

NOSAMS Facility

METHOD: Acid-base-acid (ABA) pretreatment of organic carbon samples
EFFECTIVE DATE: 1/25/2023
APPROVED BY: Dr. Roberta Hansman

PURPOSE: To remove exogenous carbon from plant/wood/charcoal samples prior to radiocarbon analysis using a series of acid-base-acid leaches. The acidification removes any inorganic carbon from the sample, and organic acids are removed with repeated leaching in a weak base solution. A final acidification ensures complete removal of base and inorganic carbon.

INTRODUCTION: Unprocessed plant-based materials undergo a series of acid-base-acid leaches designed to remove inorganic carbon and/or base-soluble organic acids that may have originated from surrounding sediments prior to combustion and may contaminate the sample with carbon of a different age. This process necessarily removes some sample mass. Plant/wood materials collected live that have not been buried in sediments do not need such rigorous chemical pretreatment and may be acidified only. Bulk organic sediment samples undergo acid pretreatment only, unless there is a large plant component that would remain after the acid treatment such as peat or mangrove sediments. If samples are very small and/or friable, the client may be contacted to discuss pretreatment options before processing to decide on the best approach. Large samples may be broken up and subsampled to collect a representative sample.

MATERIALS & APPARATUS

- 1.2 M HCl
- 0.5 M NaOH solution
- Milli-Q H₂O
- Centrifuge tubes (50 ml, Teflon)
- Nitrile gloves, weigh paper and clean utensils (spatula, forceps etc.)
- Glass pipettes
- Water bath (60 °C)
- Drying oven (50 °C)
- Quartz fiber filters (pre-baked)
- Filtration apparatus
- Centrifuge

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PROCEDURE:

NOTE: This procedure describes the pretreatment of samples done manually at NOSAMS. Samples may also be processed on a semi-automated Organic Carbon Pretreatment System, but the chemistry is the same.

Estimate % organic carbon of sample and calculate the sample amount needed for 3 mg of carbon. This is a conservative mass to be pretreated (if possible) to make sure the remaining sample is sufficiently large enough to produce 1 mg C. Weigh the sample and transfer to 50-ml Teflon centrifuge tube. Wear gloves and use only clean utensils to weigh the sample. Record the weight of the sample.

Add approximately 5-10 ml (enough to cover the sample) of 1.2 M HCl to the centrifuge tube and loosely cap.

Place in 60 °C water bath for 1 h.

Remove the sample from the water bath and centrifuge (2300 rpm for 3 min).

Carefully decant or pipette off the supernatant and discard.

Rinse the sample well (at least 3 times) with Milli-Q water.

Add approximately 5-10 ml (enough to cover the sample) of 0.5 M NaOH to the centrifuge tube and loosely cap.

Place in 60 °C water bath for 1 h.

Observe the color of the NaOH solution after 1 h, noting any discoloration (brown to black). Centrifuge, and pipette or decant the supernatant from the centrifuge tube. Repeat base treatment at least 3 times, and up to 10 times, until the solution becomes clear or there is no change from rinse to rinse.

Once the solution is clear or the maximum number of rinses is reached (10), rinse the sample well (at least 3 times) with Milli-Q water.

Add approximately 5-10 ml (enough to cover the sample) of 1.2 M HCl to the centrifuge tube and loosely cap.

Place in 60 °C water bath for 1 h.

Centrifuge, and decant or pipette the acid solution from the tube. Rinse the sample with Milli-Q water to neutralize (until pH of 5-6).

Filter the sample onto a quartz fiber filter that has been pre-baked at 650 °C for 1 h. Rinse the sample well on the filter with Milli-Q water.

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Transfer the filter with sample to a small clean baked glass petri dish, cover, and place in 50 °C drying oven. Allow the sample to dry for 24-36 h or until completely dry.

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, pretreatment method and number of rinses, and comments.