

HOW LONG WILL 100ml (OF WATER) LAST ?

We assume the following:

Volume = 100ml

Pressure = 100psi

Nozzle = 30microns

Turbo pumping speed = maximum ~1400 l/s for N₂ – we guess ~1000 l/s for water

(Note: only below ~7.5E-4 Torr most turbo pumps operate at their maximum pump capacity)

Assume that the gas expands into 4E-4 Torr (0.0532Pa) pressure and that this pressure is stable.

This implies that the pump removes at most 1000 l/s of 4E-4 Torr pressure (water) gas.

In this pressure regime the amount of gas produced by a substance is given by:

$PV=nRT$. $R=8.3144621 \text{ J/(K}\cdot\text{mol)}$, $T=373 \text{ K}$, $n=100\cdot p/(\text{molar mass})$ (for water this is $100\text{g}/(18 \text{ g/mol})=5.5 \text{ mol}$),
 $P=0.0532 \text{ Pa}$. With these values we get $V=320,623 \text{ m}^3=3.21\text{E}8 \text{ l}$.

Divide V by the pump speed to get 321,000 s.

Divide by 3600 s to get hours:

Duration minimum = 89.16 hrs (Water).

Divide by 24 hrs to get days:

Duration minimum = 3.7 days (Water).

To get the pump time for your liquid, find the number of moles m in the liquid reservoir and multiply this number by $m/5.5$.

PUMP DOWN OF THE DRY LIQUID-TO-GAS MANIFOLD: Start-up Phase

Preparation !!!

In case you need to evacuate the >>tank<< make sure you have the valve $V_{\text{evac-tank}}$ open during the pump down of the chamber. **If you forgot to evacuate during the pump down phase you need to make sure that the “hidden bypass” and the bypass between the chamber and the 2nd stage is closed; open $V_{\text{evac-tank}}$ very slowly.**

Get the Liquid-to-Gas Manifold ready

1. Close V_{drain}
2. Close C_{aux}
3. Close and lock V_{transfer}
4. Close and lock $V_{\text{gas-manifold}}$
5. Close $C_{\text{reservoir-drain}}$ or V_{seed} and V_{supply} in case you use the optional liquid and gas supply arm
6. Close C_{aux}
7. Close V_{bypass}
8. Close V_{protect}
9. Open V_{blocking}
10. Open $V_{\text{evac-line}}$

Start “chem-lab” diaphragm pump in controller rack

11. Open $V_{\text{three-way}}$ to pump
12. Pump for 1 minute
13. Open V_{nozzle}
14. Pump for 1 minute and check Pressure Gauge (should go down)
15. Close $V_{\text{three-way}}$

Make sure the chamber fore-vacuum is running (especially scroll at 2nd stage)

16. Open V_{bypass} slowly only when the manifold and the reservoirs are dry !
17. Open V_{protect}
18. Let it pump for 10 minutes

START/STOP HEATING

INSIDE THE CHAMBER

Use heat controller on Controller Rack

Heat the gas line first

set desired temperature to 120deg C: **DO NOT GO HIGHER IN TEMPERATURE !!!**

- turn on controller unit (switch in the back)
- select thermocouple and temperature units (1.: press “↵” for 3 sec. , press “↶” till you get “Inpt”, press “▲” or “▼” till you get “J”, only if “J” is flashing hit “↵”. 2.: Press “↶” till you get “tpun”, press “▲” or “▼” till you get “C”, only if “C” is flashing hit “↵”.)
- enable PID mode (press “↶” till you get “ctrl”, press “▲” or “▼” till you get “PID”; only if “PID” is flashing hit “↵”)
- set desired temperature (press “↵”, press “▲” or “▼” and then confirm with “↵”)
- turn on the heater controller (flick the switch labeled “heater controller on” up; check if fans are running)
- start PID controller (press “↶” till you get “r-S”, press “▲” or “▼” till you get “run”, only if “run” is flashing hit “↵”)
- set digital trimmer to restrict max. current (set dip switch to “10”)

Heat the nozzle next

set desired temperature to 125deg C: **DO NOT GO HIGHER IN TEMPERATURE !!!**

=> see instructions above

To stop heating:

- stop PID controller (press “↶” till you get “r-S”, press “▲” or “▼” till you get “stop”, if flashing hit “↵”)
- set digital trimmer to restrict min. current (set dip switch to “00”)

See page 8 for the heating outside the chamber

MAKE A LIQUID-TO-GAS JET

Prepare the Chamber

1. Close all chamber bypasses
2. Set Vacuum Gauge controller to appropriate ranges
(Jet Dump: 1E-7, Chamber: 1E-7, 2nd Stage: 1E-6, Source: 1E-4)

Prepare the Liquid-to-Gas Manifold

3. Reservoir Heater should be cold (set it to 20deg C); remove sleeve & cool it down with a “water bucket” if needed;. **Note: you cannot and should not transfer liquid into the hot reservoir (safety issue !).**
4. Close V_{nozzle}
5. Close V_{protect}
6. Close V_{blocking}
7. Close $V_{\text{three-way}}$
8. Close and lock V_{evac}
9. Close V_{seed}
10. Close V_{supply}

Option A to fill the Reservoir: Prepare the Transfer Arm of the Manifold

11. Close V_{transfer}
12. Close C_{aux}
13. Close $C_{\text{supply-drain}}$
14. Open V_{drain}
15. **Wear your Personal Protective Equipment (Gloves, Glasses etc.):** Fill liquid into small metal funnel slowly to avoid bubbles which can clog the filling mechanism; stop when liquid reaches desired level in glass indicator funnel.
16. Close V_{drain}

Transfer the Liquid

17. Open V_{transfer} slowly and watch the level in the glass indicator funnel drop to desired level
18. Close and lock V_{transfer}

See next page for Option B to fill the Reservoir =>

Option B to fill the Reservoir: Prepare and use the Syringe

11. Close and lock V_{transfer}
12. **Wear your Personal Protective Equipment (Gloves, Glasses etc.):** Attach the needle to the syringe and suck the (toxic or hazardous) liquid (150ml max) from a beaker in a fume hood.
13. Close V_{syringe} and detach the needle
14. Connect Syringe assembly to V_{supply} (pink nut)

Pump out the connector between V_{supply} and V_{syringe}

15. Open V_{blocking}
16. Open $V_{\text{evac-line}}$
17. Open $V_{\text{three-way}}$ to the diaphragm forepump
18. Pump down for 10 seconds and check Pressure Gauge (should go down)
19. Close $V_{\text{three-way}}$
20. Close $V_{\text{evac-line}}$
21. Close V_{blocking}

Transfer the Liquid

17. Open V_{syringe} and slowly press the plunger of the syringe down to the desired milliliter value
18. Close V_{syringe}
19. Close V_{supply}
20. The syringe may be disconnected at V_{supply} (pink nut); if so cap V_{supply}

See next page for instruction how to start the jet =>

continue: MAKE A LIQUID-TO-GAS JET

Make a (cold) jet

1. Open V_{blocking}
2. Open V_{nozzle} slowly (2 to 3 seconds)

Flush out the gasline when needed (when seeding a buffer gas may be used instead: see Seeded Jet section):

3. Close V_{blocking}
4. Open $V_{\text{evac-line}}$
5. Open $V_{\text{three-way}}$ to exhaust for 3 seconds
6. Open $V_{\text{three-way}}$ to the forepump and pump for 3 seconds (not longer to avoid water pollution of the pump !)
7. Close $V_{\text{three-way}}$
8. Close $V_{\text{evac-line}}$
9. Open V_{blocking}
10. Start outside Heaters (set to desired temperatures; for the reservoir it is 170deg C max. It is recommended to go up in steps like 50, 80, 100, 120, 150, 170deg C): see next page for instructions
11. Watch the vacuum gauges at the controller rack


START/STOP HEATING - continue

OUTSIDE THE CHAMBER

Use heat controllers next to Gas Manifold

Heat the gas line first

set desired temperature to max 125deg C: **DO NOT GO HIGHER IN TEMPERATURE !!!**

- first press the “reset” button on the yellow GFCI plug to supply 110VAC
- turn on the unit (red button)
- **watch out: Unit may immediately starts running if somebody didn't dial it down before – act fast to stop it**
- (unit is preset to deg C units and J thermocouples)
- select your heating temperature (1.: press “


Heat the reservoir next

Keep it cold for the transfer of the liquid; you cannot and should not transfer liquid into the hot reservoir

(safety issue !). Set to desired temperature after transfer (165deg C max). It is recommended to go up in steps like 50, 80, 100, 120, 150, 165 deg C...

=> see instructions above

To stop heating:

- put temperature to “20” temperature (1.: press “

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STOP AND START JET IN VACUUM CHAMBER

Stop the Jet

1. Close V_{blocking}
2. Open $V_{\text{evac-line}}$
3. Open $V_{\text{three-way}}$ to exhaust slowly (should take 3 seconds)
4. Open $V_{\text{three-way}}$ to diaphragm pump and pump for 10 seconds (not longer since condensables are bad for the diaphragm pump) , then close $V_{\text{three-way}}$ again
5. Do NEVER pump wet reservoir or gas lines via the bypass to the second stage (it will pollute and damage the scroll forepump).

Restart the Jet

1. Close $V_{\text{three-ways}}$
2. Close $V_{\text{evac-line}}$
3. Open V_{blocking}

SEED JET WITH NOBLE GAS: USE OF THE OPTIONAL GAS SUPPLY SYSTEM

Prepare the Gas Supply System

1. **The reservoir should be empty and dry and cold**
2. Connect the diaphragm pump to gas supply system
3. Close V_{blocking} and V_{transfer}
4. Open C_{drain} and connect gas supply system to reservoir
5. Connect the gas supply system to the regulator of the gas bottle
6. Close V_{exhaust}
7. Open V_{seed} , any valves in front of the gas regulator, and the regulator itself to pump to the main valve of the bottle (which should be closed)
8. Open V_{pump} and pump for 5 minutes

Make a seeded Jet

9. Close V_{seed} , V_{pump} , and the regulator (keep any valve in front of the regulator open)
10. see **MAKE A LIQUID-TO-GAS JET**
11. **Note:** It is recommended to start with a low reservoir temperature after the liquid transfer like 50deg C and observe the vacuum gauges
12. To avoid the liquid being pushed through the nozzle apply He buffer gas with a pressure that is higher than the seed gas via $C_{\text{dry-out}}$: Open $V_{\text{dry-out}}$ to dial in the pressure while $V_{\text{evac-line}}$ and V_{nozzle} are open after this section has been pumped out; note the ion gauges need to be preset for this jet. Now open V_{blocking} and close $V_{\text{evac-line}}$.
13. Open the gas bottle and dial in a pressure of ~30 to 50 psi. Then open V_{seed} and watch the gauges. Wait for the He to get out (~10 to 15 minutes)
14. Increase temperature of the liquid and pressure of the gas in an alternating way (20 to 40deg C & 20 psi steps)
15. For a water jet of 170deg C at the reservoir the Ar pressure is supposedly ~120 psi
16. Feel the Knee with your finger (**caution:** warm to hot): You likely will feel a temperature gradient which can be moved to the left and right with increasing the temperature of the reservoir and increasing the gas pressure

Stop the seeded Jet

17. Close V_{seed} and the gas bottle
18. See **STOP AND START JET IN CHAMBER** or **STOP ENTIRE LIQUID-TO-GAS JET OPERATION OR MAKE A REFILL**
19. Open C_{exhaust}
20. Open V_{exhaust} slowly to drain the gas (should take 2 to 3 seconds)
21. Close V_{exhaust}
22. Open V_{pump} and pump for 1 minute
23. Close the gas regulator and V_{pump}

STOP ENTIRE LIQUID-TO-GAS JET OPERATION OR MAKE A REFILL

Stop Jet in Chamber

1. Close V_{seed}
2. Close V_{blocking}
3. Open $V_{\text{evac-line}}$
4. Open $V_{\text{three-way}}$ to exhaust slowly (should take 5 seconds)
5. Open $V_{\text{three-way}}$ to pump and pump for 15 seconds, then close $V_{\text{three-way}}$ again
6. Close V_{nozzle}

Re-transfer Liquid back to Supply Reservoir (for non hazardous liquids only !)

7. Close V_{blocking}
8. Close V_{drain}
9. While the Reservoir and the gas line is hot slowly open V_{transfer} and monitor the liquid level in the glass funnel. Close V_{transfer} when the level stopped rising and no more bubbles make it to the surface.

If your Chemical is hazardous or toxic:

10. Do not re-transfer the liquid but open $V_{\text{evac-line}}$ and open $V_{\text{three-way}}$ to exhaust
11. Monitor with the ion-gauges and the COLTRIMS spectrometer if the jet is fading. You can close $V_{\text{three-way}}$ and check if the source chamber is falling or rising after a while; if it is still rising there is still liquid – if it is stable or falling you can now pump on it.
12. You may want to heat up the reservoir if it safe to accelerate the process. With 100 deg C it can take 2h for ~100ml.
13. Alternatively use Syringe to suck out the reservoir: Open V_{supply} open V_{syringe} and pull the plunger as far out as possible. Close V_{supply} close V_{syringe} . Detach syringe (pink nut) and bring it to a fumehood to drain it. Cap V_{supply} .

Drain (and refill) the Reservoir

13. Stop the reservoir heater, i.e. set the heater to 20degC (not higher !) and monitor the cool-down process. Wait for the temperate to go down to <30deg C. You may want to use a bucket with water to cool down the reservoir after carefully removing the heater sleeve (**caution:** hot on the inside).
14. In case you want to refill the reservoir close V_{nozzle} , open V_{blocking} and $V_{\text{evac-line}}$ and open $V_{\text{three-way}}$ to the pump and pump for 20 seconds, then close $V_{\text{three-way}}$, $V_{\text{evac-line}}$ and V_{blocking} and transfer more liquid using the transfer arm or the syringe.

See next page =>

continue: STOP ENTIRE LIQUID-TO-GAS JET OPERATION OR MAKE A REFILL

In case you DO NOT want to refill:

- Step 1: Clean the gas lines

15. Keep Heaters of outside and inside gaslines on
16. Close V_{blocking}
17. Open $V_{\text{evac-line}}$
18. Open $V_{\text{three-way to forepump}}$
19. Pump out as long as you can (>1day if possible)

- Step 2: Pump out the Reservoir

20. Make sure V_{nozzle} is closed
21. Wear your PPE (Gloves, Glasses, etc.): Open $C_{\text{reservoir-drain}}$ and drain the liquid into a beaker
22. Open $C_{\text{dry-out}}$ and attach dry gas with reasonable flow which you can control by opening $V_{\text{dry-out}}$
23. Open $V_{\text{evac-line}}$
24. Open V_{blocking}
25. Close $V_{\text{three-way}}$ to the pump just a little bit to give some gas ballast; watch for liquid coming out of the pump exhaust
26. Let it run for 2 to 12 hours
27. Now detach dry gas and close $V_{\text{dry-out}}$, $C_{\text{dry-out}}$ and $C_{\text{reservoir-drain}}$. You can now pump out the reservoir by opening $V_{\text{three-way}}$ for at least 3 minutes. Optional: Close $V_{\text{three-way}}$ afterwards, open V_{protect} and V_{bypass} to pump with scroll.

CLEAN-UP PROCESS

Clean out the Supply Vessel

1. Close V_{transfer}
2. Wear your PPE (Gloves, Glasses, etc.): Open $C_{\text{supply-drain}}$
3. Open V_{drain} and empty liquid into a beaker
4. Attach a dry gas to $C_{\text{supply-drain}}$ and dial in a decent flow by opening $V_{\text{dry-out}}$
5. Let it run for 2 to 12 hours

Clean the Syringe

1. Wear your PPE (Gloves, Glasses, etc.)
2. Empty the syringe in the fume hood by retransferring as much liquid into a beaker as possible
3. **Note:** There is likely still <5ml of liquid in the syringe even if the plunger is all the way in.
4. Open the syringe in the fume hood by taking out the plunger back-stop (using a screwdriver to take of the 6 little screws) and drain all fluids
5. Clean syringe with isopropanol using a soaked tissue and long tweezers (note: don't scratch the stainless steel surface)
6. Use silicon (vacuum) grease to lightly lubricate the two O-rings on the plunger

Regenerate the Forepump(s)

In case you polluted the forepumps with any condensable gases open the gas ballasts or detach forepump and let it pump a small amount of (dry) air, i.e. you need to restrict the flow with either a small pin hole in a Kwik-Flange plastic cap or a screw with a washer in the (red water) hose or similar.