I. SOLID SAMPLE PREPARATION METHODS & TIPS

A. EA Solid Sample Preparation

1. DRYING METHODS

a) Oven Drying – This should be done at NO MORE than 60°C and for NO LONGER than 2 weeks. Do not try to increase temperature above 60°C to speed up drying time; it may volatilize lighter organics in your sample!

b) Freeze-Drying – Viable option for those who have access to a freeze drier. Particularly useful for samples harder to dry, like a chunk of animal tissue. Freeze-dried samples are often easier to grind and produce a finer powder.

2. STORAGE

- Keep your samples safe in a container that seals well. Ziploc bags, scintillation vials, micro-centrifuge tubes, centrifuge tubes, petri dishes, etc. are a few examples of cost-efficient containers that seal well. The key is that the sample containers SEAL and are CLEAN!

3. GRINDING AND HOMOGENIZING METHODS

a) Mortar and Pestle – sometimes, if the material is stubborn, you can use liquid nitrogen. Just pour out a little bit to submerge your sample and make it brittle while you grind.

b) Homogenizers or motorized grinders. Can be expensive, but often produce a very fine, homogenous powder.

c) Basically, anything you can think of that you can keep clean and avoid contamination and will give you a fine powder. Some people have used coffee grinders and other types of amalgamators.

(The goal of grinding up samples is to have a homogeneous powder so that when scooping out 0.1 mg to run in the EA, you are still getting a complete representation of your original sample. Also, as you grind, make sure you keep your instruments clean. We use methanol or ethanol on our forceps, spatulas, and mortar and pestle to dissolve any organics and avoid cross-contamination between samples.)

4. ACIDIFICATION

a) If you want TOTAL carbon (inorganic + organic), this doesn’t pertain to you. If you are interested in ONLY organic carbon and do not want your data to include any contribution from inorganic carbon (i.e. sand, calcareous shells, secretions or infrastructure, a little crustacean stuck to a blade of macroalgae), you will need to acidify your sample.
Pipetting a small amount of 6% sulfuric acid or 1 N HCl works well. With solid samples, it is suggested that you acidify ground samples and allow them to dry before placing in tin boats, as the acid will cause some degradation of your boat. The amount will vary depending on the carbonate content, but you want to be consistent within a sample set while using as little acid as possible. Some methods may suggest rinsing with a small amount of de-ionized water following acidification, but due to possible loss of material, we do not recommend this. Instead, just re-dry your sample.

B. Weighing and subsampling

When your sample is inserted into the EA, it is wrapped up in a tin or silver capsule. Tin capsules come in various sizes. The capsules we consistently use and are very familiar with are made by Costech (http://www.costechanalytical.com/costech/default.shtml) and come in the following sizes (in mm): 3.5x5, 4x6, 5x9, 9x10. Do not use anything larger, as it may not fit in the EA! The tin capsules are cheaper and sufficient for most needs. Silver may be more desirable if you are treating your sample with acid, as it is more robust.

The following is a guide to weighing samples. There is no “right” way to do this, so adapt this method to suit your sampling style.

1. Make sure all of your utensils and surfaces are clean! I clean everything with ethanol and wipe it dry with a kimwipe. Useful tools include a stainless steel spatula, forceps (straight and curved), and a clean flat surface.
2. Place a tin capsule (straight from the box…we don’t usually find contamination or a difference between methanol cleaned capsules and unaltered capsules) on a scale. Tare the capsule.
3. Remove capsule from scale and place a small amount of material into capsule. Place on scale to see tentatively how much material you put in. Remove or add material until it is in the desired range.
4. Once you have the amount you need, use a forceps to pinch closed the top of the capsule as you remove it from the scale. Fold the top twice to secure the sample. Next, flatten the capsule and fold into a smooth, compact ball or square with forceps and spatula. My samples usually look like tight, compact squares or rectangles with NO MATERIAL LEAKING.
5. Place in secure container and make sure you know what is where and how much there is! For example, I used microtitre plates (96 wells). Use the supplied Prepped EA Sample Submission Form to log your samples.
6. We suggest using an index card placed over the wells before securing the plate cover. This can prevent samples slipping into other wells during shipping.
II. WATER SAMPLE PREPARATION FOR DISSOLVED INORGANIC CARBON (DIC).

A. METHOD I

1. Contact the lab manager and provide the number of samples to be analyzed.

2. The lab will ship prepared vials. Each vial will arrive capped, containing 0.1mL of 85% phosphoric acid, and flushed with helium gas.

3. Using a gas tight syringe, inject 1 mL of sample water in the vial. Label the vial with sample identifier.

4. Wrap the top of each vial in plastic paraffin film to fortify the seal during return shipment.

5. Return the vials in the same packaging as they arrived to the address listed below.

   Iowa State University
   Attn: Suzanne Ankerstjerne
   Dept of Geological & Atmospheric Sciences
   253 Science I
   Ames, IA  50011

B. METHOD II

Supply treated (i.e., mercuric chloride) bulk samples (~5mL) in tightly sealed vials. There is an additional fee for disposal of poisoned samples.