**METHOD:** Aqueous extraction of dissolved inorganic carbon

**EFFECTIVE DATE:** 2/7/2023

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**PURPOSE:** To extract dissolved inorganic carbon (DIC) as CO₂ from aqueous samples via water stripping.

**INTRODUCTION:** Inorganic carbon dissolved in samples such as seawater or groundwater (DIC) is released via acidification and then sparged from an aqueous sample with a nitrogen carrier gas in a process referred to as “water stripping” (WS). Samples are either collected in a vessel that accepts a specialized “stripping probe” using a ground glass neck, or the sample is transferred from the collection bottle to a stripping vessel in a glove bag filled with nitrogen. Inorganic carbon species dissolved in the water (i.e., CO₂(aq), H₂CO₃, HCO₃⁻, and CO₃²⁻) are converted to CO₂ by acidifying samples with 1 ml 85% H₃PO₄ phosphoric acid introduced via a septa-seal fitting on the probe. The resulting CO₂ is circulated via the carrier gas through a closed system at 0.6 atmospheres until all of the inorganic carbon has been converted to CO₂ and trapped. Under slight vacuum, the gas passes through an isopropanol dry-ice slush to remove water and the CO₂ is collected in a liquid nitrogen trap. Due to the partial pressure of the system, the nitrogen gas is not trapped in liquid nitrogen, but the CO₂ is.

**MATERIALS & APPARATUS**

- Custom glass water-stripping probes
- Chemglass valves
- Rubber septa
- Apiezon “M” grease
- Glove bag
- High purity nitrogen gas
- Glass sample bottles (100- or 500-ml) with 29 mm OD ground glass joint
- Liquid nitrogen and dewars
- Vacuum line with glass sample manifold
- 85% H₃PO₄
- Glass syringe

**PROCEDURE:**

*Probe and sample preparation*

Assemble probes by inserting lightly greased valves, o-ring, and septa into position and applying grease in zigzag along ground glass probe joint.
Place probes in glove bag with nitrogen hoses placed into the top valve of each probe to flush. Working in nitrogen-purged glove bag, carefully transfer sample (if necessary) to glass bottle with 29 mm OD ground glass joint.

Insert probe into bottle opening, rotate several times to ensure a good seal, and close bottom then top manual valves.

**Prepare vacuum line for sample extraction**

Place samples onto vacuum line, pump down for roughly 2 h, and leak check the line. Flush probes and line with N₂ carrier gas.

Acidify samples by injecting 1 ml 85% H₃PO₄ through septa using glass syringe.

Fill large LN₂ dewars, and pre-fill all dewars to cool the loop trap, baratron region, and storage manifold. Open valves to large LN₂ dewars.

Zero baratron display and begin automated run procedure.

**Automated run**

During the automated run, nitrogen gas is bubbled through a submerged frit on the attached probe into the acidified sample. The CO₂ resulting from the acidification of the DIC sample is stripped from the aqueous solution and entrained in the inert N₂. Sample CO₂ is cryogenically separated from the N₂ carrier gas and transferred to individual storage ports in the glass sample manifold after removal of water and quantification in a calibrated volume.

**RECORDS:** Information specific to the processing of each sample is recorded in a notebook and in the NOSAMS relational database including operator, unique receipt number, quantity of CO₂ extracted, and comments.

**REFERENCES:**