COMPASS Coastal Observations, Mechanisms, and Predictions Across Systems and Scales	COMPASS Field Sampling Protocol	
Date Created: 2-7-2025 Date Updated: 5-13-2025	Creator Name(s): Evan Phillips Editor Name(s): S. Wilson	Version: 2

Gas Well Construction and Sampling

Objective: To monitor profiles of soils greenhouse gas (methane and carbon dioxide) concentrations across the terrestrial aquatic interface from gas wells.

Contents:

I. Experimental Design: The gas wells are installed across the COMPASS synoptic site transects. In the Chesapeake Bay this includes sites: GCW, SWH, MSM, and GWI. At each site there are three zones; UP, TR, and WC (at SWH also SWAMP). In each zone, three replicate clusters (A, B, C) of gas wells are positioned at three depths (10, 20, 45cm). The gas well is positioned so that the center of the well is at the specified depth, the wells are 6cm in length so they span roughly 3cm above and below the target depth, similar to the lysimeters.

The wells serve as a pocket of air similar to how a canoe can create an air pocket when it is upside down in water that, over time, equilibrates with the soil. The equilibrated air can then be sampled and analyzed for gases of interest. In this study we are focused on methane and carbon dioxide, but nitrous oxide and oxygen could also be measured.

Some caveats associated with these wells: We are making the assumption that the air in the well is in equilibrium with the soil and represents the in situ gas concentrations. Whatever gas is removed from the well must be replaced with ambient air in order to maintain the air pocket, this is why equilibration time between samplings is critical. For the synoptic sites, there is at least a month of time between samplings of the gas wells. Testing of a gas well's oxygen concentrations over time in the GCReW wetland showed equilibration within a week.

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II. Personal Protective Equipment: Close-toed shoes and long pants are required at all times when working at COMPASS sites. Work gloves are also recommended for the installation portion of this protocol. Protective eyewear and knee pads are available. Sampling the gas wells includes the use of glass vials and needles. Be aware of sharps in between use and store them in the proper container.

III. Construction:

A. Materials:

- 2inch PVC Sched 40 - PVC072000200HA
- 2inch PVC caps (flat top) - PVC 00132 0800HD
- Swagelok fittings - 2713C27EA
- Black UV resistant ¼" OD tubing - 2VDL1
- Stopcocks – MED DYNJSC3MPFLS
- Colored tape
- Heat shrinks

B. Construction:

- Cut 2" PVC into 6cm long pieces
- Prime and Glue Caps to the 2" pieces
- Using a drill press; make holes in the top of the flat caps
    i. You want the hole to be a bit smaller than the Swagelok so that when you screw the Swagelok fitting into the cap it will be very tight
- Screw the Swagelok fitting into this hole
- Cut tubing to desired length and label with colored tape based on depth
    i. 10cm deep well: red tape,
    ii. 20cm deep well: orange tape,
    iii. 45cm deep well: black tape,
- Attach tubing to Swagelok and tighten fitting
- Attach stopcock to tubing
    i. Put a piece of heat shrink over tubing; push to the side

ii. Using heat gun; carefully heat sampling end of tubing
iii. Push slip-tip end of the stopcock into the warmed hard tubing
    - The warming makes the hard tubing more malleable so that you can fit the stopcock into it
iv. Push the heat shrink up over the connection between the tubing and the stopcock
    - Using heat gun, shrink the heat shrink to seal the connection

B. CGas well dimensions

- 5cm diameter
- 6cm deep
- Total internal volume = 117.81 cm3 (roughly 118 mL)

IV. Installation:

A. Materials:

- Prepped gas wells (stopcocks attached)
- 2” gauge auger
- 2” bucket aguer
- Folding measuring tape
- Bucket
- Tarp
- PVC pole (~1m long; 1 1/2” wide)
- Soil Knives / spatulas for scooping
- Driveway stakes
- Zip ties
- 60mL Syringe
- Electrical tape
    i. red, orange, black
- Sharpie
- Gloves

B. Procedure:

- Make sure agars & PVC are marked to appropriate depths
- Using gauge agar, take initial core to required depth
- Keep that core intact to the side

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- Check the depth with the folding ruler
- Expand the hole with the bucket agar slightly
- Put PVC over tubing on the gas well
- Use the PVC to push the gas well down into the hole
- Check on the PVC that the depth is correct.
- Carefully remove the PVC
- Hold the tubing
- Put some material from the bottom of the bucket agar back into the hole on top of the gas well
- Remove the core from the gauge agar and slide it back into the hole
- Tamp the top of the core
- Use material from the top of the bucket agar to seal the top if needed
- Attach tubing to driveway stake to keep it up off the ground
- Use the syringe to check and see if you are getting water when pulling sample up from the well. Then ADD 180mL (3x 60mL syringes full) of ambient air to the gas well and close the stopcock.

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V. Sampling

A. Materials:

- Protocol
- Datasheets
- Sharpie
- Pencil
- 60mL syringes
- Labeled and prepped exetainer vials (12mL)
- Needles (size)
- Luer lock stopcocks
- (In tool bag) Quick Connects & Scissors

B. Laboratory Prep

- Use exetainer tubes with new septa
  - https://www.labco.co.uk/products/standard-exetainer-cap/product/557-exetainer-12ml-round-bottom/category_pathway-15
- If not using new tubes, then tubes should be DI rinsed and dried.
- Vials are flushed with N2 gas for 2mins
- Vials are evacuated
  - Hand evacuation with locking 60mL syringe, pull back and lock the syringe, allow to sit for 10 secs; OR
  - Hand evacuation with non-locking 60mL syringe, hold for 10 secs; OR
  - 5mins with vacuum pump
- *Add note for needle size
- Label tubes
- Pack in vial boxes for sampling trips in order
  - boxes labeled for order

Gas Well Construction and Sampling

C. Field Procedure:

1. Check the zone (UP, SWAMP, TR, WC), gas well cluster ID (A, B, C) and the color/ depth ID (10, 20, 45cm) matches the exetainer you are about to sample from
    a. **Red = 10 cm, Orange = 20cm, Black = 45cm**
2. Connect 60mL syringe to the gas well with stopcock
3. Open stopcock
    - allowing connection from gas well to syringe, not ambient air
4. Pull out 20mL out of the gas well (should be gas)
    a. IF there is water - make a note on the datasheet
    b. Push 180 mL of ambient air into the gas well to reset
    c. Do not take sample, record volume put into well
5. Close gas well stop cock to the well and release gas in syringe to ambient air
    - This is to clear out the line to the gas well
6. Reconnect to the gas well stopcock
7. Pull out another 40mL of the gas well for sample
8. Close gas well stopcock to the well and to the syringe
9. Connect a needle to the syringe
10. Open stopcock and discard 20mL of the sample to the ambient air
11. Put the remaining 20mL into an evacuated exetainer
    - If needed, vials can be field evacuated using a syringe. To do this, use a locking syringe and pull 60mL of air from the exetainer and allow it to sit with the syringe locked for 10-15 seconds then repeat
12. Put the exetainer into the box of vials
    - We typically put collected vials upside down to help indicate it contains sample
13. Fill syringe with 60mL of ambient air
14. Connect to the gas well and push the ambient air down into the well
15. Close well stopcock to the gas well
16. Record the time, the volume of the sample, and whether the well was refilled on the datasheet
17. Move to next well
18. Also collect ambient air samples as 20mL into an exetainer to capture ambient air concentrations in each zone (1 air sample per zone: UP, TR, WC, SWAMP)
19. After completing a zone, and at the end of sampling, double check that the datasheet has been filled out correctly and that the time zone has been indicated.

VI. References:

VII. Notes:

In 2023, when the wells were installed we sampled from water using the shaky method of equilibrating the water (20mL) with air (20mL) as described below and was noted. In 2024, we stopped doing this and noted if there was water, but did not sample. The reason for stopping the collection of samples from water was that the gas and water extracted gas samples are difficult to compare and are fundamentally different measurements.

Extracting Gas Samples from Water

- Using a 60mL syringe, pull and discard roughly 30mL of air/water from the well, to clear the lines
- Pull water sample slowly (to minimize bubbles) to the ~25 mL mark on the syringe. Write down the time on the datasheet that you took this sample (or set a timer).
- Turn the syringe pointing upwards and tap to get rid of any bubbles; open the syringe and push out any air bubbles.
- Eject the excess water sample, leaving exactly 20 mL of water sample in the syringe.
- Holding the syringe pointing upward, introduce 20 mL of atmosphere (draw the plunger down to the 40 mL mark). Close the syringe and shake for 2 minutes.
- After shaking for two minutes, turn the syringe upside down and slowly eject the water from the syringe (away from the wells), leaving only the air inside of the syringe. Close the syringe and flick the end of the syringe with your finger to get rid of any remaining water in the stopcock
- Attach a 0.45 uM filter to the stopcock on the syringe and a single use needle.
- Take the exetainer (check label to make sure it is the right one), uncap the needle, and pierce the septa on the exetainer with the needle, pushing the needle most of the way in to the exetainer. Open the stopcock, and slowly but firmly push the sample into the exetainer.
- Tip: rest the syringe plunger on a firm surface, such as your leg, and push down on the syringe to eject the sample into the exetainer. It can be hard to inject the sample, so this makes it a bit easier and uses your leg or a surface as leverage.
- Replace well with 180mL of ATM air