The collection and preparation of samples for particulate load determination

Production Summary

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History of Procedure

Issue	**Date Issued**	**Changes**	**Changes made by**
Number One	**19/03/04**	**Original**	

Introduction

Suspended particulate results are an important part of calculating transport and fate of contaminants. **NOTE:** The weight of suspended particulate material can now be calculated as mgl^{-1} using weights of the unused filter paper, used paper and volume of sample filtered.

Scope

This SOP describes the procedure to be followed in the collection and filtration stage of the sample. It does not include the pre or post processing of samples. (For this, refer to SOP no. EQ-BGC-005A, EQ-BGC005B, EQ-BGC-005C, EQ-BGC 005D and EQ-BGC-005E).

Training

Operator must have been trained thoroughly in the correct use of niskin bottles and the principles of sample collection. The order in which samples are collected from a niskin bottle is of the utmost importance. The correct order is: dissolved oxygen, salinity, nutrients, suspended load or particulate load and phytoplankton. Operator must be trained thoroughly in GLP (good laboratory practice). It is advisable for the operator to read this procedure thoroughly before starting the process and run through the first filtration with an experienced operator.

Apparatus

1. Filter forceps
2. Funnel, 300 ml, ground glass seal
3. Glass base & tubulated cap
4. Ground joint flask, 1 litre
5. Anodised aluminium spring clamp
6. Vacuum/Pressure Pump, 220 v, 50 Hz
7. Clamp stand
8. Clamp
9. 500 ml wash bottle
10. 1 litre measuring cylinder
11. Cyclopore track etched membrane 0.4 μm Polycarbonate (Hydrophilic) diam:47 mm filters (pre weighed)
12. De-ionised water

Procedure

Sample collection at source

1. Samples for suspended load analysis must be taken from the Niskin bottle immediately or as soon as possible, to avoid the settling of suspended material within the bulk sample.
2. When taking the sample from a Niskin bottle or similar, collect the sample directly from the tap without using tubing of any kind.
3. Rinse the poly bottle & insert thoroughly with sample and discard.
4. Repeat step 3.
5. Fill bottle to top, place insert in bottle and screw on cap.
6. Label the bottle clearly with sample details, such as station number, depth, site code etc.
7. Where possible the sample should be filtered immediately to avoid particulate matter sticking to the sides of the poly bottle.

About the volume to be filtered

A constant volume of sample is collected at each station and the volume of sample to be filtered will depend on the amount of suspended load in the water. There may be samples that have a very high-suspended load value (usually estuarine samples) and it is not necessary to filter as large a volume as for those samples with low suspended load. Indeed, in the case of samples with high suspended load, it is not a good idea to filter too large a volume, as any particulate may either stick to the glassware or become dislodged from the filter paper once dried.

For coastal seawater samples a volume of 800-1000 ml is usually sufficient to provide a measurement of the suspended load concentration.

If 1000 ml takes too long to filter it may be necessary to reduce the volume of further samples by degrees in order to prevent a backlog of samples during periods of intensive sampling. In the Wash and the Humber it is common to filter as little as 100, 50 or even 15mls of sample.

SAMPLE PREPARATION

8. Note that the Nuclepore membrane filters are to be used shiny side up only. This should be the way that they are orientated in the petri dish.
9. Nuclepore filters are supplied ready to use with only the weight of the filter written on the petri dish. Do not place filter in anything other than the petri dish it was supplied in, as the weights of the filters are all different.
10. Number the papers sequentially starting at number one. Filter samples in the order of bottom to surface as this helps greatly in the speedy processing of data. DO NOT NUMBER PAPERS THAT ARE NOT REQUIRED FOR USE ON THE CURRENT CRUISE.
11. Fill in log sheet with sample details (see appendix I for example). Write cruise, station number and the sample depth on the petri dish to avoid any possible confusion at a later date.

12. Ensure that the filter rig to be used is clean. If in any doubt rinse the 300ml glass funnel thoroughly with deionised water, but not through the glass sinter as this blocks the holes.
13. Place filter paper, shiny side up, carefully on the sintered disc of the glass filter support using the Millipore forceps provided. Carefully clamp the 300ml glass funnel in place so that it covers the edge of the filter paper, taking care not to tear the filter.
14. Shake sample vigorously to totally re-suspend particles.
15. Rinse measuring cylinder thoroughly with an aliquot of sample from the poly bottle and discard. This will remove any particulate matter that may have entered the measuring cylinder between samples.
16. Pour the sample into the measuring cylinder by inverting poly bottle completely and shake bottle whilst pouring to ensure material is kept in suspension rather than settling in the bottle. Note the volume on the log sheet and petri dish.
17. Pour sample into 300ml glass funnel and cover both funnel and measuring cylinder with large petri dishes to avoid contamination. Continue adding more sample to the filter funnel, (remembering to shake the measuring cylinder before pouring out more sample).
18. Rinse out the measuring cylinder with 50ml de-ionised water and pass through the filter as well to remove any particles stuck to the sides.
19. Once all of the sample has been transferred to the filter funnel, as soon as the volume of the sample reaches approx. 150 ml gently rock the filter rig in a circular movement to dislodge any suspended material that may have settled on the neck.
20. Just before the filter paper is about to be sucked dry, using the 500ml wash bottle provided, squirt about 50ml of de-ionised water into the filter funnel, directing the water around the funnel walls to wash any particulate matter that has stuck to the glass funnel onto the filter. When the filter is about to be sucked dry for the second time repeat the procedure with another 50-ml rinse of de-ionised water. NOTE: *the purpose of this rinsing stage is to remove salt deposits as well as particulate matter still stuck to the glass funnel.*
21. NOTE: The length of time required to filter 2 x 50 ml deionised water rinses can take some considerable time if the filter paper has been allowed to be sucked dry during filtering. It may be necessary to reduce the amount of deionised water used to 2 x 25ml aliquots
22. Allow the filter paper to be sucked dry.
23. Turn off valve to filter unit. Remove paper carefully with forceps provided and place in petri dish making sure that the paper insert is removed first.
24. Replace the lid and seal with elastic band then return to the foil tray that the papers are supplied in.
25. Empty filtrate from conical flask to avoid spillage during next filtration.
26. Rinse the 300ml funnel and measuring cylinder thoroughly with de-ionised water to avoid contamination of subsequent samples.
27. Secure all components before walking away from the filter rig to avoid unnecessary breakages.
28. Do not store measuring cylinders on the rusty clamp stands.

TREATMENT OF USED FILTER PAPERS

29. Carefully store the used papers in aluminium trays making sure that they remain upright. If papers are disturbed or tipped over particulate may fall of the paper, invalidating the result.
30. Return filter papers and log sheets to EQ-BGC nutrient chemist in lab 155.

Appendix I.

SUSPENDED LOAD LOG SHEET **CRUISE :** _____ **SHEET NO :** _____

STN No.	SAMP DEPTH M	DATE COLLECT	duplicate sample no.	SITE CODE	COMMENTS	FP NO	WT OF FP	VOL FILT MLS