PILODIST®

laboratory & process technology

Operation Manual

PD 250 BB



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1 Figure PD 250BB

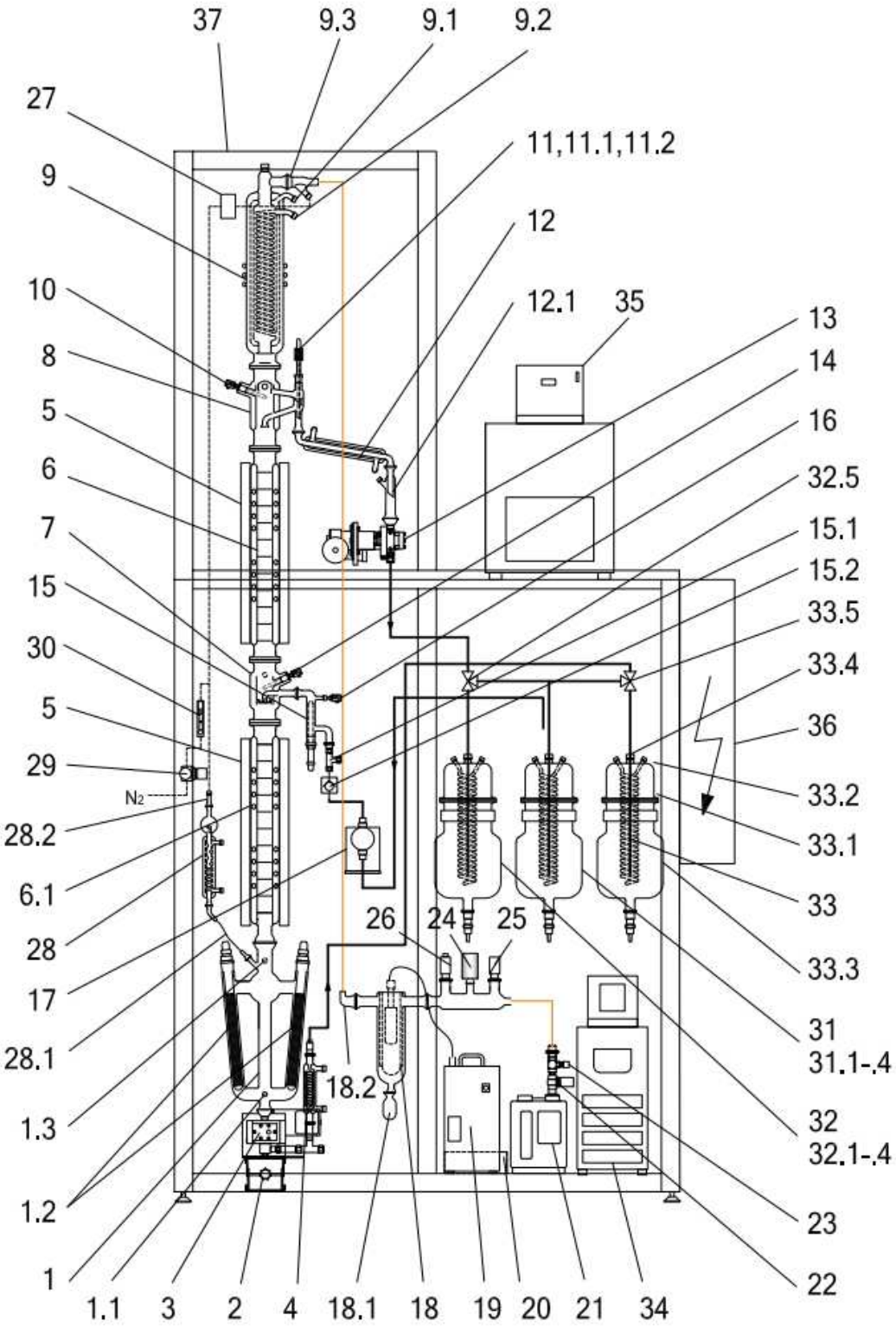


Fig. 1: Drawing of PD 250BB

Legend to Fig. 1

1	reboiler
1.1	residue temperature sensor
1.2	reboiler heater
1.3	residue level sensor
2.	discharge pump lift
3	discharge gear pump for bottom product take off
4	residue discharge bottom product cooler
5	column mantle heaters
6	packed column upper part
6.1	packed column lower part
7	feed segment
8	distillation head with reflux divider
9	condenser
9.1	cooling liquid temperature sensor outlet
9.2	cooling liquid temperature sensor inlet
9.3	vacuum connection piece
10	head temperature sensor
11	valve plunger
11.1	solenoid coil
11.2	valve plunger hood
12	distillate cooler
12.1	distillate line
13	distillate discharge pump
14	column temperature sensor
15	feed heater
15.1	T-piece
15.2	check valve
16	feed temperature sensor
17	feed pump
18	cold trap
18.1	condensate trap
18.2	vacuum connection piece
19	immersion cooler
20	transformer
21	vacuum pump
22	vacuum valve
23	vacuum throttle valve
24	vacuum sensor
25	ventilation valve
26	pressure relief valve
27	differential pressure sensor
28	safety cooler
28.1	connection line safety cooler - evaporator
28.2	connection piece safety cooler - diff. pressure sensor
29	nitrogen inlet valve
30	nitrogen flow meter
31	feed vessel (raw product)
31.1	feed charge balance
31.2	upper part of feed vessel
31.3	drain valve of feed vessel

31.4 glass lines feed vessel
32 distillate receiver vessel
32.1 distillate receiver balance
32.2 upper part of distillate receiver vessel
32.3 drain valve of distillate receiver vessel
32.4 glass line receiver vessel
32.5 2/3-way-valve
33 residue collection vessel
33.1 residue charge balance
33.2 upper part of residue collection vessel
33.3 drain valve of residue collection vessel
33.4 glass line residue collection vessel
33.5 2/3-way-valve
34 thermostat (receiver vessels)
35 thermostat (condenser)
36 control cabinet
37 mounting frame

2 Introduction

The new windows based PD250BB control software is an user friendly and self-explaining software.

It is based on the strategy of easy operation and high flexibility.

In this manual the software screens will be described to give the operator an overview of the handling and the functions.

The software has been already installed on the PC system. To start the PD250BB Software, just use the Icon on the windows screen.

All parameters and data shown in this manual are just samples and cannot be used for data preparation for a distillation run.

3 Main Page

The main page is displayed after opening the PD250BB software.

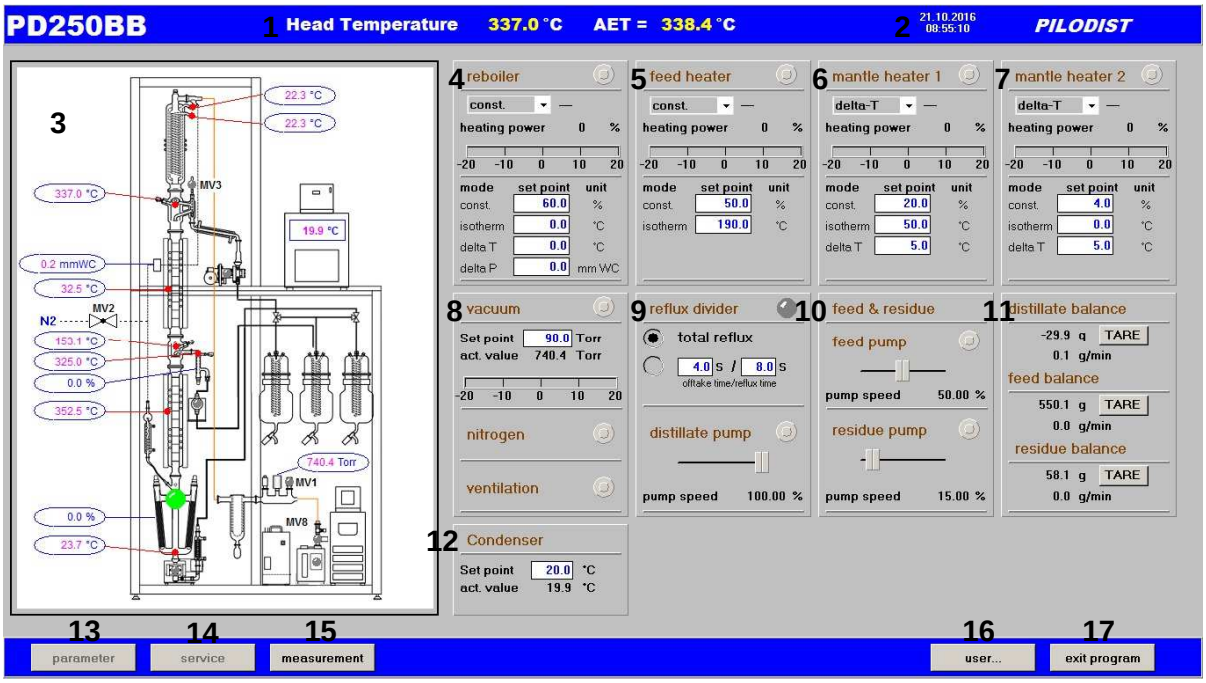


Image 1: Main Page

1. **Head Temperature** shows the measured temperature and the calculated atmospheric equivalent temperature at the head of the column.
2. Displays the **Date** and **Time**.
3. **Signal Display** of the PD250BB unit. (See chapter 3)
4. **Reboiler:** Control of the reboiler heater temperature. (See chapter 4)
5. **Feed Heater:** Control of the feed heater temperature. (See chapter 4)
6. **Mantle Heater 1:** Control of the mantle heater 1 temperature. (See chapter 4)
7. **Mantle Heater 2:** Control of the mantle heater 2 temperature. (See chapter 4)
8. **Vacuum:** Control of the pressure in the unit. (See chapter 5)
9. Distillate discharge control: Control of the **reflux divider** and the **distillate pump**. (See chapter 6)
10. **Feed & residue:** Control of the feed and residue pump. (See chapter 7)
11. **Balances:** Control and display of the distillate, feed and residue balance. (See chapter 8)
12. **Condenser:** Control of the main thermostat. (See chapter 9)
13. **Parameter:** Button to enter the parameter area. Only accessible after logging into the restricted area. (See chapter 10)

14. **Service:** Button to enter the service area. Only accessible after logging into the restricted area. (See chapter 11)
15. **Measurement:** Button to show the graphical representation of different measurements. (See chapter 12)
16. **User:** Button to activate the restricted areas. (See chapter 13)
17. **Exit program:** Button to exit the PD250BB program. (See chapter 14)

4 Signal Display

The signal display displays the values or states of the different analogue (numbers) and digital (letters) signals.

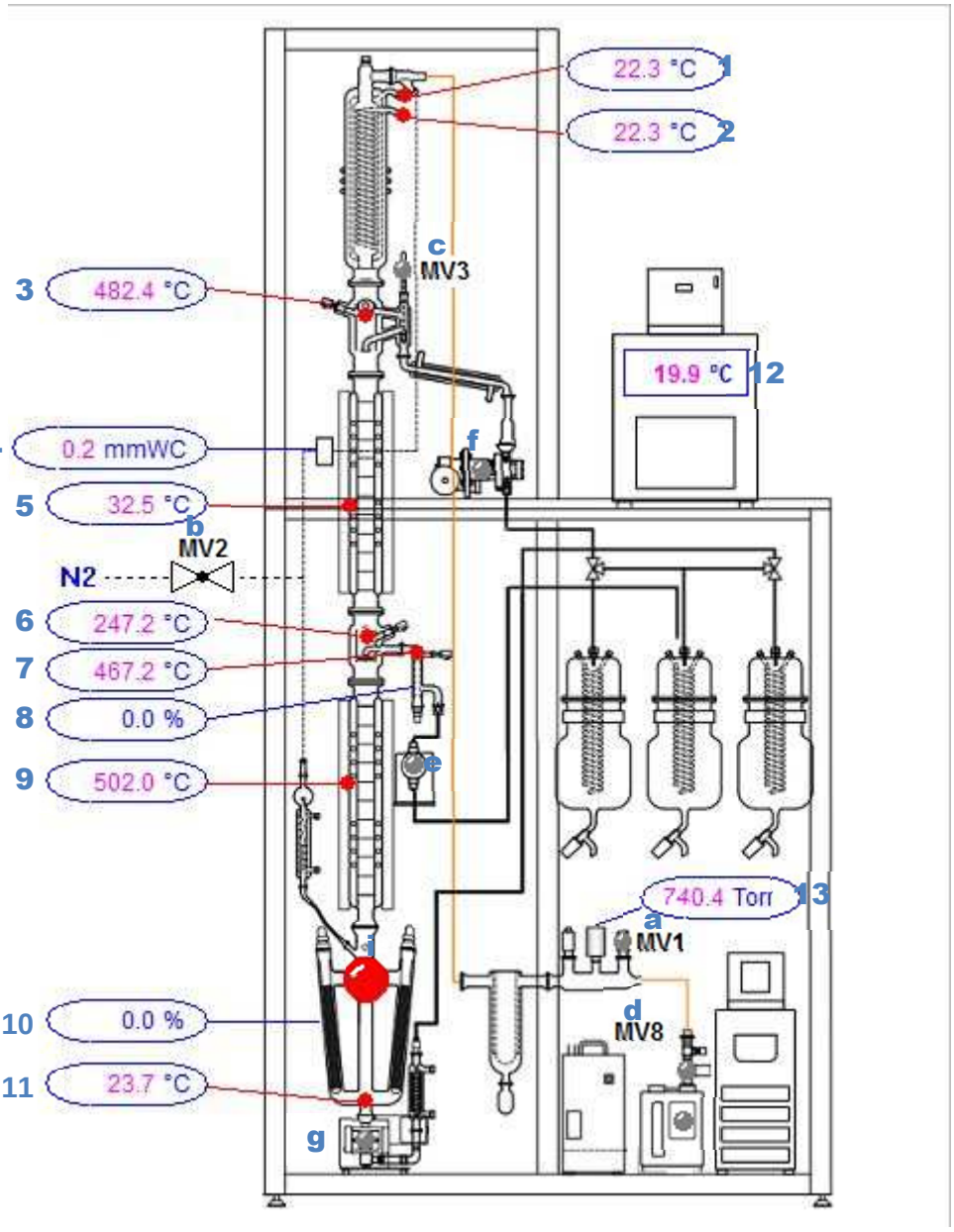


Image 2: Signal Display

4.1 Analogue Signals:

1. Cooling liquid temperature at the main condenser outlet. [°C]
2. Cooling liquid temperature at the main condenser inlet. [°C]
3. Vapour Temperature at the head of the column. [°C]
4. Differential pressure between the reboiler and the condenser. [mmWC]
5. Mantle heater 2 temperature. [°C]
6. Column temperature [°C]
7. Feed temperature which enters the system. [°C]
8. Feed heating power. [%]
9. Mantle heater 1 temperature. [°C]
10. Reboiler heating power. [%]
11. Residue temperature. [°C]
12. Temperature exiting the thermostat. [°C]
13. Pressure inside the unit. [Torr]

The value of the analogue signals is displayed in blue. 0.0 In case the program is not able to determine the value of the signal it is displayed in pink. 0.0

4.2 Digital Signals:

a. MV1 - Ventilation valve
b. MV2 - Nitrogen inlet valve
c. MV3 - Reflux divider
d. MV8 - Vacuum valve
e. Feed pump
f. Distillate pump
g. Residue pump
h. Vacuum pump
i. Residue level detection

The digital signals are displayed in a dark green if its state is inactive MV1 and light green if its state is active MV1. In case its state is indeterminable, the display light is grey MV1.

5 Heater

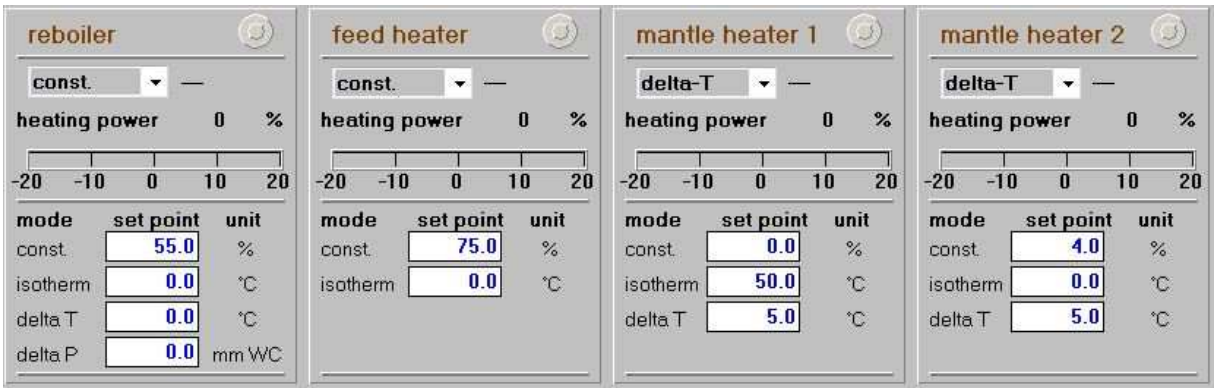


Image 3: Heater Control

The PD250BB is equipped with four electrical heaters: the *Reboiler*, the *Feed Heater*, *Mantle Heater 1 and Mantle Heater 2.*

The different heater controls can be switched on by pressing its proper button on the top - right side of the panel. When the heaters are activated, the button will be 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 three different modes: the *Constant* heating mode, the *Isotherm* heating mode and the *Delta-T* heating 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.*

Reboiler

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

$$\Delta T = T_{Reboiler} - T_{Column}$$

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

$T_{Reboiler}$ = *Flask Heater* temperature

T_{column} = Column temperature

Feed Heater

The purpose of the *Feed Heater* is to preheat the sample entering the column. The *Feed Heater* only heats with the *Constant* and *Isotherm* heating mode.

Mantle Heater 1

The purpose of the *Mantle Heater 1* is to decrease the energy loss of the vapour inside the lower column. The *Delta-T* heating mode for the *Mantle Heater* regulates the heater so that the difference of the column temperature and the *Mantle Heater* temperature corresponds to the *Delta-T* set point.

$$\Delta T = T_{Sample} - T_{Mantle1}$$

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

T_{Column}= Column temperature

$T_{Mantle1}$= *Mantle Heater 1* temperature

Mantle Heater 2

The purpose of the *Mantle Heater* 2 is to decrease the energy loss of the vapour inside the higher column. The *Delta-T* heating mode for the *Mantle Heater* regulates the heater so that the difference of the Head temperature of the column and the Mantle Heater temperature corresponds to the Delta-T set point.

$$\Delta T = T_{Head} - T_{Mantle2}$$

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

T_{Head}= Head temperature in the column

$T_{Mantle2}$= *Mantle Heater 2* temperature

6 Vacuum Control

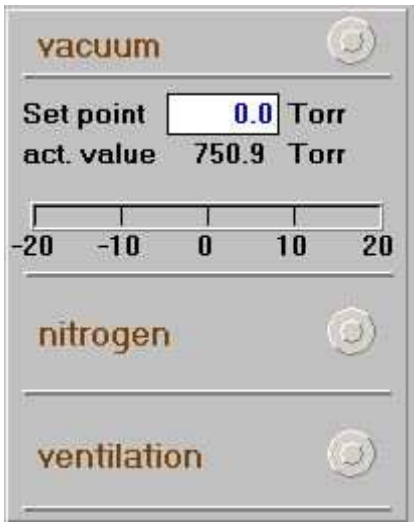


Image 4: Vacuum Control

The ***vacuum pump*** can be switched on by pressing the button on the top - right side of the panel. When the *vacuum pump* is activated the button will be blue. During operation, the program will control the desired vacuum value by opening and closing a magnet valve, located between the vacuum pump and the unit. The set point can be entered by clicking into the white box and tipping the desired pressure. The absolute pressure inside the system is displayed below the set point.

The fine regulation to reduce the fluctuation of the value has to be done via the vacuum throttle valve at the unit. The unit can be ventilated manually through the ventilation valve. The bar in the control display shows the difference between the set point and the actual value. If the bar is red, the pressure in the unit is higher than the set point therefore the vacuum valve stays open so that the pump is able to create vacuum inside the unit to the moment the desired pressure is established. Ones the pressure is below the set point the bar will be blue and the vacuum valve closes so that the pump is not able to increase the vacuum in the unit.

The ***nitrogen flow*** can be switched on by pressing the button on the right side. When the *nitrogen flow* is activated, the button will be blue. A light nitrogen flow can be blown into the reboiler to prevent flaring up the residue. The nitrogen flow is regulated at the flow meter.

The ***ventilation*** can be switched on by pressing the button on the right side. When the *ventilation* is activated, the button will be blue. The purpose of the ventilation valve is to isolate the unit from the atmospheric pressure so that vacuum can be created inside the unit. The ventilation valve is open when the ventilation signal is activated. During an atmospheric run the ventilation valve should be open.

7 Distillate Discharge Control

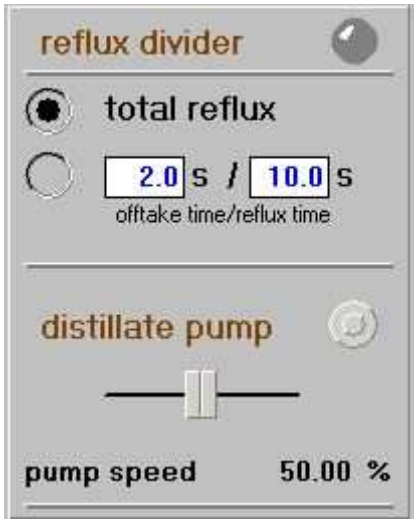


Image 5: Distillate Discharge Control

The distillate is extracted from the column via two mechanisms. The first mechanism is the extraction of the distillate from the column with the ***reflux divider***. The reflux divider enables the condensed vapour to flow back into the column, so that the desired degree of separation can by established, or into the distillate pump to discharge the distillate into the distillate receiver vessel. By default the reflux setting is set at *total reflux* which means the 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 open), which is the first number, and the reflux time (the time the reflux divider is closed), which is the second number.

The second extraction mechanism is the ***distillate discharge pump*** which pushes the distillate into the distillate receiver vessel. The distillate discharge pump can be activated by pressing the round button on the right. When the pump is activated, the button will be blue. The extraction rate of the discharge pump can be regulated with the slider below. The pump speed is controlled with a percentage of its maximum speed. By clicking on the number the percentage can also be inserted with the keyboard. The distillate discharge pump should always be regulated with a higher flow rate than product is extracted with the reflux ratio so that the entire product is discharged directly into the discharge receiver vessel to determine the distillation rate with the balance system. The flow rate settings are the same as the residue pump.

8 Feed & residue panel

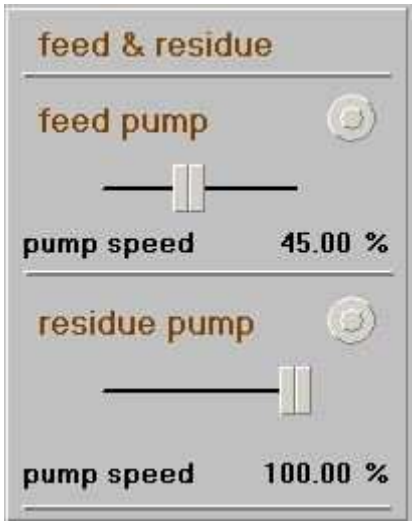


Image 6: Feed & Residue pump control

The ***feed and residue pump*** are regulated in the same manner than the distillate pump. (See chapter 6) The feed pump is able to insert the sample with a maximum flow rate of 88ml/min at 100.00%. The residue discharge pump extracts the product with a flow rate of ml/min at 100%

9 Balance



Image 7: Balance Control

The ***balance control*** displays the weight of the feed vessel, the distillate and residue receiver vessel. The weight displays can be tared by pressing the TARE-buttons on the right side. After filling or emptying the vessels, the user needs to wait 180 seconds before pressing the TARE-button so that the balance can register the complete weight. The flow rate into or out of the vessel is calculated and displayed. The sum of the distillate rate and the residue rate is equivalent to the feed rate in case the unit is in an equilibrium state.

10 Condenser

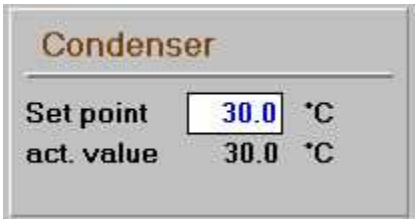


Image 8: Condenser control

The PD250BB software is able to control the temperature set point of the ***main thermostat*** which cools the condenser and the distillate lines. The desired temperature can be set by clicking into the white window, inserting the value and then confirm with ENTER. The value below is the actual temperature inside the thermostat.

11 Parameter

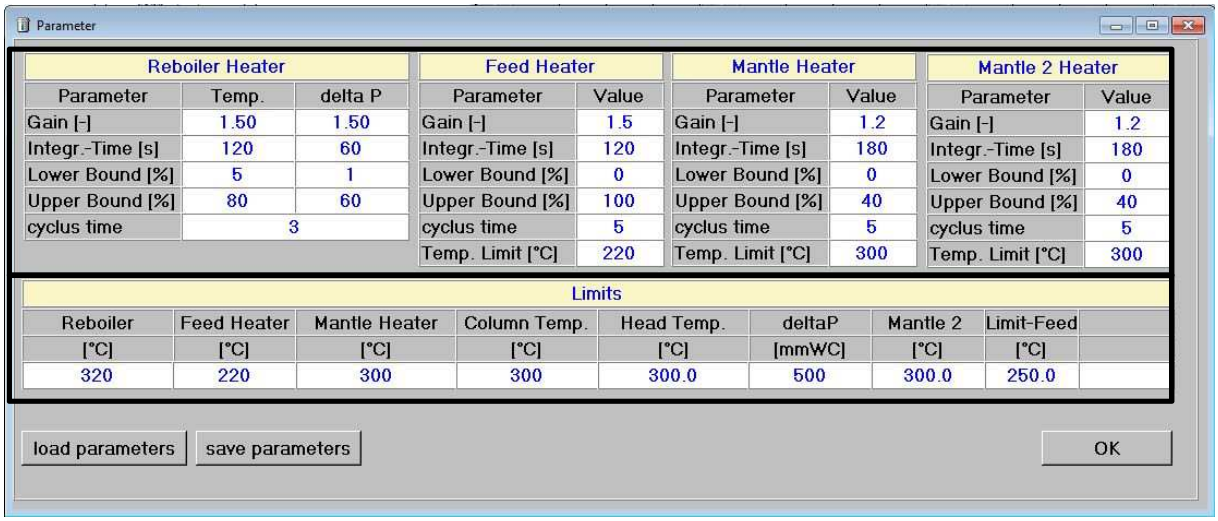


Image 9: Parameter Section

The Parameter window can only be opened when logged in as administrator with the User log-in button (See chapter 14.).

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 (See chapter 5. *Constant* heating mode).
- **Temp. Limit:** In case the heater reaches the *Temp. Limit*, the heater will heat constantly with this limit temperature.

The limits (2.) are security parameters in case the heaters, for any reason, go higher than its *Temp. Limit* and reaches the *Alarm Limit* temperature, then the unit will stop the distillation. The same procedure happens when the residue temperature, the observed head temperature, the column temperature and the atmospheric equivalent temperature in the column head reach their limits.

11.1 Load/Save Parameters

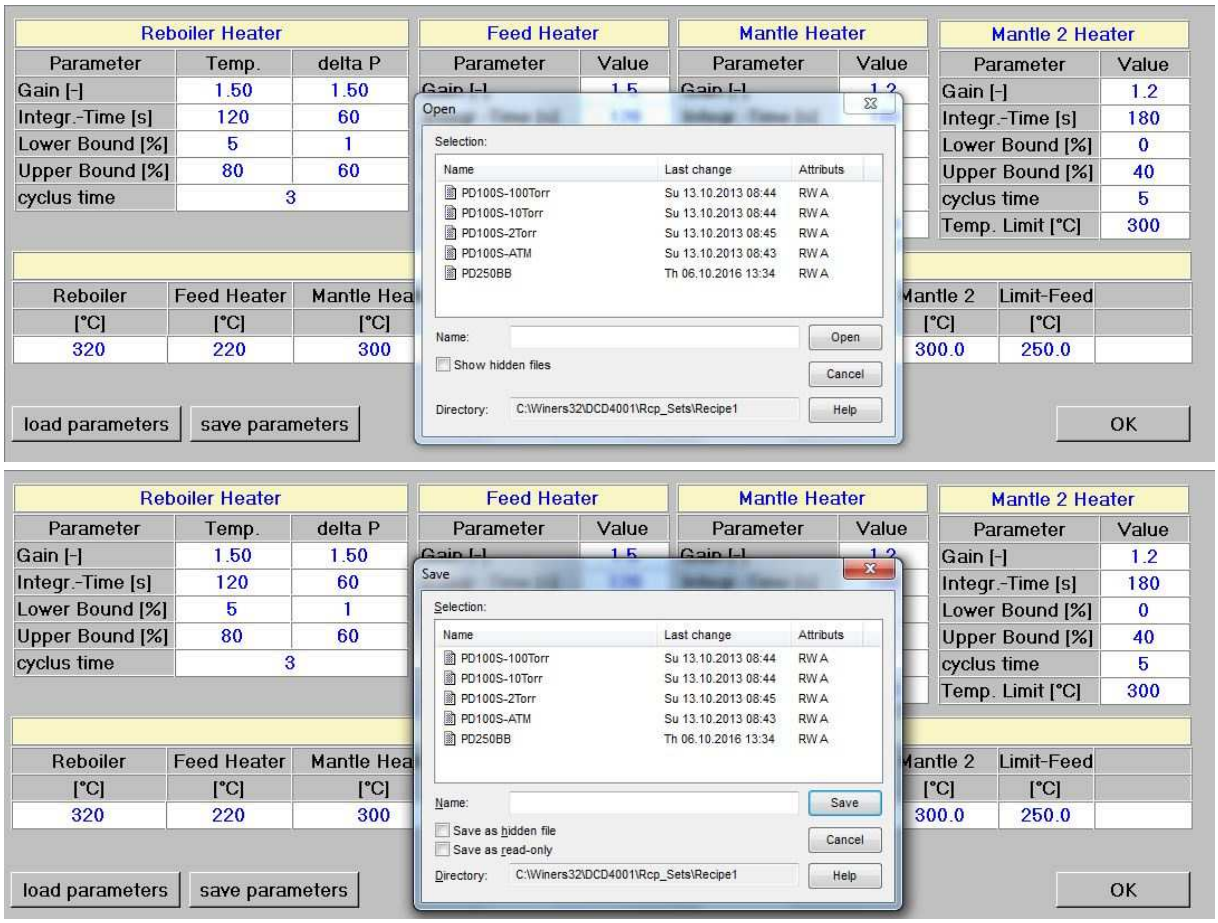


Image 10: Load/Save Parameters

The parameters can be stored as recipe and recalled by using the *Save Parameters* and *Load Parameters* buttons.

12 Service

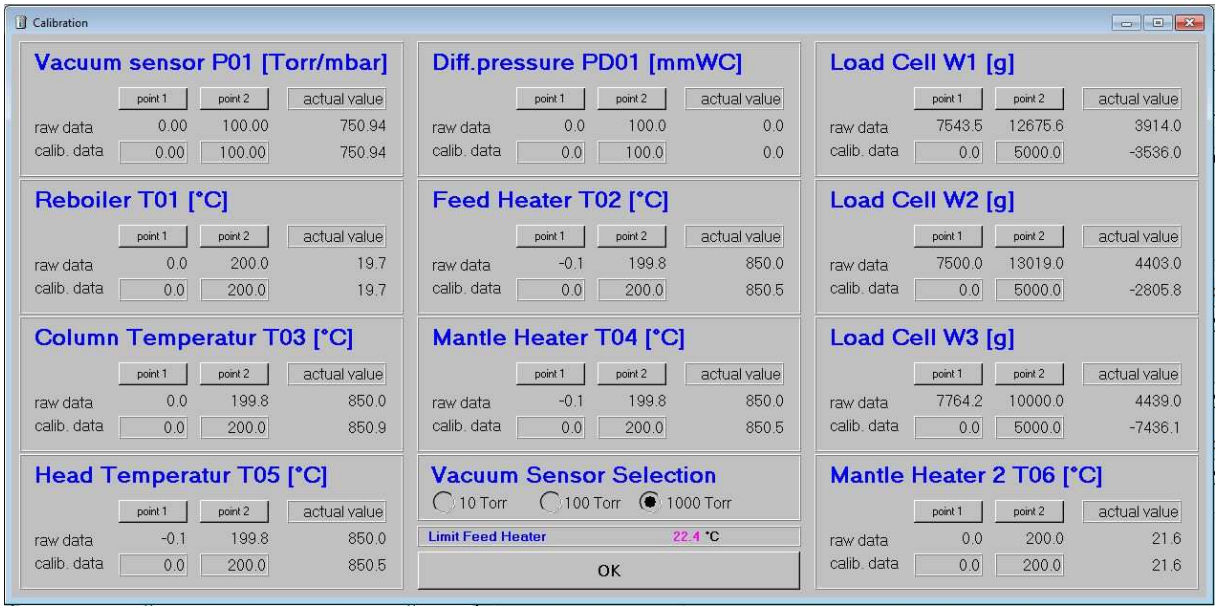


Image 11: Calibration Area

The service area allows doing calibration work. To operate in this area it is required to be logged in as administrator, by using the *User log-in* button.

The user is able to calibrate the vacuum sensor, the differential pressure sensor, the three load cells and the different temperature sensors in the heaters, the temperature sensor in the head of the column, the column temperature sensor, the feed temperature sensor and the residue temperature sensor.

The vacuum sensor is calibrated with an external vacuum testing kit. For further instructions, consult the proper calibration manual. It is also calibrated by disconnecting the vacuum sensor and then press the *"point 1"* button ("calib data" = 0.00) because the pressure inside the unit would be 0 Torr the vacuum sensor would send 0V to the control unit. For the second point the user needs to put 10,000V at the signal entry of the vacuum sensor and press the *„point 2" button* ("calib data" = 1000.00 or 1.0000), in this case the 1000.00 Torr or 1.0000 Torr, depending on the sensor, are simulated.

We do not recommend doing the second part of the calibration without consulting the *PILODIST GmbH* service staff.

To calibrate the temperature sensors: 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.

The load cells are calibrated similar to the temperature sensor, the zero point can be set by taking everything from the balance and pushing the "*point 1*" button. It is important to give the balance enough time so that the value of the balance can stabilize. Now place an exact weight onto the balance and set "*point 2*" in the calibration menu.

In case of a different vacuum sensor, the service technician can change the settings to the appropriate sensor. The limit feed heater is an additional information for the technician.

13 Measurement

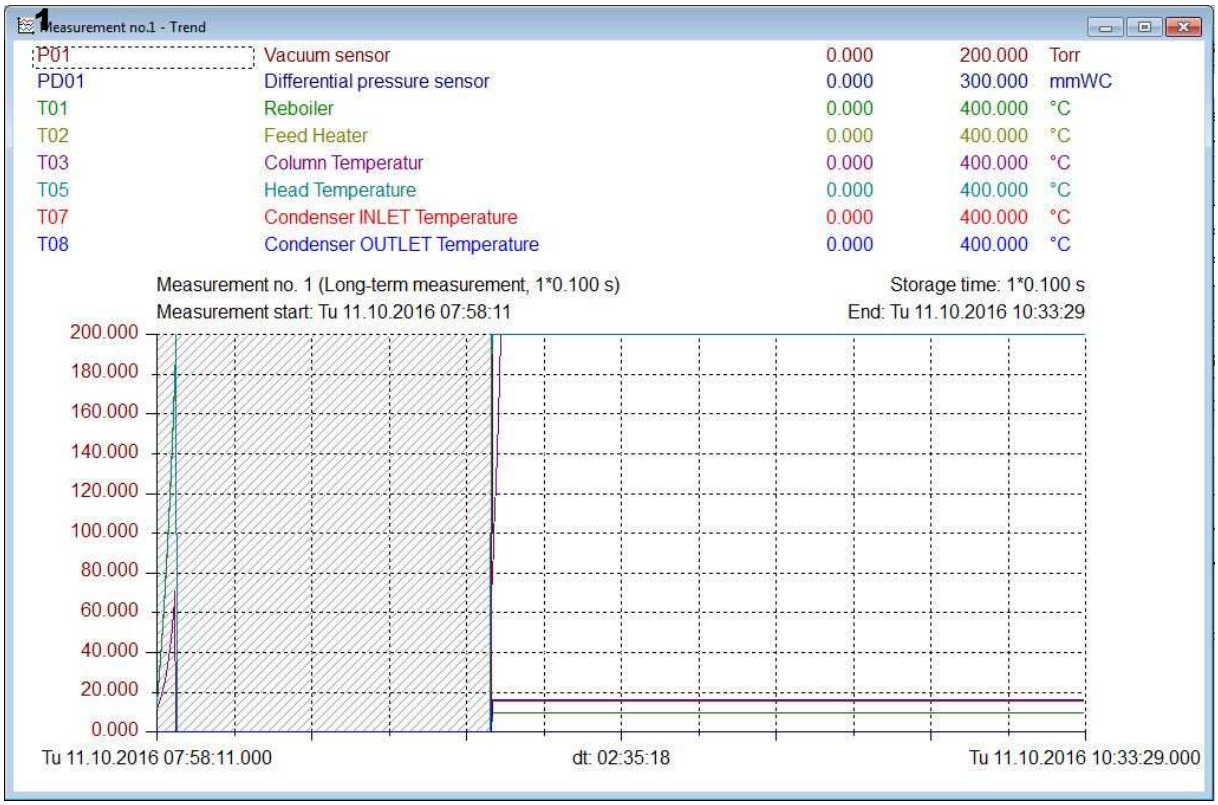


Image 12: Data Curves

The trend curves for the temperatures or the pressures can be shown via pushing 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 column below number 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 howl measurement will be shown, etc.

14 User



Image13: Log-in window

The buttons *PARAMETER* and *SERVICE* are deactivated by default. To activate those buttons the user has to log himself in as administrator. Therefore the user has to press the *USER* button and the log-in window (image 13) will open.

User: Pilodist

Password: 4711

15 Exit program

By pushing the exit button the PD250BB software will be closed and the system will be set to standby mode.

16 Start of the Unit

Starting the unit is a complex procedure, the different heaters and pumps influence the distillate so every component needs to be regulated appropriately to get the desired result.

16.1 General Preparations

1. Switch on the system with the main switch.

2. Start the PC.

3. Start the software with the “PD250BB” shortcut on the desktop.

4. Check that the thermostats (34 and 35, Fig.1) are switched on. If the main thermostat is still in the “rOFF”-mode, please close the software with the “Exit Program”-button and restart. To activate the second thermostat; select the desired temperature and press ok. For further information consult the proper thermostat manual.

5. Check that all required media is available (nitrogen)

6. Check the emptiness and cleanliness of the receivers.

7. The cold trap (18.1, Fig. 1) must be clean and filled with dry ice if no immersion cooler (19, Fig. 1) is used. In case of an electrical immersion cooler, it must be filled with a nonfreezing alcohol (please check the concentration of water caused by moisture frequently).

16.2 Sample preparation:

1. Check the tare weight of the receivers.

2. Fill the feed vessel with the desired sample (32, Fig.1)

3. Rinse the unit with the sample so that the leftovers from the previous run can be extracted to not influence the results of the next run. To rinse, the unit insert some of the sample into the unit via the feed pump and extract the sample leftover mixture with the residue pump and collect it into a separate container by disconnecting the inlet tube of the residue vessel. The residue reflux valve (33.5, Fig. 1) needs to be set to fill the residue receiver vessel.

4. After the unit is rinsed, reconnect the inlet tube to the residue receiver vessel. Switch the reflux valves so that the distillate and the residue would flow back into the feed vessel.

5. Fill the reboiler with the sample. The optimal level for the sample is just at the beginning of the horizontal glass pipe (see Fig. 2) which is approximately 1500ml.

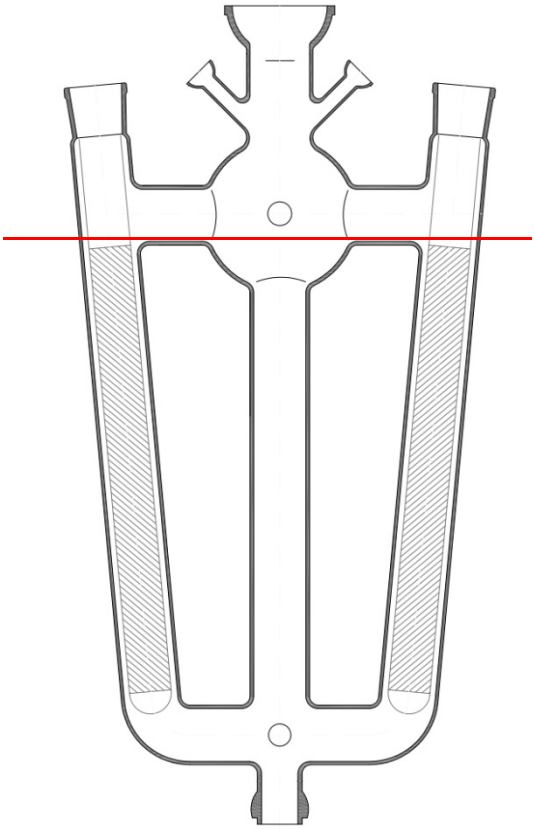


Fig. 2: Reboiler

16.3 Distillation

The appropriate values for the heaters and pumps to start the distillation depend on the application and the experience of the operator; those values need to be determined individually. A certain amount of time is required to start up the distillation.

1. If needed, start the vacuum pump and set the pressure to the desired value.

2. Start the reboiler heater with a low percentage and observe the reaction of the sample.

3. The moment the residue temperature stops rising and no vapors are observed, increase the heating power of the reboiler. A steady creation of bubbles and a constant circulation of the sample can be observed when the right amount of heating power is inserted into the reboiler. When the liquid sample begins rising into the column too much energy is put into the sample and the operator needs to reduce the heating power.

4. Wait as long as the head temperature increases.

5. Switch on the column heater with a *Dealt-T* of 5°C.

6. If the amount of condensate is not the desired quantity increase the heating power slightly the increase the amount of condensed vapor.

7. Wait that the column stabilizes. The column is stable when the head and column temperature does not change anymore.

8. Switch on the feed heater. The feed heater temperature should be set at 5°C to 10 °C below the head temperature.

9. Switch on the feed pump. At the beginning the flow rate should be low.

10. Switch on the residue pump with an extraction rate so that the level of the sample in the reboiler does not increases or decreases. The residue will flow back into the feed vessel.

11. Wait as long as the column stabilizes.

12. Start the offtake with the desired reflux ratio and turn on the distillate discharge pump so that all the distillate can flow back to the feed vessel. Decrease the power of the residue pump so that the reboiler will not be emptied.

13. Regulate the heaters, the pumps and the reflux divider to get the desired result. The flow rates in the software are indicators to regulate the feed and discharge pumps. The flow rate of the feed should be the same as the flow rate of the distillate and residue.

14. The moment the desired products are extracted from the unit, switch the reflux valves so that the distillate can flow into the distillate receiver vessel and the residue into the residue receiver vessel.

16.4 Stop distillation

1. The distillation is finished when the feed vessel is empty.
2. Stop all the heaters.
3. Stop the discharge pumps.
4. Empty the distillate and residue receiver.
5. Wait to the moment the sample in the reboiler reaches an appropriate temperature.
6. Discharge the reboiler by switching on the residue pump and collect the sample in the residue receiver or another container.

16.5 Washing Run

After each complete distillation a washing run needs to be performed. The washing run is an atmospheric distillation with toluene or xylene.

1. Fill approx. 50% of solvent in the feed vessel (31.0, Fig. 1).
2. Prepare the system in the same manner as a normal distillation process.
3. Start a distillation at atmospheric pressure.
4. The unit will run as long as the desired amount of sample is collected in the receivers.
5. After the cooling phase discharge the reboiler.
6. Pull vacuum to dry the unit. The set point should be between 5 – 10 Torr.
7. Uninstall the condensate trap (18.1 Fig. 1) of the cold trap (18, Fig. 1) and remove the waste.
8. Reinstall the condensate traps (18.1, Fig. 1) and check the freezing point (< -40°C) of the alcohol in the cold trap. Exchange if necessary.