

NOSAMS: Organic Carbon

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Overview



Dana Gerlach prepares to transfer an aliquot of Primary Standard Oxalic Acid CO₂ to a graphite

reactor. (Woods Hole Oceanographic Institution)

are collected can contribute modern contamination.

If you are submitting organic carbon sample types such as plant, wood or charcoal, make sure that you have properly cleaned and separated any non-plant, wood or charcoal type material from the sample. Pretreatment provided in our labs will remove inorganic carbon and leaves behind the acid-insoluble organic components, but will not remove other organic contaminants like sediment OC or quartz grains included with your sample.

It's obviously best to date clean, pure materials rather than a mixture of components. Separation or purification of the component you want analyzed may be performed by gently cleansing samples with distilled water, sieving and using a tool like a wet "picking" brush or tweezers under microscopic or magnified examination to remove unwanted components.

If extraneous material is included with your submitted sample that contains organic carbon, the radiocarbon result can potentially be contaminated by younger or older carbon. For example, modern roots or rootlets that penetrate to levels where sediments



Extracted bone collagen in a glass tube. The collagen will be weighed and combusted. (Woods Hole Oceanographic Institution)

preserved or contains a lot of humic material or dirt, the process may take a few weeks longer than the average turnaround. Process used [Organic Combustion](#).

Bone Collagen

Approximately 0.5 gram of bone material is required to extract enough collagen for a standard AMS sample. This is approximate since conditions for preservation vary and generally, the older the bone, the less collagen is extracted. Due to the labor-intensity of the extraction, if an inadequate amount of collagen is obtained from a sample for AMS, we will bill for partial processing up to the point of the collagen extraction.

Currently we are sending our bone samples to an outside specialist for the extraction of collagen, and in turn we combust the collagen and convert to graphite here in our Sample Prep Lab. The collagen extraction method used is referred to as EDTA (ethylenediaminetetra-acetic acid). It takes generally no more than 30 days to get the collagen and we try our best to keep the total turnaround time to a minimum, but in some cases, the decalcification of bone can take longer. For instance, if the bone is poorly



A charcoal sample submitted for AMS analysis. (Woods Hole Oceanographic Institution)

Charcoal

The carbon content of charcoal can vary significantly. However, under good conditions, 40% is a reasonable estimate of the organic carbon by weight, therefore approximately 1 milligram of clean charcoal in good condition should suffice for a standard analysis.

Often, charcoal becomes broken and disseminated in surrounding soil matrixes. Separation or purification of the charcoal component may be as simple as scraping away the outer surfaces, but if disseminated, can be performed by gently cleansing samples, sieving and using a wet "picking" brush under a microscopic or magnifier to collect the charcoal fragments. Then, if further cleaning is necessary, gently sonicate samples with distilled water and carefully draw off the supernate. Repeat until clean. Dry, weigh and transfer to clean glass vials. If a mixture of disseminated material is submitted, then the mixture will be combusted; our procedures do not include hand-picking out the fragments of charcoal.

The pretreatment of charcoal is the same as that of plant/wood and involves a series of heated acid-base-acid leaches to remove the mobile phases of organic carbon (fulvic and humic acids) and any inorganic carbon.

If you request small sample analysis, keep in mind that the transfer of material, the pre-treatment of charcoal and a 10% split of CO₂ for in-line stable $\delta^{13}\text{C}$ analysis will further reduce the available carbon. You may request to forego pretreatment (on the submittal form) or assume a $\delta^{13}\text{C}$ value in order to conserve carbon. Process used [Organic Combustion](#).

Compound-specific

These are typically collected by submitters with a PCGC, or preparative capillary gas chromatographic system (supplemented with a liquid-chromatographic system). Compound-specific samples are usually small samples (less than 100 µg C) requiring a solvent transfer to a combustion tube. Techniques to handle such small samples were initially developed at NOSAMS specifically to aid the development of compound-specific radiocarbon analysis of compounds like fatty acids, sterols, plant waxes, lignin phenols, PAHs, PCBs, and lipids.

Training is available for the PCGC technique. Compound-specific samples may be prepared and isolated by NOSAMS staff with fees determined upon request. ([contact NOSAMS](#)) Process used [Organic Combustion](#).

Dissolved Organic Carbon

Analysis of Dissolved Organic Carbon (DOC) by UV extraction of CO₂ is available in our Sample Prep Lab. Typically investigators acidify and/or filter the sample prior to submission (pH of ~2.5 +/- 0.3) or they send the samples frozen to the facility. Unless prior arrangements and a shipping account number are provided, remaining sample and the containers will be disposed of after analysis.

The rate of sample analysis for DOC is one per day so expect longer turnaround times than for other processes. Samples containing less than 150 micrograms of carbon will be analyzed only by arrangement with the staff chemist (amcnichol@whoi.edu).

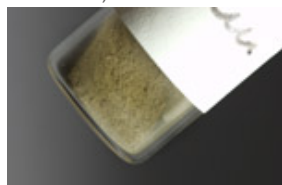


Plant/wood

Clean all non-plant/wood material from the sample using distilled water and thoroughly dry in a low temperature oven (50° C). Weigh and place in a clean, labeled vial. A general rule-of-thumb is to estimate 40% carbon by weight for plant/wood or charcoal. Some weight is potentially lost due to pre-treatment of plant/wood which involves the base extraction of mobile humic and fulvic substances and acid removal of inorganic carbon. If stable isotope analysis is requested, we will take approximately a 10% split of the CO₂ produced for in-line analysis.

If the sample is clean and in good condition, you could estimate that a regular-sized sample would weigh a minimum of approximately 1 mg. Process used [Organic Combustion](#).

Leaves in a vial for AMS. (Woods Hole Oceanographic Institution)



Sediment (total organic)

The organic carbon content of sediments vary considerably. We must know the measured % organic carbon content to gauge how much sample is required for AMS analysis. Too much material may cause the combustion tube to explode and sample loss; too little material will result in an insufficient amount for AMS analysis. Please indicate on the submittal form whether you have measured the %OC or not. If not, we can determine it during analysis.

Sediment sample. Carbonates will be removed and the organic carbon component analyzed with AMS. (Woods Hole Oceanographic Institution)

Sediment (Corg) samples typically undergo the acid pretreatment only, to remove inorganic carbon before combustion. We also offer vapor-phase acidification when requested. If your sediment contains plant material like peat, then we recommend the same treatment as plant/wood samples to remove any mobile humic or fulvic components. If the sediment contains a peat component, please indicate your preference for acid only or an acid-base-acid series of leaches on the submittal form.

Submit dry sediment samples in well-labeled, clean glass or plastic containers. We assume samples are homogeneous. Some submitters dry and grind sediment samples prior to submission to ensure homogeneity. Process used [Organic Combustion](#).

Aerosols (total organic)

Particulates collected on quartz filters can be combusted at high temperature (850 deg C) together with the sample. If pyrex filters are used, the temperature must be reduced (550 deg C) which could result in incomplete combustion of total organic carbon. Please provide the carbon loading (in milligrams C) in order for us to assess how much of the sample/filter to pack into a combustion tube. It's best to receive the filters flat rather than packed or rolled to fit into vials or containers. Past clients have used cleaned/baked aluminum foil to double wrap the sample and filter, freeze and send to us. Pre-combust foil at 550 degrees for one hour before using. Aerosols are kept cool and dry to avoid bacterial growth.

Other - Textiles, black carbon etc.

Please contact us regarding the particulars of other types to determine handling and fees. Process used [Organic Combustion](#).

When is pre-processing required for organic carbon samples?

The key distinction is between "ready-to-burn" and all other organic carbon samples. If a sample requires only mechanical or solvent-based transfer from the submission container to a combustion system, it's "ready to burn." If it requires pre-treatment, the higher fee is applied.

A typical example of chemical pre-treatment is the acid-base-acid extraction used to remove carbonates and organic acids from sediments and related materials (wood, charcoal). Upon request, vapor-phase acidification is also available.

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Mail: Woods Hole Oceanographic Institution, 266 Woods Hole Road, Woods Hole, MA 02543, USA.

E-Contact: info@whoi.edu; press relations: media@whoi.edu, tel. (508) 457-2000

Problems or questions about the site, please contact webdev@whoi.edu