ALChemia Solutions

Explorer Options

Vacuum kit for
Essential oils and hydrolates

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1. GENERAL INFORMATION

1.1. Vacuum distillation (in brief)

The Explorer's vacuum kit has been developed to distill essential oils (EOs) and hydrolates under reduced pressure. Lower pressure in the still means a lower distillation temperature, which results in less degradation (fewer off-notes, burnt notes, hydrolysis, oxidation, etc.). This can be especially beneficial when distilling high-quality essential oils and hydrolates.

However, note that vacuum distillation may also result in a lower EO yield and/or require a longer distillation time. Typically, vacuum distillation is suitable for plants that are usually distilled quickly, contain fragile fragrances or molecules, and/or tend to develop off-notes during standard distillation. On the other hand, if the plant you are distilling doesn't match any of these criteria, vacuum distillation might not be the best method. Still, one key point remains: the only way to be sure is to test it.

1.2. System overview

This kit is designed for use with the *Explorer* and requires a *Nano* to collect the hydrolate, as well as a vacuum pump. The vacuum kit consists of several parts serving three main functions:

- Protecting the Explorer tank against the vacuum
- Controlling and regulating the pressure throughout the system
- Separating essential oils and ensuring a tight connection between the *Explorer* and the *Nano*

The *Explorer* tank is protected from vacuum pressure by a height-adjustable pillar mounted on a tripod base. **This pillar must be set to the correct height for your *Explorer* before the first use** (see instructions below). **It prevents the flat bottom and lid from collapsing under vacuum and must be used in the *Explorer* tank every time you distill under vacuum.**

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When distilling under vacuum, the whole system is under vacuum: the main tank, the insulated column, the condenser, the EO separator and the receiver for the hydrolate. To control the vacuum, thus the distillation temperature, you must first and obviously use a suitable vacuum pump. A good choice is a diaphragm pump used with a rotary evaporator (contact us for advice). The vacuum pump is connected at the top of the *Nano*, but the system is designed so that the pressure is automatically equalized in all the parts of the unit. To regulate the vacuum at the desired value, we use a needle valve on the vacuum line to create a controlled leak. If the leak is minimal, the pressure goes down. If we let

more air entering in the system, the vacuum will be limited. To protect the system, we also added a safety vacuum-relief valve which should slowly open when the vacuum reaches 40 kPa in the still.

Finally, the majority of the pieces you will find in this kit are designed to assemble a special florentine flask that create a tight connection between the *Explorer* and the *Nano*. You will find below a step-by-step guide to assemble everything.

1.2. Parts list

1.2.1. Nano

- A 16.5 L tank and its lid with four M5 screws, nuts, and thumb nuts
- A large EPDM O-ring (already installed between the tank's flange and the lid)
- A straight tube in tube condenser with two 90° elbows, 3/8'' barbed fittings and clamps
- A tri-clamp tee and a tri-clamp 90° elbow
- A 1.5'' cap and four clamps + white PTFE gaskets
- A grid with three 5'' / 12.5 cm and three 6'' / 15 cm screws
- A stainless wire with a piece of microfiber fabric to clean the condenser
- A drain valve (mini ball-valve 3/8'') and a 8'' / 20 cm tube with an elbow
- A stainless condenser spiral
- A replacement lid screw, nut, and thumb nut and Teflon tape

1.2.2. Vacuum kit

- The pillar assembly comprising a tripod basis, the pillar, and the pilar head
- A special grid that can be used with the pillar (you will have to use the 6'' / 15 cm or 8'' / 20 cm stainless screws that come with the grid of the *Explorer*)
- An online thermometer with a gasket and a clamp
- A 1.5'' tri-clamp elbow, a 1.5'' tri-clamp tee, 2 gaskets and 2 clamps
- A vacuum gauge with a vacuum hose fitting (1/4'') on a tri-clamp cap with a gasket and a clamp
- A 1.5'' / ≈ 6'' long flow sight glass, a gasket and a clamp
- A stainless tubing (1 & 1.5'') welded section (main part of the EO separator)
- A drain valve (3/8'') on a tri-clamp cap with a gasket and a clamp
- A safety vacuum-relief valve and a vacuum hose fitting on a tri-clamp cap with a gasket and a clamp
- A 90° tri-clamp barbed fitting (3/8'') with a gasket and a clamp
- A straight tri-clamp barbed fitting (3/8'')
- A clear PTFE tube (10 mm) already connected to the 2 barbed fittings 3/8'' + 2 spare tubes
- A brass needle valve on a stainless cross with two vacuum hose fittings and a tri-clamp cap

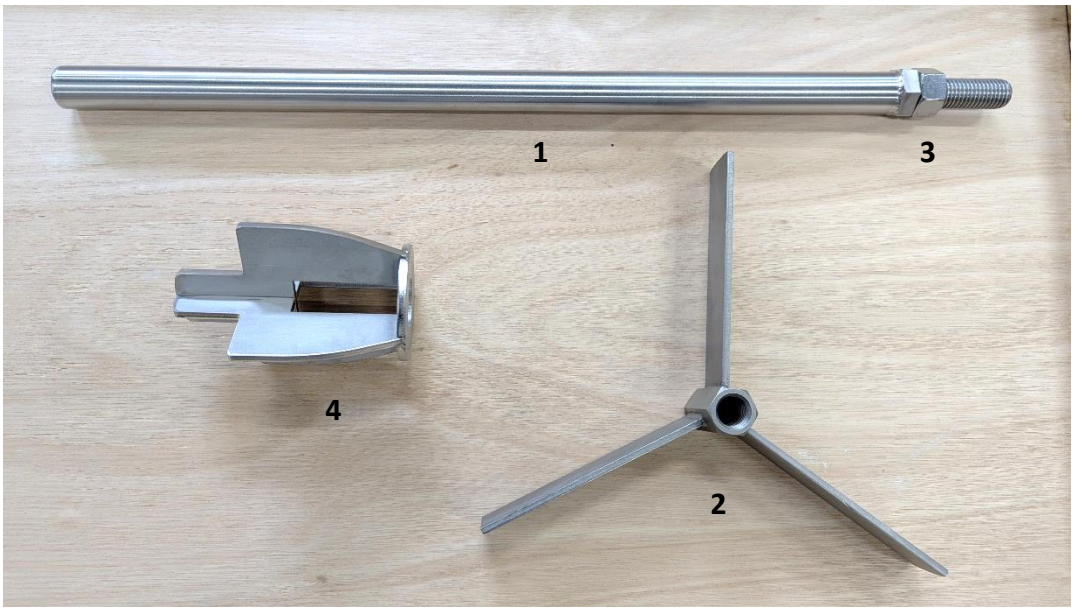
- Two sections of vacuum hose, one straight and one with a tee

2. SETTING UP YOUR VACUUM STILL

2.1. Setting up pillar height

The first thing to do when you want to use your *Explorer* in the vacuum configuration is to set the exact right height for the pillar. This must be done only once before the first use, but **the pillar must be used each time you distill under vacuum**, whatever you distill, by steam or hydrodistillation.

The pillar (**1**) has a threaded section at the bottom (a welded screw). Before screwing it on the tripod basis (**2**), screw the stop nut (**3**) completely (picture 1), then screw the pillar on the basis about halfway.

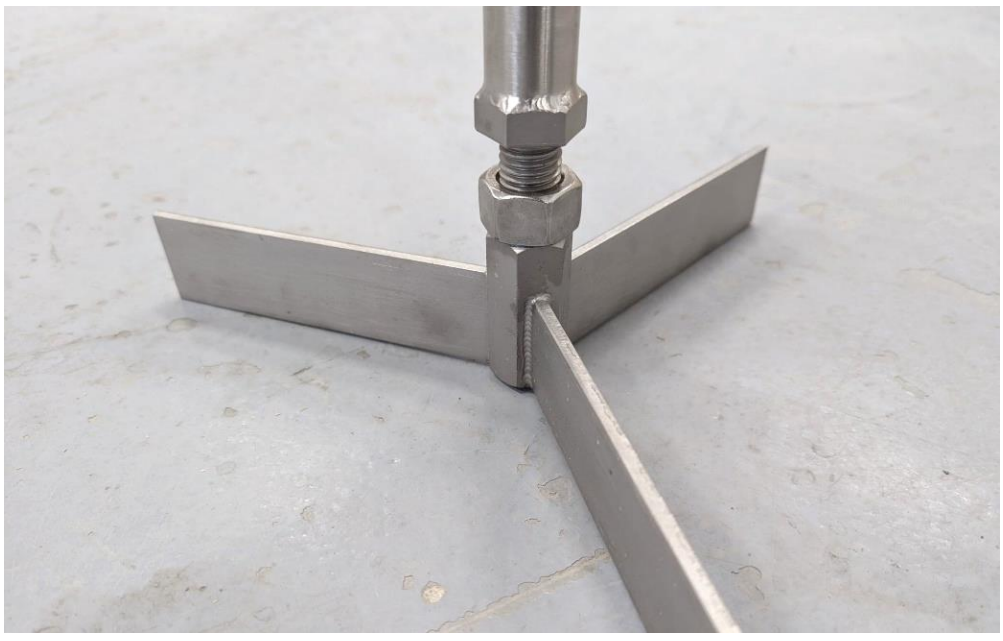


Picture 1: Pillar assembly composed of the pillar itself (**1**), a tripod basis (**2**), a stop nut (**3**) on the threaded section of the pillar and the pilar head (**4**).

Put the pillar assembly in the empty *Explorer* tank and the pilar head (**4**) on the pilar then screw or unscrew the pilar on the basis so that the pilar head is leveled with the O-ring of the *Explorer*. The easiest way to check and adjust is to use anything flat like a ruler and make sure it sits on the pilar head AND on the O-ring on both sides of the tank (see picture 2). **This step is very important for the integrity of your unit and should be done carefully!**

Picture 2: Pillar assembly in the tank. The pilar height must be adjusted so that anything flat rests on the pilar head AND on the O-ring on both sides of the tank.

Once you have the right height, carefully remove the assembly from the tank and block the position of the pilar against the basis with the stop nut (see picture 3) then try again as previously to check you have the right height and try it with the lid.

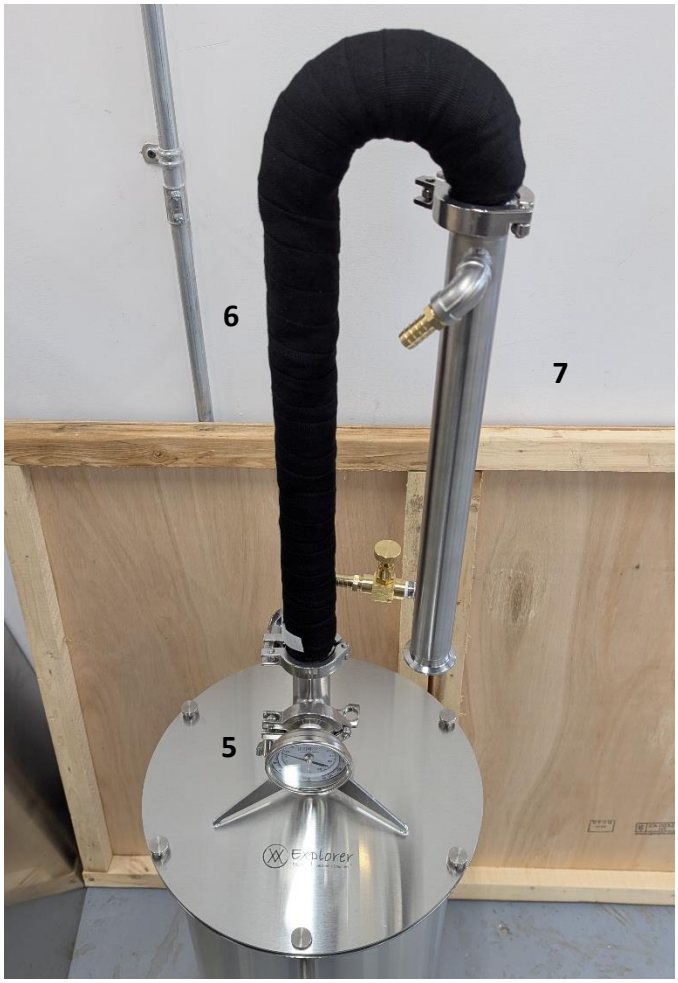


Picture 3: screw the stop nut against the basis to save the pillar height.

2.2. On the *Explorer* side

Once the pillar height is set and if you are about to distill a plant under vacuum, this is the time to add some water in the still, then put the grid in the still through the pilar, load your plant material and put the pilar head back, then close and screw the lid (in this section, we primarily describe how to set the vacuum kit but more detailed information about vacuum distillation will be given below).

In the standard configuration of the *Explorer*, the insulated column is directly connected to the lid and the condenser is connected to the downward section of the column. The vacuum configuration is very similar except that you will have to add the in-line thermometer between the lid and the column as shown in the picture 4. From the lid to the outlet of the condenser, you should have the in-line thermometer (**5**), the insulated column (**6**), and the condenser (**7**), these three parts being connected by 3 with black EPDM gaskets and clamps. The nozzle used in the standard configuration of the *Explorer* will not be used here.



Picture 4: adding the in-line thermometer on the *Explorer.*

2.3. On the *Nano* side

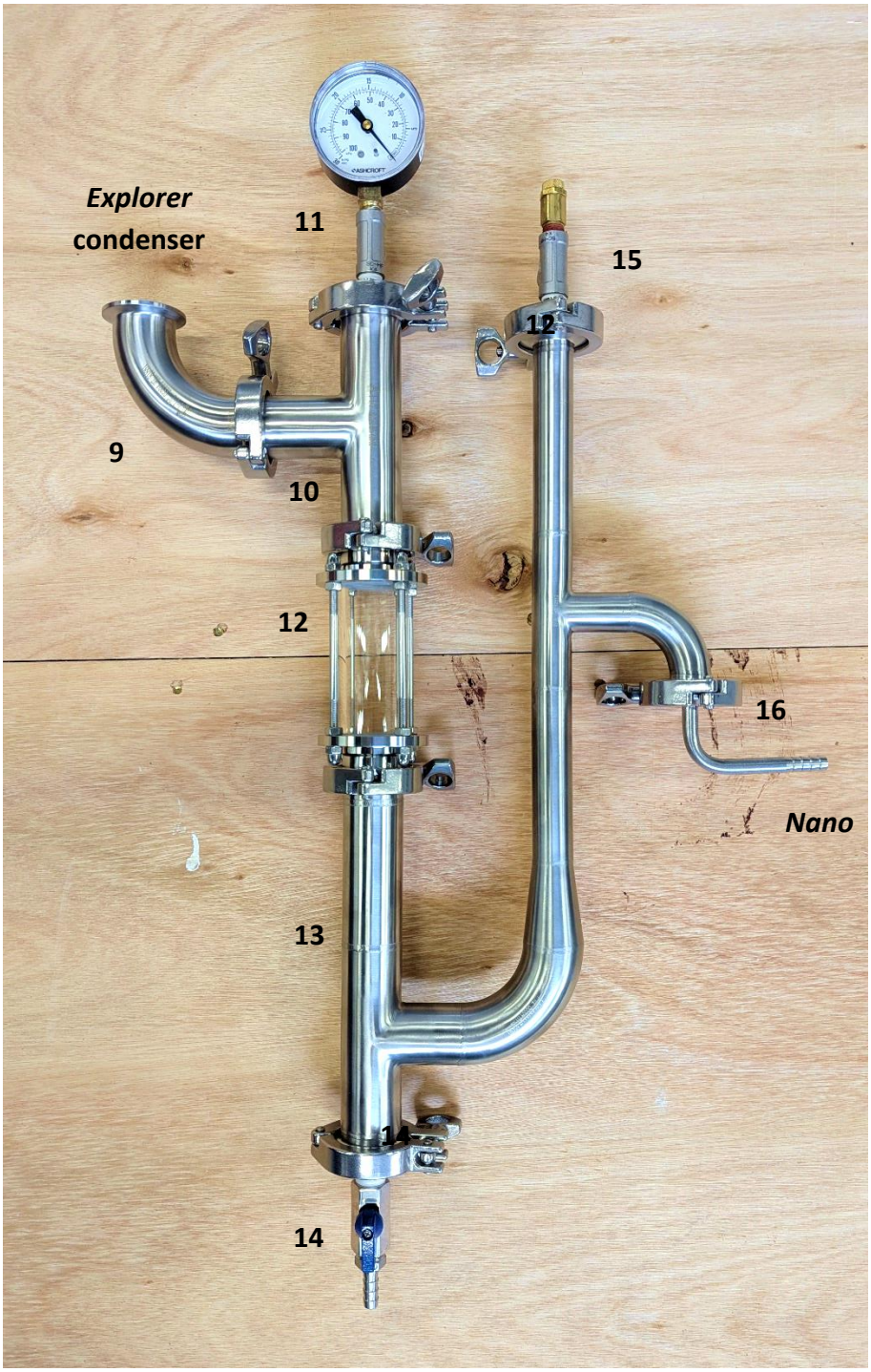
As explained in the overview, here the *Nano* is not used as a still but as the receiver for the hydrolate, thus some parts that came with the *Nano* will not be used here, namely the grid, the condenser, and the tri-clamp cap. The only modification you will have to do compared to the classic configuration of the *Nano* (when used as a still) is to replace the cap at the top of the tri-clamp tee by the piece shown in the picture 5. We will call this assembly the leak regulation valve (**8**). Note that the two pieces of vacuum hose will be connected to the piece.

Picture 5: Setting up the *Nano.*

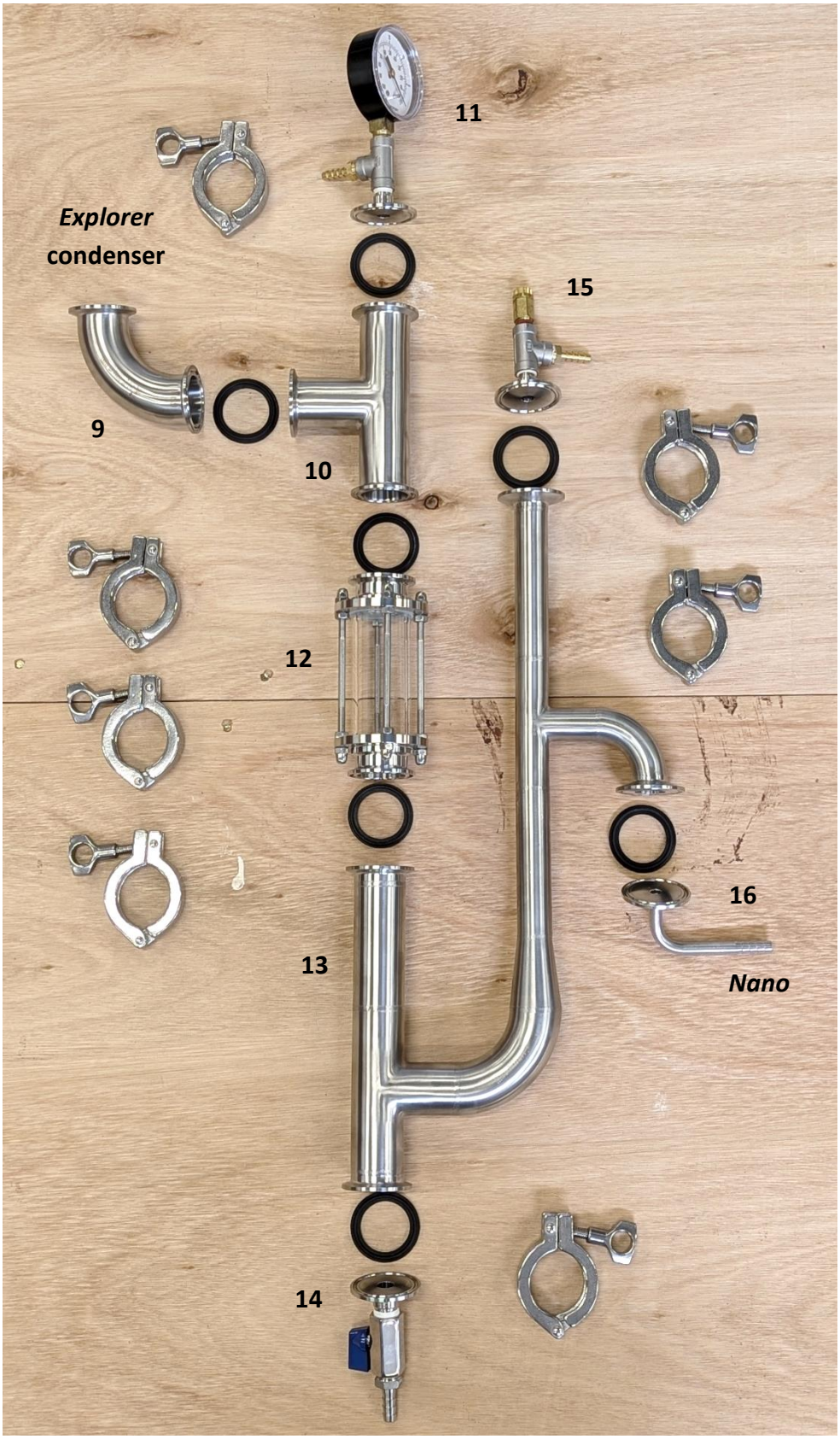
2.4. Assembling the vacuum florentine flask

Assemble the florentine flask as shown in the picture 6 and 7 (exploded view). From the *Explorer* condenser to the *Nano*, you should find: a 90° TC elbow 1.5'' (**9**), a TC tee 1.5'' (**10**), upward you have

the vacuum gauge assembly with a vacuum hose fitting (**11**), downward you have the flow sight glass (**12**), then the welded tubing section (**13**), at the bottom you have the drain valve assembly (**14**), at the top the safety vacuum-relief valve assembly with a vacuum hose fitting (**15**), and finally on the side you have a 90° tri-clamp barbed fitting 3/8'' (**16** – when you receive the vacuum kit, this piece is already connected to a piece of clear PTFE tubing).



Picture 6: Assembling the vacuum florentine flask.



Picture 7: Assembling the vacuum florentine flask (exploded view).

When you receive the Kit, you will find black gaskets and white gaskets. The white ones are made of PTFE and the black ones of EPDM. Both can be used and have pro and cons (this is why we put the two types). The white ones (PTFE) are the most chemically resistant; they withstand almost any chemicals (thus any EO), but they are pretty hard, and a good tightness may be difficult to achieve with them. The black ones (EPDM) may not be as resistant as the white ones, and they may swell with some EO but they are must softer, and a good tightness will be easier to achieve with them. What we recommend is to put the black ones at any fitting before the condenser and everywhere you only have vapors or water on the picture above (**11**, **14**, **15** & **16**) and try the white ones where you have some liquid essential oils (**9**, **10** & **12**). If you have some issues to hold a good vacuum, try the black ones everywhere.

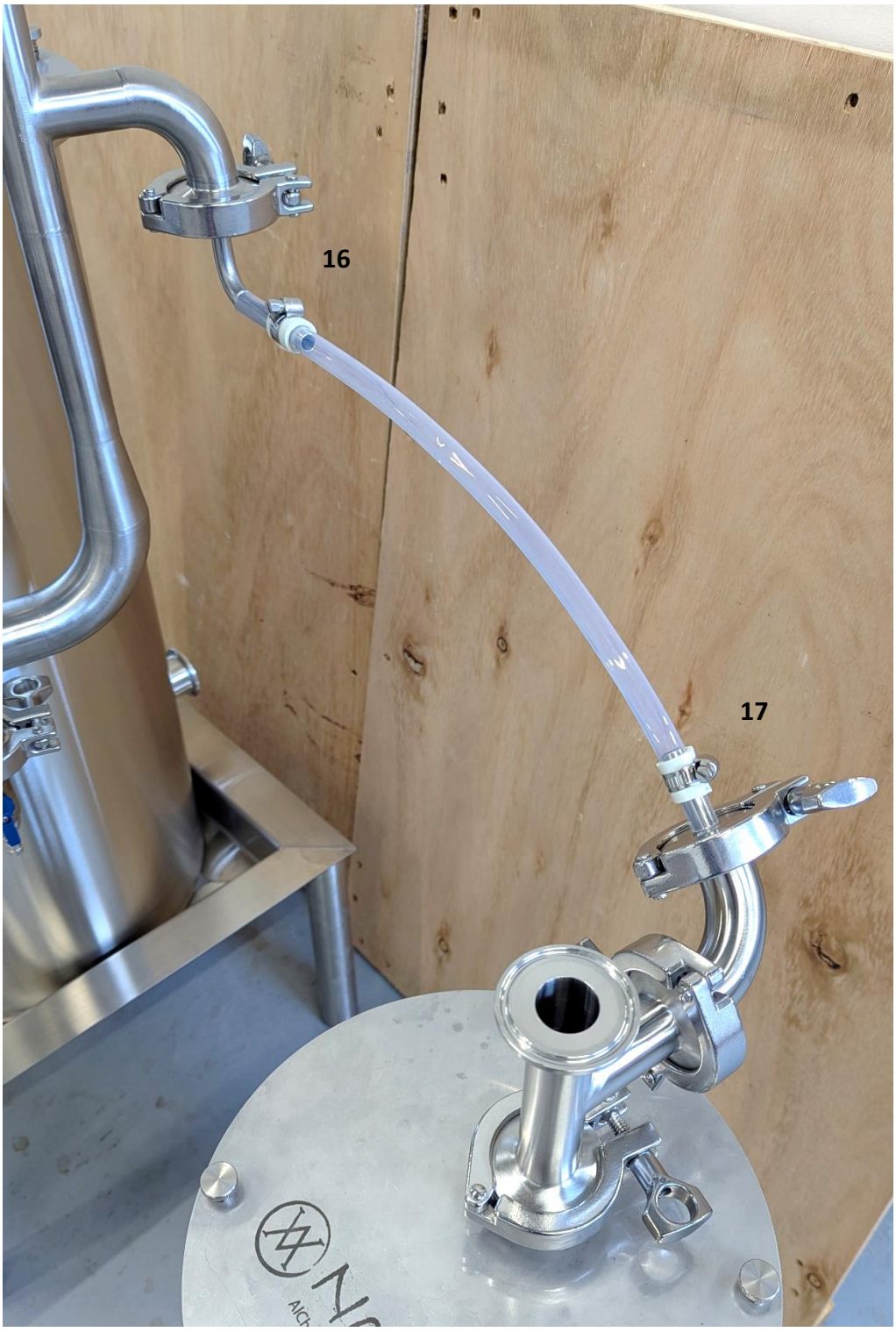
2.5. Installation of the vacuum florentine flask on the *Explorer*

Once the florentine flask is ready as in the picture 6 and 7, connect the tri-clamp 90° elbow 1.5'' (**9**) to the condenser of the *Explorer* (**7**) with a PTFE gasket and a clamp from the *Explorer* (those used with the nozzle in the standard configuration). The separator is quite heavy, especially full of water, so we strongly recommend adjusting the orientation of each part so that the flow sight glass (**12**) and the main welded section (**13**) are close and even rest on the flange and lid (see picture 8). This will provide a much better stability of the system. Note that the glass itself of the flow sight glass should not rest on the flange and lid, but one of the 4 lateral screws can (and should as mentioned above).

Picture 8: resting of the vacuum florentine flask against the *Explorer* flange and lid.

2.6. Connection of the florentine flask to the *Nano*

The connection of the vacuum florentine flask to the *Nano* is achieved by using a clear PTFE tube (picture 9). This PTFE tubing will already be installed on 2 barbed fittings when you receive the vacuum kit. The bended barbed fitting (**16**) goes on the vacuum florentine flask and the straight one (**17**) goes on the *Nano* (as in the picture). Be careful to not bend this tube too much. It is semi-rigid and will squeeze if you bend it too much. Also note that the PTFE tubing should be downward between the florentine flask and the *Nano*. If necessary, you will have to lift the *Explorer* up..



Picture 9: connecting the vacuum florentine flask to the *Nano*

2.7. Connecting the vacuum hose between the still, the receiver and the pump

To get an even vacuum everywhere in the system, the vacuum hoses must be connected as in the picture 10. The vacuum pump (not shown here) is connected to the hose leaving the *Nano* at the bottom right of the picture 10.

Picture 10: connecting the vacuum hoses

2.8. Blocking the cohobation tube of the *Explorer*

The whole system being used under vacuum, the cohobation tube of the *Explorer* (**18**) must be blocked during the vacuum distillation. This can be achieved using the piece you see on the picture 11. On one side it has a rubber stopper that fits into the cohobation tube, then a ball valve and upward, you have the same barbed fitting as the ones on the condenser. If you need to add some water during the vacuum distillation, a piece of hose can be plugged here, then put the end of the tube in a container of water, open the valve and the water will be suctioned into the tank. Immediately close the valve once the desired amount of water has been added.

Picture 11: Blocking the cohobation tube

3. Distilling under vacuum

3.1. Five points to never forget for safety

- DO NOT USE YOUR *EXPLORER* OR *NANO* UNDER VACUUM IF THEY ARE DAMAGED OR IF THEY DO NOT HAVE A PERFECT CYLINDRICAL SHAPE
- NEVER DISTILL UNDER VACUUM WITHOUT THE PILLAR IN THE *EXPLORER* SET AT THE RIGHT HEIGHT
- DO NOT APPLY MORE VACUUM THAN -60 KPA (if you observe the vacuum is lower than -60 kPa at the first use, stop everything, especially the pump and the heat source, and contact us)
- DO NOT APPLY VACUUM IF THE WATER INSIDE THE TANK IS ALREADY HOT, the water would overboil
- ONCE A VACUUM DISTILLATION IS FINISHED, IMMEDIATELY CLOSE THE VACUUM PUMP, SLOWLY BREAK THE VACUUM USING THE VALVE ON THE COHOBATION TUBE AND UNPLUG THE VACUUM PUMP

3.2. Water level in the tank

Even if you can add some water in the tank during a vacuum distillation, we suggest starting with enough water for the whole distillation + 2 to 3 litres as the plant may absorb some water (more or less depending on the plant). As a reminder, if you distill by steam distillation and use the 8'' (20 cm) screws with the grid, you can fit 10 L of water below the grid then you could distill for 2 to 3 hours at 2.5 L/hour without adding water but we would still recommend to check the amount of water left in the tank during the last hour (see below) or add 2-3 L to be sure. If you want to start the distillation with 15 L of water in steam distillation, this is possible, but you would have to lift the grid up. Two options are possible; you can replace the 3 screws of the grid by 3 x 25 cm 1/4''-20 Stainless threaded rod (not included) or cut 3 x 40 cm copper tube (1/2'' – not included either) you will put in diagonal in the tank before the grid. The grid will then sit on the top end of the 3 tubes. This would give you about 22 L of free space below the grid and you could easily poor 15 L of water in the tank without soaking the plant.

3.3. Distilling under vacuum

<u>Preparing the distillation</u>

If you mainly distill for the EO, you can add clean water in the florentine flask before the distillation. If you distill for the hydrolate, don't add water in the florentine flask but steam the whole system for at least 30 minutes to avoid hydrolate contamination (just remove the vacuum gauge from the florentine flask and install the cap that comes with the *Nano* before steaming the unit).

Once you have the pillar with the right height, the grid at the desired height and the right volume of water in the tank, you can pack the plant as usual (as homogeneously as possible and more or less

packed depending on the plant). Then close the still and install all the parts as described above (Pictures 1 to 11) as well as the refilling assembly on the cohobation tube (picture 12).

Starting a vacuum distillation

Once you are ready, tighten all the tri-clamps and hose clamps, and test the whole system for the vacuum: switch the vacuum pump ON, close (screw completely and unscrew 1 turn) the vacuum regulation valve on the vacuum hose and see if the vacuum stabilises around -60 kPa. If it does not reach this relative pressure, check the lid, all the tri-clamps, hoses, hose clamps, valves (must be closed of course), refilling assembly and sight glass. If everything is properly installed and tightened, the pressure should reach -60 kPa within a few minutes (ne needed time depends on your pump and leaks). If it does, adjust the pressure as desired for the whole distillation (between -10 and -50 kPa) and start heating the still. It is important to do this in this specific order: **DO NOT APPLY VACUUM IN THE SYSTEM IF THE WATER IS ALREADY BOILING, the water would overboil**. Vacuum first then boil the water! If the water is warm but below 60°C, you can apply the vacuum but if it is above 60°C (if you cannot keep your hand on the side of the still below the water level), do not apply quick vacuum.

If you lose some vacuum a few minutes after the beginning of the distillation (not impossible with warming gaskets), tighten all the tri-clamps again as well as the hose clamps and the lid screws.

During a vacuum distillation

If you want to get more vacuum during the distillation, cut the heat source and screw the leak regulation valve very slowly before starting the heat again. You may observe (when the pressure is decreasing) a vigorous boiling, but it will not last long if the heat source is off. When the boiling is calm again, the heat source can be restarted.

If you want to get less vacuum during a distillation, do not change anything on the heat source, slowly open the leak regulation valve up to the desired pressure. Adjusting the pressure up is much faster that adjusting it down. The boiling will stop for a few seconds or minutes but will start again as soon as the whole system reaches the new boiling point.

During the distillation, and especially after 2-3 hours, it is important to check if you still have enough water in the still to not run dry. The simplest way is to slightly open the valve on the cohobation tube and listen the sound it makes at the bottom of the still (the vacuum pump can be turned off for this if it is too noisy). If you hear a bubbling sound, you still have at least 2-3 L of water in the still. **If you**

do not hear any bubbling sound, you are about to run dry and you must add more water immediately. You can add between 5 and 15 L depending on the grid height.

If you want to refill the unit with water, you can use a water hose of the same size as the one used for the condenser and suction water into the tank (see section 2.8). **Do not let the valve open more than necessary, here you want to let the least amount of air entering in the still**.

Stopping a vacuum distillation

At the end of the distillation, stop heating the still and switch the pump OFF. Unscrew the lid thumb nuts and break the vacuum by slowly opening the valve on the cohobation tube. **Be careful, unscrew it slowly! The pressure must rise slowly in the still to not force out the essential oil out of the florentine flask**. This way of breaking the vacuum is especially recommended if you distilled for the hydrolate or plan to keep it as the air entering in the system (potentially contaminated), would pass through the hot water and plant material and microorganisms would be stopped by the plant material or killed by the heat. We believe there is less risk of contaminating the hydrolate using the method. Once the vacuum is completely broken, unplug the vacuum hose from the pump.

The hydrolat can be taken from the *Nano* using the drain valve at the bottom of the tank (keep the lid on to reduce the risk of contamination) and the essential oil (with some hydrolat) can be taken from the florentine flask using the drain valve at the bottom of the welded section.

Contact us if necessary

Vacuum distillation is a very interesting technic for plant distillation as you work at a lower temperature and potentially have less chemical degradation. But this is not the easiest way to distill. If you need help, validation, or assistance, please contact us.

Troubleshooting

The main difficulty when starting a distillation under vacuum is the unwanted leaks you may have at any fittings. If everything is assembled and you have some issues to reach the desired vacuum (and if you are sure of your vacuum pump), check all the valves and try to screw a bit harder every tri-clamp fitting. If you feel you don't have enough strength to screw everything manually, you can use a tool to screw a bit more the tri-clamp but do not go too hard either to not break them. As explain at p. 10, you can also use the black EPDM gaskets everywhere instead of the white PTFE gaskets. They are less resistant but softer and give a better sealing. Finaly, you can check the safety vacuum-relief

valve. It should not suction much air below -50 kPa but if your vacuum pump is weak, you might need to adjust it. **Please contact us if you have to do this.**

Annexe 1: Water boiling (°C) point vs absolute pressure in the still (kPa)

Pressure (kPa)	Water BP (°C)
101.3	100
94.4	98
87.8	96
81.5	94
75.7	92
70.2	90
65.0	88
60.2	86
55.6	84
51.4	82
47.4	80
43.7	78
40.2	76
37.0	74