METHOD: Acidification of inorganic carbon samples
EFFECTIVE DATE: 1/23/2023
APPROVED BY: Dr. Roberta Hansman

PURPOSE: To convert inorganic carbon to CO_2 by acidification under vacuum in preparation for subsequent reduction to solid carbon and radiocarbon analysis.

INTRODUCTION: Typical sample types prepared for radiocarbon analysis via acidification include shells, foraminifera, otoliths, stalactites and stalagmites, coral and sediments containing carbonate species. Samples should be submitted as free from extraneous material such as adhering sediment or other discrete particles that could contribute carbon of a different age than the intended sample.

Acid etching or pre-leaching is available upon request. The etching process is used to remove the surface layers of the sample which are potentially remineralized or contaminated with carbon of a different age. Samples are etched immediately prior to acidification to prevent/minimize adsorption of atmospheric CO_2. IAEA C-1 blanks require etching to reach the lowest background levels, but for small or powdery samples, etching may remove too much of the sample. Therefore, etching is not recommended for powdery or delicate samples (foraminifera) or for very small sample sizes.

Using our custom glass side-arm reaction vessels, the acid is placed into the side-arm, away from the sample then the vessel is attached to a vacuum line and the air is pumped out. One evacuated, the vessel with sample + acid is removed from the line, the acid tipped to reach the sample and the vessel placed in a heated water batch to speed evolution to CO_2.

MATERIALS & APPARATUS

- 100% H_3PO_4
- 1 N organic-free HCl
- Distilled H_2O
- Clean beaker (50-100 ml)
- Custom glass side-arm reaction vessel
- Nitrile gloves, weigh paper and clean utensils (spatula, forceps etc.)
- Water bath (60 ˚C)
- Drying oven (50 ˚C)

PROCEDURE: Prepare side-arm vessels for a batch of 10 samples comprised of 7 unknowns, 2 secondary standard carbonate materials and 1 process blank (IAEA C-1 or TIRI-F). Vessels are cleaned and baked in 500 ˚C oven prior to use. Intersperse the standards and blank, varying the process order each time.

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Visually inspect the samples and note any discoloration on the Chain of Custody sheet (COC). Notice whether the sample requires etching on the COC or if there are any special processing requirements.

Wear gloves and use only clean utensils to weigh the sample. Split or subsample if necessary to produce an ideal sample of 1 mg extracted carbon.

Stoichiometric acid-etching: Calculate the strength and amount of HCl to add to the sample to achieve the desired amount of material to leach. Record the weight of the sample.

NOTE: The %HCl will need to be determined and depends on the size of the sample. Use enough acid to completely cover the sample.

Pipette the determined amount and concentration of HCl into each tube using an acid-cleaned pipette tip.

Label the tube with lab tape. Do not use a Sharpie pen on the tube (it may come off the bath).

Loosely cap the sample and leave until the pH is neutral which indicates the reaction is complete. Using a glass pipette, check with pH paper by pipetting a drop or two on the paper. When complete, pipette as much of the acid waste out of the tube as possible. Dry the sample in a 50 ˚C oven.

Non-stoichiometric etching: For IAEA C-1 Carrara marble, and other robust carbonates, a simple etch is performed by adding enough 1 N HCl acid to cover the sample in a clean petri dish for 30 to 60 s, then flushed repeatedly with distilled water until neutral, then dried at 50 ˚C.

For acidification:

Record the weight of the sample.

Place the sample into a labeled side-arm vessel, aiming to get the entire sample at the bottom of the tube. Tap or use clean tools to make sure the sample is at the bottom of the vessel.

For pure carbonate samples, add 2 ml of 100% phosphoric acid to the acid arm of the vessel. For TIRI-K, other sediments, or impure carbonates, use 2 ml 85% phosphoric acid. Place a water trap (slush) on the acid arm if 85% phosphoric acid is used to remove excess water.

Place the vessel with sample and acid on the vacuum system and perform routine pumping out and leak checking. Remove from the line, dispense the acid into the vessel and gently gyrate the tube in the acid so that the acid completely meets with the sample. Place reactors in a 60 ˚C water bath overnight.

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, mass, method, and comments.