Setup for Collecting gas for ^{14}C of soil respired CO_2 *in situ*

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Background

- There are lots of ways to do this and the best one depends on a lot of things!
  - Existing field setups to utilize/new installations
    - Gas well profiles are awesome but a lot of effort.
    - Dynamic chamber systems might be worth tapping into for high frequency/repeated measurements, especially with automated flasks.
    - Static chamber lids can be easily constructed for a variety of collar sizes and experimental setups.
  - Number of samples to be collected
    - Tons of samples or backpacking might warrant using MSC traps instead of flasks.
    - Field sites close to the lab might allow for airbags instead of flasks.
    - Field sites close to vehicle parking make flasks easier.
  - Field logistics
  - Research questions and objectives
    - Stable isotope measurements require more care to avoid fractionation

Outline of Steps

1. Identify setup/make chambers if needed.
2. Measure CO_2 flux rates or have an estimate from a previous measurement.
3. Purge/scrub chamber or well system of CO_2.
   - Or take a t0 sample if not possible to scrub volume
4. Close and let CO_2 accumulate to 3-4 times ambient for a 1 L flask (higher for smaller, can go lower for larger sampling volumes).
5. Collect headspace/well gas into sampling vessel.

1. Field site infrastructure

- Can you use existing flux chamber systems?
  - Licor automated chambers are easy to tap into.
  - Licor survey chambers aren't so easy – might want to build static chamber lids for gas sampling.
- Let new collars equilibrate if you need to use them.

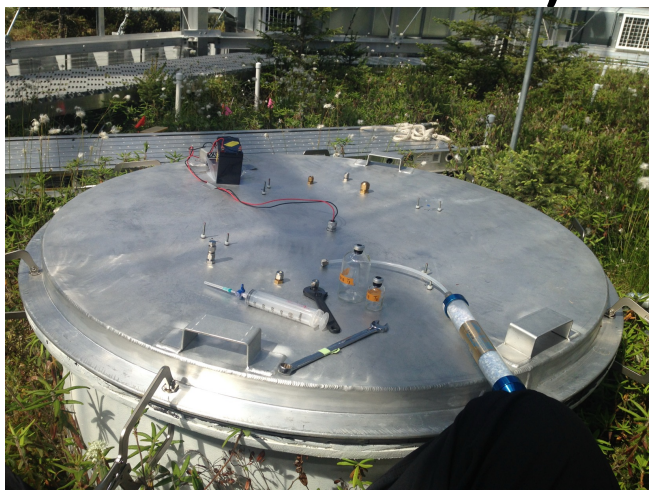
LEFT: Disconnecting Licor auto-chambers and tapping into existing lines with quick connect bulkheads is straightforward (here at TRACE in Puerto Rico).

1a. Constructing Static Chamber Lids

PVC lid for small PVC collars

LEFT: 24 cm diameter collars used with Picarro. RIGHT: 15 cm and 30 cm diameter collars used with Licor survey chambers.

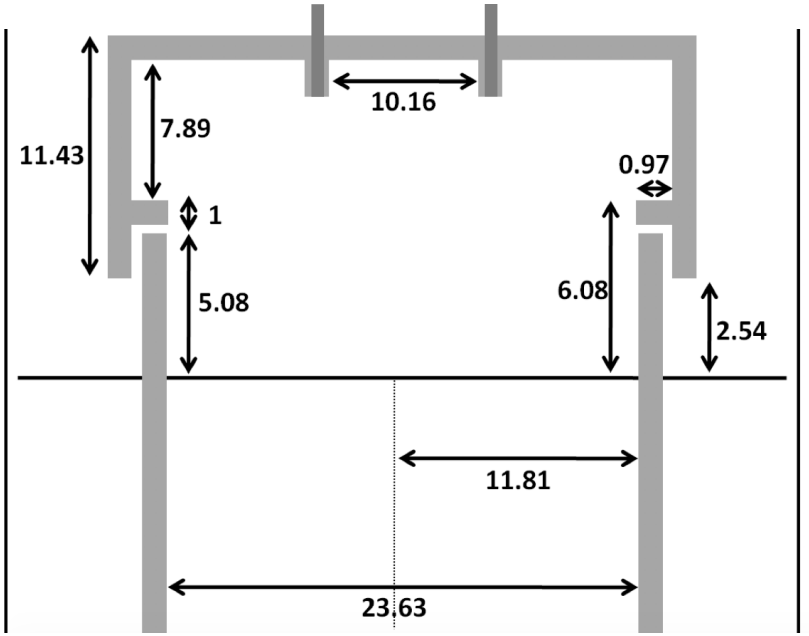
Steel lid for large collars (1 diameter at SPRUCE)



Lids should have:

- At least 2 sampling ports to allow headspace volume to be purged and for samples to be collected
- Tight-fitting seal to exclude atmosphere – foam gasket, clamps if necessary for collar design
- **NO high VOC adhesives!** Silicone sealant and silicone gaskets and septa are preferable.
- Large chambers should have fans to circulate air inside chamber for long periods of accumulation (e.g., SPRUCE lids for CH4 isotopes).

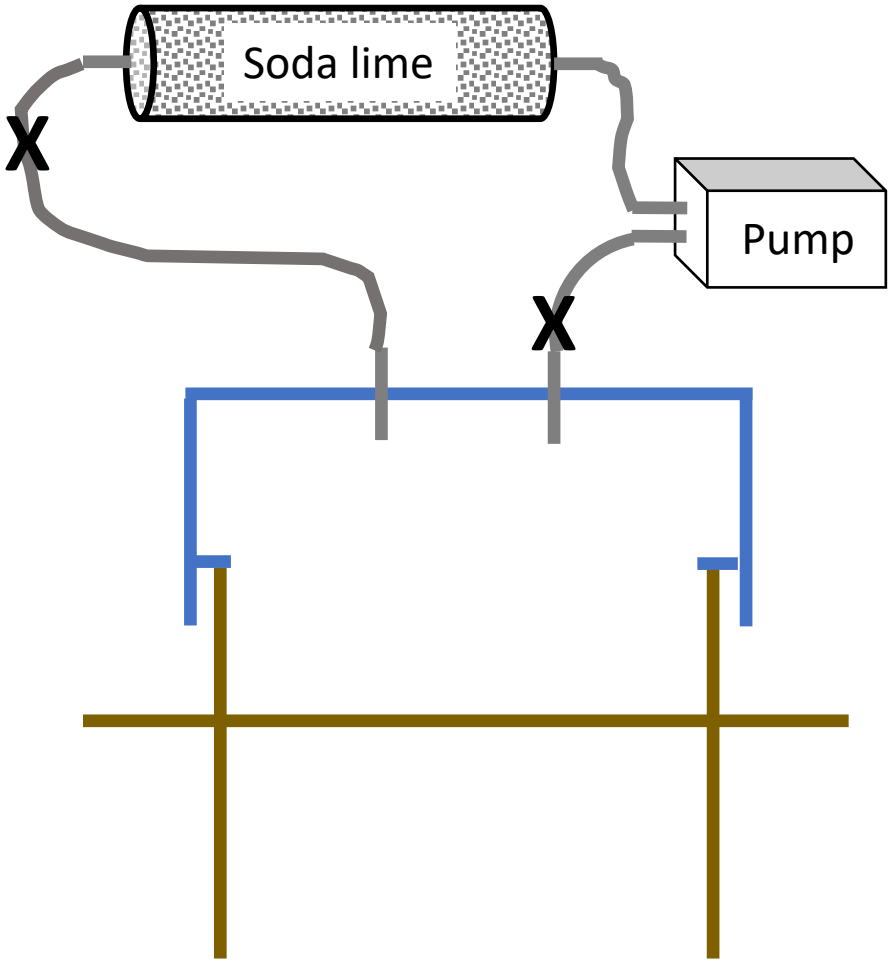
Ex. 24 cm d Picarro chamber lid (Nobel Foundation)



2. Estimate time required for CO_2 to accumulate

- Based on:
  - Measured or estimated flux rate
  - Volume to be sampled
  - Volume of system
  - Length of time for sampling
- Objective is to have close to 1 mg C in sampling vessel (**definitely** more than 0.25 mg).
- For 1L flasks, outfitted with a flow controller, accumulate at least 2000 ppm CO_2. (This is enough for CAMS, but maybe not higher size requirements by other AMS labs!)

3.Purge/scrub CO_2 from headspace volume



1. Connect air pump and soda lime trap to chamber ports in one loop.
2. Allow air to recirculate for as long as it takes to purge 3-4 x headspace volume
   - E.g., Nobel lids are 7.2 L, my pump is 1 Lpm, I pump for _ minutes
3. Close valves (black X's). Disconnect disconnect pump. Leave tubing with closed valve connected. Remove soda lime trap.
   - OR SKIP VALVE AND PUT CAPS ON both CHAMBER BULKHEADS! (will have a little leak when swapping tubing)

If not possible (like SPRUCE high volume chambers) :

- Time 0, background air sample for ^{14}C and pCO_2
- Final air sample for ^{14}C and pCO_2
- use a mixing model to determine respiration end member

Scrubbing a 30 cm diameter soil flux collar chamber made from PVC at SWELTR (McFarlane et al., in review).

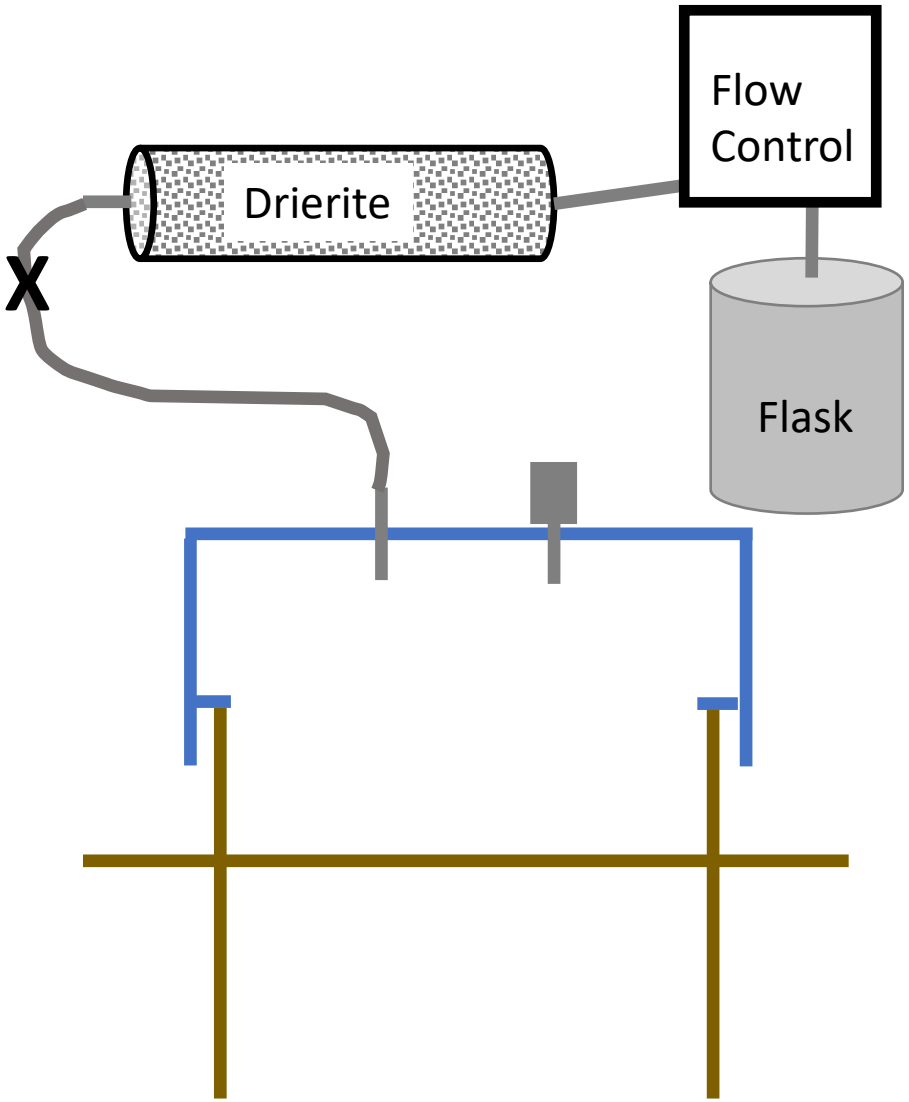
4. Close and let CO_2 accumulate

- For however long is anticipated to allow >2000 ppm accumulation

Accumulating CO2 in a 15 cm diameter soil flux collar chamber made from PVC at SWELTR (McFarlane et al., in review).

About to sample! No Drierite trap used in the above photo (Oklahoma in a dry summer), but at wet sites one is needed between the chamber lid and the flow controller to reduce water vapor. A water trap is also needed if using a molecular sieve trap (it will also trap water, not just CO2).

5. Collect headspace - Flasks



1. Connect water trap, then flow controller, then evacuated flask to closed off tubing connected to chamber fitting.
   - Flow controller is there to avoid mass dependent isotopic fractionation during filling. This eliminates need to leave system open long enough for isotopic equilibrium.
2. Open valve to water trap.
3. Open flask. Flow controller will slowly fill to just below atmospheric pressure.
4. Close flask when vacuum guage on flow controller reads -5” Hg.
   - 10 minutes with the 1 L flask and 80 ml/min flow controller). If it takes longer, flow is restricted and air is likely being pulled from outside of the collar (deeper or laterally).
   - Can use a capillary tube instead of the flow controller (less expensive and simpler, but no pressure guage – have to calculate fill time to sub-atmosphere).

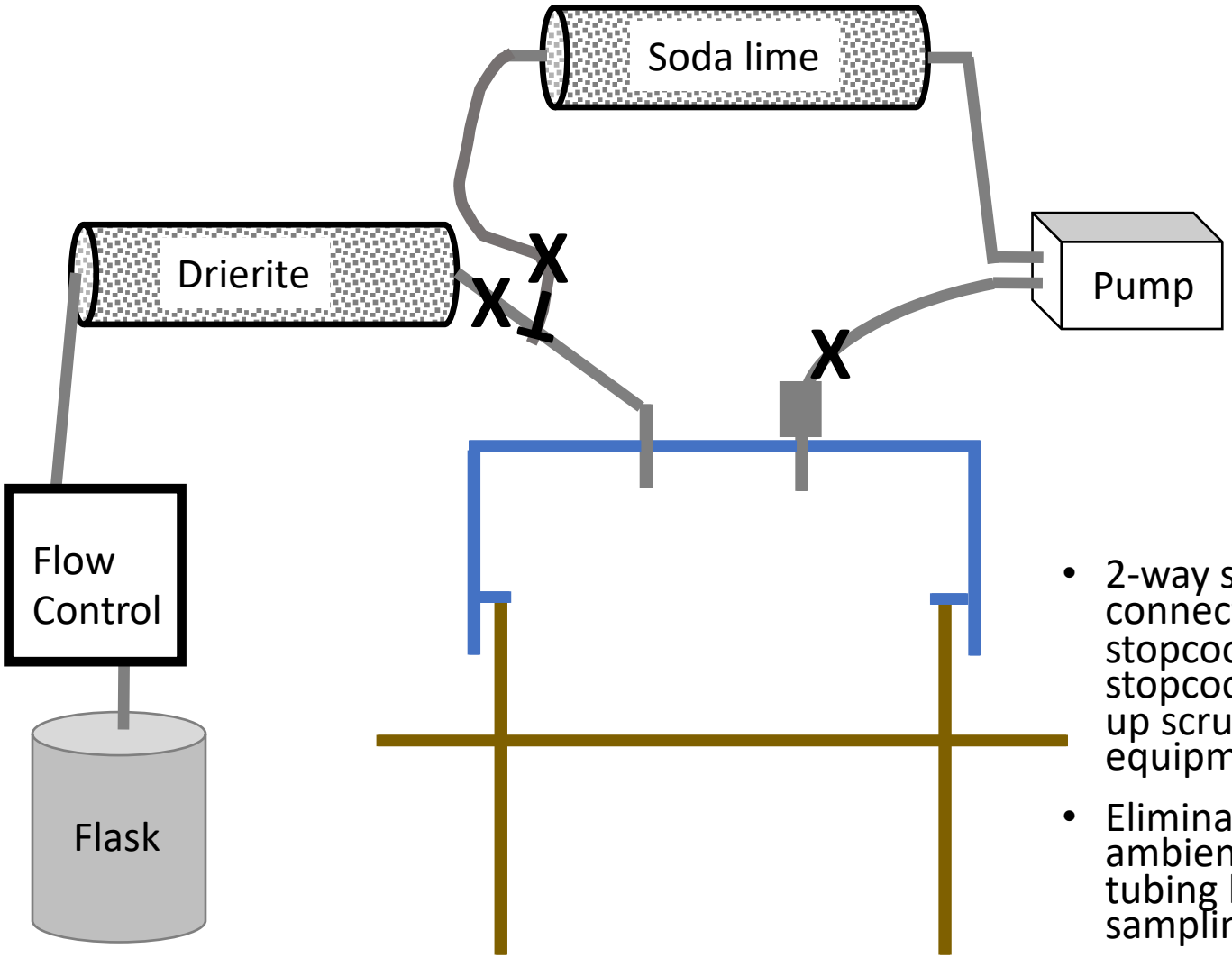
Restek flow controllers

- I use the Restek SS Soil Gas Sampler Kit with 0.0040” orifice size with 1L stainless flasks most of the time.
- https://www.restek.com/p/22932
- Check out the document for instructions and details in how to use them and select the correct one for your application

Modifications – Septa Seals

- Swagelok caps can be replaced with end caps for tube fittings (fittings with a hole for ¼” tubing).
- A septa can be put inside the fitting and a needle used to pierce the septa, either on the chamber lid bulkhead fittings or the flasks.
- This is helpful if you want to collect smaller volume headspace samples for pCO_2 or IRMS for stable isotopes.

Modifications – Bypass Loops



- 2-way stopcocks and t-connectors (or one 3-way stopcock and one 2-way stopcock) can be used to set up scrub and sampling equipment at the same time
- Eliminates small amount of ambient air from swapping tubing between scrub and sampling set-ups