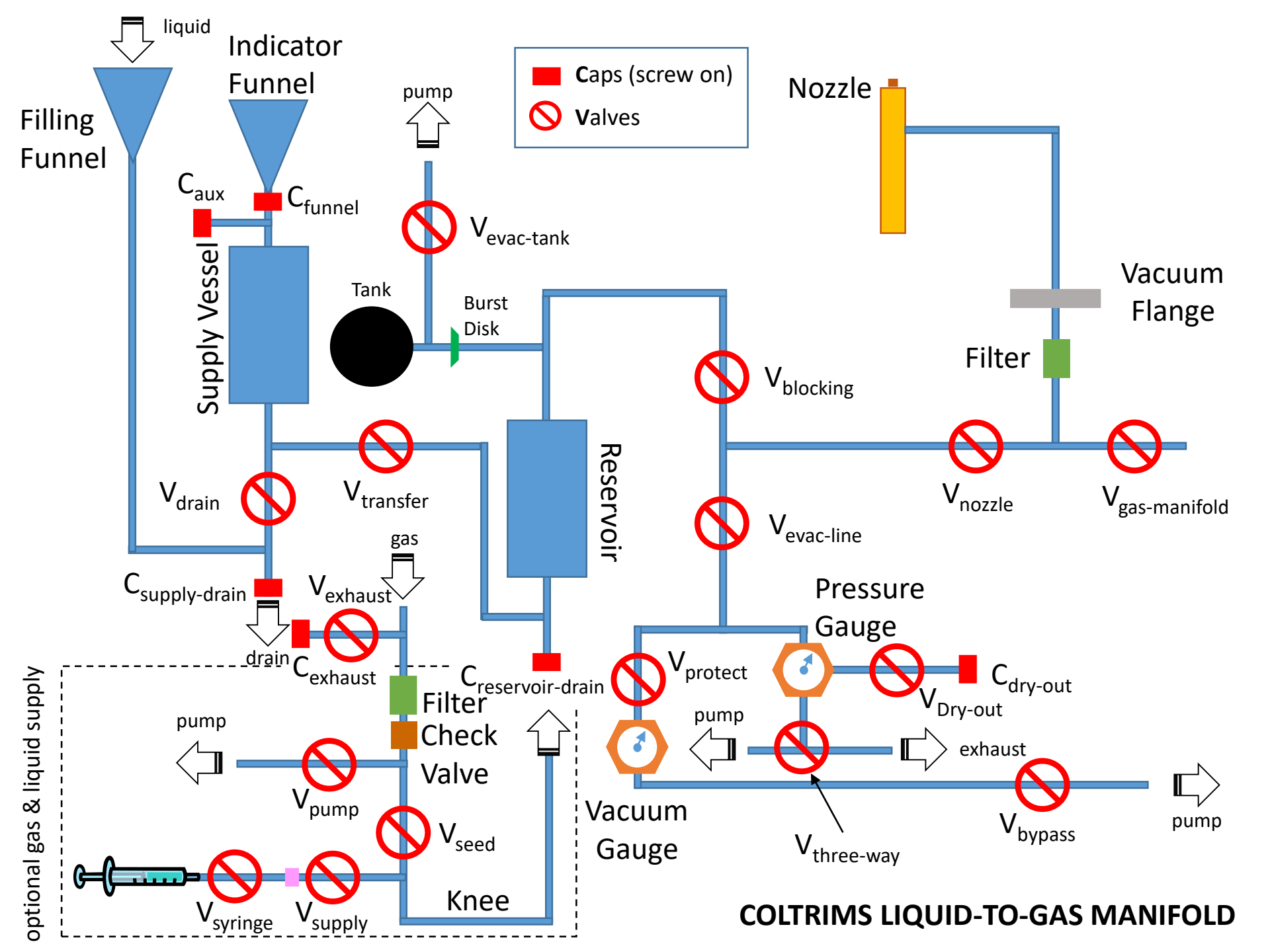
liquid
Indicator Funnel
Filling Funnel
pump
Caps (screw on)
Valves
Nozzle
C_{aux}
C_{funnel}
$V_{evac-tank}$
Supply Vessel
Tank
Burst Disk
Vacuum Flange
Filter
$V_{blocking}$
V_{drain}
$V_{transfer}$
Reservoir
V_{nozzle}
$V_{gas-manifold}$
$V_{evac-line}$
gas
$C_{supply-drain}$
$V_{exhaust}$
Pressure Gauge
drain
$C_{exhaust}$
$C_{reservoir-drain}$
$V_{protect}$
$C_{dry-out}$
$V_{Dry-out}$
Filter
Check Valve
pump
pump
exhaust
V_{pump}
V_{seed}
Vacuum Gauge
$V_{three-way}$
V_{bypass}
pump
$V_{syringe}$
V_{supply}
Knee
optional gas & liquid supply
COLTRIMS LIQUID-TO-GAS MANIFOLD

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 N_2 – 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 J/(K*mol), T = 373 K, n = 100*p/(molar mass) (for water this is 100g/(18 g/mol) = 5.5 mol), P = 0.0532 Pa. With these values we get V = 320,623 m^3 = 3.21E8 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 $V_{evac-tank}$ 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 $V_{evac-tank}$ 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 $V_{gas-manifold}$
5. Close $C_{reservior-drain}$ 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 $V_{evac-line}$

Start "chem-lab" diaphragm pump in controller rack

11. Open $V_{three-way}$ 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 $V_{three-way}$

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" to "15", i.e. 10 to 15% of 5A; **the lower the better! Recommended: "10" for nozzle and "13" for gasline**)

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 $V_{three\text{-}way}$
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 $C_{supply\text{-}drain}$
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 $V_{evac-line}$
17. Open $V_{three-way}$ to the diaphragm forepump
18. Pump down for 10 seconds and check Pressure Gauge (should go down)
19. Close $V_{three-way}$
20. Close $V_{evac-line}$
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 $V_{evac\text{-}line}$
5. Open $V_{three\text{-}way}$ to exhaust for 3 seconds
6. Open $V_{three\text{-}way}$ to the forepump and pump for 3 seconds (not longer to avoid water pollution of the pump !)
7. Close $V_{three\text{-}way}$
8. Close $V_{evac\text{-}line}$
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 ”▭”, 2.: change red value with “▲” or “▼”, 3: confirm with “↵”, 4: leave menu with pressing ”▭” twice)

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 ”▭”, 2.: change red value with “▲” or “▼”, 3: confirm with “↵”, 4: leave menu with pressing ”▭” twice)
- verify that the heating stops and the temperature goes down
- turn off the unit (red button)
- press the “test” button on GFCI plug to cut the AC power

STOP AND START JET IN VACUUM CHAMBER

Stop the Jet

1. Close $V_{blocking}$
2. Open $V_{evac\text{-}line}$
3. Open $V_{three\text{-}way}$ to exhaust slowly (should take 3 seconds)
4. Open $V_{three\text{-}way}$ to diaphragm pump and pump for 10 seconds (not longer since condensables are bad for the diaphragm pump) , then close $V_{three\text{-}way}$ 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 $V_{three\text{-}ways}$
2. Close $V_{evac\text{-}line}$
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 $C_{dry\text{-}out}$: Open $V_{dry\text{-}out}$ to dial in the pressure while $V_{evac\text{-}line}$ 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 $V_{evac\text{-}line}$.
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 $V_{evac\text{-}line}$
4. Open $V_{three\text{-}way}$ to exhaust slowly (should take 5 seconds)
5. Open $V_{three\text{-}way}$ to pump and pump for 15 seconds, then close $V_{three\text{-}way}$ 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 $V_{evac\text{-}line}$ and open $V_{three\text{-}way}$ to exhaust
11. Monitor with the ion-gauges and the COLTRIMS spectrometer if the jet is fading. You can close $V_{three\text{-}way}$ 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 $V_{evac\text{-}line}$ and open $V_{three\text{-}way}$ to the pump and pump for 20 seconds, then close $V_{three\text{-}way}$, $V_{evac\text{-}line}$ 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 $V_{evac\text{-}line}$
18. Open $V_{three\text{-}way\ to\ forepump}$
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 $C_{reservoir\text{-}drain}$ and drain the liquid into a beaker
22. Open $C_{dry\text{-}out}$ and attach dry gas with reasonable flow which you can control by opening $V_{dry\text{-}out}$
23. Open $V_{evac\text{-}line}$
24. Open $V_{blocking}$
25. Close $V_{three\text{-}way}$ 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 $V_{dry\text{-}out}$, $C_{dry\text{-}out}$ and $C_{reservoir\text{-}drain}$. You can now pump out the reservoir by opening $V_{three\text{-}way}$ for at least 3 minutes. Optional: Close $V_{three\text{-}way}$ 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 $C_{supply\text{-}drain}$
3. Open V_{drain} and empty liquid into a beaker
4. Attach a dry gas to $C_{supply\text{-}drain}$ and dial in a decent flow by opening $V_{dry\text{-}out}$
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.