

NOSAMS Facility

METHOD: Aqueous extraction of dissolved inorganic carbon
EFFECTIVE DATE: 2/7/2023
APPROVED BY: Dr. Roberta Hansman

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., CO_{2(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.

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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:

McNichol, AP, Jones, GA, Hutton, DL, Gagnon, AR, Key, RM, 1994 The Rapid Preparation of Seawater Sigma-Co₂ for Radiocarbon Analysis at the National Ocean Sciences AMS Facility. Radiocarbon 36(2):237-246, DOI: [10.1017/S0033822200040522](https://doi.org/10.1017/S0033822200040522)