Standard Operating Procedures: Sediment Filtration and Acid Treatment

Lab Name: Rosenheim Lab

Lab Location: Marine Science Laboratories, CMS, USF Room #: 227A

Principal Investigator: Brad E. Rosenheim

Time Frame: 2-5 days (time frame is dependent upon the speed of filtration)

Before samples are run through the Ramped Pyrox System, they must be filtered and acid treated in order to remove larger particles and carbonates. If transported through the sediment column, large particles such as roots, can provide skewed ages if dated (see Appendix B in Carey Schafer's thesis). Carbonates, when not removed, show up as a distinct high-temperature peak on a Ramped PyrOx thermograph and also have the potential to bias radiocarbon ages.

1. Process	1. Mix fresh or freeze-dried sediment with 2000 mL of deionized (DI) water in a 2000 mL beaker to create a sediment slurry. - All glassware (beakers, filtration glassware) must be either pre-combusted or cleaned before use. To clean glassware, rinse with 1N HCl, DI water, methanol, and a final rinse of DI water. - Make sure to clean glassware between sediment intervals to avoid contamination. 2. Pass sediment slurry through 500 and 63-micron sieves. - Sieve catch pan can only hold 1000 mL of liquid, so sieving must be done in two steps. - The >63 μm slurry can be transferred into a separate, clean 2000 mL beaker. 3. Set up filtration apparatus as shown below (Figure 1) and put a pre-baked and weighed quartz fiber filter on the filtration post. - See furnace protocol 4. Pour 500 mL of the sieved sediment slurry into the glass frit. - The 2000 mL of sediment slurry will be divided between 4 filters with

	approximately 500 mL of slurry being filtered through each quartz fiber filter. 5. Remove the filter once ~500 mL of slurry has passed through the filter and place on a pre-combusted aluminum tray and cover with pre-combusted aluminum foil. - See furnace protocol 6. Place the tray in the muffle furnace at ~53°C for 24 hours to dry. Remove the filter once it is dry. - Dry weight can be subtracted from the initial filter weight to determine the weight of the sediment on the filter. 7. Place sediment-laden filter on the post using the same filtration set-up in Figure 1. 8. Submerge filter in 1N HCl for 30 minutes or until no bubbles are visible (no sign of reaction). 9. Rinse filter with DI water until a pH of ~6 is reached. 10. Remove filter and place on a pre-combusted aluminum tray and cover in pre-combusted aluminum foil. 11. Place the tray in the muffle furnace at ~53°C for 24 hours to dry. Remove the filter once it is dry. - Subtracting the acid-treated filter weight from the dry weight gives the weight of carbonates in the sample.
- **Hazardous Chemical** **Class of Hazardous Chemical**	1N hydrochloric acid
- **Personal Protective Equipment**	Closed toed shoes, gloves, long pants, hair pulled back, safety goggles
- **Engineering/Ventilation Controls**	All work performed in a hood
- **Special Handling Procedures** **Storage Requirements**	N/A
- **Spill Containment** **Accident Procedures**	Familiarize yourself with procedures for acid spills
- **Waste disposal**	Acid waste should be disposed of in the acid waste bin in the lab
- **Special Precautions** **Animal Use**	N/A
- **Required Approvals**	N/A

- **Decontamination**	Pre-combustion of quartz fiber filters; acid rinsing of glassware
- **Designated Areas**	Performed in a hood

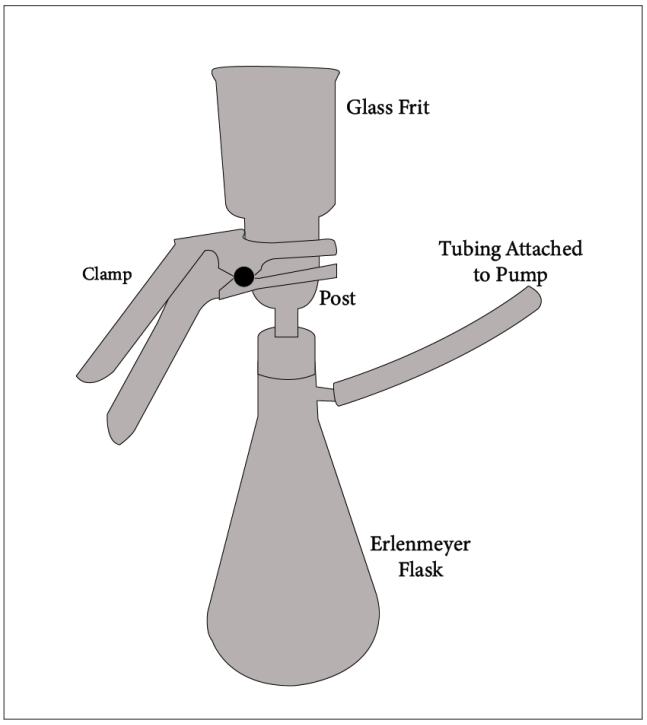

Figure 1 *Schematic of the filtration apparatus. Quartz fiber filters are placed between the post and glass frit which are secured in place with the clamp.*