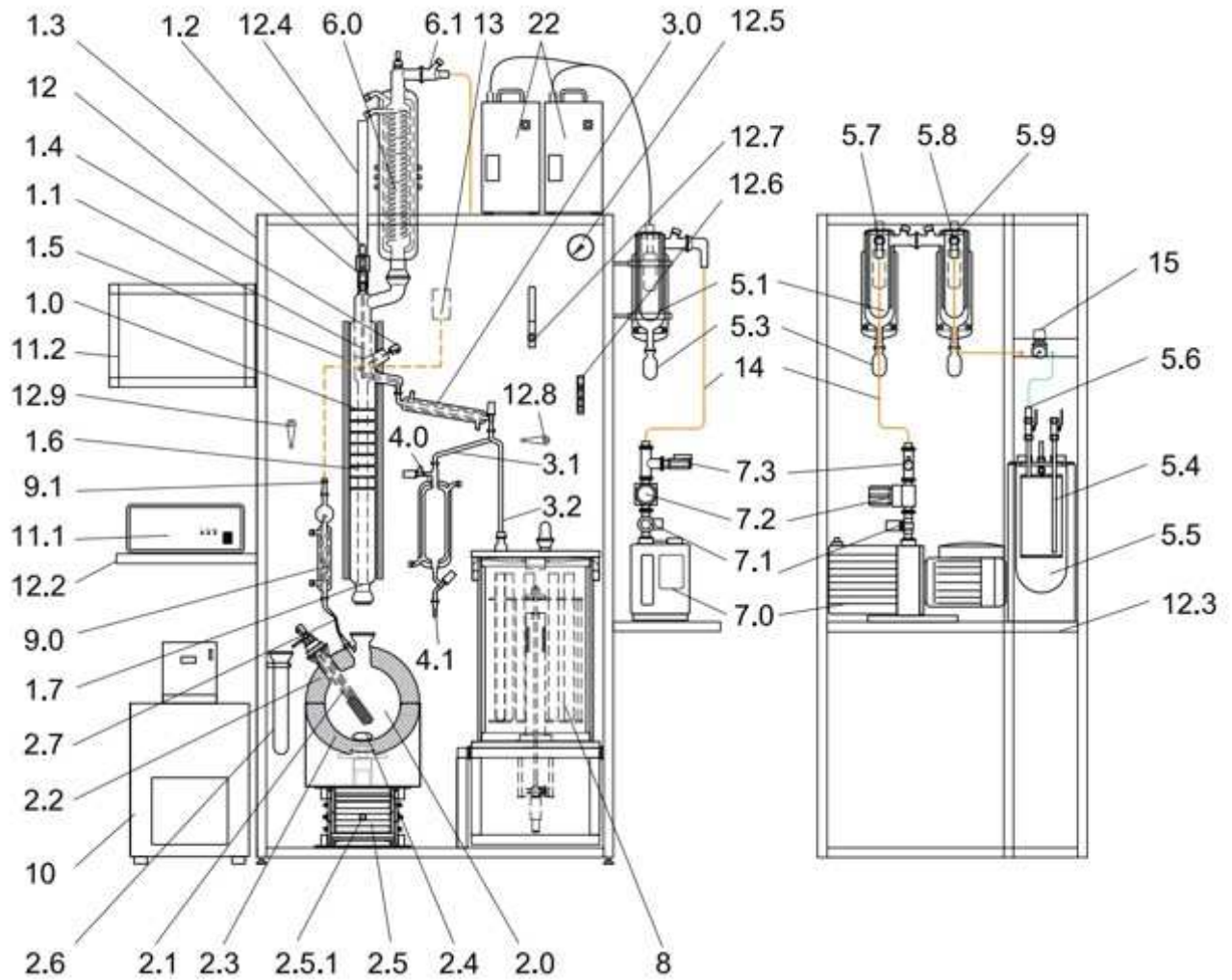
1.3.5 Connections:

Before operating the system, the carrier must ensure that the local regulations (for example for the electrical connection) are observed during installation and commissioning, when these are carried out by the user himself.

	 CAUTION
	At least one annual safety briefing (and in each case at the beginning of employment) on dangers and protective measures is required by means of the operating instructions in the language which is understandable to the person concerned. Failure to do so may result in property damage and / or injury. The safety briefing must be confirmed with a signature.
	 CAUTION
	Illuminance: The operator must ensure a sufficient and uniform lighting intensity in all areas of the system. Failure to do so may result in injury. 300 lux (checkpoints 500 lux), depending on the plant region, are recommended.
	 DANGER
	The plant may only be used in an encapsulated manner, such as a sheathing ordered by manufacturer or put into a fume hood. Failure to do so may result in serious injury or death.
	 DANGER
	The use of combustible or explosive liquids and substances are in the sole responsibility of the customer. Any resulting risks and necessary actions must be examined by the customer and executed if necessary.

1.4 PD 100 S

Picture may not show all special customized features



view from the right

Fig. 1: PD100S

Legend to Fig. 1

- 1.0 distillation column
- 1.1 valve plunger
- 1.2 valve plunger hood
- 1.3 solenoid coil for the take-off divider
- 1.4 head temperature sensor Pt-100
- 1.5 tower mantle with built-in temperature sensor,
- 1.6 column filling, Propak 316, 4 mm
- 1.7 foam breaker
- 2.0 distillation flask
- 2.1 flask temperature sensor with integrated quench coil
- 2.2 insulation jacket for the upper half of the flask
- 2.3 heating bath for flask with temperature sensor Pt-100
- 2.4 stirring device
- 2.5 lifting platform
- 2.5.1 retched handle
- 2.6 holder for the quench cooler
- 2.7 differential pressure line to flask
- 3.0 distillate cooler
- 3.1 distillate line to intermediate receiver
- 3.2 distillate line to fraction collector
- 4.0 intermediate receiver 250 ml, with cooling mantle and vacuum mantle
- 4.1 drain line intermediate receiver
- 5.1 cold traps with silvered vacuum mantle
- 5.3 condensate traps
- 5.4 gas trap made of stainless steel
- 5.5 dewar vessel for gas trap
- 5.6 hose connection
- 5.7 vacuum connection line
- 5.8 vacuum connection line
- 5.9 centering piece for immersion cooler finger (option)
- 6.0 main condenser, with vacuum mantle, cooling
- 6.1 vacuum connection line (condenser)

- 7.0 vacuum pump
- 7.1 vacuum solenoid valve
- 7.2 throttle valve
- 7.3 T-piece with ventilation valve
- 8 fraction collector (see fig. 14)
- 9.0 safety cooler
- 9.1 differential pressure line
- 10. thermostat
- 11.1 control device DCD 4001
- 11.2 touchscreen control panel
- 12 mounting frame
- 12.2 tray for control device
- 12.3 tray for vacuum pump and gas trap
- 12.4 rod for condenser
- 12.5 manometer
- 12.6 flow meter nitrogen
- 12.7 gas flow selection: Distillation - Debutanization
- 12.8 fraction collector interlock
- 12.9 tempering fluid selection: distillation - water removal
- 13 differential pressure sensor
- 14 vacuum hose system
- 15 nitrogen main valve
- 22 immersion coolers for cold traps (option)

1.5 Technical Data

Operation Temperature (flask):	Up to 350°C
Temperature Range (vapor):	Up to 400°C AET (depending on a sample)
Operation Pressure:	Ambient down to 2,0 Torr
Available Column DN:	25, 36, 50 and 70 mm
Available Flask Sizes:	2, 4, 6, 10, 20, 35 or 50 L
Charge Quantity:	up to 50% of the flask volume
Mains Supply:	230/400 V, 50 Hz, 3 phase + N + PE (5 wire) *- different types of power supply are to be discussed prior order placement
Plug type:	CEE 230/400V 16A (5 wire 3 ph + N + PE 6h) *- different plug types are to be discussed prior order placement

		CAUTION
	All glass parts are made of Borosilicate glass 3.3 class. Melting point of this glass is around 550°C. Never exceed this temperature! Otherwise injuries may result.	
		NOTE
	System is equipped with safety temperature limits preventing the flask and column temperatures exceed 400°C. Once 400°C exceeded distillation will be stopped immediately.	
		NOTE
	Heaters for flask and column are equipped with software overtemperature protection with adjustable limits, maximum allowed of 450°C. When this temperature exceeded system will discontinue power supply of all heaters. Glass never will be heated up higher than 450°C keeping safe distance of at least 100°C lower than melting point.	

1.6 Installation requirements

Dimensions: **Standalone unit**

w: 1810mm (+550mm if Water Removal option ordered)

h: 2320mm

d: 870mm

Additional Space: recommended width and depth additional 0,5m for service

Power Supply: **1x** 230/400V 50Hz 3 phase + N + PE (5-wire), 16A

2x 230V, 50Hz phase + N + PE, CEE 7/4, 16A

*- different types of power supply are to be discussed prior order placement

Power Consumption: max. 7 kW (peak consumption)

Plug Type: **1x** CEE 16A 400V 3L+N+PE 6h

2x CEE 7/4, 16 A

*- different plug types are to be discussed prior order placement

Nitrogen Connection: System is delivered with

- 6x4mm tube connection to adapt locally available nitrogen supply
- Pressure reducer is integrated in the system
- Minimum pressure 1 bar.

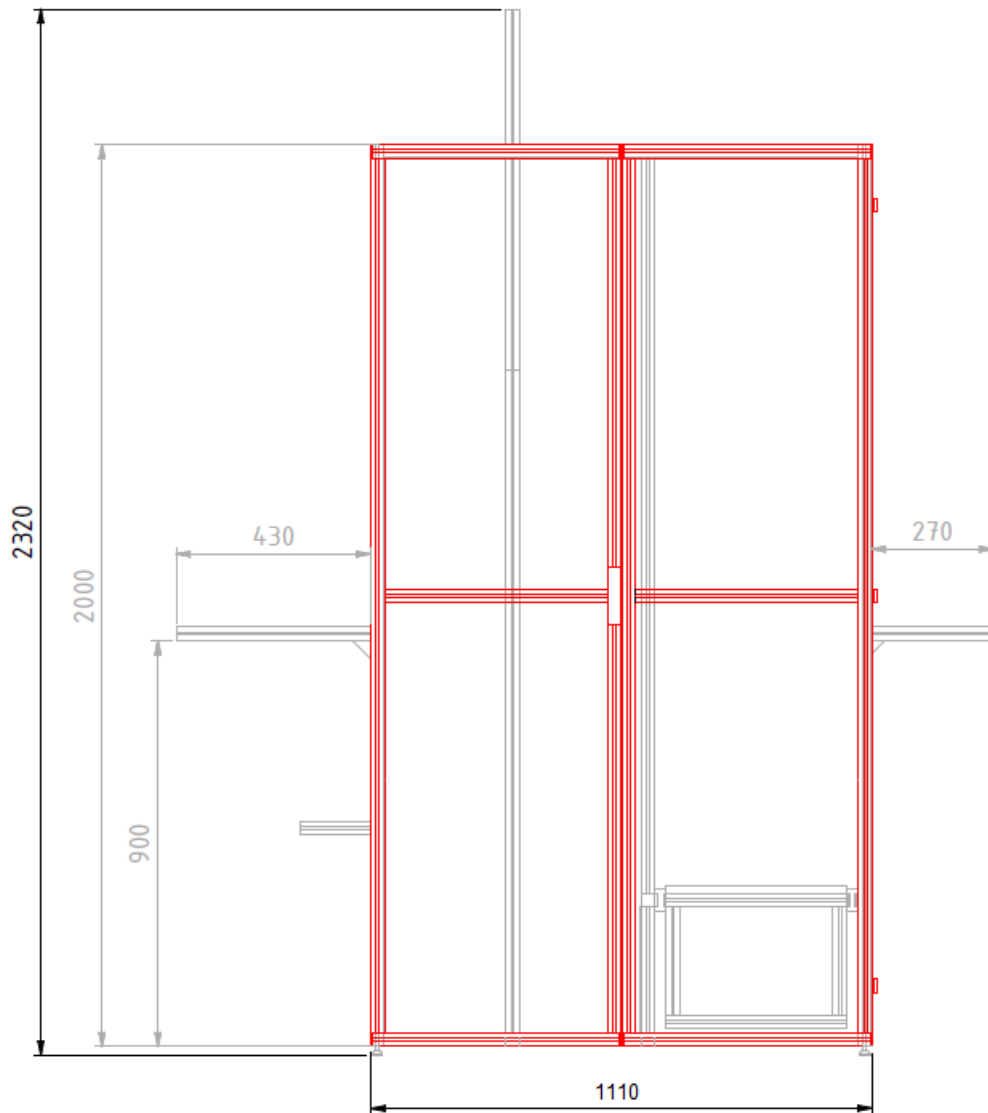
System Outlets: **1x** Vacuum Pump Exhaust - 12mm tube connection

1x System Ventilation IN/OUT – 6x4mm tube connection

Ventilation Requirements: Forced-air and exhaust ventilation system on site. Minimum air exchange rate – 10 times per hour.

Additional on-Site Requirements:

- Glycol based antifreeze liquid down to -40°C (approx 20L)
- Dry Ice for Cold traps (if no immersion cooler ordered)
- Toluene for washing run (approx. 5L)
- Sample for test run (approx. 3L)
- Ethanol/Isopropanol for Option immersion cooler (approx. 5L)



Additional space requirements

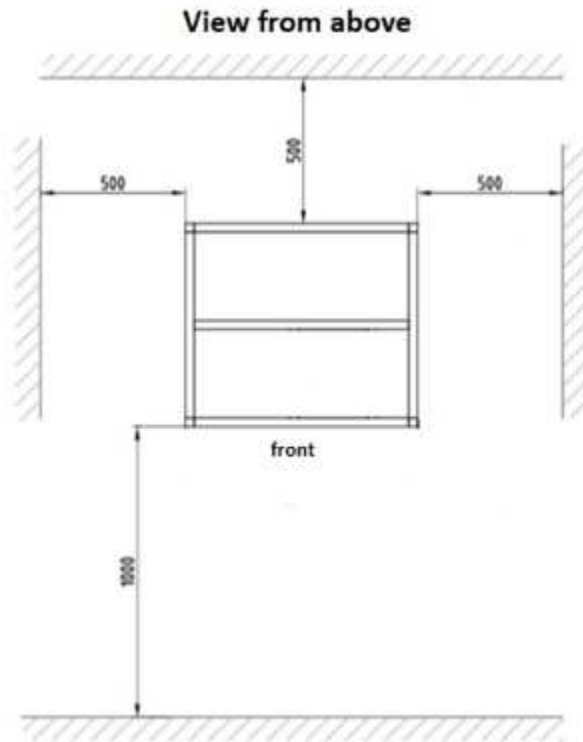


Fig. 2: PD100S Dimensions

2 Components of the distillation system

2.1 Glass Apparatus

The construction and the dimensions of the glass apparatus correspond to technical drawings. All glass parts are made of DURAN-glass.

		CAUTION
	All glass parts are made of Borosilicate glass 3.3 class. Melting point of this glass is around 550°C. Never exceed this temperature! Otherwise injuries may result.	
		NOTE
	System is equipped with safety temperature limits preventing the flask and column temperatures exceed 400°C. Once 400°C exceeded distillation will be stopped immediately.	
		NOTE
	Heaters for flask and column are equipped with software overtemperature protection with adjustable limits, maximum allowed of 450°C. When this temperature exceeded system will discontinue power supply of all heaters. Glass never will be heated up higher than 450°C keeping safe distance of at least 100°C lower than melting point.	
		CAUTION
	All glass parts are designed to operate under vacuum. Maximum allowed overpressure is 0,5 Bar higher than ambient. Otherwise injuries may result.	
		NOTE
	System is equipped with manual ventilation valve which must be always opened during STANDBY mode, ATM distillation and Washing Run. Also, the system is equipped with automatic overpressure safety valve which opens at 0,2 Bar overpressure.	

Description of the individual components:

- Distillation flask (2) with connections for the distillation column (1), nitrogen supply and for temperature measurement, complete with motorized stirrer (2.4) for avoiding boiling retardations.
- Distillation column (1) with silvered vacuum jacket and Sulzer packing.
The column is equipped with connections for flask, temperature measuring sensor, main condenser, and distillate line.
- A vacuum cold traps (5.7 and 5.8) are mounted for protection of the vacuum pump. Each of them is cooled by means of an immersion cooler (22).
- Intermediate receiver (4.0) located in front of the system and used for collecting light naphta cut.
- The receivers are located inside fraction collector.

2.2 Vacuum System

The vacuum pump (7.0) is a rotary pump with 2 stages and displacement capacity of 18 m³/h.

On the top of the pump a manual throttle valve (7.2) is installed, which responsible for the pump's capacity to the leakage rate of the system. The vacuum solenoid valve (7.1) serves for stabilising the preselected vacuum and is controlled automatically by software.

The vacuum measuring sensor (14.0) is independent of the type of gas. Measuring range from 100 down to 0,1 Torr.

2.3 Nitrogen-Flow

		WARNING
	Only connect Nitrogen (N ₂) to the N ₂ port. Do NOT connect pressurized air.	

Nitrogen on site requirements approx. 1-2 bar has to be reduced down to 0,5 bar at the integrated N₂ pressure reducer.

The N₂-flow meter (12.6) is the connection between the integrated pressure reducer and the System. Adjust the N₂-flow with the flow adjustment knob. The N₂-valve located directly below the reducer has to be closed after distillation.

		CAUTION
	Use nitrogen supply at ATM distillation when ventilation valve is opened only! Or during VAC stages when vacuum pump is running. Never fill completely closed system with nitrogen! Otherwise injuries may result.	

		NOTE
	The system is equipped with automatic overpressure safety valve which opens at 0,2 Bar overpressure keeping the system safe for operator. Check the operation conditions of this valve on a regular base. Make sure it is not blocked with product!	

Legend:

- 15.0 pressure reducer
- 15.1 N₂-flow valve
- 15.2 pressure adjustment knob
- 15.3 N₂-distributor
- 12.6 N₂-flow meter
- 12.6.1 flow rate adjustment knob

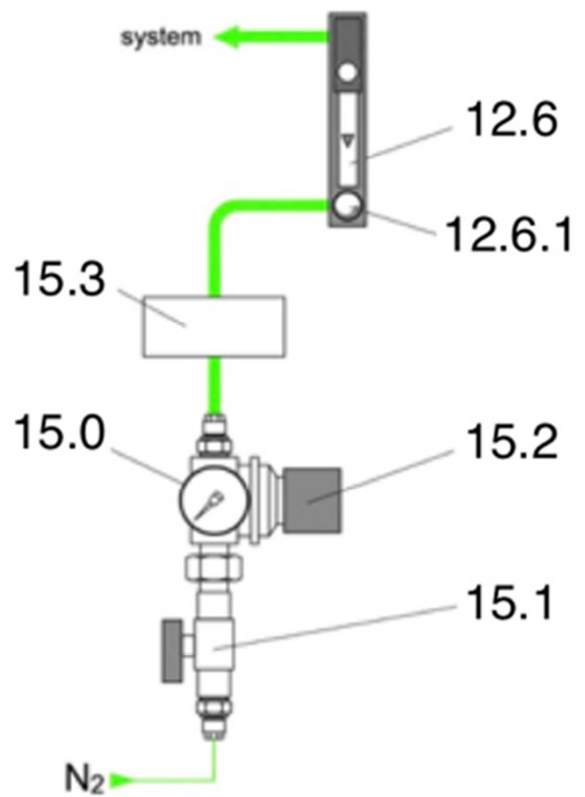


Fig. 3: Nitrogen Flow

2.4 Flask Heating

The flask heating consists of a heating hood with a heating capacity of approx. 1400 W. The insulation hood for the upper half of the flask has a power consumption of approx. 350 W. It is installed on a manually operated lifting platform with integrated stirrer drive.

2.5 Stirrer

The stirrer (2.4) speed is adjusted with the potentiometer at the stirrer drive such that the sample will be constantly stirred.

2.6 Lifting Platform

The lifting platform for the distillation flask can be lifted and lowered by means of operating the wrench located in front of lifting platform.

Use a special selector to select the direction:



Position 1 of the selector makes flask heater move up.



Position 2 of the selector makes flask heater move down.

Fig. 4: Wrench selector

		NOTE
	Make sure that flask properly and completely installed into flask heater. The flask must be lifted up very carefully to keep the glassware safe.	

2.7 Gas flow selector

Depending on actual process Gas Flow selector (12.7 fig.1) must be turned to a proper position. Debutanization position redirects the gas flow into Gas trap. During debutanization the Fraction collector interlock must be CLOSED as well.

2.8 Thermostat / Immersion cooler

Depending on system configuration (ordered options) the PD100S can be equipped with a number of different thermostats:

- Thermostat for main condenser
- Immersion cooler for cold trap
- Immersion cooler for gas trap

Each thermostat is supplied with own manual to be read before use.
Please see special operation manual service part.

2.9 Control Panel

The system is equipped with Touch Screen Windows based control panel as user interface. Control panel is equipped with special software package which allows to control the distillation process and data record.

		WARNING
	Do not turn OFF the main switch while Control Panel is running. Shut down the control panel first, then turn OFF the main switch!	

For further information, please refer to Software operation manual.



Fig. 5 Touchscreen Control Panel

Picture may not show all special customized features

2.10 Control Unit

Control unit DCD4001 located on the left side of the system and contains of all electronic components, except of sensors, and communications. Control unit is a painted steel box with sealed cover. Please, keep the cover completely closed during routine operations to avoid dust entering.

Only authorized personnel allowed to access into control unit. Use a special hexagon key to open/close the cover (key is not included).

System operates with 2 different voltage circuits:

- 230/400 V AC, 50 Hz (60 Hz on additional request).
- 24 V DC from inbuilt voltage converter.

Wiring diagrams are given in separate document “Electric Plan DCD4001” and contains all diagrams, schematics, interconnections and can be used for repair the control cabinet.

Please, contact to a manufacturer or authorized service centre prior repairing the control cabinet.

	DANGER High voltage! Never open the control unit while power supply is connected! Failure to do so will result in serious injury or death. Access only allowed for electrician or electrically instructed person.
 	NOTE Make sure that system is connected to a protective earth Failure to do so may result in injury, also by replace or confuse these connections during repairs.

3 Preparation for distillation

Before starting the distillation make sure that system is properly assembled.

Assembling of the system first time or before/after moving the instrument to different location must be done only by authorized personnel in obligatory presence of manufacturer or authorized distributor representatives.

3.1 Assembly and Commissioning

3.1.1 Assembly of the System



Please note that all glass parts should be handled very carefully and unstressed during all works.

System is delivered 50% preassembled. The framework, shelves and most of holders are already installed.

Installation procedure is to be done by authorized personnel only! All parts must be installed according to a Fig.1. Please, refer to Fig.1

Install the framework on flat and horizontal surface close to all necessary media connection points (electricity, pressurized air, nitrogen and exhaust line).

		NOTE
	The PD100S system is ONLY to be used INSIDE the laboratory environment and in a non-ATEX area.	

Using the Level check if framework is installed correctly:

- Top of the framework must be complete horizontal in all planes.
- Side panels must be complete vertical in all planes.

If not, use adjustable feet to adjust the framework position.

When framework was fixed in proper position, install the column heating mantle using the corresponding holders and introduce the column inside the mantle heater.

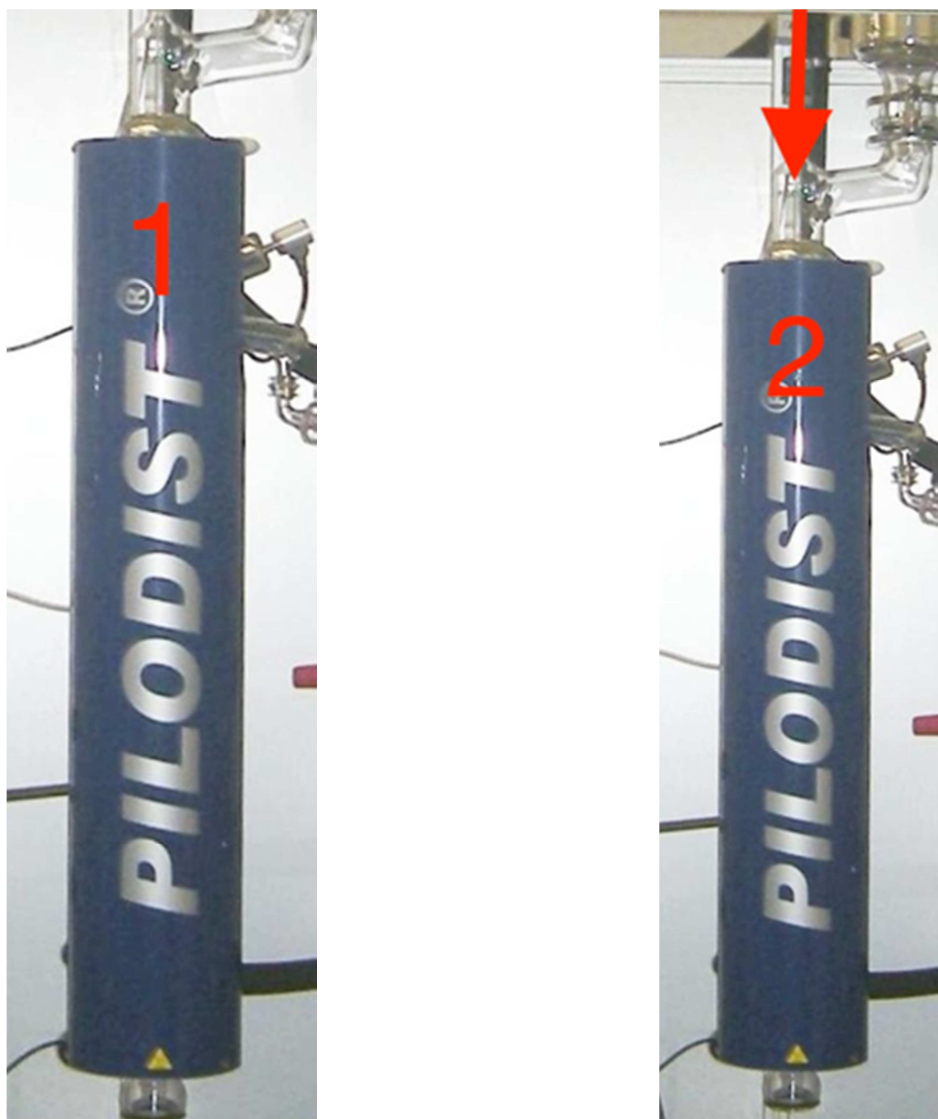


Fig. 6 Mantle heater with column

Introduce the reflux divider plunger and protective cap to a column top opening.



Fig. 7 Reflux divider

Install the main condenser and fix the connection to a column by a clamp.

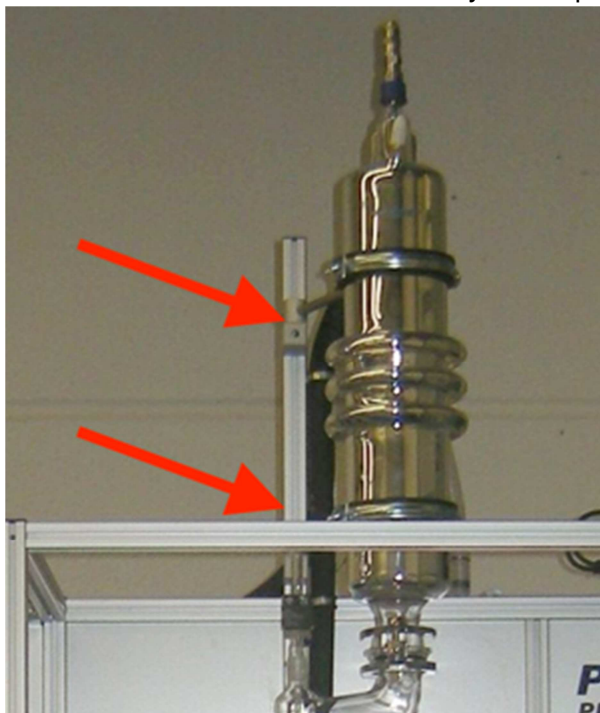


Fig. 8 Main condenser holders

Install the cold traps and condensate receivers. All interconnections must be secured by clamps.

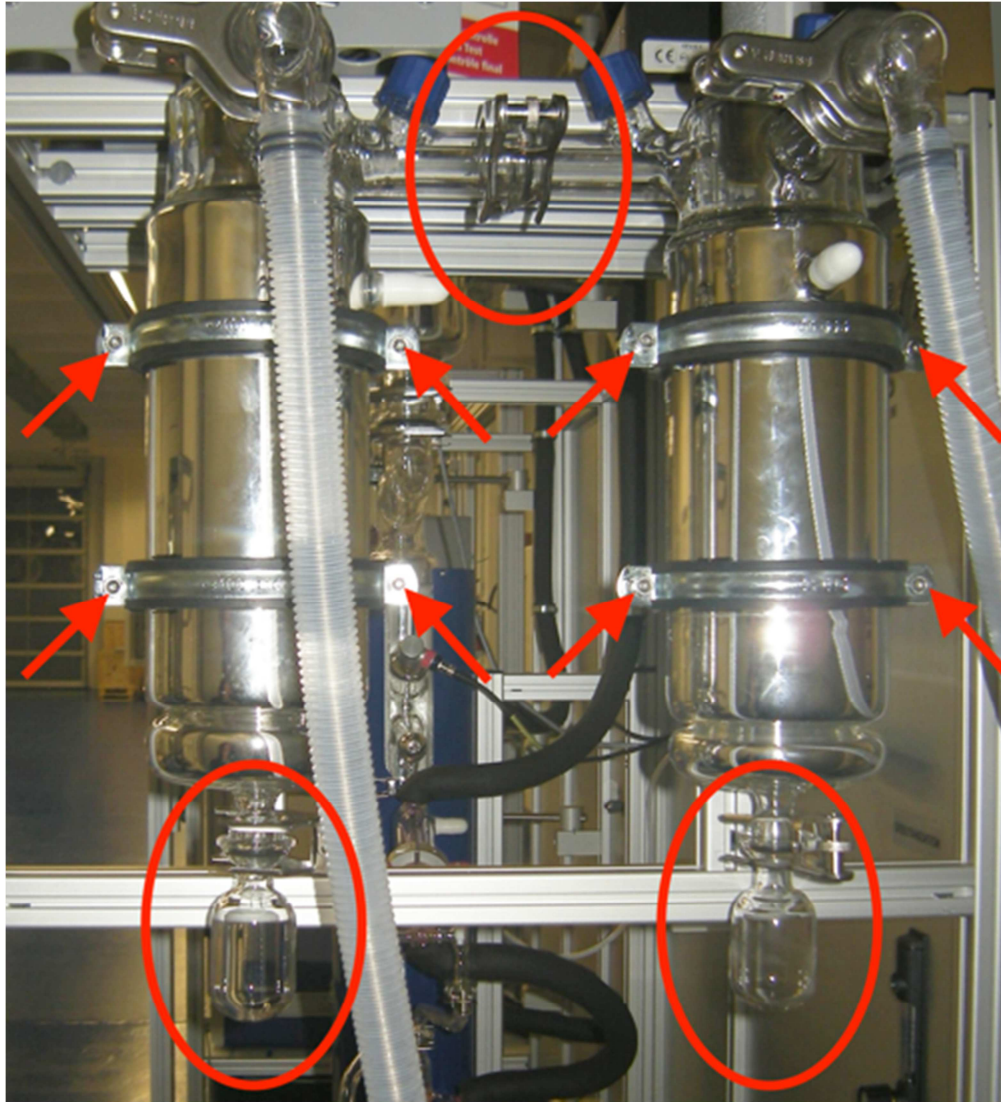


Fig. 9 Cold traps holders

Install the distillate cooler, intermediate receiver and distillate line to fraction collector.

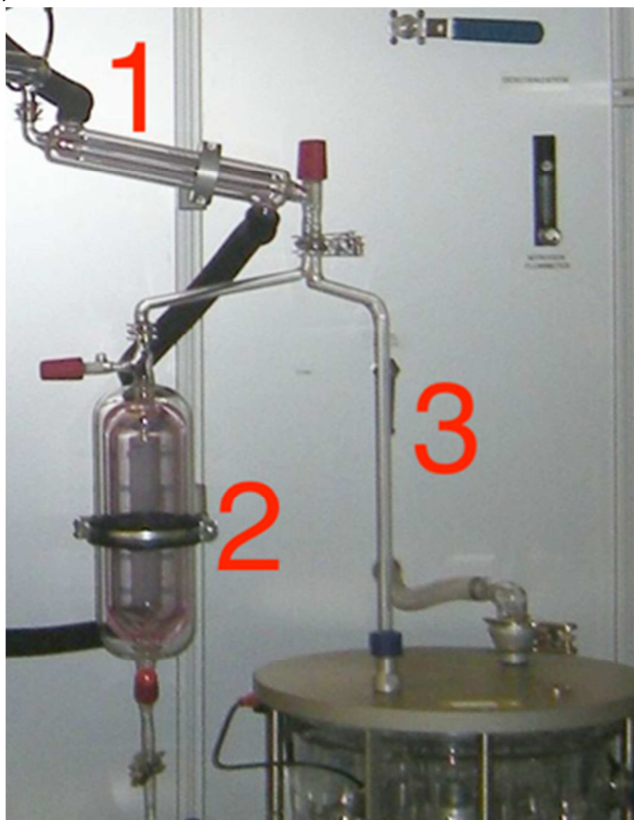


Fig. 10 Distillate lines

Install the protection cooler for differential pressure sensor on the left side of the column.



Fig. 11 Expansion cooler

Thermostats and the immersion cooler have to be positioned according to figure 1.

The vacuum pump is to be located on the right side shelf.

DCD4001 is to be located on the left side shelf.

All water and vacuum lines to be connected according to a nameplates located on each hose ending.
For further information, please refer to P&ID schematics.

Disassembling must be done in opposite way.

If system needs to be moved to a different location in future, we recommend using the marking lines on body of each glass part.

		CAUTION
	It is prohibited to change the location of the system out of the place of original installation (to different room or building for example) without pre-disassembling. Please follow the assembling procedure in opposite direction to dismount all necessary parts prior moving the system. Pre-disassemble, change of the location, setup at new location should be carried out under manufacturer/authorized distributor representatives control only!	

3.1.2 Vacuum System

The vacuum cold traps are to be put into the corresponding fastening and have to be connected to the corresponding lines. Then the solenoid valve and the throttle valve have to be connected with the vacuum pump by means of a tension ring and have to be connected to the control unit according to the socket descriptions.



Fig. 12 Vacuum pump

Immersion cooler's finger (if used) must be placed into the cold trap via centring device.

Fill the cold trap with pure ethanol to cover the cooling finger completely.

System requires 20-30 minutes after main switch was turned on to cool down the cold traps and warm up the vacuum sensors.

System is equipped with 0-100 Torr vacuum sensor to distil the samples in the range of 1-100 torr. Sensor must be installed to a vacuum distributor and connected via associated cable.



Fig. 13 Vacuum distributor

3.1.3 Temperature Measuring Sensors

The temperature measuring sensors PRT for flask and head have to be positioned according to figure 1 and have to be connected to the control unit.

Please note that head PRT must be installed using a Teflon sleeve and vacuum grease to a grinded PRT connection place in the column.

3.1.4 Fraction collector

Fraction collector is designed to operate in fully automatic mode during single stage. It means it is capable to collect different cuts into different receivers automatically according their boiling temperatures.

Fraction collector consist of:

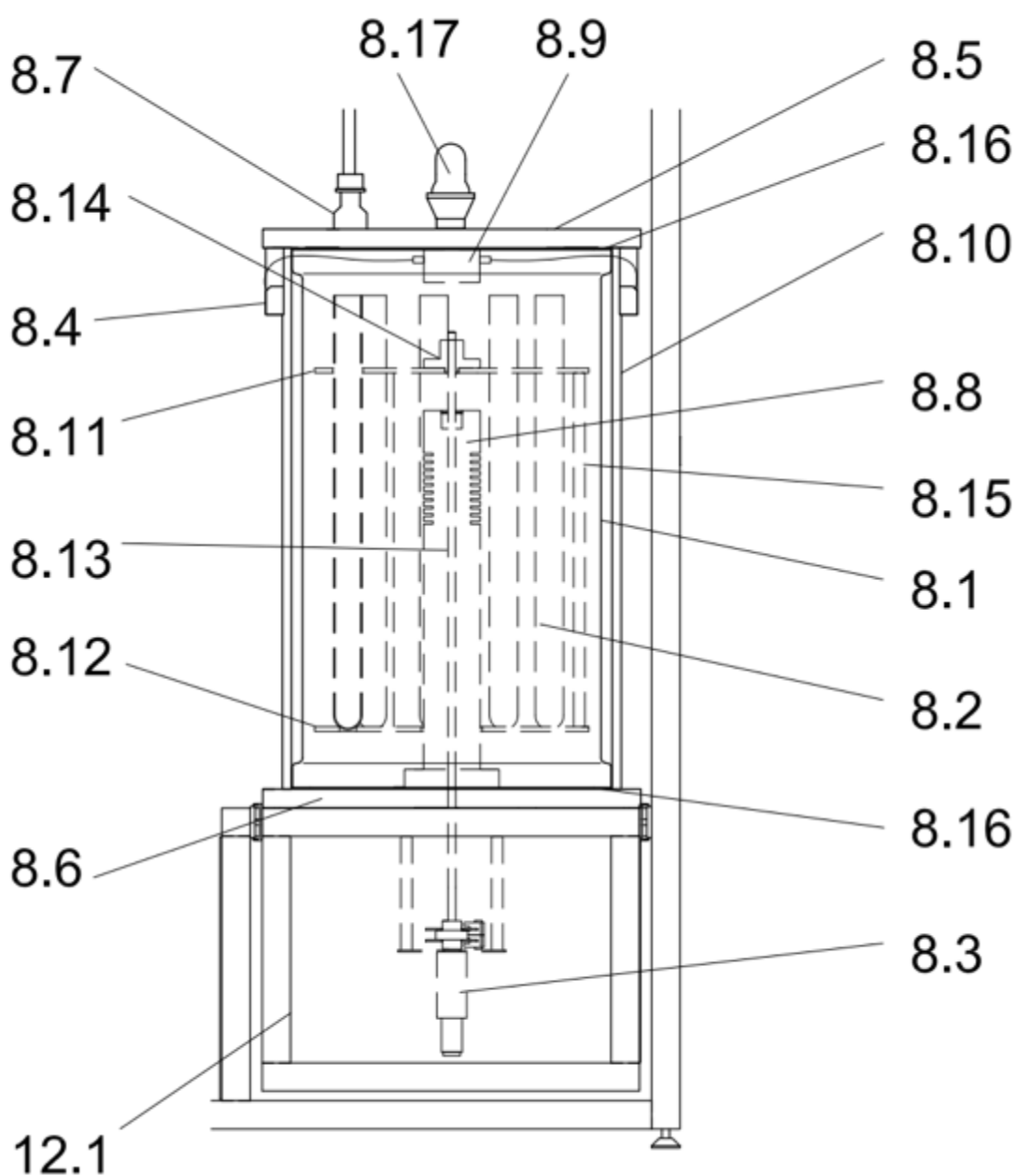


Fig. 14 Fraction collector

Legend to Fig. 14

- 8 fraction collector
- 8.1 covering hood
- 8.2 receiver glass
- 8.3 fraction collector drive
- 8.4 overfilling protection sensor
- 8.5 upper fraction collector flange
- 8.6 lower fraction collector flange
- 8.7 distillate inlet
- 8.9 connection box
- 8.10 centering rods
- 8.11 upper receiver plate
- 8.12 lower receiver plate
- 8.13 motor
- 8.14 holder flange
- 8.16 O-ring 310x8
- 8.17 vacuum adapter, glass
- 12.1 tray for fraction collector

Fraction collector tray is already preassembled.

		CAUTION
	Minimum 2 persons required for fraction collector assembly! Do not forget to install Viton O-rings between hood and both flanges!	

To do that:

1. Pull the fraction collector lock out.
2. Move the tray out from framework. Tray is installed on rails.
3. Put the glass covering hood to a lower collector flange.
4. Close the upper fraction collector flange using 4 guiding rods.
5. Connect the cable to connection box.

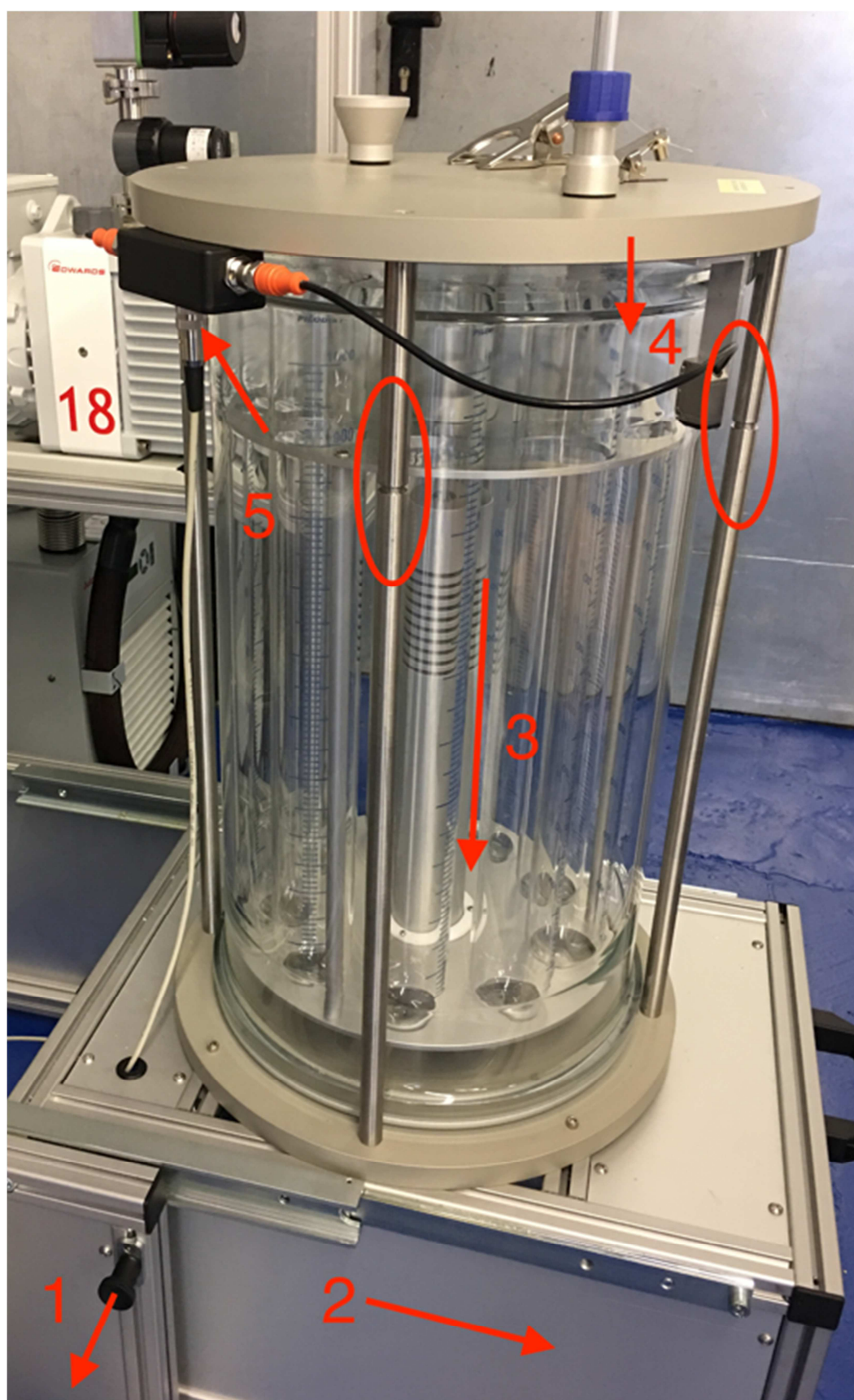


Fig. 15 Fraction collector assembly procedure

During routine operation it is not necessary to disassemble the fraction collector completely but it is necessary to replace receivers after each distillation step.

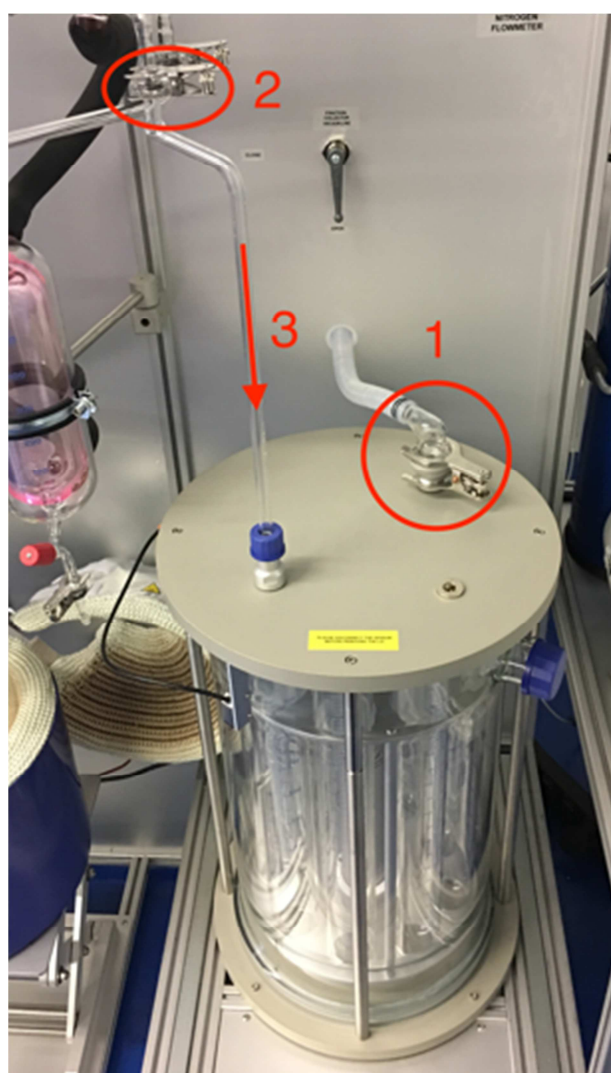
To replace the receivers, follow the procedure:

	<table border="1"><tr><td data-bbox="566 403 678 470"></td><td data-bbox="678 403 1457 470">WARNING</td></tr><tr><td colspan="2" data-bbox="566 470 1457 595">Do not disconnect any line from fraction collector while ventilation valve is closed!</td></tr></table>		WARNING	Do not disconnect any line from fraction collector while ventilation valve is closed!	
	WARNING				
Do not disconnect any line from fraction collector while ventilation valve is closed!					

1. Disconnect the vacuum line from fraction collector.

2. Disconnect the distillate line from distillate cooler.

3. Move the distillate line for several cm deeper into fraction collector to ensure some free space between upper end of line and lower end of distillate cooler.



4. Pull the fraction collector Lock.
5. Slide the collector out of framework a handle in front.
6. Disconnect the wire from connection box.
7. Open the lid and replace the receivers.



After replacing the receiver, assemble the collector with the same step but in reversed.

3.1.5 Receivers

Install 9 receivers into corresponding places in the fraction collector. Please note, that receivers must be clean and dry. It is forbidden to use receivers with dirt/dust/dark crude oil product signs on the walls. It may cause faults in overfilling protection system.

Each place has a number (1 to 9) to prevent mismatching the cuts and receivers after distillation.

3.1.6 Peripheral Devices

The control panel is to be connected to the system via inbuilt RS232 cable. Height of the control panel can be adjusted according average height of the operators.

3.2 Switching-on the System

Before switching the system on make sure that cold traps contain enough of ethanol and all thermostats are filled with associated thermostatic liquids up to a proper level.

The system is switched on by means of the mains switch. Once main switch is turned on the control panel activates. Please wait until software is started.

The glass apparatus should be dry and clean, otherwise a washing run has to be carried out.
(See software manual)

Before the distillation starts, all parameter inputs have to be checked. According to the product an adjustment of the control parameters is necessary.

It is recommended to keep the system idle running for 20-30 minutes after switching on to let cold trap cool down and warm up the vacuum sensors.

		WARNING
	Never use the system without properly working cold traps! The vacuum pump is not suitable for flammable vapours.	

3.3 Vacuum Test

In order to check the leak-tightness of the system, a vacuum test should regularly be carried out. Assemble an empty apparatus (with no sample), close the ventilation valve and activate the vacuum pump. Set point must be set to 0 Torr. Manual throttle must be fully opened. Gas flow selector must be set to DISTILLATION and fraction collector interlock OPENED. If system is capable to create vacuum lower than 1 Torr means no significant leakages exist. Otherwise find and fix the leakage.

3.4 Safety Instructions

In case of appropriate handling, i. e. correct preset of the safety limitations, e.g. flask temperature, the system will automatically shut down itself in case of a failure or after reaching a cancellation criterion. This means that the distillation process will be stopped and the cooling will start.

The cause of the failure will be indicated to the operator by means of system signal such that even hours later a distillation stop can be traced.

Please put on safety glasses when you fill the cold trap or when you work at the system during operation!

Keep protective doors (if ordered) closed during the whole run!

		CAUTION
	Column and distillate lines are made of Borosilicate glass! Do not distill concentrated alkalines (NaOH, KOH...) It may damage the glass! Do not use a hydrofluoric (HF) to clean the glass parts! It may damage the glass!	

		CAUTION
	In this distillation system sealings between flask, column and distillate lines are made of Viton® (FKM/FPM)! Do not use Acetone for cleaning the system! It will damage the sealings!	

4 Software operation manual

4.1 Main Page

Main Page is displayed after opening the Software.

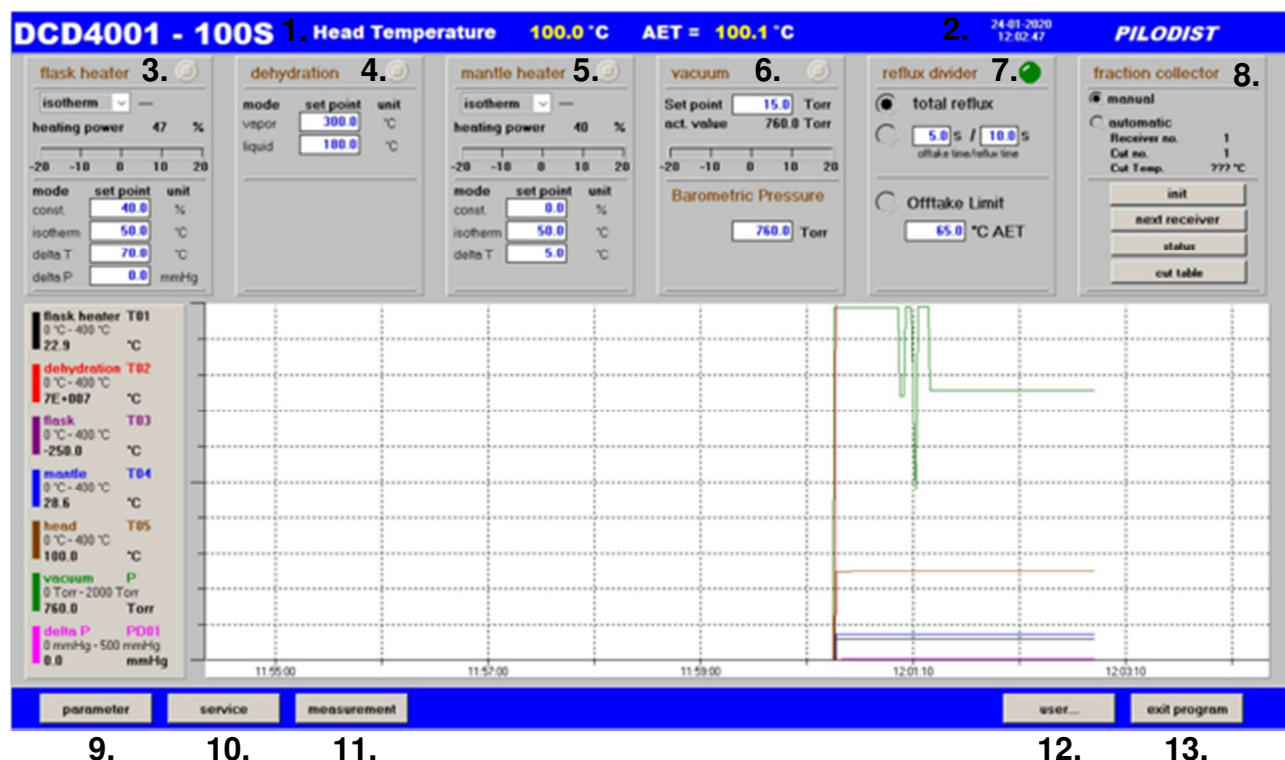


Fig. 15 PD100S Software – Main page

1. **Head Temperature** shows the measured temperature at the head of the column.
2. Displays the **Date** and **Time**.
3. **Flask Heater**: Control of the flask heater temperature. (See chapter 4.2)
4. **Dehydration**: Control of the Dehydration procedure. (See chapter 4.3)
5. **Mantle Heater**: Control of the Mantle heater. (See chapter 4.2)
6. **Vacuum**: Control of the vacuum in the system. (See chapter 4.4)
7. **Control of Reflux divider**: control of **reflux ratio**. (See chapter 4.5)
8. **Fraction Collector**: controls the fraction collector. (See chapter 4.6)
9. **Parameter**: Button to enter the parameter area. Only accessible after logging into the restricted area. (See chapter 4.7)
10. **Service**: Button to enter the service area. Only accessible after logging into the restricted area. (See chapter 4.8)

11. **Measurement:** Button to show the graphical representation of different measurements. (See chapter 4.9)
12. **User Log-in:** Button to activate the restricted areas. (See chapter 4.10)
13. **Exit program:** Button to exit the PD 100 S program. (See chapter 4.11)

4.2 Heater

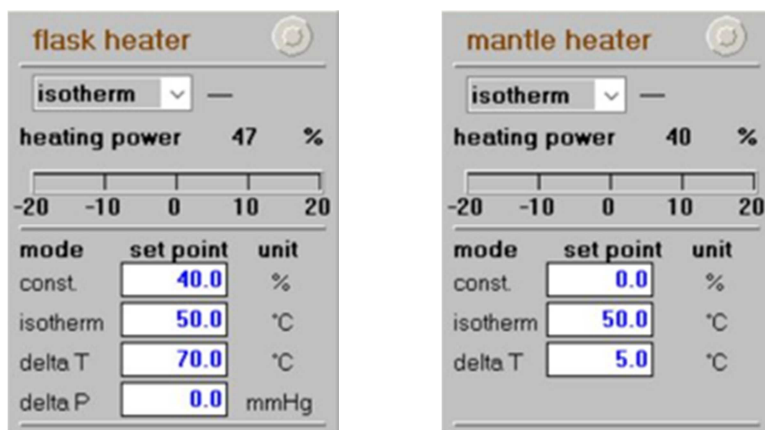


Fig. 16 Heaters

The PD 100 S is equipped with 2 main electrical heaters: *the Flask Heater and the Mantle Heater*. The heater controller can be activated by pressing the associated button on the right top corner of each controller. When the heater is activated, the button becomes blue. The bar in the control display shows the difference between the set point and the actual value. If the bar is red, the heater needs to heat more and if the bar is blue, the heater needs to cool down which means in our case, it stops heating or decreases its heating power.

The heaters can be controlled via four different modes (*-depends on heater and exact application): *Constant* heating mode, *Isotherm* heating mode, *Delta-T* heating mode and *Delta-P* mode. The *Constant* and *Isotherm* heating mode are the same for all four heaters but by choosing the *Delta-T* heating mode, the various heaters are regulated differently. The reboiler has additionally to the three other heating modes the *Delta-P* heating mode. The set point of each heating mode can be inserted in the lower half of the controller display. By clicking into the white box next to the different modes the corresponding set point can be inserted. The heating mode can be chosen by clicking on the small black triangle at the bottom of the control panel. If a mode is chosen, the controller will only read the corresponding set point. Therefore, every box can be filled out and it will not influence the chosen mode.

- **Constant heating mode:** The heater is heating with a constant percentage of its heating power. The heating power is controlled with the time. The heater will heat with full power during a period of time proportionally to the percentage and the rest of the cycle the heater is not heating.

For example: The cycle time is 5 seconds and the percentage is set to 20%. The heater will heat only for 1 second and the remaining 4 seconds it will be switched of.

- **Isotherm heating mode:** The heater is heating with a constant temperature.

- **Delta-T heating mode:** The heating regulation of this mode is different for every heater. (See below).
- **Delta-P heating mode:** The reboiler heats with the necessary power to reach the desired *delta-P*. The *delta-P* is the difference in pressure between the reboiler and the head of the column. The *delta-P* is used to determine the working point of the columns. In case of a higher *delta-P* the working point is higher in the column contrary to a lower *delta-P*.

Flask Heater

The *Flask Heater* heats the product inside the flask. The *Delta-T* heating mode for the *Flask Heater* regulates the heater so that the difference of the Heater temperature and the product temperature inside the flask corresponds to the *Delta-T* set point.

$$\Delta T = T_{\text{Heater}} - T_{\text{Flask}}$$

ΔT = the set point of the *Delta-T* heating mode

T_{Heater} = *Flask Heater* temperature

T_{Flask} = Product temperature (inside the flask)

Mantle Heater

The *Mantle Heater* heats the outer wall of the column to keep adiabatic conditions inside (protecting from heat loss). The *Delta-T* heating mode for the *Mantle Heater* regulates the heater so that the difference of the Reboiler temperature and the column temperature corresponds to the *Delta-T* set point.

$$\Delta T = T_{\text{Head}} - T_{\text{Mantle}}$$

ΔT = the set point of the *Delta-T* heating mode

T_{Mantle} = *Mantle Heater* temperature

T_{Head} = Head temperature (Top of the column)

Please note, using Delta-T mode Mantle heater will keep temperature ΔT below head temperature.

4.3 Dehydration

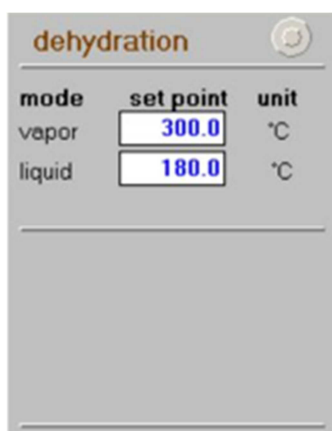


Fig. 17 Dehydration

PD100S system can be equipped with optional water removal feature.

This controller allows to stop the Dehydration process automatically according to ASTM D2892 Appendix X1.

The Dehydration controller can be activated by pressing the associated button on the right top corner of each controller. When the heater is activated, the button becomes blue. Once controller is activated system checks the limits set in this controller and automatically turns the heater off when any of these limits exceeded.

There are 2 windows to set the STOP limits: Vapor and Liquid. Set required values for Max. Vapor temperature and Max. Liquid temperature to be used as STOP limits.

Activate this controller prior dehydration procedure. It will be deactivated automatically when finished.

4.4 Vacuum

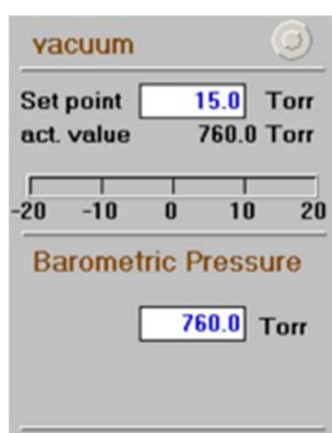


Fig. 18 Vacuum

The Vacuum controller operates with 2 different controls: Vacuum and Barometric pressure.

To apply a vacuum to the system:

1. Click to "Set point" and type required pressure in the system.
2. Activate the controller. Vacuum pump will start.
3. Adjust Vacuum Throttle manually.

4. When set point reached an automatic vacuum valve will keep the vacuum close to set point.

If nitrogen flow through the column is required, open and adjust the flow via nitrogen flowmeter.

		NOTE
	At different pressure levels the nitrogen flow must be adjusted carefully. Do not exceed 0,5 L/min flow, otherwise vacuum set point will not be reached.	

To bring the system to ambient pressure after distillation deactivate the Vacuum controller (vacuum pump will stop), let system cool down and open the ventilation valve.

		CAUTION
	Prior distillation at ambient pressure, make sure that ventilation valve is opened. Otherwise system can be destroyed with overpressure from inside.	

Barometric Pressure window is used to calculate correct AET while distillation at ambient pressure is being performed. Check the Barometric pressure via external Barometer (not included) and type this value into Barometric Pressure window. This way system will cut the sample according preset AET temperatures in Cut table without a need to calculate the corrections manually.

4.5 Reflux Divider

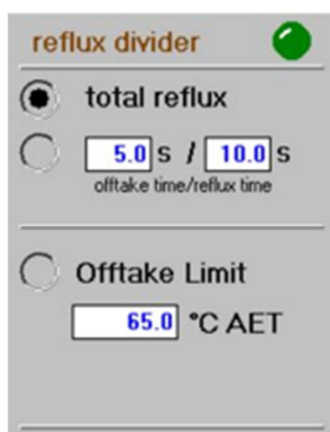


Fig. 19 Reflux Divider

The distillate is extracted from the column via the **reflux divider**. The reflux divider enables the condensate from main condenser to flow back into the column, so that the desired degree of separation can be established, or into the distillate receiver. By default, the reflux setting is set at *total reflux* which means all distillate flows back into the column. By clicking onto the grey circle next to the reflux ratio, the offtake will be started with the integrated reflux ratio. The reflux ratio

consists of the offtake time (the time the reflux divider is opened), which is the first number, and the reflux time (the time the reflux divider is closed), which is the second number. To change the reflux ratio just click to associated window and input a new value.

		CAUTION
	Prior activating the reflux divider make sure that correct path is opened at distillate cooler: to intermediate receiver or to fraction collector!	

4.6 Fraction collector

PD100S Can be equipped with 2 different fraction collectors: 9 or 18 receivers. Both of them are controlled with the same controller. Operation procedures are similar for both collectors.



Fig. 20 Fraction collector

Fraction collector controller has 2 modes of operation: Manual and Automatic.

Manual mode allows to change the receivers at any time on operator's wish.

Automatic Mode changes the receivers according to preset cut temperatures in Cut table without any input from operator's side.

For easy observation of fraction collector status working in Automatic mode there is a small list of 3 main parameters shown:

- **Receiver no.:** displays actual receiver is being used by a system at the moment;
- **Cut no.:** displays actual cut number is being collected at the moment;
- **Cut Temp.:** displays next AET to change the receiver.

Press **INIT** button to set the fraction collector to position #1.

		NOTE
	It may take some time to start the Initialization procedure due to safety check performed by software. Please wait at least for 10 seconds before pressing INIT button again.	

		WARNING
	Never operate with fraction collector while upper lid is opened. It may cause injury!	

Press **NEXT RECEIVER** button to move the collector to next position.

		NOTE
	It may take some time to change the position of the fraction collector due to safety check performed by software. Please wait at least for 10 seconds before pressing NEXT RECEIVER button again.	

Press **STATUS** button to check the status of the fraction collector. A status window will appear.

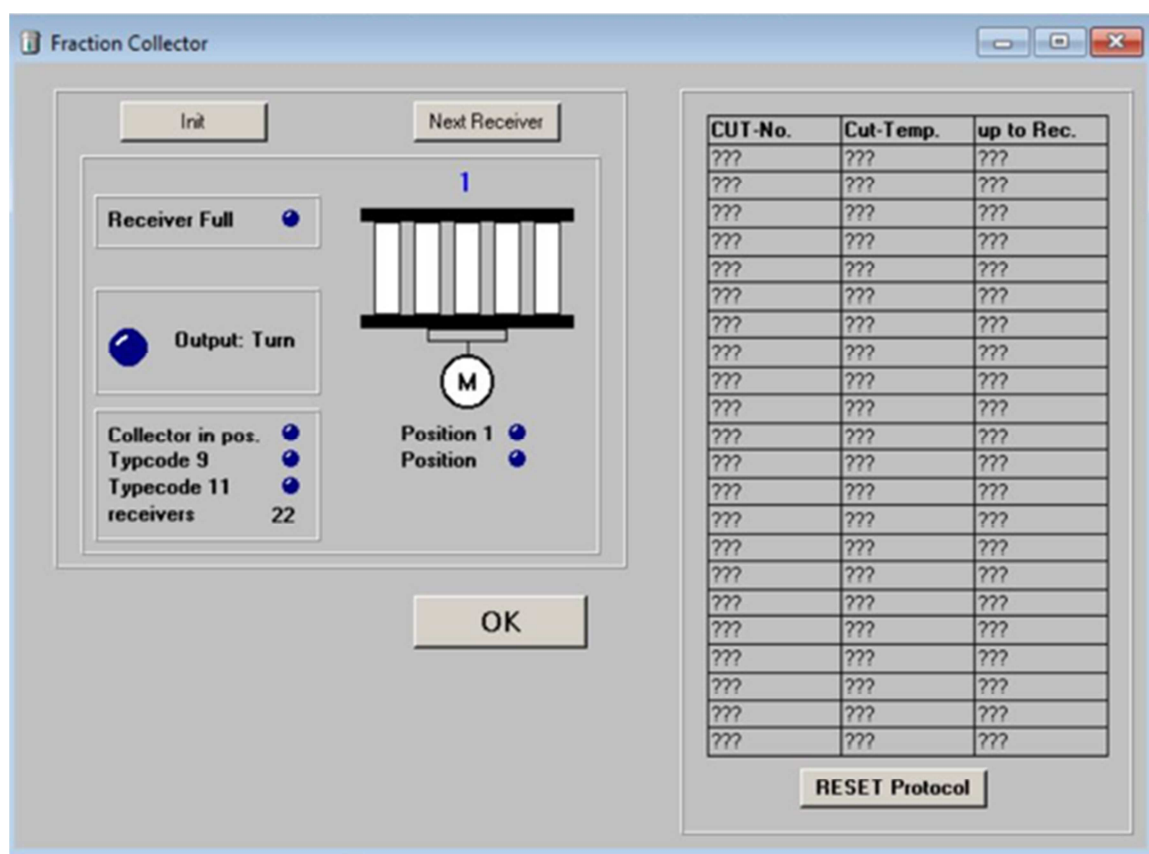


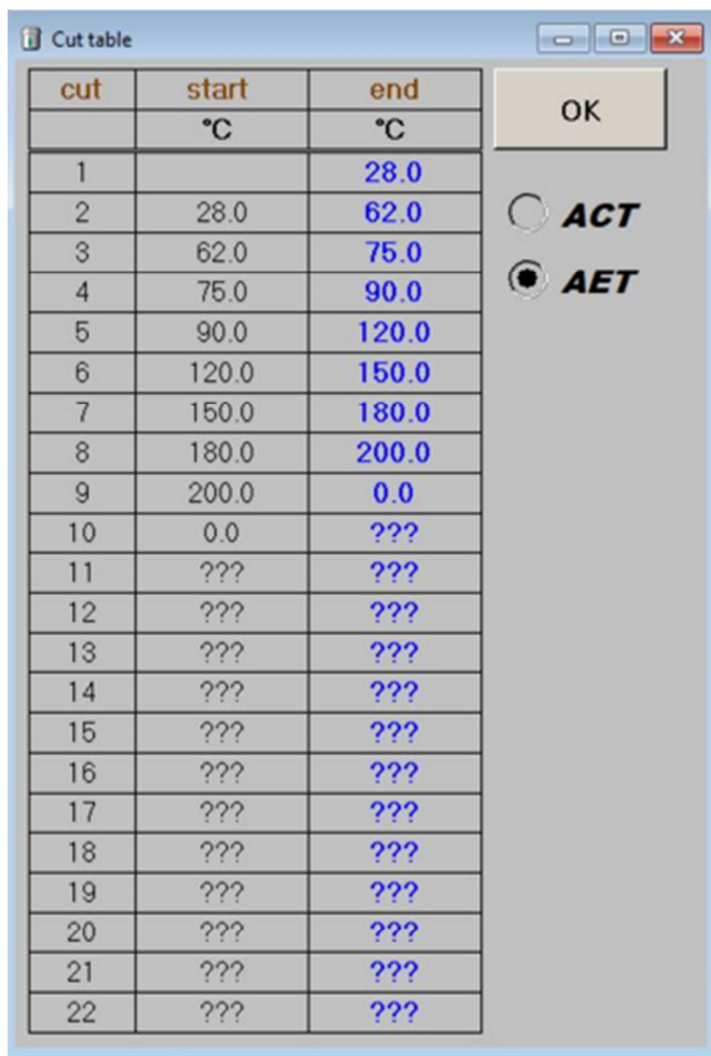
Fig. 21 Fraction collector status

Here You can find all service information of fraction collector: actual position, status of overfilling protection system, signal to turn (motor), INIT position (Position 1). All these signals are given in digital way: once signal is in HIGH status associated LED changes its color from Dark Blue to Light blue.

On the right side of this window you can see actual cut table loaded into fraction collector memory.

		CAUTION
	Always RESET PROTOCOL prior doing next distillation. It will exclude any troubles during the run in automatic mode.	

Press **CUT TABLE** to adjust cut temperatures to be collected into different receiver in automatic mode. A small window will appear.



The 'Cut table' window displays a table with 22 rows. The first three columns are 'cut', 'start', and 'end', all with units in °C. The 'cut' column contains numbers 1 through 22. The 'start' column contains values from 28.0 to 200.0, then 0.0, and then three question marks. The 'end' column contains values from 28.0 to 200.0, then 0.0, and then three question marks. To the right of the table is an 'OK' button and two radio buttons labeled 'ACT' and 'AET'. The 'AET' radio button is selected.

cut	start °C	end °C
1		28.0
2	28.0	62.0
3	62.0	75.0
4	75.0	90.0
5	90.0	120.0
6	120.0	150.0
7	150.0	180.0
8	180.0	200.0
9	200.0	0.0
10	0.0	???
11	???	???
12	???	???
13	???	???
14	???	???
15	???	???
16	???	???
17	???	???
18	???	???
19	???	???
20	???	???
21	???	???
22	???	???

Fig. 22 Cut Table

Here you can choose which temperatures will be used to cut the sample: AET or ACT. Required temperatures to be filled here. Please note, once fraction collector is switched to Automatic mode, system will collect all distillate to Receiver #1 until the end boiling point of Cut #1.

		NOTE
	Always put 0 as the end boiling point of last cut. This is a signal for system to finish the distillation process. System will automatically deactivate reflux divider, stop heating, but will keep vacuum pump running to let flask and column cool down.	
		CAUTION
	Never exceed number of available receivers inside fraction collector while filling the cut table. Once all receivers were used, system will automatically stop the distillation process!	

4.7 Parameter

Press **PARAMETER** button and window will appear.

1.

Flask Heater		
Parameter	Temp.	delta P
Gain [-]	1.50	1.50
Integr.-Time [s]	1000	60
Lower Bound [%]	5	1
Upper Bound [%]	80	60
cyclus time	5	

2.

Mantle Heater	
Parameter	Value
Gain [-]	1.2
Integr.-Time [s]	180
Lower Bound [%]	0
Upper Bound [%]	40
cyclus time	5
Temp. Limit [°C]	300

3.

Limits						
Flask Heater	Dehydration	Mantle Heater	Flask Temp.	Head Temp.	deltaP	Final-Cut
[°C]	[°C]	[°C]	[°C]	[°C]	mmHg	[°C]
450	300	300	400	250.0	300	400.0

Timer	
distillate cooler drain time [s]	10
Fraction collector check time [s]	30
Alarm shut down delay time [s]	3

load parameters save parameters OK

Fig. 23 Parameter

The Parameter window can only be opened when logged in as administrator with the **User log-in** button.

In this area, the heater parameter can be set. The heater parameters (1.) are the same for every heater.

- **Gain:** *Gain* is the parameter which regulates the heating intensity of the heater.
- **Integr.-Time:** The *Integral Time* adjusts the heating intensity of the heater so that heater is able to reach a constant temperature which was set.
- **Lower Bound:** The *Lower Bound* limits the minimal power with which the heater heats.
- **Upper Bound:** The *Upper Bound* limits the maximal power with which the heater heats.
- **Cyclus time:** the *Cyclus time* is the time of one heating cycle.
- **Temp. Limit:** In case the heater reaches the *Temp. Limit*, the heater will heat constantly with this limit temperature.

The limits (2.) are safety parameters: in case of temperature, for any reason, go higher than its *Temp. Limit*, then the unit will stop the distillation.

Please note, that final cut temperature limit is given in AET!

The Timers (3.) are used for proper fraction collector operation:

- Distillate cooler drain time is used to let all distillate get into final receiver prior fraction collector will turn. It is allowed to raise this time delay for very viscous distillates and reduce for light distillates.
- Fraction collector check time is a frequency which fraction collector is checked by software;
- Alarm shut down delay is used for proper interpretation of any alarm signal during the run. It means that Alarm signal must be active constantly for time period listed here. Do not change it without a need.

4.8 Service

Press **SERVICE** button and window will appear.

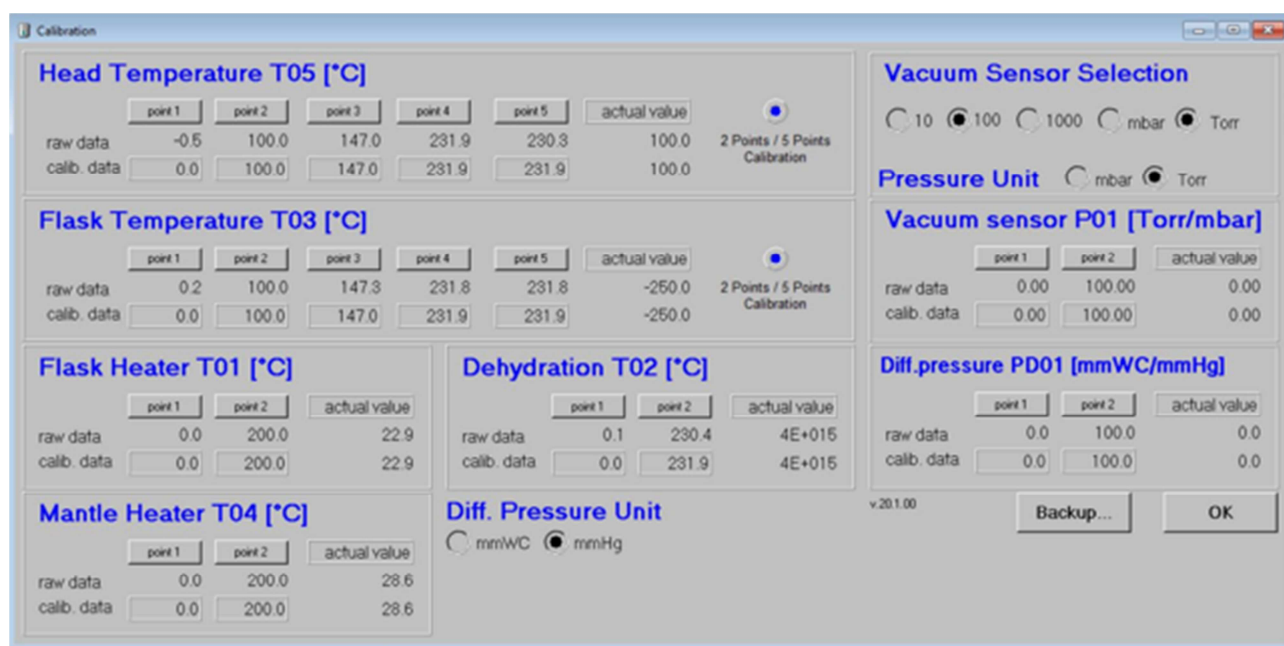


Fig. 24 Service

The service area allows to do a calibration work. To operate in this area, it is required to be logged in as administrator, by using the *User log-in* button (See chapter 4.10).

The user is able to calibrate the vacuum sensor, the differential pressure sensor, different temperature sensors in the heaters, the temperature sensor in the head of the column, flask temperature sensor and so on.

The vacuum sensor is calibrated with an external vacuum testing kit. For further instructions, consult the proper calibration manual.

We do not recommend doing the Vacuum sensor calibration without consulting the PILODIST GmbH service staff.

To calibrate the temperature sensors inbuilt to heaters: Disconnect the PRT you want to calibrate. Plug in a calibration resistor, which simulates 0°C, into this socket. Now click on the button that is marked with "point 1", the value of the resistor will be assumed ("calib data" = 0). Now change the resistor with a resistor which simulates 200°C, and repeat the procedure with the „point 2" button ("calib data" = 200). If a Heater is not used, change the calibration data to "0°", because an open PRT connection means for the control system 850°C which is higher than the limits in the *Parameter* window.

Head temperature sensor and flask temperature sensors are calibrated against melting points of different pure compounds according to ASTM D2892 test method. Additional equipment is required to repeat this kind of calibration. Please, contact to authorised distributor or manufacturer for further information.

In case if a different vacuum sensor used, the service technician can change the settings to the appropriate sensor.

		CAUTION
	Service menu is responsible for proper measurements! Never let unauthorized personnel change the calibrations!	

4.9 Measurement

Press MEASUREMNT button and window will appear.

1.

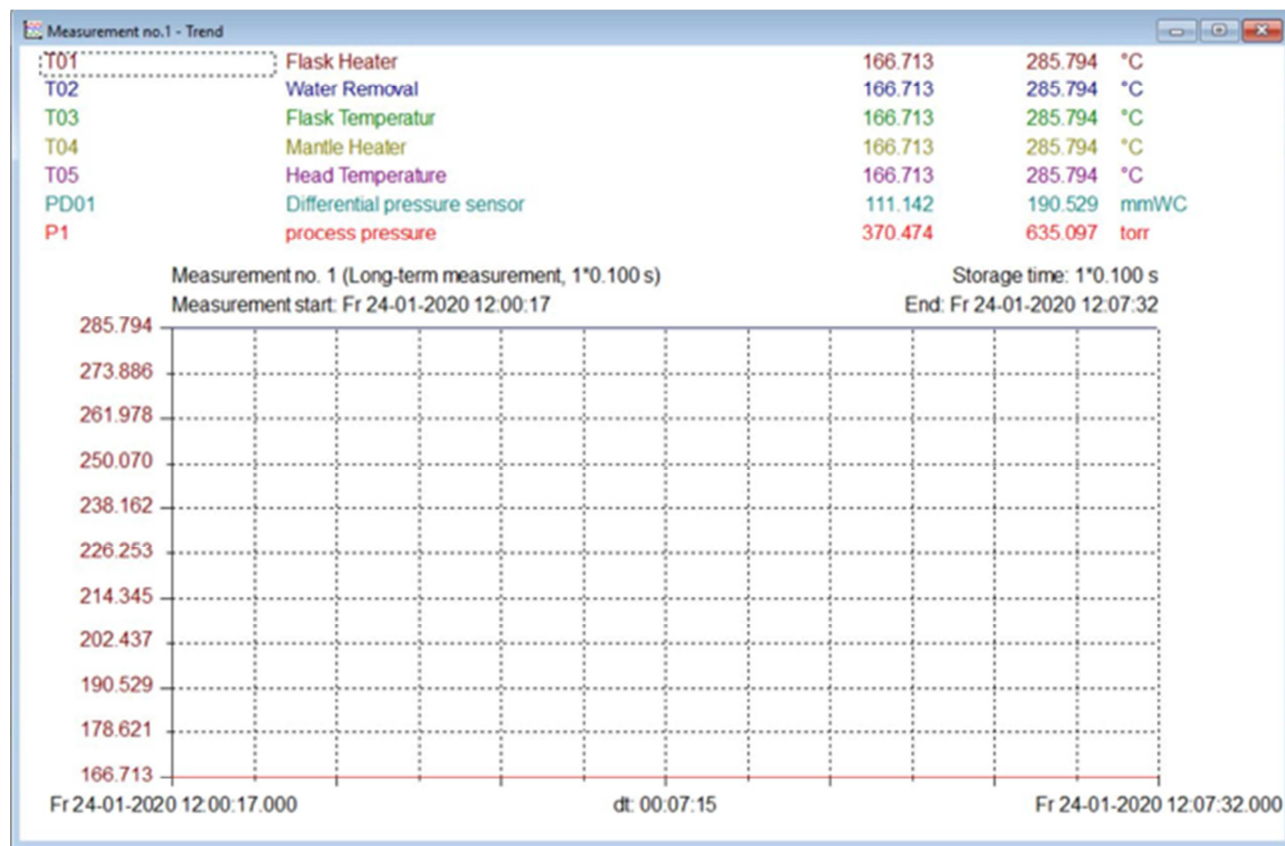


Fig. 25 Measurement

The trend curves for the temperatures or the pressures can be shown via pressing the measurement-button in the bottom row of the main screen. The desired signal needs to be selected to be able to read its data from the graph. To select a signal the user needs to click on the desired signal in the list (1.).

By pressing the right mouse button, a small window will be shown where the user is able to modify the view of the curves. He can choose a specific time range or zoom in by using *Cut Area...*, by using *Origin* the whole measurement will be shown, etc.

4.10 User Log-In



Fig. 26: User Log-in

The buttons *PARAMETER* and *SERVICE* are deactivated by default. To activate these buttons the user has to log himself in as administrator. Therefore, the user has to press the *USER* button and the log-in window (Fig.18) will open.

User: Pilodist

Password: 4711

Type User name and Password to appropriate fields and press OK.

4.11 Exit program

By pressing the Exit button, the PD 100 S software will be closed and the system will be set to standby mode. You can use a PC in a regular way.

5 Distillation

Distillation process according to ASTM D2892 is divided into 4 obligatory steps:

- Debutanization (evaporation and collection of light dissolved gases, commonly C2-C4)
- ATM Run (distillation at ambient pressure)
- VAC 100 Run (distillation at vacuum level of 100 Torr)
- VAC 99-2 Run (distillation at vacuum level between 99 and 2 Torr). It is recommended to distill the sample at 2 Torr to reach 400°C AET without significant cracking.

All stages are performed in the same way except the vacuum equipment, which is used only at VAC stages.

		NOTE
	Best distillation parameters, like heating power, for different samples must be defined by routine operation. Results of distillation are very related to correct parameters.	

5.1 Step-by-Step guide for PD100S:

5.1.1 Flask

1. Check the tare weight of the empty flask (complete with stirrer bar) on the external balance and record it.
2. Fill the sample into the flask (approximately 50% of the flask volume) and record the gross flask weight.
3. By difference between empty and gross weights you can calculate the weight of sample charge.
4. Insert the flask into the flask heater and install the quench cooler.
5. Use the lift to drive the flask to the column. To do this, use the flask ramp and lift up the flask, with your free hand support correct alignment flask connection nozzle and column. As soon as the sealing ring of the column touches the flask, stop the drive, put the ball-joint clamp on the connection and tighten it.
6. Make sure that the stirring bar can rotate freely. To do this activate the stirrer drive by knob located on the front side of the flask heater. If the stirrer bar is touching the quench cooler, correct its position.
7. Place the insulation heater on the upper part of the flask.

5.1.2 Receivers

Check the tare weight of the empty receivers on the external balance and record them.
Note: The filling weight must also be determined after distillation.

5.1.3 Traps

Determine the tare weight of the cold trap receivers and tare weight of gas trap. Record them.

5.1.4 Fraction collector

Install clean, dry and weighted receivers into fraction collector carousel. Assemble the fraction collector as mentioned above and connect it to a distillate cooler.

5.2 Software Settings:

All software adjustments must be done prior any distillation started.

1. Click "INIT" on the fraction collector controller. Fraction collector will turn to Position #1.
2. Click "STATUS" on the fraction collector controller. In opened window press "Reset Protocol" to clear internal memory of fraction collector. Close "STATUS" window.
3. Click "CUT TABLE" on the Fraction collector controller. Enter your desired fraction temperatures in the table starting from 2nd cut. First light cut is usually to be collected into tempered intermediate receiver to reduce losses caused by evaporation at room temperature.

For example, operator needs to collect next cuts:

15-62;
62-75;
75-100;
100-150;
150-200.

Due to first cut is very light and has a boiling temperature lower than ambient, to reduce losses caused by evaporation, it must be collected to intermediate receiver. Fraction collector to be used from second cut. So, cut table must be filled like:

___-75;
75-100;
100-150;
150-200;
200-0.

Do not forget to place 0 at the end of last cut (f.e. 200-0). That is how system detects the end of stage and automatically switches the flask heater off.

The typical temperatures and pressures are the following:

ATM: Atmospheric distillation up to 180 - 200 °C AET

VAC1: vacuum distillation at 100 torr up to 280 - 300°C AET

VAC2: vacuum distillation at 2 Torr up to 380 - 400°C AET

Last cut at each stage must be chosen in such way that 310°C in the flask (liquid temperature) never exceeded. Otherwise, most samples will start cracking.

4. Once last cut from cut table was taken out, the system will automatically stop heating and will cool down.
5. Before starting any distillation, Reset the fraction collector protocol.

5.3 Debutanization and ATM distillation

1. Cool down thermostat to a temperature lower than -20°C.
2. Introduce the cooling finger into Dewar vessel of Gas trap and turn it on. Make sure that gas trap is completely immersed into ethanol (isopropanol). Both valves must be opened on a gas trap. Gas line must be connected to Gas trap INLET.
3. Turn the Gas Flow selector to **Debutanization** position.
4. Turn the Fraction collector interlock to **Close** position.
5. Make sure that Ventilation valve is opened.
6. Activate the stirrer bar and adjust the stirring speed.
7. Open the nitrogen flow using a flowmeter. Adjust flow to 0,3-0,5 l/min.
8. Activate mantle heater in Delta-T mode with 0-5°C set T difference.
9. Activate flask heater in Delta-T mode with appropriate heating rate. Heating rate must be high enough to receive first distillate in the neck between column head and main condenser in 20-50 minutes after start.
10. Check the head temperature. If it was dropped down lower than +15°C turn the thermostat off and wait until head temperature will reach +15°C again.
11. Turn the thermostat ON and check head temperature. If it was dropped down lower than +15°C turn the thermostat off and wait until head temperature will reach +15°C again.

12. Repeat the steps 7 and 8 until head temperature will not drop below +15°C with working thermostat. At this point debutanization process is finished and ATM distillation can be started.
 13. Make sure that distillate flow selector at the end of distillate cooler is set to intermediate receiver and not to fraction collector.
 14. Activate Reflux Divider. It will start operating immediately. Distillate of the first cut will be collected in intermediate receiver.
 15. Check the distillation rate in ml/min (or ml/hour) using a volume graduation and stopwatch. . Adjust a heating rate if necessary.
 16. Please note, once end boiling temperature of first cut is reached, operator must change the direction of distillate flow from intermediate receiver to fraction collector. To exclude any possible troubles caused by missing the correct switching point, you can activate "Offtake Limit" at Reflux Divider controller with end boiling point of the first cut. Once this limit is reached, reflux divider will be automatically switched to Total Reflux, but flask heater will continue to heat. Take-Off process will be just paused. This feature is to ensure that operator will never miss this switch point.
 17. Change the distillate flow direction at the end of distillate cooler to fraction collector.
 18. Switch the fraction collector to Automatic mode. Reflux divider will start the take-off immediately. All distillate will be collected from now into fraction collector receivers according to Cut table. Once end boiling point of each cut is reached, the take off will be paused for 15-20 seconds and fraction collector will change its position to next receiver. Then take-off will be continued.
 19. Check the distillation rate in ml/min (or ml/hour) using a volume graduation and stopwatch from time to time. Adjust a heating rate if necessary.
 20. Once Head temperature reached 60-100°C, switch gas flow selector to Distillation mode and fraction collector interlock to OPEN position.
 21. Once head temperature reached 65°C it is allowed to raise the thermostat temperature up to 0...+5°C.
 22. When last cut from cut table is distilled out, the system will discontinue the take off and turn the flask heater off automatically. If external thermostat for forced flask cooling is used – activate it. Otherwise, keep the system cooling until flask temperature is decreased lower than 90°C at least.
 23. Close the nitrogen flowmeter.
 24. Open the fraction collector and weight the receivers. Calculate and record the cut weights.
 25. Remember: first cut, collected to intermediate receiver must be transferred out and weighted in a precooled completely sealed receiver. It is to stored only in a freezer.
-

26. Close both valves on a gas trap and disconnect the gas line. Weight the gas trap and calculate the weight of Gas cut.

5.4 VAC Distillation.

1. Adjust thermostat to a proper temperature in such way to exclude any solidification of the distillate. Recommended temperatures for VAC1 (100 Torr): 20-30°C; For VAC2 (2 Torr): 60-70°C.
2. Close the ventilation valve.
3. Distillate flow must be switched to fraction collector.
4. Make sure that both immersion cooler fingers are completely immersed into cold traps filled with ethanol/isopropanol (if used). Turn the immersion coolers on.

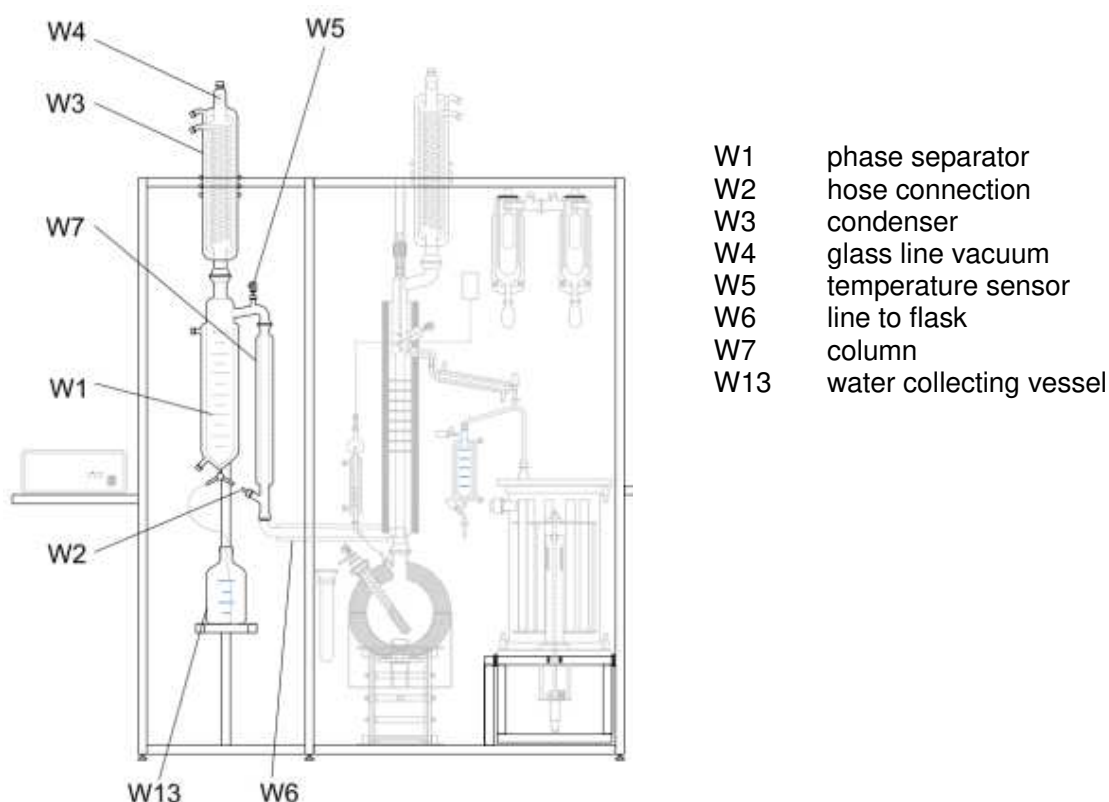
Otherwise, fill the cold traps with dry ice.

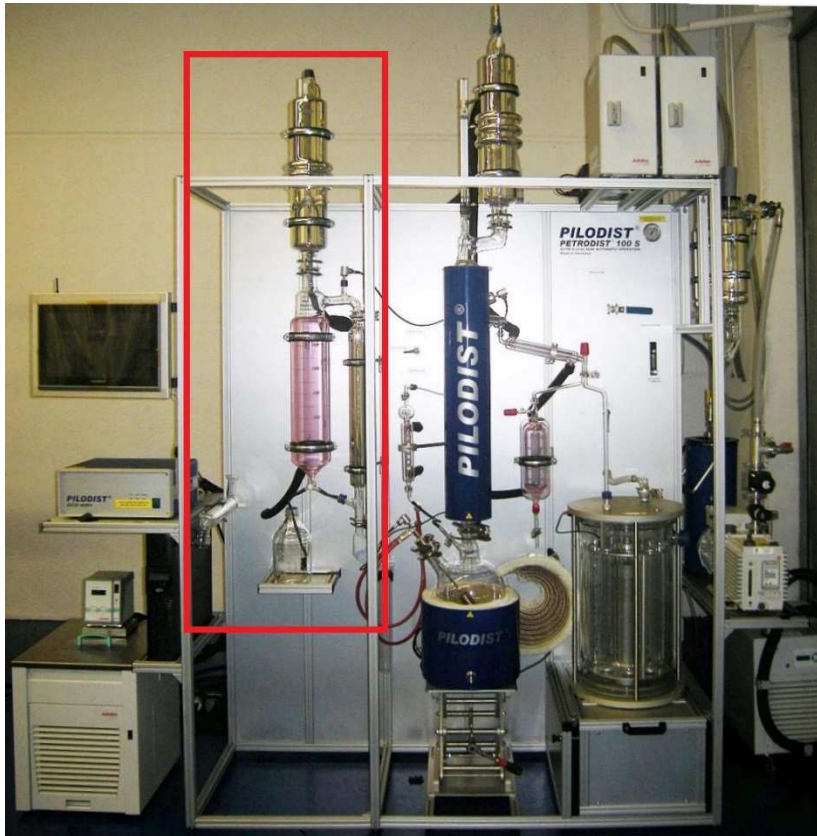
5. Close the Vacuum throttle completely by rotating it clockwise.
6. Put required pressure value into vacuum setpoint window of Vacuum controller.
7. Make sure that Gas flow selector is switched to Distillation position and fraction collector interlock is OPENED.
8. Activate Vacuum controller. Vacuum pump will start immediately.
9. Open the vacuum throttle gently to such level, that system enable to reach the vacuum set point and bounce around it. Please, keep patience doing it first time. In result of wrong adjustment, you will get significant pressure instability.
10. Open and adjust the nitrogen flow on a flowmeter to 0,2-0,3 l/min.
11. Adjust the position of vacuum throttle if necessary.
12. Activate flask heater in Delta-T mode with appropriate value.
13. Activate Mantle heater in Delta-T mode with 0-5°C T difference.
14. Wait for the first distillate in the column head.
15. Keep column refluxing until stable conditions (pressure drop, head temperature) are reached, or at least 10 minutes.
16. Switch fraction collector to automatic mode. Take off process will start immediately.
17. Check the distillation rate in ml/min (or ml/hour) using a volume graduation and stopwatch from time to time. Adjust a heating rate if necessary.

18. When last cut from cut table is distilled out, the system will discontinue the take off and turn the flask heater off automatically. If external thermostat for forced flask cooling is used – activate it. Otherwise, keep the system cooling until flask temperature is decreased lower than 90°C at least.
19. Close the nitrogen flow.
20. Once the flask was cooled down, deactivate the vacuum controller and open the ventilation valve gently.

		WARNING
	Never try to disassemble the system which is under vacuum! It may cause injury or death! Always check the analogue vacuummeter to ensure that system is at ambient pressure every time before touching the instrument. Analogue vacuummeter must show 0 if inside pressure is equal to ambient!	

5.5 Water removal (optional)





Water removal part of instrument is used to reduce a water content in crude oil sample to appropriate level required by ASTM D2892.

8.5 ASTM D2892: "If the water content exceeds 0.3 % volume, the sample shall be dehydrated prior to fractional distillation."

In case if this option was ordered (separately) the dehydration part will be integrated into the system frame. This part consists of:

- Additional low separation efficiency column;
- Additional condenser;
- Water trap, double jacketed, with separation valve;
- Adapter, made of glass, to connect the flask to additional column.
- Set of water lines;
- Set of gas lines;
- Additional vapor temperature sensor.

The Dehydration procedure to be performed prior regular distillation.

The Dehydration procedure principle is based on evaporation of light naphtha cut together with water through the low separation efficiency column up to 130°C vapor temperature maximum. Water, together with naphtha, is condensed and collected in water trap in emulsion state. Due to very high difference in densities and very low inter solubility coefficient between water and light hydrocarbons, the emulsion is separated essentially to 2 non-mixing layers: water layer, located in the bottom, and hydrocarbons layer, located on the top. Then water discarded and regular distillation can be performed.

To do so, please, follow the procedure:

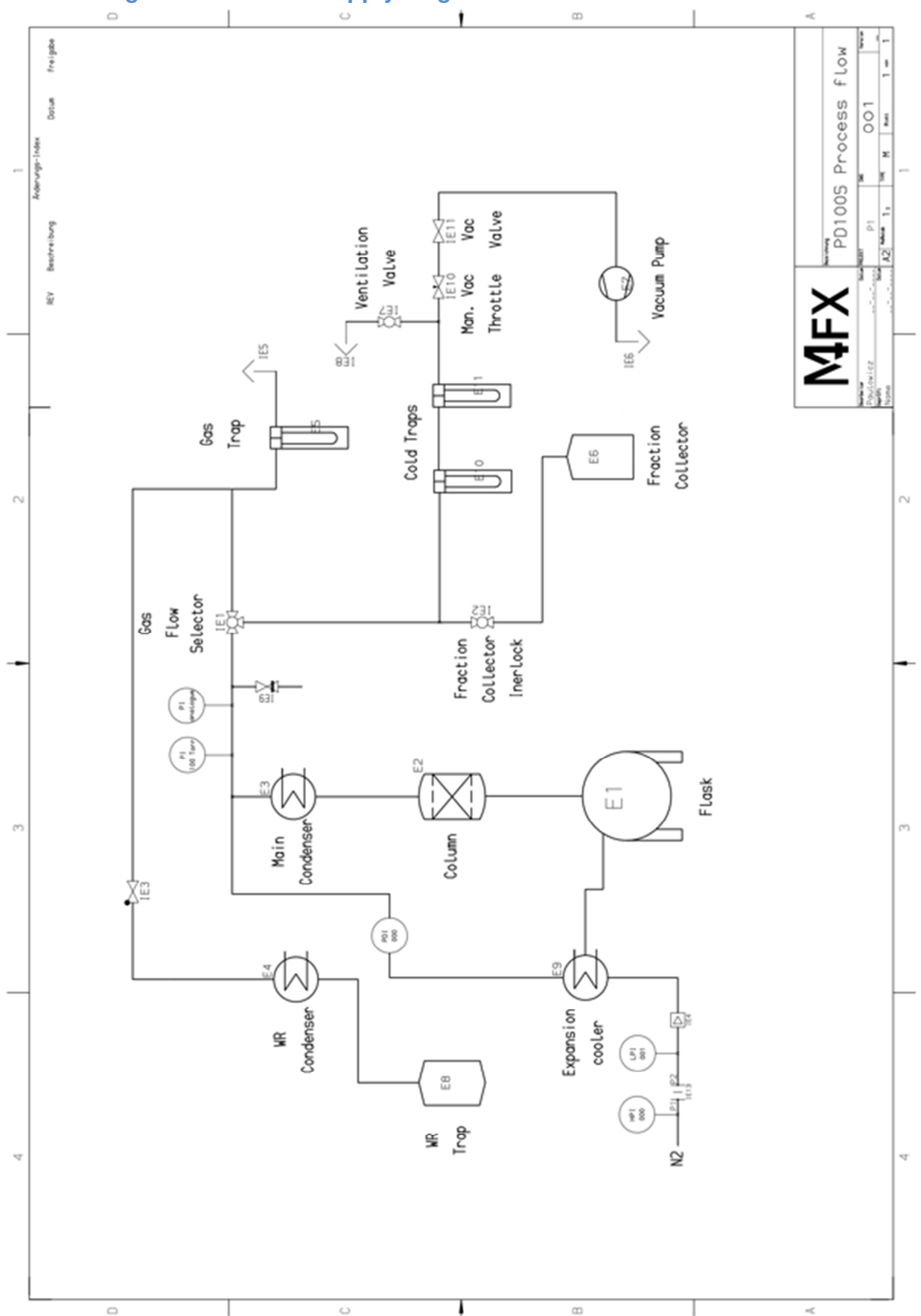
1. Prepare the instrument according 5.1.1 to 5.1.4 steps of this manual, but instead of connecting the flask to main column, connect it to special glass adapter to dehydration column.
2. Using the DEHYDRATION controller in the software set maximum head temperature of vapors to 130°C.
3. Maximum liquid temperature must be set to any level (*depending on a sample properties), but not higher than 310°C.
4. Change the position of water flow selector to DEHYDRATION.
5. Change the position of gas flow selector to DISTILLATION.
6. Change the position of fraction collector interlock to CLOSED.
7. Make sure that separation valve located at the bottom of water trap is completely closed.
8. Introduce the cooling finger into Dewar vessel of Gas trap and turn it on. Make sure that gas trap is completely immersed into ethanol (isopropanol). Both valves must be opened on a gas trap. Gas line must be connected to Gas trap INLET.
9. Adjust a nitrogen flow to 0,3-0,5 l/min.
10. Cool down the main thermostat not lower than +5°C.
11. Activate the FLASK HEATER controller with appropriate ΔT .
12. Activate the DEHYDRATION controller.
13. The dehydration procedure will be performed automatically and when 130°C of vapor temperature reached, the system will automatically stop heating (deactivate flask heater controller).
14. Cool down the flask to a temperature of +5°C using quench cooler and tap water supply (or external thermostat if used).
15. Make sure that emulsion in water trap is separated and You can clearly see the border between water and oil layers. If not, you can change a main thermostat temperature to higher value to speed up this process. But do not raise it too much, because of possibility to lose some hydrocarbons due to very low boiling temperatures of some of them.
16. Using the dividing valve let the water flow out from the water trap to separate vessel (part of supply).

17. When all water is drained, change the flow to a flask and return the naphtha back to flask. But do it when flask was already cooled down only. Otherwise naphtha will be evaporated immediately.
18. Disconnect the glass adaptor from the flask and connect the flask to a main column.
19. Continue distillation in regular way from Debutanization step (5.3 of this manual).

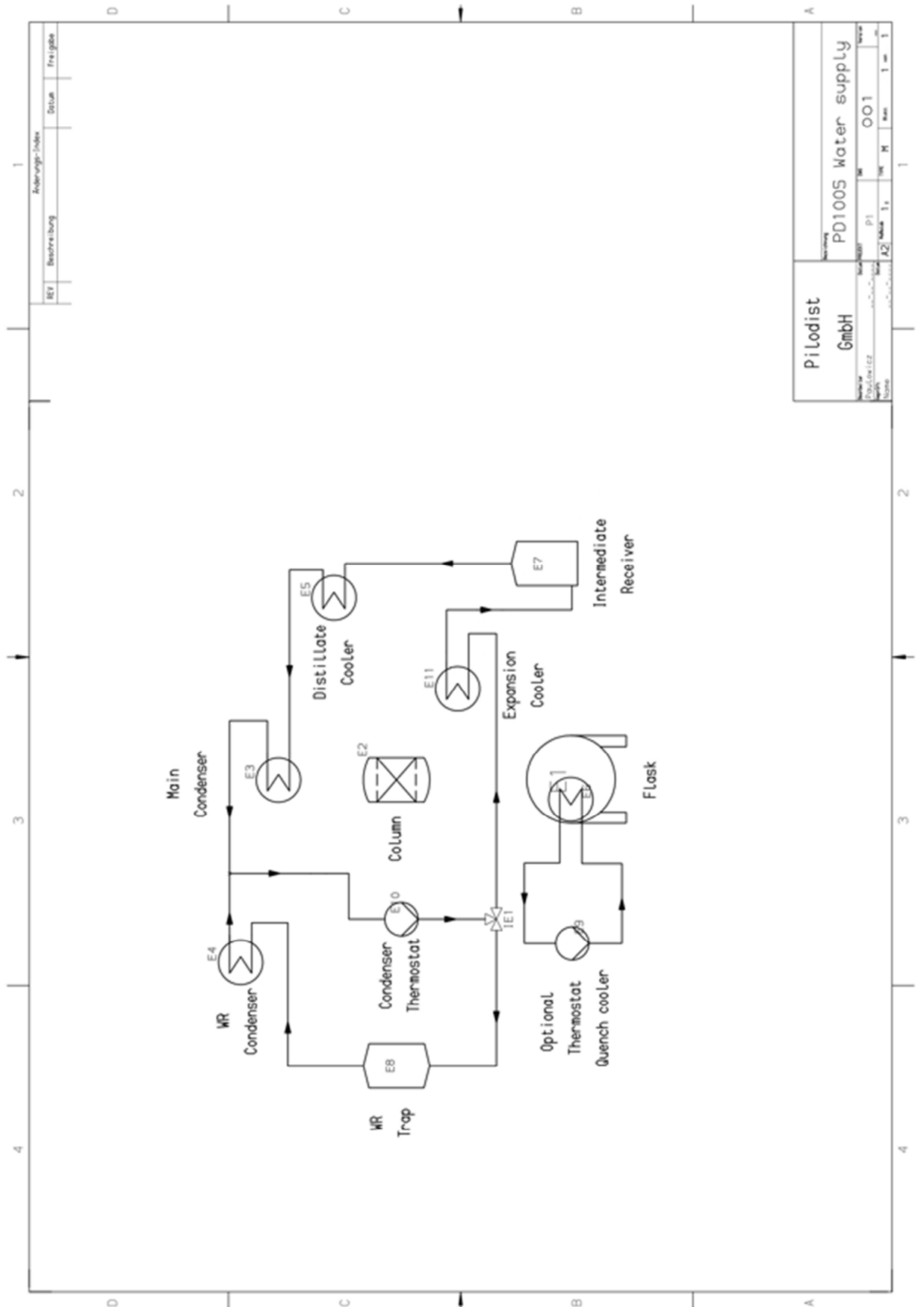
6.1.1 Process flow diagram



6.1.2 Nitrogen and vacuum supply diagram



6.1.3 Water supply diagram



6.2 Service before utilization

1. Any service must be performed only when system is in standby mode or turned off. Do not make any service during the run or while system is hot. Wait until system will cool down.

		WARNING
	Do not open the control cabinet if system is turned on. Turn it off completely, disconnect the power supply and then maintenance of control cabinet can be performed.	

2. Damage check of glass-flask and glass-receivers is strongly recommended before each test. In case of damaged glass found exchange it with a new one. Even minor cracks can falsify the results or malfunction for example no vacuum or set off the alarms. Breaking glass can lead to harm and injury of the health!

		WARNING
	The glass equipment must be inspected very carefully and before each run. In case is any scratch or damage discovered change the part immediately!	

3. Defective sealings have to be exchanged.
4. Control of the liquid level in the cryostats and the cold trap. Check of the water content of the cooling liquid (recommended Ethanol) in the cold trap with a densitometer and refill the cooling liquid when the water content exceeds 10%. Freezing water in the cooling liquid may damage the cold traps and the isolation of the gas trap.

		CAUTION
	The cold trap reaches a temperature below -40°C. Do not touch the cold trap during the operation.	

5. Power supply and nitrogen supply check must be performed on a regular base. If necessary exchange the nitrogen storage tank or fix the leakage on the supply tubes to regain the required pressure. For the nitrogen at least 1,5 bar are needed. Concerning the water for the quench cooler the pressure should not exceed 1,0 bar because of the resistance of the silicon tubes.

6. The vacuum pump oil should be renewed every 12 months (with high workload, refill the pump 2 times per year). Depending on the soiling rate the exchange interval should be decreased. For that the pump needs to be switched off, unplug the power supply and disconnect the vacuum connection from the distillation system. Then place the pump on the working table and open the oil drainage screw under the oil level gauge. Collect the old oil with a beaker and close the oil outlet. Fill new oil into the pump through the oil inlet on the top of the pump up to the desired level. The proposed vacuum oil for the pumps is Edwards *"Ultra Grade Oil 19"*.
7. The fraction collector and the bottling system should be checked regularly by its functions and cleanness. If carousel doesn't move, make sure that coupling between motor and drive shaft is not damaged.
8. Service and maintenance on additional equipment:
Concerning the additional equipment like the cryostat, the UPS, the immersion cooler, balances and vacuum pump consult the maintenance and service manual of the respective producer.

6.3 Installation and calibration instructions

6.3.1 Adjustment of the fraction collector

The receiver has to be positioned exactly under the inlet distillate line to fraction collector. In case of a small deviation of the position the receiver can be adjusted by changing the angle of the carousel, fixed by screw to a drive shaft. To do so, carousel can be rotated against drive shaft for better position. Be assured that the light beam of the overfilling protection sensors should always be guaranteed.

6.3.2 Calibration service

Log into the service area with:

User: pilodist

Password: 4711

Go to Calibration page.

6.3.3 PRT calibration

To calibrate the PRTs you may need an additional equipment: ice water, melting oven for Sn and Pb.

Switch to the service area. Immerse the PRT you want to calibrate into the calibration area (ice water). Wait until temperature will stabilize. Now click on the button that is marked with "point 1", the value of the correction will be assumed. ("calib data" = 0). Now change the calibration area to Sn or Pb and repeat the procedure with the "point 2 " button ("calib data" = melting point of Sn or Pb).

6.3.4 Vacuum sensor calibration

Usually vacuum sensors no need to be recalibrated. But zero point adjustment is required from time to time.

Special test kit to connect an additional reference vacuum sensor with digital display to the PETRODIST system for testing and adjusting the system vacuum sensor zero point.

Content

- Reference Sensor (accuracy 0.25% of reading)
- Vacuum Sensor Cable (or power supply)
- Y-distributor with sealing
- GL25 adapter with sealing and screw cap

Procedure

Mount the *Y-distributor* (Fig. 1) with the *KF25 connection* directly to the vacuum pump.



Fig. 7: Y-distributor

Mount the system sensor into the *GL25 adapter* (Fig. 2) and connect it to one of the two upper connections of the *Y-distributor*. Then connect the *Micro Pirani reference sensor* (Fig. 3) onto the other distributor connection.



Fig. 8: GL25 adapter



Fig. 9: Micro Pirani reference sensor

Tighten all GL25 screws smoothly.

Connect the sub-D male plug from the *Vacuum Sensor Cable* to the sub-D female plug, which is connected to the control cabinet. Then connect the two sub-D female plugs of the *Vacuum Sensor Calibration Cable* to the proper vacuum sensors on the *Y-distributor*.

Start the vacuum pump with the unit's software where the set point is to set to 0 Torr. When the vacuum has been stabilized and the value on the *Pirani reference sensor* is below 0,01 torr.

MKS Sensor:

Adjust the zero point of the vacuum sensor with a small screw driver by turning the small screw on top of the sensor. Adjust the sensor so that the value in the unit's software shows 0 torr.

PILODIST Sensor:

In the service area → Calibration 1 → Vacuum sensor 100 [Torr] push the button "point1".

Remount the sensor back to the system and keep the reference sensor clean and capsuled.

Now the zero point adjustment is done.

6.4 Change of column filling material

Before exchanging the column filling the column needs to be cleaned with a washing run and dried with a vacuum test. The next step is the disassembling of the reflux divider. Firstly, the electromagnetic magnet needs to get removed so that the protection cap and the reflux divider can be carefully pulled out of the column. Now the head temperature sensor can be removed carefully and should be stored in a protection tube. Disconnect the distillate cooler. Disconnect and move the main condenser to a side.

Afterwards the column can be carefully lifted out of the mantle heater and put down onto proper bedding. The foam breaker inside the column not pressed in too tight so that it can be easily removed with a flat plier with a turning movement.

The old filling should be collected in a collecting vessel. Make sure that there is no old packing left in the column. Now the new filling can be put into the column. Then the foam breaker can be pressed into the column so that it is fixed tight enough and does not fall out of the column.

The integration of the column is the same procedure as the disassembling but in backwards.

7 Trouble Shooting

The system is equipped with an online self-diagnostics feature. This feature monitors the condition of the system and if any trouble appeared it sends a pop-up message on the main screen. Safety limits are fully adjustable by authorized representative of end-user. Make sure that these limits have correct values.

		WARNING
	Do not try to solve any errors until system is completely cooled down and then ventilated. It may cause injury!	

POSSIBLE ERRORS

Message	Description	Reason	Possible action
Maximum Flask heater temperature reached	System detected that flask heater temperature is higher than safety limit.	Flask heater is too hot.	Wait until flask heater will cool own.
		Flask heater temperature sensor is damaged.	If shown value is around 850°C, flask heater must be replaced.
Maximum Dehydration temperature reached	System detected that dehydration PRT temperature is higher than safety limit.	Dehydration column is too hot.	Wait until column will cool own.
		Dehydration temperature sensor is damaged.	If shown value is around 850°C, sensor must be replaced.
Maximum Mantle heater temperature reached	System detected that mantle heater temperature is higher than safety limit.	Mantle heater is too hot.	Wait until Mantle heater will cool own.
		Mantle heater temperature sensor is damaged.	If shown value is around 850°C, sensor must be replaced.
No power supply	Power supply failed.	No power supply	Check the power supply.
		RCD breaker detected the leak.	Check all power connections and reset the RCD breaker.
Maximum Flask temperature reached	System detected that flask temperature is higher than safety limit.	Flask is too hot.	Wait until flask will cool own.
		Flask temperature sensor is damaged.	If shown value is around 850°C, flask temperature sensor must be replaced.
Maximum Head temperature reached	System detected that head temperature is higher than safety limit.	Column is too hot.	Wait until column will cool own.
		Head temperature sensor is damaged.	If shown value is around 850°C, head temperature sensor must be replaced.

		NOTE
	All solenoid valves in the system are normally closed! If NO POWER SUPPLY error appeared and due to this system was turned off it will stay closed under vacuum. Let it completely cool down first and then turn it on again.	

Errors listed below have an adjustable limits! Visit SERVICE menu of the PD100S software to adjust these limits according to local requirements:

- **Flask temperature too high**
- **Vacuum loss during distillation**
- **Temp. diff. flask-head is too high**
- **Max. evacuation time exceeded**

		WARNING
	Do not exceed 400°C of liquid temperature! It may cause strong cracking of the product and damage the flask as well as vacuum equipment!	