Additional information on the dripper design

Supplies

- Syringe pump: Harvard Apparatus / Modified 702019 – LEADSCREW & 1/2 NUT = 40 THREADS PER INCH / part 982577
- Heating plate: Cole-Parmer / Jenway stirring hot plate with temperature probe / part C-84200-00
- Evaporating flask: 1L glass flask from Cole-Parmer
- Syringes: Cole-Parmer / Hamilton gastight syringe 0.25-2.5 cc
- Three-way valves: The Lee Company 860-399-6281 / 3-way valve, barbed fitting 12V / part LFRX05000050BC
- Large capacity molecular sieve: Praxair / moisture trap / part PRXSG6191-1, 400cc 1/4" (note: we had mixed results with this product: of the three traps we used, two dried air down below 10 ppm but one could not dry air below 50 ppm.)
- Small capacity molecular sieve: SGE / moisture trap / part number 103480 (note: this trap can produce dry air below 0.5 ppm.)
- Mass flow controller: Omega / MFC model FMA5416 / power supply: FMA545PW / connector cable: FMA545C
- Needle valves: Swagelok / metering valves / part SS-SS4
- Dry air generator: Twin Tower Engineering / Dry air package dew point -100oC / part 51785

Continuous dripping versus interrupted dripping

The Harvard Apparatus syringe pump carries two syringes in opposite directions (infuse versus refill). It reports the pump direction via its serial port which is used to control the three-way Lee valves. Because the two Lee valves operate in opposite directions, the dripping configuration (see the attached drawing) provides continuous water flow to the evaporation flask. Such continuous and stable “dripping” rate is desirable if frequent calibration is required (ie every min).

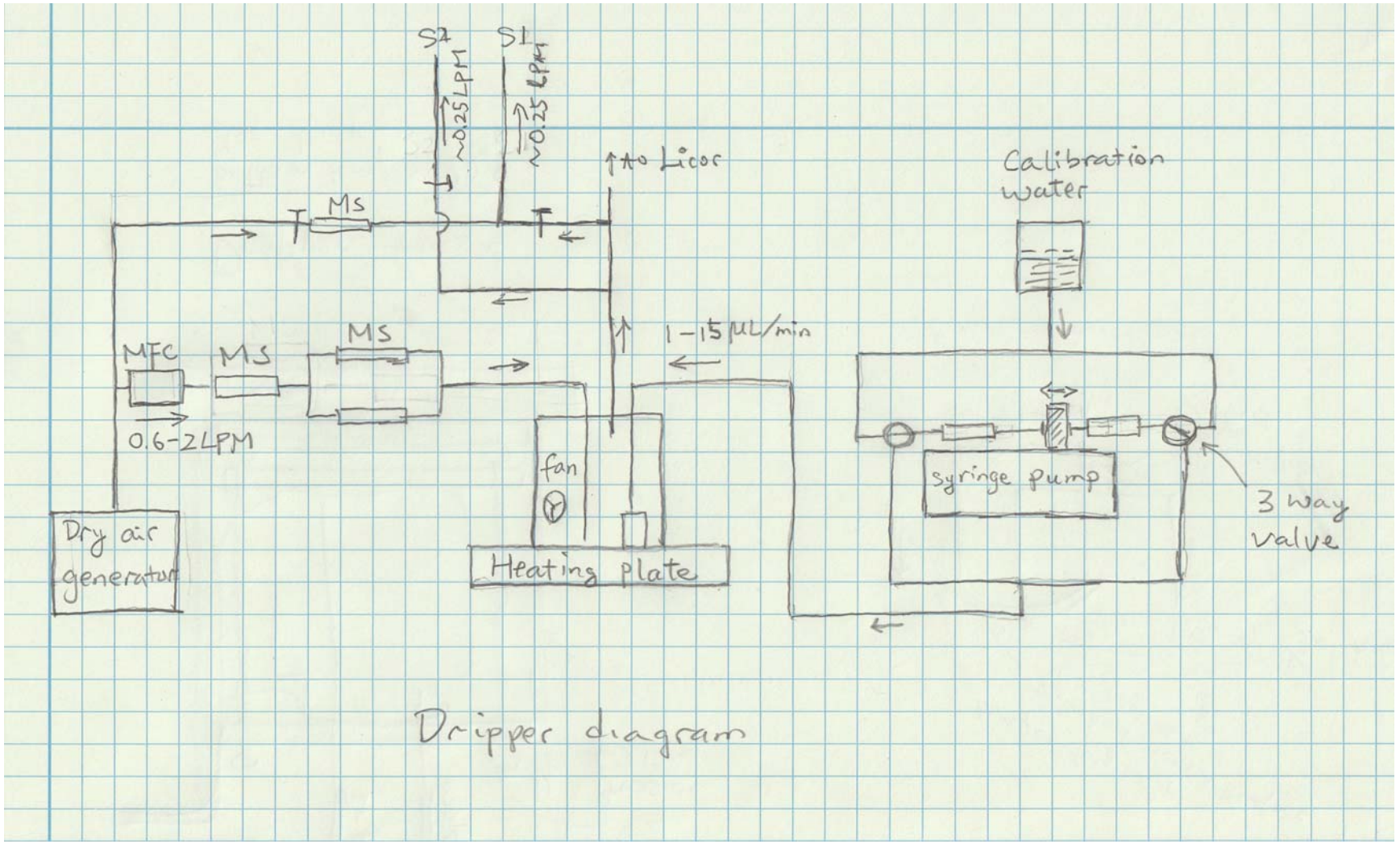
If calibration is done once every few hours, continuous water delivery may not be necessary. In that case, you can use a less expensive syringe pump (ie SyringePump.com/NE-1000 single syringe pump) with a 3-way check valve to accomplish infuse and refill operation. (You can get free check valve samples from Qosina.) Water delivery is interrupted periodically when the pump reverses from the infuse to the refill mode. The NE-1000 pump allows you to specify a refill rate that is much higher than the infuse rate, minimizing down time to ~ 60s.

Dripper instability

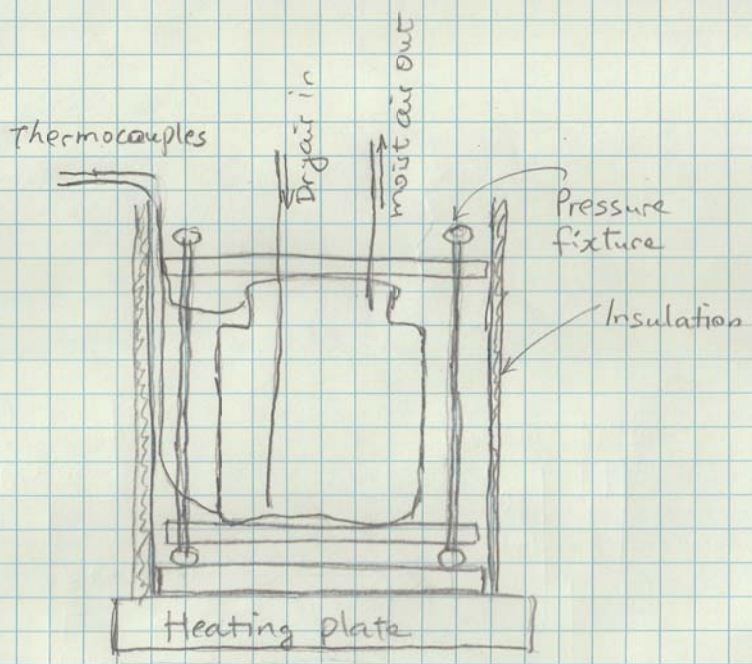
In theory, the configuration shown in the attached diagram should provide steady water delivery. However, degassing of the calibration water often results in bubble formation in the delivery tube. When these bubbles move to the evaporating surface, the mixing ratio of the moist air exiting the evaporation flask will drop abruptly. When this happens, the isotopic ratio can deviate from the calibration value by up to 10 per mil (18O).

When the syringe pump changes direction, flow will stop for a brief period of time. Use of electric valves instead of check valves cuts down this dead period (to 10-60 s depending on syringe size and delivery rate) but does eliminate it completely.

Finally, we find that when dripping rate is < 3 uL/min, the dripper will occasionally go through periods of oscillation in an unpredictable manner. Because the humidity target is achieved by adjusting the dripping rate, the dry air flow rate, or both, low dripping rate can be avoided by supplying dry air at a high flow rate. (If you use bottled dry air, however, this may not be a good option.) Also use of small syringes helps the dripper to improve its stability in low humidity conditions.

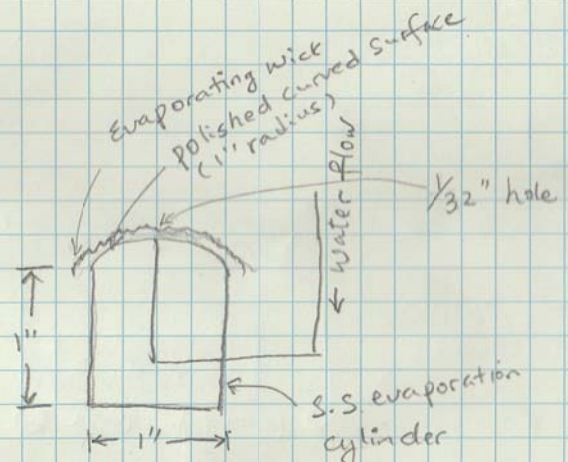


Dripper diagram



Dripper flask

bottom temp ~ 70°C
top temp ~ 65°C



Evaporation block