REGIONAL SEAS

UNITED NATIONS ENVIRONMENT PROGRAMME August 1987

Guidelines for determining inputs of inorganic contaminants into estuaries

Reference Methods For Marine Pollution Studies No. 41

Prepared in co-operation with

IOC *IAEA*

UNEP 1987

Na.88-5224

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PREFACE

The Regional Seas Programme was initiated by UNEP in 1974. Since then the Governing Council of UNEP has repeatedly endorsed a regional approach to the control of marine contamination and the management of marine and coastal resources and has requested the development of regional action plans. The Regional Seas Programme at present includes ten regions and has over 120 coastal States participating in it. (1), (2)

One of the basic components of the action plans sponsored by UNEP in the framework of the Regional Seas Programme is the assessment of the state of the marine environment and of its resources, and of the sources and trends of contamination, and the impact of contamination on human health, marine ecosystems and amenities. In order to assist those participating in this activity and to ensure that the data obtained through this assessment can be compared on a world-wide basis and thus contribute to the Global Environment Monitoring System (GEMS) of UNEP, a set of Reference Methods and Guidelines for marine pollution studies are being developed and are recommended to be adopted by Governments participating in the Regional Seas Programme.

The methods and guidelines are prepared in co-operation with the relevant specialized bodies of the United Nations system as well as other organizations and are tested by a number of experts competent in the field relevant to the methods described.

In the description of the methods and guidelines the style used by the International Organization for Standardization (ISO) is followed as closely as possible.

The methods and guidelines, as published in UNEP's series of Reference Methods for Marine Pollution Studies, are not considered as final. They are planned to be periodically revised taking into account the development of our understanding of the problems, of analytical instrumentation and the actual need of the users. In order to facilitate these revisions the users are invited to convey their comments and suggestions to:

International Laboratory of Marine Radioactivity
International Atomic Energy Agency
c/o Musee Oceanographique
MC98000 MONACO

which is responsible for the technical co-ordination of the development, testing and intercalibration of Reference Methods.

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(1) UNEP: Achievements and planned development of the UNEP's Regional Seas Programme and comparable probrammes sponsored by other bodies. UNEP Regional Seas Reports and Studies No. 1 UNEP, 1982.

(2) P. HUIM: A Strategy for the Seas. The Regional Seas Programme: Past and Future UNEP, 1983.

This draft issue of the Reference Method for Marine Pollution Studies No. 41 was prepared in co-operation with the Intergovernmental Oceanographic Commission (IOC) of Unesco and the International Atomic Energy Agency (IAEA). It includes comments received from the joint IOC/UNEP Group of Experts on Methods, Standards and Intercalibration (GEMSI) of GIPME, and from a number of scientists who reviewed and tested the method. The assistance of all those who contributed to the preparation of the draft issue of this reference method is gratefully acknowledged.

CONTENTS

1. SCOPE AND FIELD OF APPLICATION

This publication describes sampling and sample preparation procedures suitable to obtain uncontaminated samples for the purpose of determining river inputs of inorganic contaminants into estuaries. Emphasis is placed on heavy metal contaminants but procedures are suitable, with appropriate modifications for other inorganic contaminants. For example, the collection of samples for mercury may require modifications of handling procedures.

2. REFERENCES

WINDOM, H.L., SMITH, R.G. and MAEDA, M. (1985) The geochemistry of lead in rivers, estuaries and the continental shelf of the southeastern United States. Mar. Chem., 17, 43-56.

3. PRINCIPLES

River water samples are collected at the most down-river point where no estuarine influences effect results. Samples are collected using a peristaltic pump and separated into aqueous and particulate phases for contaminant analysis.

As is the case of all trace contaminant analyses, meticulous care is required to prevent contamination of the sample and in addition to the precautions described in this method, great personal attention is required to minimize sample handling, contamination by smoke, hands, hair, dust, talc from gloves, etc. and to avoid all contact of the samples and reagents with skin and metalic objects.

4. REAGENTS

4.1 10% HCl(v/v). 1 part reagent grade concentrated hydrochloric acid mixed with 9 parts deionized-glass distilled water (4.2).

4.2 Deionized glass distilled water.

4.3 Reagent grade nitric acid.

4.4 Water distilled in quartz sub-boiling still. Milli-Q water may be an acceptable substitute but should be thoroughly tested by blank analysis for the determined parameters.

4.5 Reagent grade chloroform.

4.6 Sub-boiling redistilled nitric acid.

5. APPARATUS

5.1 Current meter with direction sensor and deck readout.

5.2 Peristaltic pump. Type chosen will depend on logistics (e.g. power source) and the size of the pump head will depend on the volume requirements of the project.

5.3 Silicone sampling tube - 50 feet (or longer depending on water depths to be sampled) of silicone tubing sized to fit peristaltic pump head (5.2). An approximately 20 cm length of rigid teflon tubing is fitted to one end of the silicone tubing (see Figure 2).

5.4 40 to 50 liter plastic pail with lid.

5.5 Plastic bags (20 cm x 10 cm x 5 cm). To cover ends of sampling tube (5.3) and for 47 mm filter holders (5.9).

5.6 Rubber bands to secure ends of plastic bags.

5.7 Pressurized filtration system (Figure 3) consists of: nitrogen gas tank, gas pressure regulator, in-line gas filter, 5 to 10 liter conventional polyethylene carboy, teflon tubing, two teflon valves and teflon compression fittings to connect teflon tubing to system components and to 142 mm and 47 mm filter holders (5.8, 5.9).

5.8 Teflon lined holder for 142 mm Nuclepore filters.

5.9 Polycarbonate (Swinlok or Swinnex or Sartorius equivalents) holders for 47 mm Nuclepore filters.

NOTE: These filter holders are shipped with Luer connections.These should be removed with a fine tooth saw. This will leave a threaded nipple that will accept 1/4 inch N.P.T.(National Pipe Thread) fittings allowing the filter holders to be attached to the filtration system.

5.10 Plastic bags large enough to cover carboy of filtration system (5.7) and to place 142 mm filter holder (5.8) in between use.

5.11 Large glass beaker for acid bath. Must be large enough to hold (5.8).

5.12 Hotplate.

5.13 Laminar flow clean cabinet.

5.14 Silicone o-rings to fit (5.9) filter holders.

5.15 Ultrasonic bath.

5.16 Sample bottles. Either FEP teflon or CEP polyethylene. Size 0.5 to 1.0 liter depending on analyses to be made.

5.17 Plastic bags large enough to contain bottles (5.16).

5.18 142 mm diameter Nuclepore filters (0.4 µm pore size).

5.19 47 mm diameter Nuclepore filters (0.4 µm pore size).

5.20 Small vacuum pump with side arm flask reservoir.

5.21 Small (ca 5-10 ml) polyethylene vials with polyethylene caps for storing filters (5.19).

5.22 Inlet tube attachment assembly (Figure 2).

5.23 Polypropylene rope to use as hydrowire (sufficient length for depth to be sampled).

5.24 Sampling rope weight covered with plastic bag.

5.25 500 ml graduate cylinders.

5.26 Plastic tweezers.

5.27 Styrofoam ice chest or refrigerator.

5.28 Analytical balance, 100g capacity with a precision of at least 0.1mg. The balance should have provision for dessication within the weighing compartment (a suitable dessicant can be provisionally held in a cloth bag taped to the compartment wall). For the weighing, an ionizing source should also be placed within the weighing compartment.

5.29 Glass petri dishes of about 6 cm i.d.

5.30 Small thermostatted drying oven.

5.31 Dessicator.

6. LOCATION OF SAMPLING SITE

6.1 Longitudinal Location

To determine the input of contaminants to estuaries by rivers it is important to locate river sampling sites in such a way that: (1) estuarine processes have no influence and (2) the site is in the down-river extreme location at which condition 1 is still satisfied. For the purpose of defining this location consider the diagrams in Figure 1 which depict schematically cross-sections of the lower reaches of a river system as it interacts with tidal saline waters. 1a depicts the river cross-section during periods of maximum river discharge when the point at which a detectable rise in the concentration of chloride is observed furthest down the stream at point C. 1b depicts the riverine cross-section during the times of minimum river flow when the point at which a detectable rise in the chloride concentration is observed at its maximum up-stream location, B. During these extremes in conditions, the flow of water at point A is down stream at all depths. Any location further down-stream from A experiences up-stream flow at some time during the period between the extremes depicted in 1a and 1b.

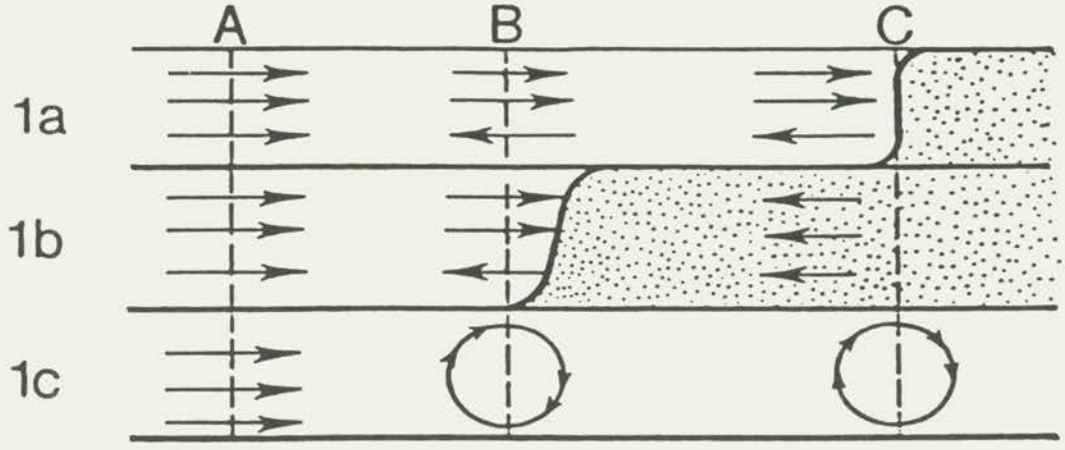

Figure 1: Schematic diagram of River/Estuary Boundary

For the purposes of estimating gross riverine fluxes of materials to estuaries the river boundary must be chosen at A. This is clearly evident by considering diagram 1c which indicates that material, particularly particles, could be transported through a stream cross-section more than once at any location below point A.

The riverine boundary should always be chosen at point A, the down river-most position where downstream unidirectional flow occurs from top to bottom. The location of this point is best determined by current direction surveys conducted at times of low river discharge and rising spring tide. For this purpose a simple current direction meter with deck readout (5.1) is required.

6.2 Cross-Channel Location

In general, for rivers having mean discharges of the order of 100 m^3/sec or less, sampling from mid-channel is probably adequate for determining contaminant inputs. For rivers having larger mean discharges the cross-channel location of the sampling site may require a preliminary survey of the cross-sectional variability of dissolved and particulate major elements (e.g. soluble Na, Ca, Mg and particulate Al) to assess inhomogeneity. Depending on the results of such a survey several cross-channel sampling locations may be required.

In determining the cross-channel location of sampling sites consideration must also be given to contaminant sources just upstream. Contaminants released from sources near the sampling site may not be distributed cross-channel similar to the major elements. Thus, sampling sites should be located to assure that such sources will not be missed.

6.3 Sampling Depth

Vertical inhomogeneity in particulates require that samples at various depths be collected to adequately determine contaminant transport. To best account for vertical inhomogeneity top to bottom integrated samples should be collected. This is explained below (9).

7. FREQUENCY OF SAMPLING

Rivers exhibit pronounced seasonal and storm-period variations in discharge and in chemical concentration and composition, especially with regard to sediment-associated constituents. The nature and extent of these fluctuations further differ between years, events, seasons and stations. If detailed records (hourly intervals) of river chemistry are available from continuous monitors or time-interval samplers, or discharge-interval sampling is employed to assess flow-proportional mean concentration for the study period, the computation of chemical flux (load, transport) presents little problem, providing inhomogeneity within the stream cross-section has been properly taken into account.

Detailed river chemistry data are, however, relatively rare and contaminant flux estimates commonly have to be made by combining continuous records of river discharge with chemical information derived from relatively infrequent spot samples, the number of which may be based on logistical and/or fiscal considerations. There is, therefore, no simple recommendation that can be made regarding the frequency with which samples should be taken for the purpose of estimating contaminant inputs to estuaries by rivers. The following suggested steps, however, will be useful in establishing an appropriate sampling frequency.

1) Obtain river discharge records. These are often available from government hydrological offices. The longer the period for which records are available the better. Instantaneous measurements with a current meter (5.1) after sample collection, may help interpret chemical data but the measurement of river flow itself is not a trivial matter, requires long time series and is beyond the scope of the present manual.

2) If possible calculate the mean discharge for each two week periods over the entire year.

3) Plot biweekly mean discharges in relation to time.

4) Choose sampling times based on changes in discharge. For example, during periods of uniform discharge (usually dry seasons) samples can be collected less frequently to characterize river chemistry. As discharge increases and decreases over the period of maximum runoff samples should be collected more frequently to provide information on composition versus discharge.

Any other information that may be helpful in assessing a priori how river chemistry changes temporally should be incorporated into the selection process for establishing sampling frequency. For example, information on sediment transport would be useful. Likewise any information on the temporal variability of contaminant releases should be incorporated in the sampling design.

Clearly, the more often a river is sampled the greater will be the accuracy of contaminant input estimates. Sampling twice a month, however, is probably the maximum frequency recommended for the stated purpose as long as major discharge variations are taken into consideration. The utility of increasing this frequency is a small gain in the reliability of the flux estimates but not a substantial one.

8. PRESAMPLING PREPARATION

8.1 Cleaning of Sampling Apparatus

The sampling apparatus consists of a peristaltic pump (5.2) and sampling tube (5.3). To clean the sampling tube fit it into the peristaltic pump head and put both the inlet and outlet ends of the tube into a clean plastic pail (5.4) containing about five liters (or more if necessary depending on length of sampling tube) of 10% HCl (4.1). Turn on pump and let acid circulate through tubing for about 15 min.

Remove outlet and inlet of the sampling tube from acid and pump dry. Replace acid in pail with deionized distilled water (4.2). Repeat the steps described above to rinse tube with distilled water.

After tubing has been pumped dry cover ends with plastic bags (5.5) and secure with rubber bands (5.6). Remove sampling tube from pump head and coil into emptied plastic pail (5.4) and cover until needed.

8.2 Cleaning of Pressurized Filtration System

All teflon tubing and fittings of the pressurized filtration system (5.7) should be soaked overnight in 10% HCl (4.1) then rinsed with deionized distilled water (4.2). The polyethylene carboy/sample reservoir should be cleaned following procedures described below (8.4) for polyethylene sample bottles. Connect system and flush with deionized distilled water.

NOTE: Keep all open ends of teflon tubing (where it connects to filter holders) covered with plastic bags (5.5) and store polyethylene carboy in large plastic bag (5.10) until assembled for use.

8.3 Cleaning of Filter Holders

a. Teflon faced, 142 mm diameter filter holders (5.8): The filter holder is disassembled, rinsed with deionized distilled water (4.2) and soaked over-night in concentrated nitric acid (4.3) in a large glass beaker (5.1) on a hotplate (5.12) at 60°C. It is then rinsed sequentially with deionized water and quartz distilled water (4.4), then air dried in a clean cabinet (5.13). The filter holder is then stored in a plastic bag (5.10) until needed.

b. Nuclepore (Swinlok) polycarbonate 47 mm diameter holders (5.9): The filter holders are dismantled and o-rings replaced with ones made of silicone (5.14). They are then cleaned for 1 hour in an ultrasonic bath (5.15) with a dilute solution of laboratory detergent. The filter holders are then rinsed three times with deionized distilled water (4.2) and ultrasonicated for two hours with 10% HCl (4.1). After rinsing thoroughly with deionized distilled water the filters are ultrasonicated for 30 minutes with deionized distilled water. The filter holders are air dried in a clean cabinet (5.15), loaded with precleaned filters (see 8.5) and then placed in plastic bags (5.5) until needed.

8.4 Cleaning of Sample Bottles

a. Teflon (FEP) (5.16): New bottles must be initially rinsed with a small amount of organic solvent such as chloroform (4.5) to remove any oil or grease present in the bottles. The bottles are then rinsed with deionized distilled water (4.2), filled with concentrated nitric acid (4.3) and loosely capped. The bottles are placed in a large beaker (5.11) of concentrated nitric acid maintained at 60°C for three days (used teflon bottles require only an over-night soak in the acid bath). The bottles are removed from the acid and the acid contents poured into a container for reuse. The bottles are rinsed and filled with deionized distilled water and placed on a hotplate at 60°C for at least 24 hours. The bottles are then emptied, rinsed again and filled with quartz distilled water (4.4) and acidified with 1 ml/l of sub-boiling redistilled nitric acid (4.6). The bottles are stored in plastic bags (5.5) until use.

b. Polyethylene (CEP) (5.16): Initially new bottles should be rinsed with chloroform as described above for teflon bottles. Bottles are filled with 10% HCl (4.1) and heated on a hotplate at 60°C for at least 3 days. The bottles are then rinsed and filled with deionized distilled water and placed on a hotplate for at least 24 hours. The bottles are then rinsed and filled with quartz distilled water, acidified with sub-boiling redistilled nitric acid (1 ml/L) and bagged as described for teflon bottles.

8.5 Cleaning of Filters

a. 142 mm diameter Nuclepore filters (5.18): These filters are soaked in 10% HCl overnight just prior to use. After loading in holder (5.8), the filters must be rinsed carefully with quartz distilled water to remove all of the acid before filtration (see section 10).

b. 47 mm diameter Nuclepore filters (5.19): These filters are ultrasonicated (5.15) with 10% HCl for five minutes and ultrasonicated again with quartz distilled water. The filters are then loaded into the polycarbonate filter holders (5.9) in a clean bench using plastic tweezers (5.26). After loading the filters are rinsed with quartz distilled water using a vacuum pump (5.20) to draw the water through the filters.

8.6 Cleaning Polyethylene Vials (5.21)

Soak vials in 10% HCl (4.1) overnight, rinse with deionized water and dry in clean cabinet (5.13). Keep in plastic bags.

9. SAMPLE COLLECTION

Upon arrival at the sampling site the silicone sampling tubing (5.3) is installed into the peristaltic pump (5.2) allowing enough length of tubing on the outlet side to reach the polyethylene carboy of the pressurized filtration system (5.7). Attach the end of the silicone sampling tubing (5.3) having the rigid teflon tube to the inlet tube attachment assembly (5.22, Figure 2) and secure onto the polypropylene hydroline (5.23) approximately 70 cm above the plastic covered weight (5.24). The assembly and sampling tube will act as a vane orienting the inlet of the tube upstream.

Station keeping should be achieved in such a way as to disturb bottom sediments as little as possible and thus avoid resuspending particulate matter. Anchoring may be necessary in swiftly flowing rivers where resuspended sediments are rapidly dispersed, but should be avoided where flow is sluggish and where stations can be held under the boat's power or by drifting.

After arrival on station remove plastic bag from inlet of sampling tube (5.3) and lower hydroline until weight just touches bottom and then raise approximately 30 cm so that the sampling tube inlet is 1 meter off the bottom. Remove plastic bag from outlet side of sampling tube and pump water for several minutes to flush. Switch off pump and place outlet of sampling tube into carboy of pressurized filtration system (5.7; Figure 3) and pump for a set period of time. Switch off pump, raise sampling tube inlet one meter. Flush sampling tube and then collect sample into carboy for same period of time. Repeat this at one meter intervals to the surface. Pumping time period at each depth should be based on total volume required to supply samples needed for final analyses.

After collection of appropriate sample volume (sufficient to obtain at least duplicates of all samples), raise inlet of sampling tube out of water and pump dry. Cover ends with plastic bags and place excess tubing in plastic pail (5.4) and replace cover until needed at next station.

NOTE: Collection of samples for most organic contaminant compounds can be accomplished as directed above. The reservoir in which the sample is pumped, however, should be a precleaned glass reservoir.

10. PHASE SEPARATION AND SAMPLE PRESERVATION

After an appropriate amount of river water is loaded into the polyethylene carboy of filtration system (5.7) it is tightly capped and the whole system is pressurized to about 0.5 to 0.75 atms. over pressure. To collect particulate samples, water is passed through the 47 mm filters (5.19) until they clog and the flow slows considerably (this usually occurs after about 200 to 500 ml have passed the filters). The volume is collected, measured with a graduated cylinder (5.25) and recorded. The filter holders, with filters intact, are removed after turning off pressure using valve (Figure 3) and rinsed by passing quartz distilled water through them using a vacuum pump (5.20). If clean hood is available, remove filters from filter holders using tweezers (5.26) and place in plastic vials (5.21), label and place in plastic bags and place in ice filled cooler or refrigerator (5.27). If a clean cabinet is unavailable, place filter holder, with filter in place, in a plastic bag, label, place in a cooler and return to laboratory before transferring to plastic vials.

Samples for soluble contaminant analysis are obtained by collecting water passing the 142 mm filter (5.18) directly into sample bottles (5.16). Prior to collecting the sample, however, allow approximately 100 ml of water to pass the filter to flush it first. After suitable samples are collected in sample bottles (5.16) add 1 ml of sub-boiling redistilled nitric acid (4.6) per liter of sample. Place bottle into plastic bag (5.17) and label.

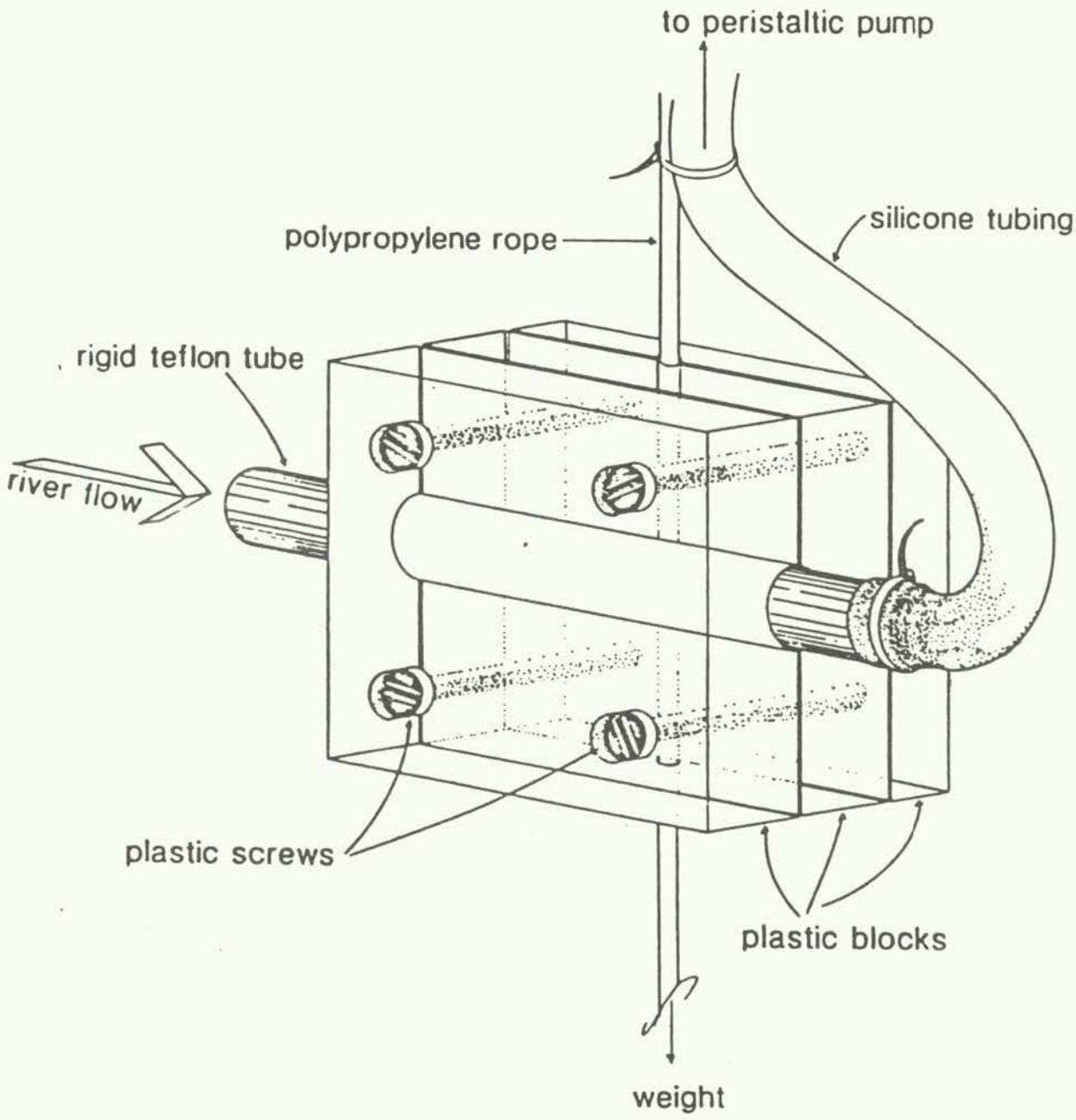

Figure 2: Sampling Tube Attachment Assembly.

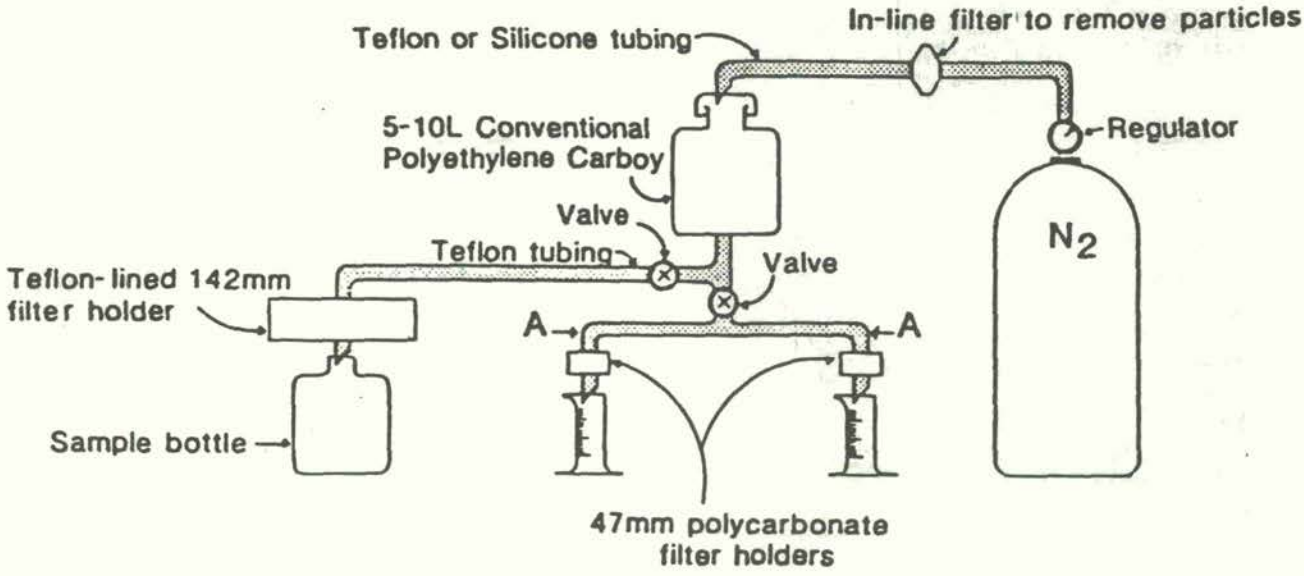

Figure 3: Schematic Diagram of Pressurized Filtration System.

In turbid, organic rich waters the large filters may clog before sufficient sample is collected. If this happens, turn off pressure using valve (Figure 3). Discard used filter and load a new one using plastic tweezers (5.26) and follow procedures described in (8.5).

Between samples the filter system should be rinsed and flushed with dionized distilled water.

11. GRAVIMETRIC DETERMINATION OF SUSPENDED MATTER CONCENTRATION

For calculating the flux of particulate contaminants, some workers prefer to express their data in terms of particulate mass (µg metal/g). The gravimetric determination of suspended matter is intrinsically a simple procedure but requires considerable care when handling and weighing filters and samples.

Filters (5.19), prepared as in 8.5 b and rinsed with at least 250ml of quartz distilled water are placed in Petri dishes (5.29) (previously cleaned as in 8.3 a.) and dried (5.30) to constant weight at 110°C. It is important to handle all filters with tweezers (5.26) and cool and store them in a dessicator (5.31). The filters may become considerably electrostatically charged during drying and give unreliable weights on some balances unless these are efficiently earthed or, ideally, include an ionizing source. Dried filters may then be placed in clean polycarbonate filter holders (5.9, 8.3) and stored in plastic bags.

Depending upon data application, 100-200ml samples for gravimetric determinations can either be drawn off at each sampling level, or as the first duplicate sub-samples from the integrated sample carboy. Some suspended matter tends to sediment very quickly and it is important to thoroughly agitate the contents of the carboy when the sub-sample is taken. The samples are filtered and rinsed as in 10, and returned to Petri dishes for drying at 110°C to constant weight. Suspended particulate load is calculated from the weight difference and the volume of water filtered.

12. SAMPLING AND SAMPLE PREPARATION PROTOCOL

Fill in the sampling and sample preparation protocol (Table 1) giving full details in every column. This protocol should be attached to the reports of analyses of all contaminants in the samples.

The following guidelines should be kept in mind when completing the protocol (the numbers refer to those used in Table 1).

1.2 Some reference to landmarks, river mile or some other feature should be given to accurately locate sampling site.

1.3 Station code should be that adopted by your institution. This code should reflect river, sampling site and date and should be incorporated into sample labels (see below).

1.5 Some description should be given of the method of sampling site selection keeping in mind the discussion in section 6 above.

3.2 This should be provided if known. Results of river gauge may, however, not be available at the time of sampling and may have to be added later.

4.1 On some occasions discrete samples from different depths may be required. If this is the case, sample depth should also be incorporated into the sample label.

5.1 All filters should be assigned a code number which will be used throughout the gravimetric analysis (see also note under 6.1).

5.2 Provision is made here for up to three reweighings (to achieve constant weight).

5.5 This is calculated from:

$$\frac{[(\text{Filter + particulate weight}) - (\text{Filter weight})] \times 1000}{\text{volume filtered (ml)}} \quad \text{g/l}$$

6.1 Each replicate sample should be labeled including station code, depth and replicate (i.e. a, b, etc.).

6.2 Water volume refers to the amount of water filtered and is required to calculate concentrations of contaminants transported in particulate phase.

6.3 Give a description of how samples were handled referring to section 10 above.

7.2 Give the volume of each sample collected since different volumes may be required for different analyses.

7.3 Indicate the amount of sub-boiling redistilled nitric acid (4.6) used.

Table 1: Sampling and Sample Preparation Protocol

1. Sampling Location

   1.1 River:_____

   1.2 Sampling site:_____

   1.3 Station code:_____

   1.4 Station depth:_____

   1.5 Method of selecting sampling site:_____

   _____

1. _____

2. Date of Sampling: Day_____; Month_____; Year_____

3. River Discharge

   3.1 Mean annual:_____m^3/sec.

   3.2 At time of sampling:_____m^3/sec.

   3.3 Instantaneous current measurements:

   3.3.1 Time of measurement_____h._____min, duration_____min
   3.3.2 Depth of measurement_____m below surface
   3.3.3 Current speed_____cm/sec.
   3.3.4 Current direction_____degrees.

4. Sample Depth

   4.1 Discrete sample depth:_____

   4.2 Integrated sample depth interval:_____

5. Gravimetric analysis

   5.1 Filter label a:_____ b: _____

   5.2 Filter weights a1:_____g b1: _____ g

   a2:_____g b2: _____ g

   a3:_____g b3: _____ g

   5.3 Volume filtered a: _____ml b: _____ ml

   5.4 Filter + particulate a1:_____g b1:_____ g
   weights

   a2:_____g b2:_____ g

   a3:_____g b3:_____ g

   5.5 Suspended sediment load a:_____g/l b:_____g/l

6. Particulate Fraction

6.1 Sample label a:_____ b:_____

6.2 Water volume a:_____ml b:_____ ml

6.3 Handling and storage:_____

_____

_____

_____

7. Dissolved Fraction

7.1 Sample label a:_____ b:_____ c:_____

7.2 Sample volume a:_____ ml b:_____ ml c:_____ml

7.3 Acidification a:_____ ml b:_____ ml c:_____ml

7.4 Handling and storage_____

_____

_____

_____

8. Full address of the institution carrying out the sampling and sample preparation:

_____

_____

_____

_____

9. Name(s) and signature(s) of the person(s) who carried out the sample preparation:

_____

_____

_____

_____

Date: _____

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