^{32}Si Sample Processing for Risø Beta Counter

At Sea:

1. Rinse a 250ml square PC bottle (305ml to brim) three times with sample water then fill to brim. Place bottle in bottle carrier and keep covered with dark plastic bag to protect from light.
2. In radiation area: Rinse pipette tips (both 10-100µl and 100-1000µl) 1x with 1N HCl and 3x with Nanopure water to clean it. Separate the bottles for ambient [$Si(OH)_4$] (i.e. profile) and enhancement (i.e. +20µM [$Si(OH)_4$]) experiments.
3. To Enhancement bottles ONLY: using 100-1000µl pipetter to inject 320µl of 5mM sodium metasilicate solution into the bottle (+20.4 uM), cap and invert several times.
4. To ALL bottles: using the 10-100µl pipetter, draw/expel ^{32}Si stock several times to wet tip with isotope (isotope sticks to plastic) then inject 75µl of ~0.1µCi/ml ^{32}Si stock into all bottles, cap, invert several times to mix and record the time of injection.
5. Place injected samples back in bottle carrier and cover with dark plastic. Take carrier out to deck and place bottles in incubator, be sure to place bottles from specific sampling light depth into the corresponding light-depth mesh bag. Incubate for 24hours.
6. Prior to filtration, rinse all filtration towers in the Nalgene bath (cubic container) with dilute HCl (~10% or less) in order to keep background ^{32}Si activity low. Filtration towers can be left inverted in the acid bath for a few hours. After acid rinse, wash towers with Nanopure to remove HCl, by dipping into a series of plastic beakers (filled with Nanopure). After rinsed, replace the towers in the manifold. If any significant amount of time will pass between rinsing the towers and filtering the samples (e.g. >1 hour), cover all rinsed towers with a plastic Ziploc bag in order to keep towers clean.
7. Collect bottles from incubator – keep in bottle carrier covered with dark plastic. Filter each sample through a 0.6µm DTTP 25mm filter (Millipore Brand).
8. Prior to sample running dry, rinse inside of the incubation bottle with an 0.2µm-FSW, vigorously shake and pour FSW rinse into the corresponding filtration tower (i.e. this is to rinse any diatoms sticking to the interior of the bottle). Use enough volume to allow bottle interior to be coated by FSW upon shaking, a 2-3 second squeeze of the FSW-squirt bottle should be sufficient. Do this rinse, shake, and filter step 2 or 3 times.
9. When the sample goes dry, rinse the tower with 3 small FSW rinses using a squirt bottle to rinse any radioactive particulates that may cling to tower walls. Record local time and date when sample goes dry and turn off vacuum to tower position.
10. Place filter on a pre-labeled planchette particle side up (remove the locking ring from the planchette), flat and centered on the disc. The underside of the planchette and the lid of the small petrie dish should both be labeled with event #, and bottle ID. Let the filters air dry in covered bin (use small white basket or larger clear basket, both of which have holes for airflow). While drying, make sure to store filters in a location that does NOT have a lot of airflow (e.g. from foot traffic, near a vent, near a door).
11. Once filter is dry: center the planchette in the small petrie dish and lay a circle of mylar over it. Place the locking ring over your finger and place that finger on the centre of the planchette to hold the mylar in place. Using the other hand, push the locking ring down over the mylar, lifting your finger off the mylar as you push the ring down so that the mylar will stretch across the planchette forming a smooth cover over the filter. Place the lid on the petrie dish and stack the petrie dishes into a "burrito" which you can then wrap with foil to keep together.
12. Rinse the sampling bottles 3x with Nanopure (MilliQ) prior to next use. Rinse towers in HCl bath (see step 6) prior to next filtration.
13. When filtration waste reservoir is full, empty into 20-50L rad waste containers (provided by USCB EH&S radiation safety people). Use the squirt bottle to rinse the towers down with Nano after filters have been removed.

In Lab:

14. Samples must sit for 100 days to reach secular equilibrium before counting.
15. Calibrate the Risoe Low-level beta GM multicounters using the standards that came with the machines. Place the known planchette in the far end position, #5 and select calibrate on the software.
16. Carefully unroll the burrito and place the samples in the counter rack – position 1 is closest to the finger hole.
17. Fill out the sample names on the software and select “Start Counting”.
18. Counts for 32Si are set to 12 2hr counts for a total of 24hours.
19. Repeat for all samples.