

NOSAMS Facility

METHOD: Pretreatment of sediment for organic carbon ^{14}C analysis
EFFECTIVE DATE: 1/25/2023
APPROVED BY: Dr. Roberta Hansman

PURPOSE: To remove inorganic carbon from bulk sediment samples in preparation for radiocarbon analysis of the organic carbon component.

INTRODUCTION: Dried sediment samples are frequently received for total or bulk organic carbon radiocarbon analysis. When the sediment sample type is indicated on the submission form, the samples are examined to evaluate their composition and determine the appropriate pretreatment plan. Most sediments are pretreated with repeated acid leaches to remove inorganic carbon (carbonates). Some sediments are submitted with embedded plant material, such as peat, and require treatment with base to remove exogenous organic acid components. In this case, the client may be contacted to discuss treatment options. For some sediment samples such as fine sediments collected on a filter, black carbon or pollen extract samples, acid fuming may be used to remove inorganic carbon prior to combustion. This method is described elsewhere.

MATERIALS & APPARATUS

- 1.2 M HCl
- 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 (47 mm, baked)
- Filtration apparatus
- Centrifuge

PROCEDURE: Weigh approximately 1-1.5 g of sediment material onto weigh paper 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.

NOTE: Darker sediments tend to be higher in % OC than lighter sediments. A greater sample mass may be required for lighter sediments.

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Add approximately 15-20 ml of 1.2 M HCl (~1/3 up the centrifuge tube) to the sample and agitate the sample by hand to mix thoroughly. If the sample is light in color, it may be high in carbonates which will bubble with the addition of the acid. If this occurs, repeatedly add a small amount of the HCl until vigorous bubbling stops.

Place the tube in the 60 °C water bath and incubate 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.

Add another 15-20 ml 1.2 M HCl and repeat acid treatment (incubate, centrifuge, decant) two or more times until all inorganic carbon has been removed from the sample (i.e., no more bubbling of the sample occurs). If necessary, break up the sediment in the tube with a clean glass rod in between leaches to completely disseminate.

Rinse the sample with Milli-Q water at least 5-6 times, centrifuging and decanting between each rinse.

NOTE: Fill the tube with Milli-Q to just below the neck of the tube, leaving a small head space. Fine particle samples may require a period of time to settle after centrifuging and prior to decanting.

After 5 or 6 rinses, do not centrifuge the sample. Leave the sample overnight in Milli-Q to further neutralize. The next day make sure the sample is close to neutral with a pH of 5-6.

Filter the sediments onto a Whatman QMA filter that has been pre-baked at 650 °C for 1 h. Most sediments require the large filter rig with a 47-mm diameter filter.

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.