



Version 4

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Bubble strip aqueous gas sampling V.4

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In Development

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Chemistry Method Development Community

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ABSTRACT

The "bubble strip" method has been shown to be an effective means for sampling a gas phase in equilibrium with water (Kampbell et al., 1998). Here, a method optimized for sampling large quantities of equilibrated gas (~60 mL) is demonstrated. This has applications for advanced isotopic analyses, which often require large sample sizes.

This version was used in Samail Ophiolite sampling in 2019.

Kampbell D. H., Wilson J. T., McInnes D. M. (1998) Determining Dissolved Hydrogen, Methane, and Vinyl Chloride Concentrations in Aqueous Solution on a Nanomolar Scale With the Bubble Strip Method. In *Proceedings of the 1998 Conference on Hazardous Waste Research* pp. 176–190.

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MATERIALS

NAME	CATALOG #	VENDOR
PYREX™ Reusable Media Storage Bottles 100 mL	06-414-1A	Fisher Scientific
BOTTLE CAP GASKET FOR GL45 CAPS VITON	GS-014	COBERT ASSOCIATES LAB STORE

NAME	CATALOG #	VENDOR
Cole-Parmer™ VapLock™ Solvent Delivery Caps 304 SS Port Thread Inserts three 1/4 in.-28	13-311-001	Fisher Scientific
Flangeless Nut Short PEEK 1/4-28 Flat-Bottom for 1/8 OD	LT-115X	IDEX Health & Science
Extreme-Pressure PEEK Tubing for Chemicals Opaque 0.062 ID 1/8 OD	51085K49	McMaster-Carr
Luer Adapter 1/4-28 Female to Female Luer PEEK	P-658	IDEX Health & Science
Leap PAL PartsSupplier Diversity Partner VICI Valco MinInert Syringe Valve For Luer-Tip Syringe alLow Series C and D Syringes to store Samples up to 250 PSI Valve body is PTFE With Stainless Steel stem	50-109-0255	Fisher Scientific
BD General Use and PrecisionGlide Hypodermic Needles - 23g (0.064 cm x 2.5 cm)	14-826A	Fisher Scientific

MATERIALS TEXT

Assembling bubble strip apparatus:

This should be pretty straightforward, but here are a few things to keep in mind.

- It is important to replace the standard vaplock cap gasket with a viton gasket. This reduces gas leakage / diffusion out of the bottle.
- Make sure all the fittings for the PEEK tubing are secure. They don't need to be super tight, though.
- The inlet PEEK tubing should be ~1 cm from the top of the bottle, whereas the gas sampling PEEK tubing should be as near to the top of bottle as possible without getting water in it (when cap is on bottom and water is flowing). See photo.

Other things you will need:

- Ring stand
- Sterile 60 mL syringe with luer lock tip
- Sterile 0.2 µm syringe filter
- Sterile needle (23g is good)

DISCLAIMER:

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The author emphasizes that this protocol is optimized for sampling large quantities and concentrations of gas for trace gas or isotopic analyses. Other methods such as copper tube or evacuated serum vial direct water injection sampling may be more accurate and/or well suited to the requirements of some studies.

Prepping sample vials

1d

- 1 Pre-treat blue chlorobutyl stoppers to reduce potential contaminants by boiling in 0.1 N NaOH for 45 min followed by immersion in distilled water for ~8 hours), according to procedure of Oremland et al. (1987).

Oremland R. S., Miller L. G. and Whiticar M. J. (1987) Sources and flux of natural gasses from Mono Lake, California. Geochim. Cosmochim. Acta 51, 2915–2929.

- 2 Sampling vials will be filled with a near-saturation NaCl solution which will be displaced by the sample gas. Calculate the volume needed to fill all your vials. In a large beaker, prepare a near-saturation NaCl solution that is a bit larger than the volume of your vials.



Leave plenty of extra room in the beakers with NaCl because when you autoclave in the next step, the NaCl brine can expand and overflow the beakers.

- 3 Autoclave sterilize vials, stoppers, and NaCl solution (90 minutes at 125°C).

🕒 **02:00:00 including cooldown time**

- 4 While still warm, insert stoppers into 37 mL borosilicate glass vials, and secure stoppers with aluminum crimp tops.

- 5 Wait for vials, stoppers, and NaCl solution to cool down to near room temperature.

🕒 **02:00:00**



hot stoppers don't reseat well upon puncturing with needles

- 6 Using 2 18G BD precisionglide needle per vial and a 60 mL syringe, fill the vial as completely as possible with NaCl solution.



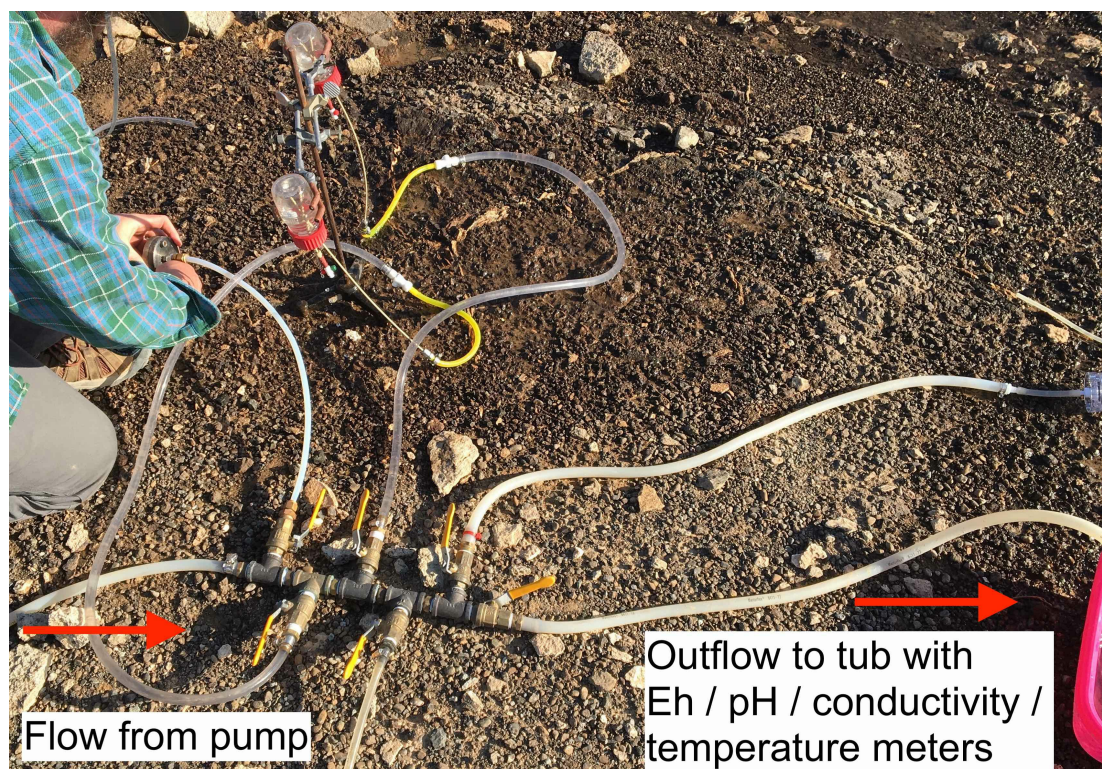
It is not essential to use 100% sterile technique for the NaCl solution because it is so salty that microbial life is inhibited in it.

Field Sampling

1h

- 7 Attach a 100 mL borosilicate glass bottle (with appropriate fittings installed - see materials) to a ring stand, cap facing up. Connect inlet tubing of bubble strip to pumped water source (i.e. manifold).
- 8 Slowly open water flow valve on manifold. Let bottle fill up with water.
- 9 Flip bottle 180° such that the cap faces downwards.

- 10 Attach a mininert stopcock to a 60 mL plastic syringe. Fill syringe with 40 mL of air.
- 11 Attach syringe to sampling port on bottle cap.
- 12 Inject 40 mL air through the sampling port into the glass bottle. Close mininert valve. The setup should look like this picture. In this example (from 2018 Oman sampling), 2 bubble strip gas samples are equilibrating simultaneously. Other tubes connected to biomass filters and/or water sampling ports are unrelated to the bubble strip.



- 13 Measure flow rate by timing the filling of a 500 mL graduated cylinder from outlet tube. Flow rate should be at least 300 mL/min.
- 14 Periodically monitor flow rate and water temperature. If flow remains at least 300 mL/min, the headspace gas should equilibrate within 30 minutes of flow, and then can be sampled. The water temperature is good to note so that Henry's constants can be adjusted for temperature.
- 15 After sufficient flow has been allowed for gas equilibration, re-attach syringe to bubble strip apparatus if it has been removed (although typically it is best to just leave syringe connected). Then, open mininert valve, and withdraw 35-40 mL of headspace gas. Close mininert valve.
- 16 Remove syringe and mininert valve from sampling apparatus. Connect a sterile 0.2 μm syringe filter and sterile 18G needle (BD precisionglide) to the mininert valve.

- 17 Open mininert valve on syringe. Inject gas through the filter and needle until the syringe reads 30 mL. Immediately stick the syringe and needle into the sampling vial through the rubber stopper. Using another sterile needle, puncture the sample vial stopper to provide an outflow for the NaCl solution. Displace 30 mL of NaCl solution with the gas in the syringe. Holding needle securely, remove syringe from vial. Store vials stopper-side down until analysis.