

UIC RF10 THIN-FILM EVAPORATOR

Operating instructions

REV No.	DATE	DESCRIPTION	EDITED	CHD.	APPD.
1.0	12.1.2022	Initial version	SRa		
1.1	23.2.2023	Updated cleaning process	SRa		

SAFETY

You should always plan your work before starting and go through all the safety issues involved. A risk analysis may be required, consult your supervisor or technical staff. Read through the material safety datasheets (MSDS) and use appropriate safety equipment and procedures.

Read carefully through the instructions. If something is left unclear, always ask for further instructions. You are responsible for the safety of yourself, the equipment, and others in the vicinity.

Be careful of chemical residues before and after the process. Clean and dry the parts thoroughly after your work!

Label clearly and store your samples appropriately. Minimum information for labels is full name, contact information, date, and chemical contents.

Handling liquid nitrogen requires additional protective gear, including protective boots, full-face shield, and thermal protective gloves for extreme colds. A dedicated, proper container for transferring liquid nitrogen must be used. Short training for first time users of the 35 L Dewar is required. Use extreme caution when transferring filled containers and preferably do not use stairs due to spilling risk.

In case of emergency, use the emergency stop button to shut down the plant. Continue according to workplace safety regulations and instructions.

Thin-film Evaporator RF10

The UIC RF10 evaporation unit is a thin-film evaporator with a 10-litre feed vessel and total volume of 47 litres. The plant is equipped with intermediate receivers with scales, and 4 litre discharge flasks.

The evaporation plant is mainly used for distillation of ionic liquid and water mixtures. Theoretically you can distil 5 kg/h feed under reduced vacuum of 1-10 mbar.

Operations are managed through three different panels: Main Control Panel, Feed Pump Control Panel and Vacuum Gauge Panel.

Main Control Panel (See Image 1) has displays for mass flow and temperature for feed, pressure and temperature between evaporator and condenser, and temperature of the evaporator bottom residue product. Operating buttons for the wiper basket and vacuum pump, in addition to mains power switches, are found on this panel.

Image 1 - Main Control Panel



The technical PID for the whole plant is included at the end of this document. It shows the components of the plant in a concise form, giving an overview of the workings of the system.

Prerequisites

Make a generic check-up of the equipment for breakages, leaks, etc. before starting up the plant. In no case are you allowed to run a damaged plant.

Check that the wiper motor oil level is adequate. The opaque oil pot is on top of the evaporator (See Image 2). If the level is low, inform technical staff for refill.

Ensure the availability of liquid nitrogen, a Dewar container (See Image 3) and protective equipment for handling and transferring it. The Dewar can be filled from a larger (35 L) container in room 137 – **Only by trained and properly protected persons**. Contact technical staff for a quick training and if the liquid nitrogen is running low.

Image 2 - Wiper basket oil pot



Image 3 - A Dewar for pouring Liquid Nitrogen



Check the heat transfer liquid levels (A3013 e.g.) are adequate for normal usage. If not, fill with proper liquids. If need be, ask technical staff for assistance.

Precautions

The feed pump is not allowed to run dry! Doing so may and will damage it. Stop the pump immediately if any grinding noises are noticeable and inform contact person and technical staff.

No solids must enter the system. Solidifying / crystallizing feed materials must not be left inside the system. System must be flushed thoroughly after distillation of ionic liquids!

Before connecting two glass parts (including also valves and glass cocks) it must be assured that the glass flanges are greased with vacuum grease. O-rings and flange-to-flange connections commonly do not require grease.

Please consider, that the wiper baskets should not run at normal operation speed without product for a longer period. Further, if the distillation process is stopped or has not been started and the temperature of the evaporator jacket is above 180 °C it should be assured that the wiper basket is rotating at least at a low rotation speed. This is to avoid inhomogeneous heating of the wiper rollers and to avoid deformation of the rollers at high temperature.

The vacuum control panel must always be switched off before the plant is set to atmospheric pressure!

Preparations

Check the discharge flasks (B2812, B2912, B4011) for any damage. Damaged flask may burst under vacuum! Ensure there is vacuum grease between sealing joints (glass to glass, glass to metal) on both necks of the flasks.

Ensure the emergency stop button on the main Control Panel is not engaged – you release it by rotating it. **Switch on the Control Voltage (24 V)** from left most mains switch on the Main Control Panel. Wait for the display panels to power up, before **switching on the main power** from the mains switch in the centre. (See Image 1)

In general, valve levers are parallel with the line/flow direction when the valve is open, and perpendicular when closed. For example, in Image 5 the valve lever is in perpendicular position, closing the valve and flow.

All valves of the plant to the atmosphere must be closed: feed vessel shut-off valve (B27101), discharge flask shut-off valves (B28101, B29101 and B40101), feed line valves (B27104, B27103), etc. The closing valve (the rotating grey outer ring) for the adjustable valve H35105 (See Image 4) should be closed as well – rotate it clockwise. Most importantly vacuum **shut-off valve B35102** (See Image 5)

Image 4 -B35105 adjustable valve



between vacuum pump (P3512) and the cold trap (CT2511) **must be closed!**

Image 5 - Vacuum Shut-Off Valve



Open the **cooling water feed** tap half-way. The tap is located next to the sink on the left-hand side of the evaporation plant. The tap for the cooling water feed is the one closest to the wall. (See Image 6)

Image 6 - Cooling Water Tap



Start the vacuum pump (P3512) from the main Control Panel. Let the pump warm up for at least 30 minutes before operating vacuum shut-off valve (B35102). You can continue with other preparations in the meanwhile.

Switch on the Cooling Unit (A3211) from its mains switch (See Image 7). It will automatically start the cooling. **Let the unit cool down to -1 °C** – this usually takes around 15 minutes. Again, you can continue with preparations in the meantime.

Image 7 - Cooling unit for the Condenser



Power up the heating unit (A3012) for the evaporator jacket from the red mains switch (See Image 8). After booting up, a graphic menu appears on the screen. Push and hold the green button on the screen to start the heating.

If there is a risk of crystallization, with for example highly concentrated ionic liquids (<15% water), the heating units A3011 and A3013 must be

used. A3011 heats up the feed line after the flow meter, and A3013 the evaporator bottom and intermediate receiver. As there is no heating earlier in feed line, the risk of clogging the flowmeter is high with feeds with low water content.

Power up the A3013 unit on from the mains flip-switch. After booting up, press *OK* to turn on/off the heating. (See Image 9)

Power up the A3011 unit on from the mains switch. Push the green button to turn on the heating. Red button stops heating. (See IMGX)

Image 8- Heating Unit A3012



Image 9 - Heating Unit A3013



If you need to change the heating unit setpoint(s), you need to enter the settings from the unit(s) (See separate chapters).

Fill the liquid trap (CT2511) with suitable coolant (liquid nitrogen e.g.) before pumping any feed into the system. Wear proper safety equipment and use the aluminium stairs to reach the trap! Pour the coolant slowly, as the large temperature difference may incur boiling and splashing.

Install the discharge flasks for concentrate (B2812) and distillate (B2912), below their respective intermediate receivers B2811 and B2911. Both flasks must be placed on their cork supports on the height-adjustable tables. When positioning the flasks, ensure there is no tension on them from the table. Install and tighten the metal clamps around both flange connections.

The discharge flask for the liquid trap (B4011) must also be installed and connected with a clamp (installation with an older clamp in Image 10).

Image 10 - Liquid Trap Discharge Flask



Close the discharge flask vent valves (B28102, B29102 and B40102). The valve is closed when the lever is perpendicular to the valve (In Image 10 valve is closed).

Open the shut-off valves (B28101, B29101) between flasks and intermediate receivers. Open the shut-off valve (B40101) between liquid trap and its flask.

After the vacuum pump is warmed up the **vacuum shut-off valve (B35102)** between the vacuum pump and the cold trap CT2511 can be **slowly opened**, and the system evacuated. **Opening the valve too quickly will allow the removed air in the system to overflow the vacuum pump oil into the filter, influencing the attainable vacuum and requiring more frequent maintenance (downtime).**

Now, **turn on the control unit** (See Image 11) of the **pressure gauges PI35104 and PI38102** to display the vacuum level between evaporator and condenser (K1111 / C2611) and behind the cold trap (CT2511).

Image 11 - Pressure gauge control panel



For **pressure adjustment** open the closing valve (the rotating grey outer ring) for the adjustable valve H35105 by rotating the grey outer ring counter-clockwise. Adjust the pressure by rotating the black adjustment knob. (See Image 4) For rough pressure adjustments, vacuum shut-off valve (B35102) can also be used.

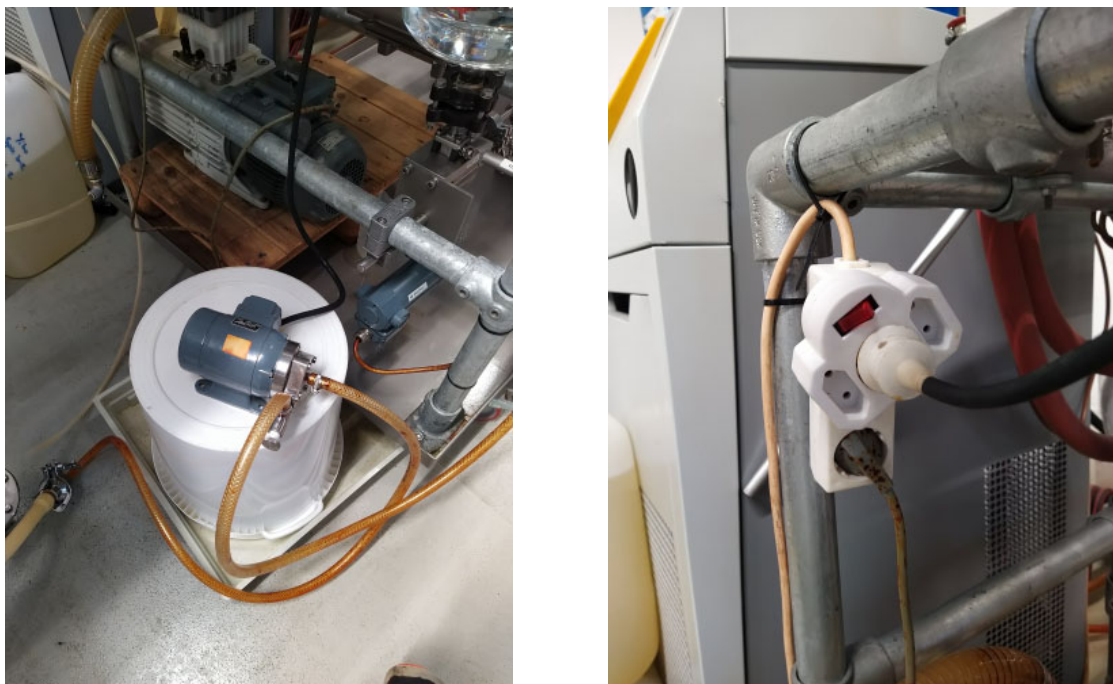
There is a natural delay before the adjustments will show in the display(s). Make small changes and wait a while before readjustment.

When the system is fully evacuated, required temperatures have been reached and the pressure is stable for about 15 minutes, only then should the distillation process be started.

Starting up the process

Transfer the feed to the feed tank (B2711) manually or with an external pump (See Image 12). Open the feed line shut-off valve (B27101).

Image 12 - Transfer Pump for Feed Tank



Start the wiper basket motor (M-P1111) by pressing ON from the main Control Panel. The speed of the wiper basket should not be adjusted. Instructions for this can be found from the original documentation.

To remove possible air out of the product pipe, the venting valve (B27104) in the feed line can be opened while the feed pump is running until the product appears. Then the valve is closed, and the product will flow into the evaporator.

From the Feed Pump Console (See Image 13) set the speed (2) and **start the feed pump** (3). For permanent operation the motor speed must be chosen lower than 700 – optimal rotation speed is 350-400.

A speed of 320 is usually equivalent to a flow rate of 3.2 – 4 kg/hr. The rate can vary during operation, so keep an eye on the mass flow (FIC 27105) on the Control Panel or the meter local display.

Image 13 – Feed Pump Console



Maximum revolutions per minute of the pump is ~100 rpm (speed set to 999). Nominal pumping speed of the pump is ~2.4 cm³ per revolution. Table below can be used as a reference for feed rates. Bear in mind that the mass flow meter gives you the closest measure.

Table 1 – Feed Pump Feed Rates per Speed Settings

Speed	Feed Rate [l/h]	Speed	Feed Rate [l/h]
0	0	500	6.91
50	0.72	600	8.64
100	1.44	700	10.08
200	2.88	800	11.52
300	4.32	900	12.96
400	5.76	999	14.40

During the process

In the very beginning of the distillation process, the condensed distillate will start accumulating first. It will take a few minutes before the

concentrate starts accumulating. With more diluted feed the distillate intermediate receiver will fill more quickly.

Refill the liquid trap when needed – approximately every 90 minutes. Pressure increase may indicate a need for a refill.

Refill the feed tank when needed. If there is some liquid in the tank and feed line, the vacuum will hold, and you do not need to stop the process or close feed shut-off valve while refilling.

Empty the discharge flasks when necessary. To **disconnect a discharge flask** for emptying while the system is running, you need to follow these steps:

Close the shut-off valve between the discharge flask and the intermediate receiver (B28101, B29101). This disconnects the flask from the system vacuum.

Open slowly the discharge flask vent valve (B28102, B29102) to normalize the flask's inner pressure to ambient pressure. Disconnect and empty the flask.

Store or dispose of the contents accordingly. Ensure the distilled water has no other chemical residues before discharging to drain.

Reconnect the flask as you did in the preparations phase (See Preparations). Ensure the vent valve is closed. **Open the shut-off valve slowly.** As the vacuum pump evacuates the volume of the flask, it will momentarily influence the vacuum!

Liquid Trap discharge flask can be detached and emptied similarly.

Shutting down

The cooling for the internal condenser as well as for the cold trap should be continued for the cleaning process. The cold trap is in operation (cooled) so that also the cold trap will be cleaned with used cleaning agent.

Do not store feed in the feed tank! You must clean the system after every use. Use the feed pump and valve B27103 to pump out the feed before the cleaning process.

Stop the feed pump from Feed Pump Console. **Close the feed line shut-off valve**. Let the feed already in the system to be processed - a few minutes should be enough. **Empty the discharge flasks** as you would during the process if they won't fit the cleaning agent or you would like to save the concentrate and/or distillate for further use.

Close the vacuum shut-off valve (B35102). **Adjust the vacuum** to 120 mbar with the valve H35105 adjustment knob and close the outer ring by rotating it clockwise. (See Image 4)

Let the vacuum pump run for another 30 minutes before switching it off. This regenerates the pump oil, removing gases and ensuring proper operation the next time.

Fill the feed tank with water (4 litres). **Open the feed shut-off valve** and **start the feed pump**. This will rinse the condenser, distillate intermediate receiver and the discharge flask. Let it run until around one litre is fed.

Stop and switch off the heating units. **Turn off the vacuum control panel**. **Slowly vent the plant** via the distillate discharge flask vent valve. Open the vacuum adjustment valve H35105 outer ring.

The water should now start rinsing the evaporator side, collecting in the condensate intermediate receiver and discharge flask. To rinse the intermediate receiver, close the shut-off valve and let the water level rise above the inner heating element. Open the shut-off valve again.

When there is less than half a litre of water in the feed tank, you can **stop the feed pump and the wiper basket**.

Switch off the cooling unit and **close the cooling water tap**.

Drain the feed line through valves B27103 and B27104. You can run the feed pump to remove the last liquids from the feed tank, but don't run it dry. Close the used valves.

Empty and clean all the three discharge flasks.

Stop the vacuum pump. Switch off the main power and then the Control Voltage.

Changing heating unit A3012 temperature setpoint

To enter the *selection menu*, tap the right most, list looking icon, on the screen. From this menu you can access the sub-menus for temperature setpoints and user-level changes. Use the angled arrow icon to go back or exit the menu.

To change the temperature set points, you must be logged in at maintenance user level. Tap in *selection menu* the person looking icon, third from left on the upper row, to enter *select Users* user level selection. Tap on middle icon, person with a spanner. In the opened *Code setters* window, enter the code 034 and tap the green icon.

With the maintenance level access, return to the selection menu, and select the *set points* menu (now without the lock icon). Select the first icon from left on the upper row. Enter the new temperature setpoint with three digits, where the third is the decimal. Confirm the change with the green icon. Exit the menu back to the main screen.

Changing heating unit A3013 temperature set-point

With the unit powered up, push on T button. User up/down arrows to adjust the whole numbers, confirm with OK. Similarly adjust the decimals.

Troubleshooting

Vacuum pump not turning on

REASONS: Blown fuse due to vacuum pump left on before powering down the plant OR no delay after turning on Control Voltage and before turning the mains switch on

FIX: Notify technical staff.

Vacuum level fluctuating

REASONS: Leakage or vacuum pump oil level low.

FIX: Check valves, connections, and possible other leakage sources. Notify technical staff.

Feed pump failure

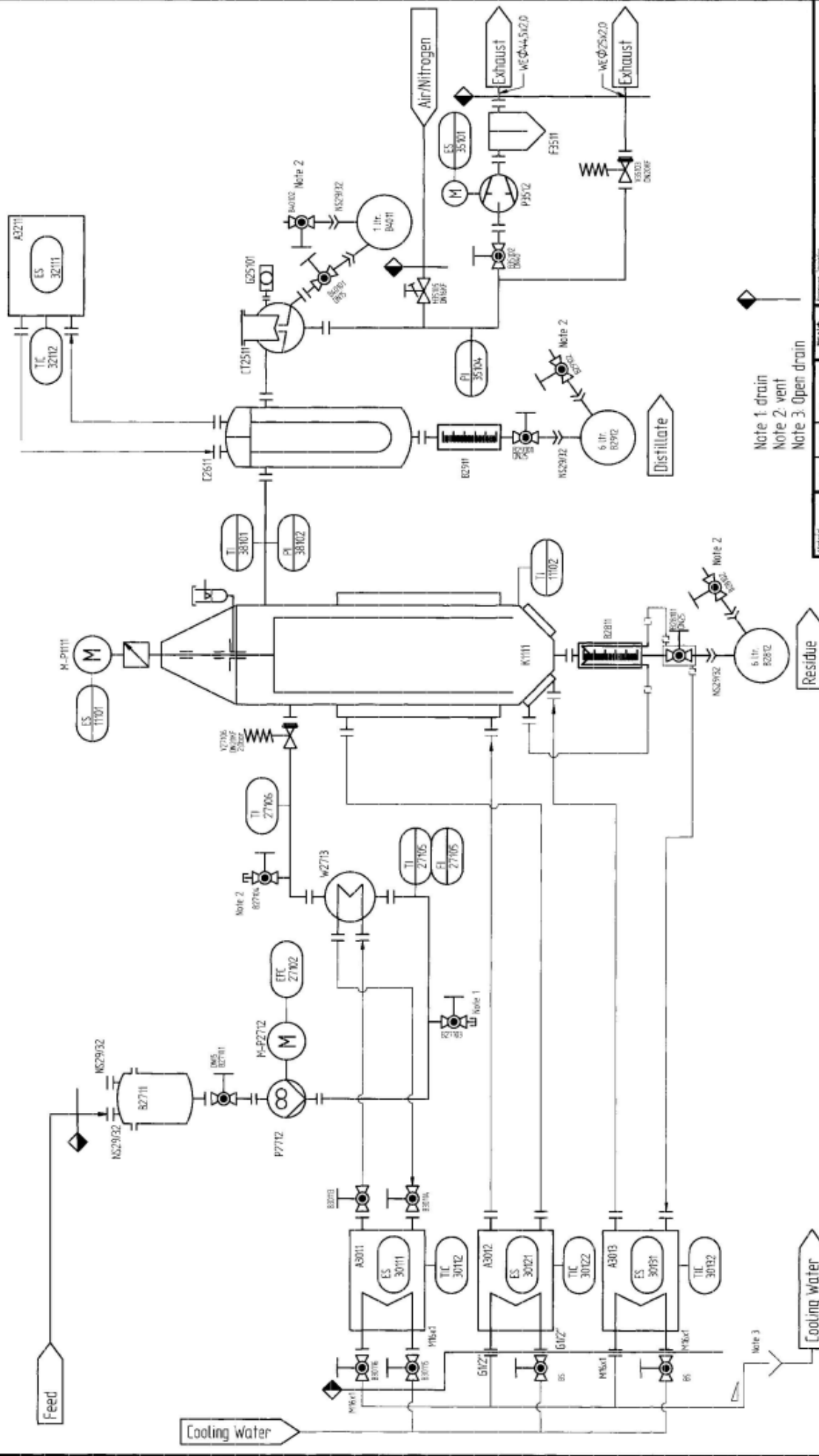
REASONS: Mechanical failure due to dry running or prolonged running at high speeds (\Rightarrow 700 rpm)

FIX: Notify technical staff.

No feed flow

REASONS: Feed pump failure or feed has crystallized inside mass flow meter.

FIX: Notify technical staff.



- Note 1: drain
- Note 2: vent
- Note 3: Open drain

Rev.	Rev. Description	Date	By
1	Initial		
2			
3			

With Venting System
 No Venting System

3D CAD

3D
 2D
 Other

Macro	Rev.	Änderung	Modification	Datum	Name

QUIC
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Supplier's Standard Cleaning Procedure

If the operation temperature of the evaporator was above 180°C, the wiper basket should not be switched off before the evaporator is cooled down to temperatures below 180 °C.

The main heating unit is cooled down by setting the temperature to 60°C.

Vacuum system should be separated (closing of valve B35102 of the vacuum system) and the plant can be vented via valve H35105 to atmospheric pressure. Keep the plant open to atmosphere to avoid overpressure in the unit.

After the last distillation run, the temperature of all heating units should be lowered as required for the cleaning. That means at least 20 °C below the boiling point of the cleaning agent.

The discharge / receiver flasks in the discharge system should be empty. Feed a suitable cleaning agent in the feed vessel and start feeding the cleaning agent and start the wiper basket at normal speed (e.g. 400 rpm).