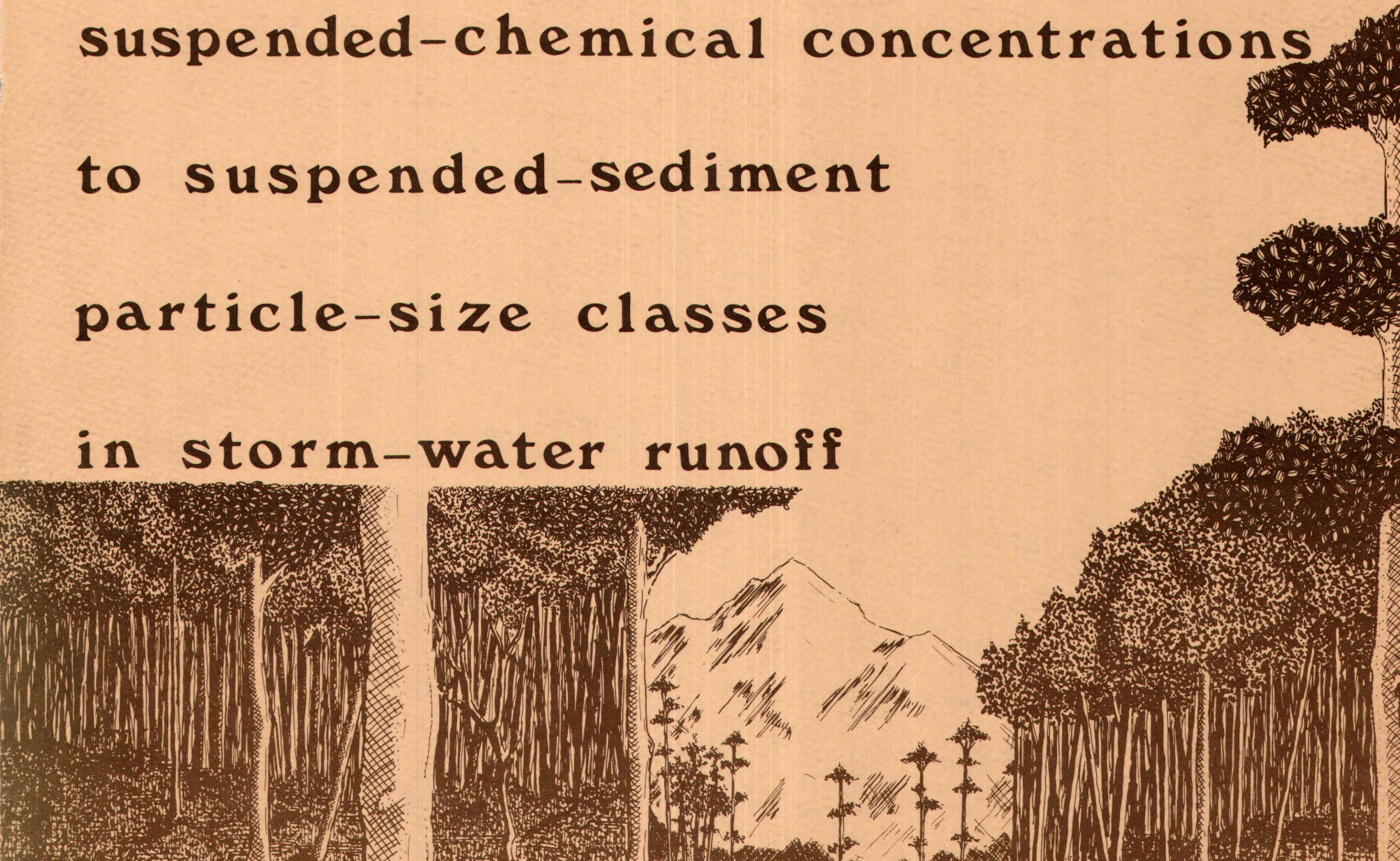
Method for relating suspended-chemical concentrations to suspended-sediment particle-size classes in storm-water runoff



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METHOD FOR RELATING SUSPENDED-CHEMICAL CONCENTRATIONS TO SUSPENDED-SEDIMENT PARTICLE-SIZE CLASSES IN STORM-WATER RUNOFF

By Joseph F. Rinella and Stuart W. McKenzie

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ABSTRACT

A method has been developed to relate suspended-chemical concentrations (associated with suspended sediments) in storm-water runoff to suspended-sediment particle-size classes. These classes are based on settling velocities in quiescent native water. This method requires processing 20 liters of water having a suspended-sediment concentration higher than 500 milligrams per liter. However, samples with suspended-sediment concentrations as low as 250 milligrams per liter may be analyzed, if sample volumes are increased to 50 liters. The time required for one person to separate suspended sediments into particle-size classes ranges from 6 to 14 hours. This report outlines procedures for processing metal, nutrient, and organic samples.

INTRODUCTION

This report outlines procedures to relate suspended-chemical concentrations (nutrients, metals, or organic constituents) associated with suspended sediments in storm-water runoff to suspended-sediment particle-size classes based on settling velocities. Knowledge of this relationship will aid in predicting the transport characteristics of chemicals in streams and in reducing the effects of storm-water runoff on receiving streams.

Two studies of the Willamette River basin, Oregon, revealed the need to define chemical and particle-size relationships. Hines and others (1977) reported that a benthal oxygen demand was contributing to low dissolved-oxygen concentrations in Portland Harbor. Not all the major sources of organic material are presently known, but a significant source may be attributed to urban storm-water runoff. Suspended-sediment concentrations of storm-water samples from seven small drainage basins near Portland generally exceeded levels expected for secondary waste-water treatment-plant effluent (Miller and McKenzie, 1978). To determine the contribution of storm water to the benthal oxygen demand in the Portland Harbor, two factors need to be defined: (1) Flow hydraulics of the harbor and (2) the settling and transport characteristics of storm-water chemicals associated with suspended sediments.

Knowledge of chemical-transport characteristics also could be used to aid in optimizing the operation of detention ponds and thereby improve the quality of storm-water runoff.

The following method quantifies the relationship between chemical concentrations and settling velocities of suspended sediments in quiescent native water (water that is in contact with the sediments at the time of sampling).

DESCRIPTION OF METHOD

Application

1. Application

1.1 This method may be used to relate suspended-chemical concentrations to suspended-sediment particle-size classes, based on settling velocities. Ideally, a water sample should have a suspended-sediment concentration higher than 500 mg/L. Samples having suspended-sediment concentrations as low as 250 mg/L may be analyzed; however, a minimum 8-hour settling period followed by supernatant withdrawal would be required to concentrate the sediments.

1.2 When using this method, suspended sediments are classified by size according to their fall velocities in quiescent native water. Quiescent native water is used to approximate the characteristics of a calm body of water, such as a lake, reservoir, or detention pond; however, these bodies of water are influenced by inflow, outflow, wind, and thermal currents. The data obtained using this method do not simulate natural streamflow conditions, where turbulence and varying degrees of sediment flocculation occur.

1.3 Particle-size classes and chemical-concentration relationships are dependent on native-water and sediment characteristics (physical and chemical). Therefore, relationships between chemical concentrations and particle sizes that are developed for one basin may not be transferable to another basin.

Because of seasonal variations in (1) the dissolved chemicals in the native water and (2) the seasonal variations in the sediment composition, relationships developed for one site may not be consistent throughout the year.

Summary of Method

2. Summary of method

2.1 Suspended sediments in native water are separated into particle-size classes and then analyzed for selected nutrients, trace metals, and organic constituents. Particle-size classes are determined by settling velocities in quiescent native water, using Stoke's law to compute particle fall time.

This method uses pipet withdrawals to separate sediments into particle-size classes. Withdrawals are made at predetermined depths. Particles having a settling velocity greater than that of the specified particle size will settle below the point of withdrawal after a certain time has elapsed. Sediments in a size class do not necessarily have diameters less than the specified size, but they do have settling velocities less than those of a quartz sphere of the specified size settling in distilled water.

2.2 To approximate settling conditions in a quiescent body of native water, (1) sediments are settled in native water, (2) sands are not separated from the sample, (3) processing includes no harsh mechanical mixing, and (4) a dispersing agent is not added to minimize flocculation.

Interferences

3. Interferences

3.1 The precision (based on indeterminate weighing errors) of a particle-size analysis decreases as the dissolved-solids concentration increases (see sec. 9, table 4). To determine the weight of a particle-size class, a subsample must be evaporated, leaving a residue of sediments and dissolved solids. The sediment weight is the residue weight less the dissolved-solids weight. As the dissolved-solids weight increases relative to the residue weight, the precision of the sediment weight decreases.

3.2 Concentrations of the total recoverable chemicals associated with a particle-size class include dissolved-chemical concentrations. As the dissolved-chemical concentration increases relative to the total recoverable chemical concentration, the precision of the suspended-chemical concentration determination associated with a particle-size class decreases.

3.3 Pipet-withdrawal procedures require warming the water sample to room temperature and maintaining this temperature for about 4 hours. The elevated temperature may alter the nonconservative constituents (nutrients and organics) by means of biological or chemical processes. To minimize errors, samples should be processed and preserved as soon as possible after collection.

3.4 Interferences associated with analyses of inorganic chemicals are discussed in a report by Skougstad and others (1979).

3.5 Interferences associated with the biochemical oxygen demand test are listed in U.S. Geological Survey Quality of Water Branch Technical Memorandum 80.28.

Apparatus

4. Apparatus

4.1 Suspended-sediment sampler and sample bottles approved by the U.S. Geological Survey (USGS) for stream sampling. For trace-metal and organic samples, the sampler should be epoxy painted and equipped with a silicone gasket and teflon or nylon nozzle.

4.2 Sample-storage bottles, 30 2-liter narrow-mouth borosilicate-glass bottles (for organic or nutrient samples), Corning[1] 1460 or equivalent; and 30 2-liter narrow-mouth polyethylene bottles (for metal or nutrient samples), Nalgene 2202-0005 or equivalent. Tare bottles to the nearest gram. Two-liter narrow-mouth bottles are convenient for handling and for pouring the sample through the cone splitter.

4.3 Funnel, polyethylene, having a top inner diameter of 6 in., Nalgene 4260-0060 or equivalent. Funnel to be used in filling the sample-storage bottle.

4.4 Sample cone splitter, described in USGS Quality of Water Branch Technical Memorandum 80.17. A bubble level is required to level the splitter.

4.5 Ten graduated cylinders, glass, 2-liter volume with 20-mL subdivisions, Kimble 20024D or equivalent. Tare cylinders to the nearest gram.

4.6 Top-loading balance, 4,000-gram capacity with a readability of 0.1 gram, Sartorius 1203MP or equivalent.

4.7 Refrigerator, capable of maintaining samples at temperatures of 1° to 4°C and having sufficient space to store 25 2-liter sample-storage bottles.

4.8 Peristaltic pump, with variable speed drive (6 to 600 revolutions per minute), Masterflex C-7553-00 or equivalent; and pump head having a flow range of 10 to 1,000 mL/min, Masterflex C-7015-20 or equivalent. Use silicone tubing, Masterflex C-6411-45 or equivalent.

4.9 Glass j-tube, attaches to peristaltic pump and is used to remove supernatant water from sample-storage bottles. The j-tube is a 12-inch-long glass tube (4.5 mm diameter) having one end bent up to form a j-shape.

4.10 Withdrawal pipet, glass volumetric, 50-mL capacity, Kimax 37000 or equivalent. Grind off the tip to enlarge the inlet-orifice diameter to 4 mm to permit withdrawal of large-diameter particles.

4.11 Movable carriage, containing the withdrawal pipet, enables convenient pipetting from all of the sedimentation cylinders (Guy, 1969, p. 25). A mechanism to lower and raise the pipet is attached to the carriage. A burette support stand may be substituted for the movable pipet carriage.

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[1] The use of brand names in this report is for indentification purposes only and does not constitute endorsement by the U. S. Geological Survey.

4.12 Plunger, (teflon) custom made to churn a 2-liter graduated cylinder. The plunger consists of a disk (2.4-cm diameter and 0.5-cm thickness) attached to a rod (1.0-cm diameter and 60-cm length). Numerous holes (0.6-cm diameter), spaced about 4 cm apart, are drilled through the disk.

4.13 Glass containers, Griffin beakers, six 250-mL and eight 400-mL capacity; and eight flat-bottom evaporating dishes having a 50-mm height and a 90-mm top inner diameter. Tare glass containers to the nearest 0.1 mg.

4.14 Three stopwatches, with readability to the nearest second. The watches will be used to time the withdrawals.

4.15 Sample bottles (for shipping), 250-mL nitric acid-rinsed polyethylene bottles for metal samples, and 125-mL glass bottles (muffle furnaced at 500°C) with an aluminum-foil cap insert (also muffle furnaced). The glass bottles are used for organic carbon and nutrient samples.

4.16 Backflush filter holder, 142-mm diameter. Geo filter 0800 or equivalent.

4.17 Stainless-steel, pressure, filter holder, 47-mm diameter, Millipore XX40040 or equivalent. (Pressure supplied by tank of water-pumped nitrogen gas. CAUTION: Oil-pumped nitrogen gas may contaminate the sample.)

4.18 Membrane filters, 0.45-micrometer pore-size Millipore HATF-142 or equivalent, for metal and nutrient samples; silver-metal membrane filters, 0.45-micrometer pore size, Selas FM-47 or equivalent (for suspended-organic and dissolved-organic carbon samples).

4.19 Centrifuge, capable of centrifuging 0.5 to 1 L of sample at 1,000 revolutions per minute. Required for dissolved-carbonaceous biochemical demand analyses.

4.20 Analytical balance, having a capacity of 166 g with a readability of 0.1 mg, Sartorius model 2003MP1 or equivalent.

4.21 Oven, capable of heating samples to 110° ±5°C. Samples should be cooled to room temperature in desiccators prior to weighing.

4.22 Desiccators, Corning 3100 or equivalent.

4.23 Shipping coolers, capable of keeping samples chilled with ice (less than 4°C) for 2 to 3 days.

4.24 Biochemical oxygen demand supplies, as listed in USGS Quality of Water Branch Technical Memorandum 80.28.

Reagents

5. Reagents

5.1 Reagents for biochemical oxygen demand, carbonaceous (CBOD): See USGS Quality of Water Branch Technical Memorandum 80.28.

5.2 Nutrient preservative: Mercuric chloride, sodium chloride tablet (10 mg of $HgCl_2$). See USGS Quality of Water Branch Technical Memorandum 80.26.

5.3 Metal preservative: Concentrated nitric acid that conforms to the specifications of the Committee on Analytical Reagents of the American Chemical Society (Skougstad and others, 1979).

5.4 Water used to prepare chemical solutions or to rinse sample containers and filters: Water shall be demineralized by passage through mixed cation-anion exchange resins or by distillation. Its specific conductance, at 25°C, must not exceed 5 umho/cm, and it shall be stored in borosilicate-glass or polyethylene bottles. Water purity must meet the specifications for type III water as established by the American Society for Testing and Materials, the College of American Pathologists, and the National Committee for Clinical Laboratory Standards (type III water specifications may be obtained through scientific-supply companies).

Procedure

6. Procedure

6.1 If the suspended-sediment concentration of a stream is estimated to be greater than 500 mg/L, follow procedure 6.1.1A. If the sediment concentration is between 250 and 500 mg/L, follow procedure 6.1.1B. Figure 1 shows a procedural flow diagram of the method.

NOTE 1: Estimation of a suspended-sediment concentration by visual inspection will require some familiarity with the sampling site. If uncertain whether sediment concentration is greater than 500 mg/L, follow step 6.1.1B.

NOTE 2: Glass sample containers should be used for storing organic samples, and acid-rinsed polyethylene containers should be used for metal samples. Either glass or regular polyethylene containers are satisfactory for nutrient samples.

6.1.1A Sediment concentrations greater than 500 mg/L: Tare 10 2-liter narrow-mouth containers to the nearest gram, and record weights on the worksheet form shown in figure 2. Fill the containers with about 20 L of depth- and width-integrated samples using a U.S. DH-48TM or other USGS approved sediment sampler (epoxy coated). To minimize sediment losses, pour contents of the sediment-sampling bottle (pint or quart) directly into one of the

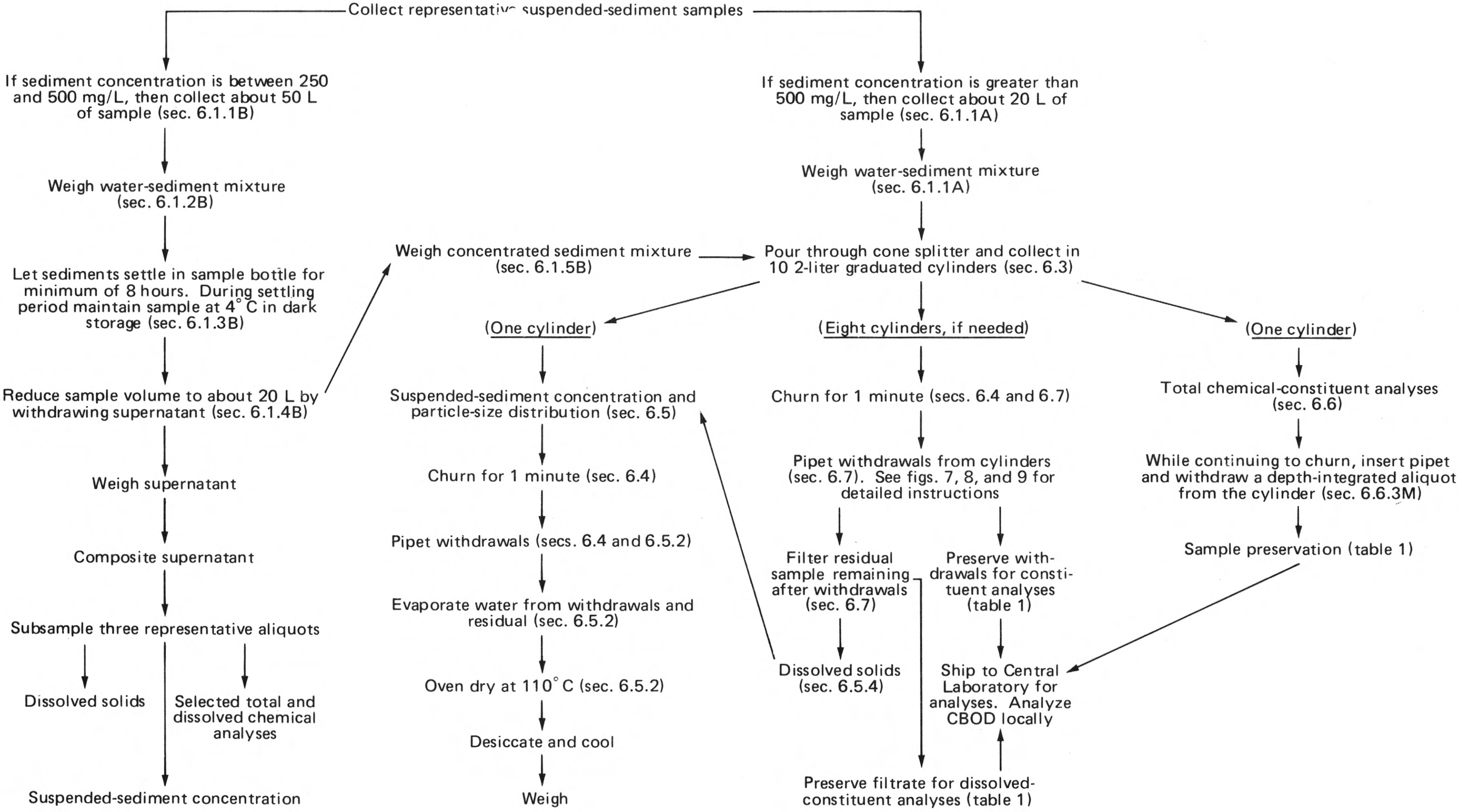


Figure 1. – Flow diagram showing procedural steps to relate suspended-chemical concentrations to suspended-sediment particle-size classes.

WORKSHEET FOR COMPUTING SAMPLE WEIGHT

[Form to be used for samples processed in section 6.1.1A]

Stream name: ______________________________

Location: ______________________________

Sampling date: ______________________ Sampling time: ______________________

Column no.	1	2	3	4	5	6	7	8	9	10
Container no.										
Gross										
Tare										
Net										

Weight of sample collected = sum of net weights in columns 1 through 10 = ______________________ grams

Figure 2.– Example of a worksheet for computing sample weight when the sediment concentration is estimated to be greater than 500 mg/L.

2-liter containers after each grab. Twenty liters of sample is adequate to analyze for only one of the following groups of chemicals: metal, nutrient, or organic. Weigh each of the containers of sample (container plus sample) to the nearest gram and record gross weights on the worksheet (fig. 2). Proceed to section 6.2.

6.1.1B <u>Sediment concentrations between 250 and 500 mg/L</u>: Tare 25 2-liter narrow mouth containers to the nearest gram, and record weights in column B of worksheet shown in figure 3. Fill the containers with about 50 L of depth- and width-integrated samples using a U.S. DH-48TM or other USGS-approved sediment sampler. To minimize sediment losses, pour contents of the sediment sampling bottle (pint or quart) directly into one of the 2-liter containers after each grab. Fifty liters of sample is adequate to analyze for only one of the following groups of chemical constituents: metal, nutrient, or organic.

6.1.2B Weigh each of the containers of sample (container plus sample), to the nearest gram, and record gross weights in column A of worksheet shown in figure 3.

6.1.3B Let the sediments settle in the 25 containers for a minimum of 8 hours in dark storage at 4°C.

6.1.4B After settling, use a glass j-tube connected by silicone tubing to a peristaltic pump and withdraw about 1.2 L of supernatant from each of the 25 containers. The 30 L of withdrawn supernatant should be composited and subsampled for analysis of sediment concentration, dissolved solids at 110°C, and total and dissolved chemical constituents (trace metal, nutrient, or organic). See table 1 for container types, sample volumes, and preservation. The supernatant should contain only those sediments having settling velocities less than those of a 0.0039-mm-diameter quartz sphere settling in distilled water (see table 2).

6.1.5B Weigh each of the 25 containers of concentrated sediments and water (including tare), to the nearest gram, and record gross weights in column C of worksheet (fig. 3). Composite the contents of the 25 containers into 10 narrow-mouth 2-liter containers. Some of the composited supernatant, weighed to the nearest gram, may be used to rinse the residual sediments from the 25 containers into any one of the 10 containers. Record the weight of rinse water on worksheet (fig. 3) in the column that is headed C-B.

6.2 Determine the weight of the water-sediment mixture, to the nearest gram, in each of the 10 narrow-mouth containers. If the sample was processed in accordance with section 6.1.1A, the weight of the water-sediment mixture in the 10 containers is the sum of the net weights in columns 1 through 10 on the worksheet shown in figure 2. If the sample was processed in accordance with sections 6.1.1B through 6.1.5B, the weight of the water-sediment mixture in the 10 containers is the sum of the weights in the column labeled C-B plus

WORKSHEET FOR COMPUTING SAMPLE WEIGHT

[Form to be used for samples processed in sections 6.1.1B - 6.1.4B]

Stream name: ______________________________

Location: ______________________________

Sampling date: ______________ Sampling time: ______________

A = Gross weight (container plus sample),
B = Tare weight (container),
A-B = Net weight of sample,
C = Gross weight (container plus sample remaining in container after supernatant withdrawal),
C-B = Net weight of concentrated sediment-water mixture after withdrawal.

Container no.	A	B	A-B	C	C-B
		Sum, W_t		Net sum	
				plus rinse	
				sum, W_c	

Total weight of sample collected = W_t = Sum of weights in column labeled A-B.

Total weight of concentrated water-sediment mixture after supernatant withdrawal = W_c = Sum of weights in column labeled C-B plus the rinse water.

Total weight of withdrawn supernatant = $W_s = W_t - W_c$.

Figure 3.– Example of a worksheet for computing sample weight when sediment concentration is estimated to be less than 500 mg/L.

the weight of the rinse water.

6.3 Pour the contents of the 10 containers through the cone splitter into 10 2-liter graduated cylinders, tared to the nearest gram (see fig. 4). A decision must now be made to analyze the 10 splits for only one of the following groups of constituents: trace metal, nutrient, or organic.

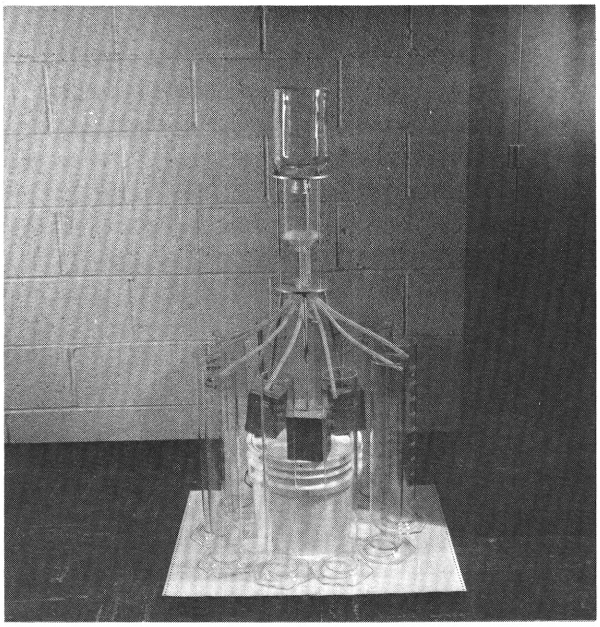


Figure 4.— Photograph showing cone splitter.

Table I.--Container types, sample volumes, and methods of preservation required for the collection of dissolved and total recoverable chemical samples

Chemical constituent	Container type	Approximate sample volume (mL)	Preservation	Comments
Cd, Cr, Co, Cu, Pb, Mn, Ni, and Zn	Nitric acid-rinsed polyethylene bottle	250	0.5-mL concentrated HNO_3	
Phosphorus	125-mL glass bottle	10	Mercuric ion (as $HgCl_2$) concentration of 40 mg/L in sample and chill to 4°C (sec. 6.6.7N)	Keep in dark storage
Kjeldahl nitrogen	do.	20	do.	Do.
Orthophosphate, ammonia, and nitrite plus nitrate	do.	50	do.	Do.
Suspended organic carbon (SOC)	Filter sample through 0.45-micrometer-membrane silver filter and place filter with sediments in petri dish	50	Chill filter to 4°C	On lid of dish, note volume of sample filtered
Dissolved organic carbon (DOC)	Place filtrate that passes through a 0.45-micrometer-membrane silver filter in a 125-mL DOC bottle	100	Chill to 4°C	
Carbonaceous biochemical oxygen demand	BOD bottle	300	Add nitrification inhibitor	Incubate sample at 20° ±0.5°C

6.4 Separating suspended sediments into selected size classes: This section outlines general steps for separating sediments into particle-size classes, using pipet withdrawals. Specific pipet-withdrawal instructions are included in sections 6.5 and 6.7.

6.4.1 Withdrawals are made using a modified 50-mL pipet connected to a peristaltic pump by silicone tubing (see fig. 5). The pumping rate should be adjusted to withdraw a subsample in 8 to 12 seconds. After withdrawal, the silicone tubing should be clamped between the pipet and pump. Releasing the line vacuum permits drainage of the subsample from the pipet into an appropriate container.

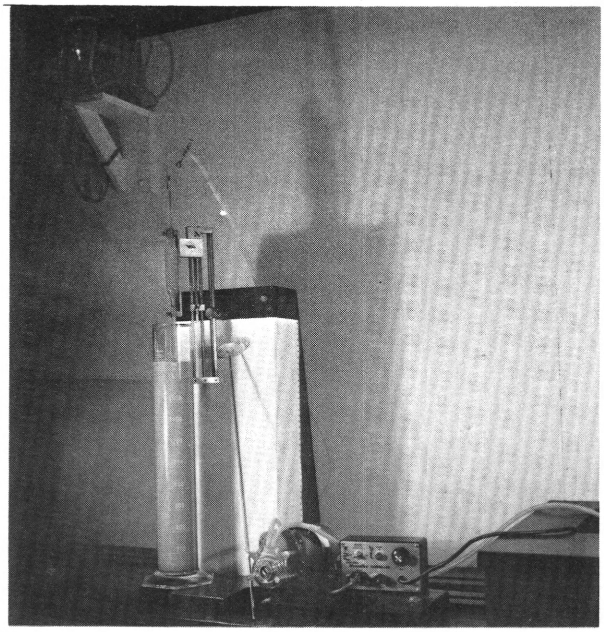


Figure 5.– Photograph showing pipet-withdrawal apparatus.

6.4.2 Withdrawals of specific volumes (depending on the chemical to be analyzed) are made at the times and depths listed in table 2. Stoke's law is used for computing the fall time.

NOTE 3: For a spherical particle (assumed to have a density of 2.65 g/cm^3 and to be settling in distilled water under a gravitational force of 980 cm/s^2), Stoke's law transforms to the following equation:

$$t = \frac{(0.1113)\ (\eta)\ (x)}{d^2}$$

where

t = the fall time, in seconds,
x = the fall distance (or depth of withdrawal), in millimeters,
d = the fall diameter of the spherical particle, in millimeters, and
η = the absolute viscosity of the distilled water, in poises.

6.4.3 Prior to withdrawals, weigh the split sample (to the nearest gram) in a 2-liter cylinder. Equilibrate the sample to room temperature and maintain it to within ±0.5°C throughout the withdrawal period. Withdrawal times for each particle-size class are based on withdrawal depth and sample temperature.

6.4.4 With a plunger, churn the split sample in the cylinder for 1 minute. Establish a uniform churning rate of about 9 in. per second. The churning disk should touch the bottom of the cylinder but not break the water surface. If the water surface is inadvertently broken, continue churning for an additional minute. When the plunger is removed from the cylinder, begin the timing for the fall of 0.0625-mm-diameter particles. Timing listed in table 2 is the mean time and is continuous once the stopwatch begins. For example, the first withdrawal (sediments less than 0.0625-mm diameter) at 20°C and 100-mm depth begins at 24 seconds and ends at 34 seconds (mean time is 29 seconds, which corresponds to the value listed in table 2). After the first withdrawal, the cylinder is not restirred nor is the time restarted. The second withdrawal (sediments less than 0.0312-mm diameter) at 20°C and 100-mm depth begins at 1 minute 50 seconds and ends at 2 minutes. Third, fourth, and fifth withdrawals contain sediments with fall diameters of less than 0.0156, 0.0078, and 0.0039 mm, respectively.

Table 2.--Timing of pipet withdrawals for selected particle-size diameters

[Particle settling in distilled water, with viscosity ranging from 0.01139 poise at 15°C to 0.007975 poise at 30°C]

Diameter of particle (mm)	0.0625	0.0312		0.0156		0.0078		0.0039	
Depth of withdrawal (mm)	100	100		100		100		50	
	Withdrawal timing								
Temperature (°C)	(s)	(min)	(s)	(min)	(s)	(min)	(s)	(min)	(s)
15	32	2	10	8	41	34	44	69	27
16	32	2	7	8	27	33	49	67	38
17	31	2	4	8	14	32	58	65	55
18	30	2	0	8	2	32	6	64	13
19	29	1	57	7	50	31	19	62	38
20	29	1	55	7	38	30	33	61	6
21	28	1	52	7	27	29	49	59	38
22	27	1	49	7	17	29	7	58	13
23	27	1	47	7	6	28	26	56	52
24	26	1	44	6	57	27	47	55	34
25	25	1	42	6	47	27	9	54	18
26	25	1	40	6	38	26	32	53	5
27	24	1	37	6	29	25	57	51	55
28	24	1	35	6	21	25	23	50	47
29	23	1	33	6	13	24	51	49	41
30	23	1	31	6	5	24	19	48	38

6.5 Suspended-sediment concentration and particle-size distribution.

6.5.1 Select one 2-liter cylinder of split sample (see sec. 6.3) for analyses of suspended-sediment concentration and particle-size distribution. For pipet withdrawals, follow procedures outlined in sections 6.4 and 6.5.2.

6.5.2 Record sediment-concentration and particle-size data on Oregon District form P-113, as shown in figure 6. Record the cylinder number, the gross weight of the cylinder (sample plus tare), and the tare weight of the cylinder on the form. Tare five evaporating dishes to the nearest 0.1 mg and record their weights in columns J through N on the form (see note 4). Withdraw a 50-mL subsample for each size class, as outlined in section 6.4, and place into separate tared evaporating dishes. Weigh each withdrawal to the nearest 0.1 g, including tare, and record gross withdrawal weights in columns J through N. Place weighed withdrawals into an oven at a temperature just below the boiling point so as to avoid spattering. After the water has evaporated, increase the temperature to 110°C for at least 2 hours. Cool the containers in a desiccator to room temperature, and then remove and weigh them to the nearest 0.1 mg. Record the gross weights (residue plus tare) in columns J through N.

NOTE 4. All glassware that is to be tared to the nearest 0.1 mg should first be oven dried at 110°C for 2 hours and then cooled in a desiccator to room temperature before weighing.

6.5.3 After withdrawals, the 2-liter graduated cylinder will contain about 1,750 mL of sample. Tare five 400-mL Griffin beakers to the nearest 0.1 mg and record weights in columns A through E (fig. 6). Pour the remaining sample into the five tared beakers, weigh to the nearest 0.1 g, and record gross weights in columns A through E. After recording gross weights, rinse the remaining sediments out of the graduated cylinder directly into any one of the beakers, using about 100 mL of distilled water. The contribution of dissolved solids from the distilled water is negligible. Place the beakers into an oven and allow the water to evaporate at a temperature just below the boiling point. Evaporation time is about 12 to 14 hours. After evaporation, increase the temperature to 110°C for at least 2 hours, then remove and cool the beakers to room temperature in a desiccator. Weigh the beakers to the nearest 0.1 mg, and record the gross weights (residue plus tare) in columns A through E.

6.5.4 Sample remaining in any one of the cylinders after withdrawals (cylinders processed in accordance with secs. 6.7.6M, 6.7.4N, and 6.7.8-0) will be analyzed for dissolved solids. Tare three 250-mL beakers to the nearest 0.1 mg. Using a membrane filter having a 0.45-micrometer pore size, prefilter 1 L of distilled water and discard; then prefilter 200 mL of sample and discard. Next filter about 600 mL of sample. Divide the filtrate into the three tared beakers and record gross sample weights (to the nearest 0.1 g) and tare weights in columns F through H (see fig. 6). Evaporate the water from each beaker at a temperature below 100°C to prevent boiling, and then increase the temperature to 110°C for 2 hours. Cool the beakers to room temperature in a desiccator, weigh the beakers to the nearest 0.1 mg, and record gross weights in columns F through H. (See section 7.1 for calculation of sediment concentration and particle-size distribution.)

6.6 Total recoverable chemical and dissolved-chemical analyses: Sections 6.6.M, N, and O outline procedures for processing samples for trace-metal, nutrient, and organic analyses, respectively.

6.6.1M Total recoverable metals and dissolved-metals analyses: Select one 2-liter cylinder of split sample (see sec. 6.3).

6.6.2M Churn the sample for l minute.

6.6.3M Continue to churn while withdrawing a 50-mL subsample using the modified pipet connected to a peristaltic pump by silicone tubing. This depth-integrated subsample should be taken from the cylinder by lowering and raising the pipet at a constant rate within the water column.

6.6.4M After the pipet is filled, place the subsample into a nitric acid-rinsed 250-mL polyethylene container.

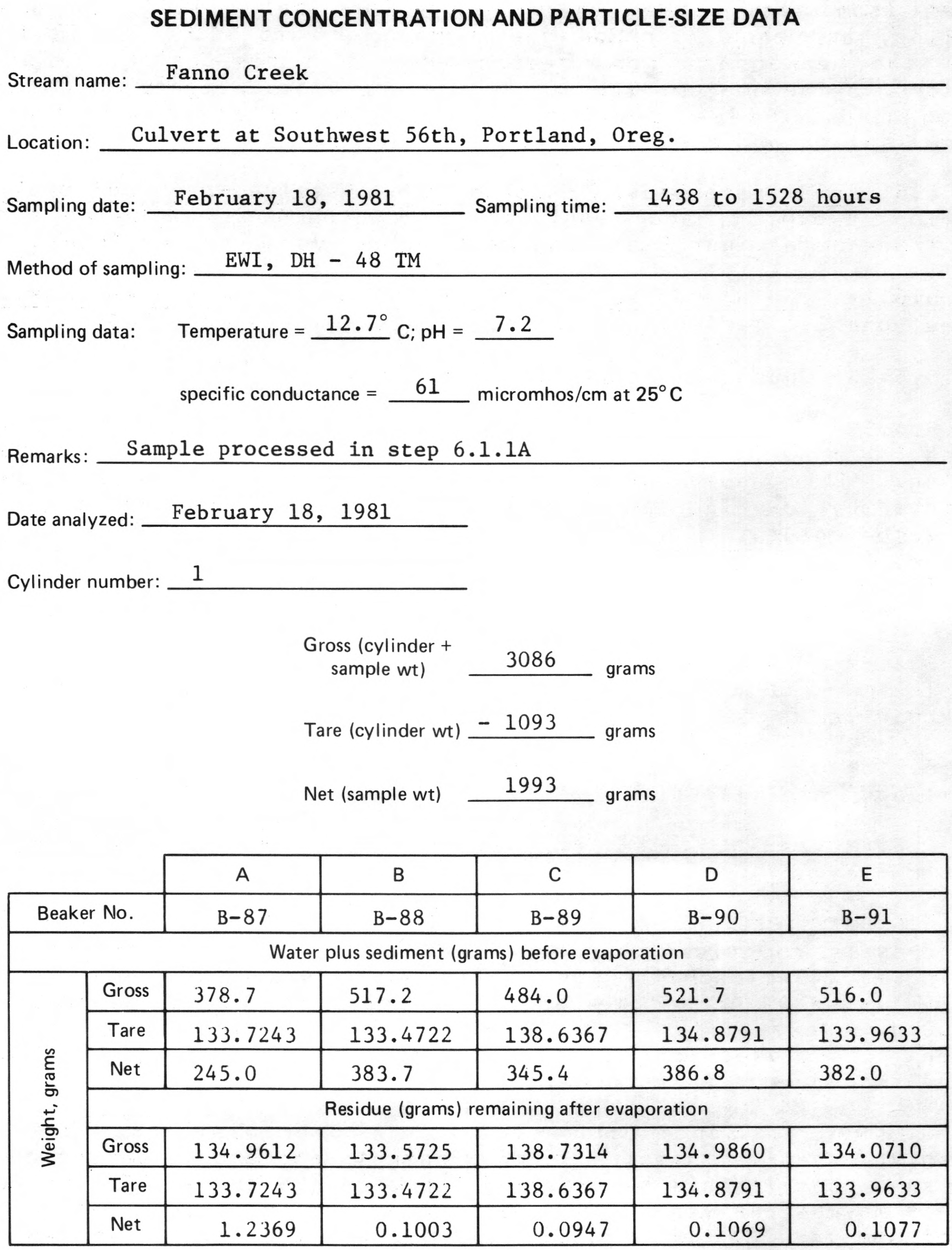
6.6.5M Repeat steps outlined in sections 6.6.2M, 3M, and 4M until the 250-mL container is full (filling will require five withdrawals).

6.6.6M Repeat above procedures until two additional 250-mL containers are filled.

6.6.7M Add 0.5 mL of reagent-grade concentrated nitric acid to each container.

6.6.8M Filter the water remaining in the cylinder after withdrawals for dissolved-metal analyses. Using a membrane filter having a 0.45-micrometer pore size (142-mm diameter), prefilter 1 L of distilled water and discard; then prefilter 200 mL of sample and discard. Next filter three 250-mL subsamples and place them into three acid-rinsed 250-mL polyethylene bottles. Add 0.5 mL of reagent-grade concentrated nitric acid to each container.

6.6.9M Send all total recoverable chemical and dissolved-chemical samples to the Central Laboratory requesting that the same constituents be analyzed for each subsample. The following metals may be analyzed with a detection level of l ug/L for each 250 mL of sample: Cd, Cr, Co, Cu, Pb, Mn, Ni, and Zn. To increase the precision of the analyses, request that all samples be analyzed at the same time rather than dissolved metals being analyzed one day and total recoverable metals on another. If additional analyses are desired, contact the chief of the Metals Analysis Section of the Central Laboratory for sample-volume requirements.



SEDIMENT CONCENTRATION AND PARTICLE-SIZE DATA

Stream name: Fanno Creek

Location: Culvert at Southwest 56th, Portland, Oreg.

Sampling date: February 18, 1981 Sampling time: 1438 to 1528 hours

Method of sampling: EWI, DH - 48 TM

Sampling data: Temperature = 12.7° C; pH = 7.2

specific conductance = 61 micromhos/cm at 25° C

Remarks: Sample processed in step 6.1.1A

Date analyzed: February 18, 1981

Cylinder number: 1

Gross (cylinder + sample wt) 3086 grams

Tare (cylinder wt) - 1093 grams

Net (sample wt) 1993 grams

		A	B	C	D	E
Beaker No.		B-87	B-88	B-89	B-90	B-91
Water plus sediment (grams) before evaporation						
Weight, grams	Gross	378.7	517.2	484.0	521.7	516.0
	Tare	133.7243	133.4722	138.6367	134.8791	133.9633
	Net	245.0	383.7	345.4	386.8	382.0
	Residue (grams) remaining after evaporation					
	Gross	134.9612	133.5725	138.7314	134.9860	134.0710
	Tare	133.7243	133.4722	138.6367	134.8791	133.9633
	Net	1.2369	0.1003	0.0947	0.1069	0.1077

Figure 6.– Sample of laboratory form, "Sediment Concentration and Particle-Size Data." (Form is available from the Oregon District.)

		F	G	H
Container No.		B-1	B-2	B-3
Filtrate wt				
Weight, grams	Gross	204.6	190.4	197.7
	Tare	59.1193	49.7648	56.4493
	Net	145.5	140.6	141.3
	Dissolved solids (110°C)			
	Gross	59.1273	49.7726	56.4569
	Tare	59.1193	49.7648	56.4493
	Net	.0080	.0078	.0076
DS wt, g / filtrate wt, g		5.5×10^{-5}	5.5×10^{-5}	5.4×10^{-5}
			Mean	5.5×10^{-5}

		I	J	K	L	M	N	O
		Total sediments J+ . . . O	Pipet withdrawals					Water + sed, net wt A+ . . . E
Fall diam, mm			0.0625	0.0312	0.0156	0.0078	0.0039	
Container no.			B-4	B-5	B-6	B-7	B-8	
Withdrawal wt, g	Gross		107.9	110.9	113.9	113.6	112.0	
	Tare		57.7613	61.1091	64.0084	63.6490	61.8527	
	Net		50.1	49.8	49.9	50.0	50.1	1742.9
Temp, °C			24	24	24	24	24	
Fall distance			100-mm	100	100	100	50	
Withdrawal time, starting			21s	1m 39s	6m 52s	27m 42s	55m 29s	Residue, net wt A+ . . . E
Residue wt, g	Gross		57.7992	61.1409	64.0306	63.6649	61.8666	
	Tare		57.7613	61.1091	64.0084	63.6490	61.8527	
	Net	1.7682	.0379	.0318	.0222	.0159	.0139	1.6465
	DS corr.	.1097	.0028	.0027	.0027	.0028	.0028	.0959
	Net sed.	1.6585	.0351	.0291	.0195	.0131	.0111	1.5506
Total concn, ppm		832						
Ppm finer than			701	584	391	262	222	

Figure 6.— Continued

6.6.1N <u>Total- and dissolved-nutrient analyses</u>: Select one 2-liter cylinder of split sample (see sec. 6.3).

6.6.2N Mark the 10-, 20-, and 30-mL levels on the outside of the modified 50-mL withdrawal pipet.

6.6.3N Tare nine 125-mL glass bottles to the nearest 0.1 g.

6.6.4N Churn the sample in the 2-liter cylinder for 1 minute.

6.6.5N Continue to churn while withdrawing three each of 10-, 20-, and 50-mL subsamples using the modified pipet. Follow withdrawal procedures outlined in section 6.6.3M.

6.6.6N Place the nine subsamples into separate 125-mL tared glass bottles, and weigh each to the nearest 0.1 g, including tare. On the bottles, record subsample weights to the nearest 0.1 g, excluding tare.

6.6.7N Add mercuric chloride to preserve each nutrient subsample. The mercuric chloride concentration (as $HgCl_2$) must be about 40 mg/L in each subsample (USGS Quality of Water Branch Technical Memorandum 80.26). Preserve subsamples in the following manner:

1. Dissolve four tablets, each containing about 10 mg of mercuric chloride, in 10 mL of distilled deionized water.

2. Preserve the 10-, 20-, and 50-mL subsamples by adding 0.1, 0.2, and 0.5 mL of the mercuric chloride solution, respectively.

3. On the bottles, record the volume of mercuric chloride solution added. Chill samples to 4°C (in dark storage) as soon as possible.

6.6.8N The water remaining in the 2-liter cylinder after withdrawals will be analyzed for dissolved nutrients. Using a membrane filter having a 0.45-micrometer pore size, prefilter (1) 1 L of distilled water and discard, and (2) 200 mL of sample and discard. Then filter three each of 10-, 20-, and 50-mL subsamples. Place the nine subsamples into separate 125-mL glass bottles, tared to the nearest 0.1 g, and preserve in accordance with step 6.6.7N. Weigh each bottle containing subsample to the nearest 0.1 g and record subsample weight on bottle, excluding tare.

6.6.9N At the Central Laboratory, the entire contents of each sample bottle will be extracted or digested for each analysis. Request analyses of phosphorus to the nearest 0.001 mg/L for the 10-mL subsamples; Kjeldahl nitrogen to the nearest 0.01 mg/L for the 20-mL subsamples; and orthophosphate, ammonia, and nitrite plus nitrate to the nearest 0.001 mg/L for the 50-mL subsamples.

6.6.10N Send the chilled nutrient samples to the Central Laboratory as soon as possible. Notify the chief of the Nutrient Section in advance as to when the samples will be arriving.

6.6.1-0 Total- and dissolved-organic analyses: For total CBOD and suspended-organic carbon (SOC) analyses, select one 2-liter cylinder of split sample (see sec. 6.3).

6.6.2-0 Churn the sample for 1 minute.

6.6.3-0 Continue to churn, while withdrawing (using the modified pipet) 20 50-mL subsamples and two 30-mL subsamples. Follow withdrawal procedures outlined in section 6.6.3M.

6.6.4-0 Place 18 of the 50-mL withdrawals into three 300-mL biochemical oxygen demand (BOD) bottles for CBOD analyses. Place remaining withdrawals (for SOC analyses) into separate 125-mL glass bottles tared to the nearest 0.1 g. Weigh the subsamples in the 125-mL glass bottles to the nearest 0.1 g, excluding tare.

6.6.5-0 For each 125-mL glass bottle, filter contents through a silver-membrane filter having 0.45-micrometer pore size. Rinse the remaining sediments from each bottle, using distilled water; also rinse the inner walls of the stainless-steel pressure filter holder. Filter the rinse water through the same filter. Place each filter with sediment into a separate plastic petri dish and record the filtered sample weight on the lid.

6.6.6-0 Refrigerate the SOC samples to 4°C and ship chilled to the Central Laboratory as soon as possible.

6.6.7-0 Analyze the CBOD samples in accordance with Quality of Water Branch Technical Memorandum 80.28.

6.6.8-0 The water remaining in any one of the cylinders after withdrawals (see sec. 6.7.8-0) will be analyzed for dissolved organic carbon (DOC). Using a silver-membrane filter having a 0.45-micrometer pore size, prefilter 25-mL of sample and discard. Then filter 200 mL of additional sample. Fill each of two 125-mL glass bottles with about 100 mL of filtrate and chill to 4°C. Ship chilled DOC samples to the Central Laboratory as soon as possible.

6.6.9-0 About 1 L of split sample remaining in one of the cylinders after withdrawals (see sec. 6.7.8-0) will be centrifuged at 1,000 revolutions per minute for 30 minutes. The supernatant will be withdrawn and analyzed in triplicate for CBOD in order to approximate dissolved CBOD.

6.7 Chemical concentrations associated with particle size classes: Procedures 6.7.M, N, and O outline steps for analyzing trace-metal, nutrient, and organic constituents, respectively.

Metal analyses (Cd, Cr, Co, Cu, Pb, Mn, Ni, and Zn) of selected particle-size classes, based on settling velocities (sec. 6.7.1M)

↓

Requires five 2-liter cylinders of split sample (sec. 6.3)

↓

Process one cylinder of split sample in the following manner:

↓

(Step 1) Churn for 1 minute (sec. 6.4)

↓

(Step 2) Make a 50-mm pipet withdrawal for each of the following particle-size classes:

< 0.0625-mm diameter
< 0.0312-mm diameter
< 0.0156-mm diameter
< 0.0078-mm diameter
< 0.0039-mm diameter
(sec. 6.4 and 6.7.2M)

↓

(Step 3) Place each withdrawal into a 250-mL acid-rinsed polyethylene container designated for a specific size class (sec. 6.7.3M)

↓

Repeat steps 1 and 2 for each of the four remaining cylinders of split sample. Composite each withdrawal into one of the containers mentioned in step 3 for a specific size class (sec. 6.7.3M)

↓

Add 0.5-mL of concentrated nitrite acid to each 250-mL-size class of sample (sec. 6.7.4M)

↓

Ship to Central Laboratory for analyses (sec. 6.7.5M and 6.6.9M)

Figure 7.– Flow diagram showing steps for determining total recoverable metal concentrations associated with particle-size classes.

6.7.1M *Metal concentrations associated with particle-size classes:* Select five 2-liter cylinders of split sample (see sec. 6.3) for analyses of Cd, Cr, Co, Cu, Pb, Mn, Ni, and Zn. Refer to figure 7 for flow diagram showing procedural steps.

6.7.2M Make five 50-mL withdrawals from each cylinder of split sample, as outlined in section 6.4.

6.7.3M All withdrawals having the same size class of sediments are composited into one 250-mL acid-rinsed polyethylene bottle. Each of the five composited sample bottles will contain 250 mL of one of the following particle-size classes: finer than 0.0625-, 0.0312-, 0.0156-, 0.0078-, or 0.0039-mm fall diameter.

6.7.4M Add 0.5 mL of reagent-grade concentrated nitric acid to each of the five bottles.

6.7.5M Send the five containers to the Central Laboratory, requesting the same metal analyses for each. Follow procedures outlined in 6.6.9M.

6.7.6M The remainder of the split sample in one of the 2-liter graduated cylinders after withdrawals may be needed to complete the work outlined in sections 6.5.4 and 6.6.8M.

6.7.1N *Nutrient concentrations associated with particle-sizes classes:* For duplicate analyses, select two 2-liter cylinders of split sample (see sec. 6.3) for phosphorus, two for Kjeldahl nitrogen, and two for orthophosphate, ammonia, and nitrite plus nitrate. Refer to figure 8 for flow diagram showing procedural steps.

6.7.2N Follow procedures for pipet withdrawals, as outlined in section 6.4, making five withdrawals from each cylinder. Withdraw the following volumes for analyses: 10 mL for phosphorus; 20 mL for Kjeldahl nitrogen; and 50 mL for orthophosphate, ammonia, and nitrite plus nitrate. Place the 30 subsamples into separate 125-mL glass bottles, tared to the nearest 0.1 g. Weigh each subsample to the nearest 0.1 g, and record the weight on the bottle, excluding tare.

6.7.3N Follow preservation, analytical, and shipping instructions as outlined in steps 6.6.7N, 6.6.9N, and 6.6.10N.

6.7.4N The remainder of the split sample in one of the 2-liter graduated cylinders after withdrawals may be needed to complete work as outlined (secs. 6.5.4 and 6.6.8N).

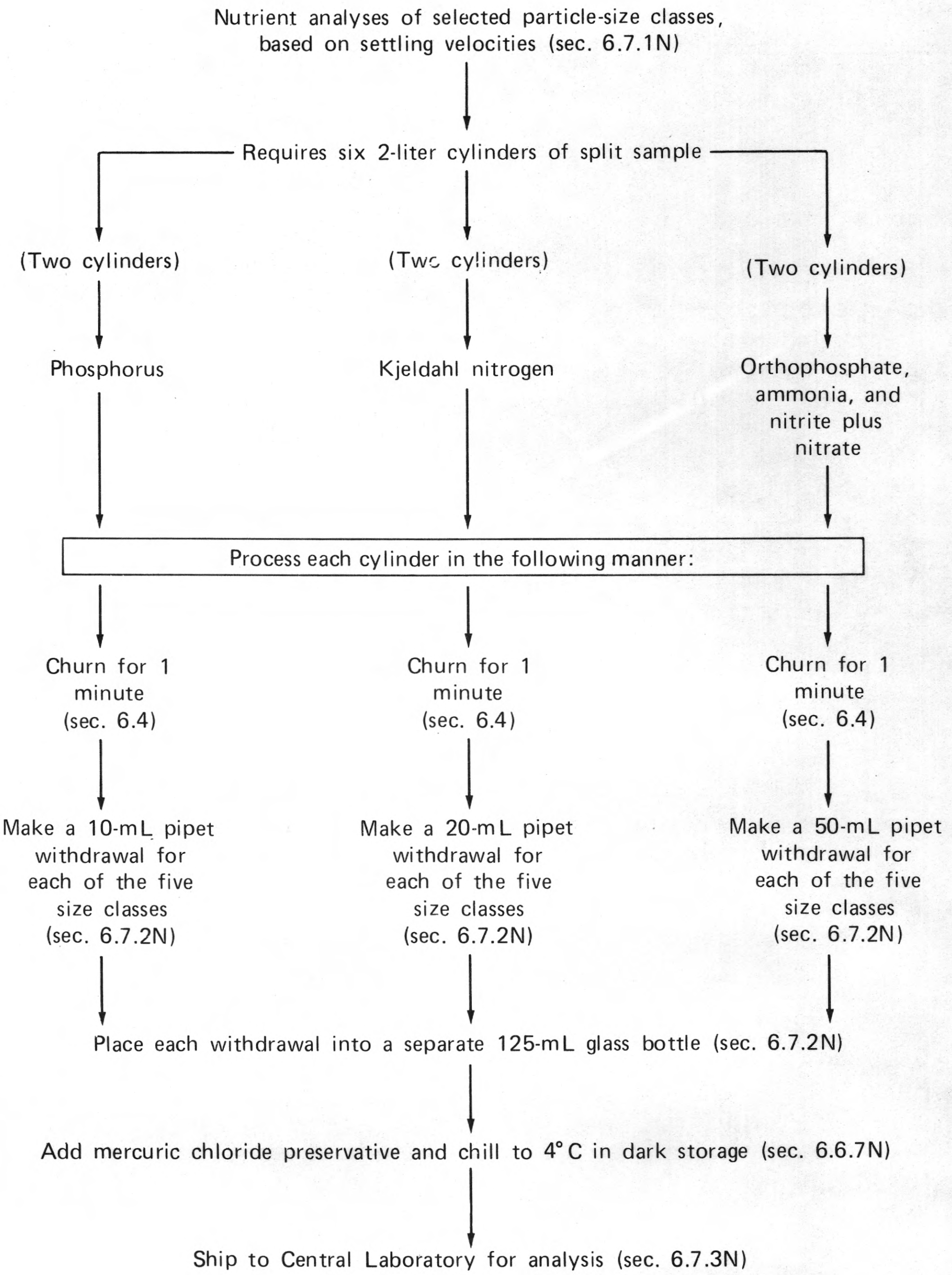


Figure 8.— Flow diagram showing steps for determining total nutrient concentrations associated with particle-size classes.

6.7.1-0 Organic concentrations associated with particle-size classes: Requires processing all eight remaining 2-liter cylinders of split sample (sec. 6.3). Sections 6.7.2-0 through 6.7.4-0 outline sample-processing procedures for SOC analyses and sections 6.7.5-0 through 6.7.7-0 outline sample-processing procedures for determining CBOD.

6.7.2-0 Select two cylinders of split sample to be analyzed for SOC, and follow procedures as outlined in section 6.4 for making pipet withdrawals. Refer to figure 9 for flow diagram showing procedural steps. Make five 50-mL withdrawals from each cylinder.

NOTE 5: If the native water is estimated to have a total sediment concentration greater than 2,000 mg/L, reduce the 50-mL withdrawal volumes outlined in sec. 6.7.2-0 to 25 mL.

6.7.3-0 Place the 10 50-mL withdrawals into separate 125-mL glass bottles, tared to the nearest 0.1 g. Weigh each bottle and subsample to the nearest 0.1 g, and record weight on the bottle, excluding tare.

6.7.4-0 Follow filtering, preserving, and shipping instructions as outlined in secs. 6.6.5-0 and 6.6.6-0.

6.7.5-0 Analyze the remaining six cylinders for CBOD. Make five 50-mL withdrawals from each cylinder as outlined in sec. 6.4.

6.7.6-0 All withdrawals having the same size range of sediments are composited into one 300-mL BOD bottle. After completion of withdrawals, each of the five composited BOD bottles will contain 300 mL of one of the following particle-size classes: finer than 0.0625-, 0.0312-, 0.0156-, 0.0078-, or 0.0039-mm fall diameter. If the composited withdrawals do not completely fill any one of the BOD bottles, add a few glass beads to raise the water level up to the neck of the bottle.

6.7.7-0 Analyze the CBOD samples in accordance with Quality of Water Branch Technical Memorandum 80.28.

6.7.8-0 The remainder of the split sample in the 2-liter graduated cylinders after withdrawals will be needed to complete work outlined in secs. 6.5.4 and 6.6.8-0.

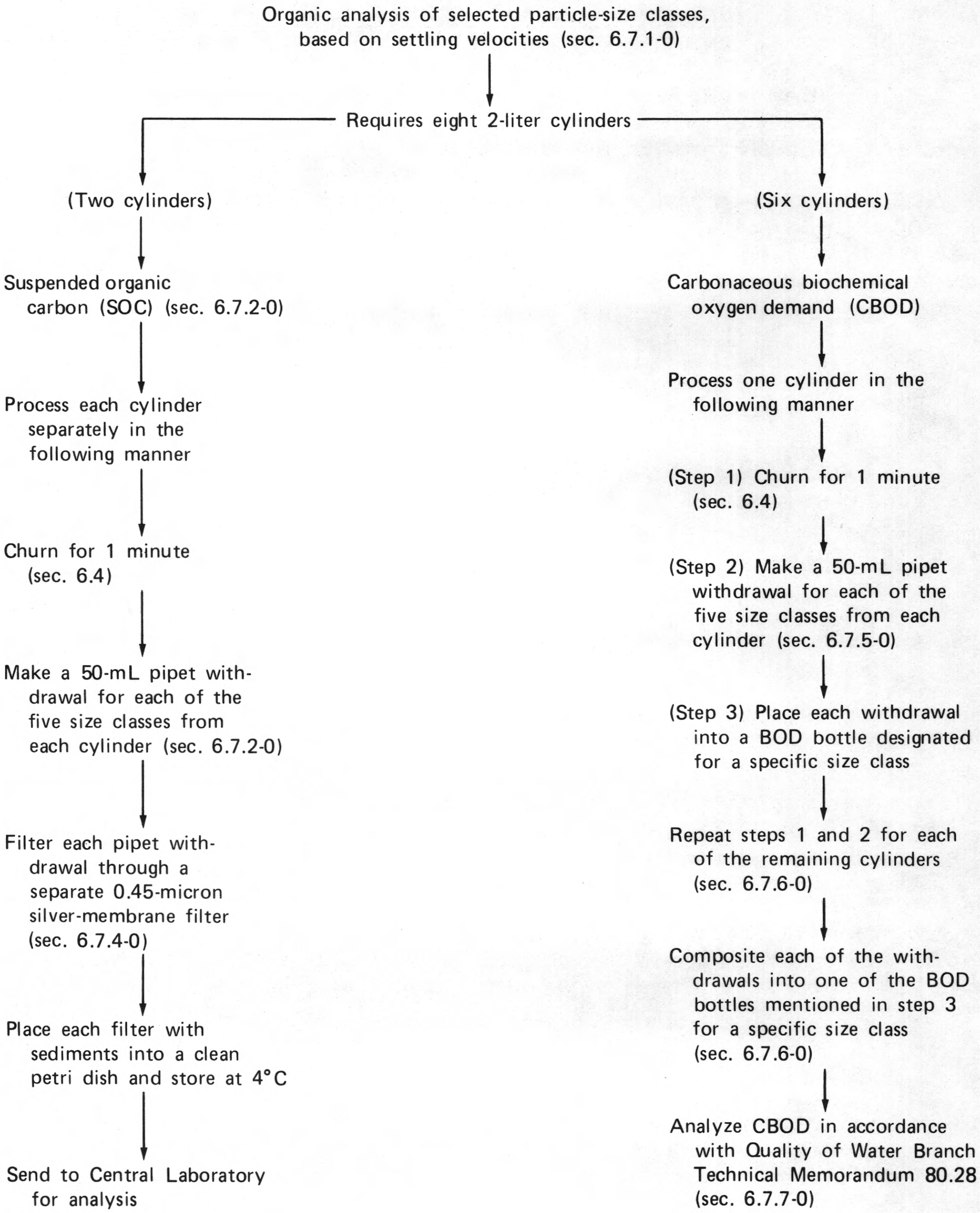


Figure 9.– Flow diagram showing steps for determining organic concentrations associated with particle-size classes.

Calculation

7. Calculation

7.1 Calculation of sediment concentration and particle-size distribution: Procedure 7.1.A outlines the steps for calculating sediment concentration and particle-size distribution in native-water samples having estimated sediment concentrations greater than 500 mg/L. (See sec. 6.1.1A for processing of native water.) Procedure 7.1.B outlines the steps for calculating sediment concentration and particle-size distribution for native-water samples having estimated sediment concentrations between 250 and 500 mg/L. (See secs. 6.1.1B through 6.1.5B for processing of native water.)

7.1.1A Complete the calculations on the laboratory form (columns A through O) shown in figure 4.

7.1.2A Compute dissolved-solids concentrations (DS wt per filtrate wt, g/g). Using data in columns F, G, and H, as follows:

$$\text{Dissolved-solids concentration (g/g)} = \frac{\text{Net DS wt, g}}{\text{Net filtrate wt, g}}$$

Record the mean dissolved-solids concentration (g/g) in the bottom row of column H.

7.1.3A Calculate the weight of residue remaining in the 2-liter cylinder after withdrawals by summing the net weights in columns A through E. Record the net residue weight (dissolved solids plus sediments) in column O in row labeled "Net."

7.1.4A Calculate the weight of dissolved-solids remaining in the 2-liter cylinder after withdrawals, as follows: Subtract the sum of the withdrawal weights in columns J through N from the sample weight in the cylinder prior to withdrawals, and then multiply by the mean dissolved-solids concentration. Record this dissolved-solids weight in column O in the row labeled "DS corr." (dissolved-solids correction).

7.1.5A Calculate the dissolved-solids weight for each withdrawal listed in columns J through N, as follows: Multiply each withdrawal weight by the mean dissolved-solids concentration to determine the dissolved-solids weight. Record these dissolved-solids weights in columns J through N in the row labeled "DS corr."

7.1.6A Add the net residue weights in columns J through O and record the sum in column I. Then add the DS corr. weights in columns J through O and record the sum in column I.

7.1.7A Calculate the sediment concentration of the sample, using data from column I, as follows:

$$\text{Sediment concentration (ppm)} = \frac{\text{Net sed. wt}}{\text{Net sample wt (in cylinder)}} \times 10^6$$

7.1.8A The data in columns J through N are used to calculate the concentrations of sediment finer than the specified diameter sizes, as follows:

$$\text{Sediment concentration finer than specified diameter (ppm)} = \frac{\text{Net sed. wt}}{\text{Net withdrawal wt}} \times 10^6$$

7.1.1B Calculation of sediment concentration and particle-size distribution for water samples processed in accordance with steps outlined in sections 6.1.1B through 6.1.5B:

This calculation requires computing (1) sediment concentration and particle-size distribution in the concentrated water-sediment mixture, and (2) sediment concentration in the withdrawn supernatant.

NOTE 6. Suspended-sediment concentrations are commonly expressed in milligrams per liter; however, in the laboratory the concentration is generally determined in parts per million (ppm) as one million times the ratio of the dry weight of sediment, in grams, to the weight of the water-sediment mixture, in grams. Measurements in ppm are esentially equal to measurements in milligrams per liter for samples with sediment concentrations of less than 15,900 ppm (Guy, 1969).

7.1.2B Follow steps 7.1.1A through 7.1.8A to compute sediment concentrations (of sample and particle-size classes) in the concentrated water-sediment mixture.

7.1.3B Sediment concentration of the supernatant is computed from data obtained using the steps outlined in sec. 6.1.4B.

$$\text{Sediment concentration (supernatant) (ppm)} = \frac{\text{Sediment wt (g)}}{\text{Subsample wt (g)}} \times 10^6$$

7.1.4B Compute the native-water sediment concentration in the sample and in specified particle-size classes, as follows:

$$\text{Sediment concentration (ppm)} = \frac{(C_c \times V_c) + (C_s \times V_s)}{V_t}$$

where

C_c = sediment concentration (of the sample or of a specified particle-size class) in concentrated water-sediment mixture, in milligrams per liter,
V_c = volume of concentrated water-sediment mixture, in liters,
C_s = sediment concentration in withdrawn supernatant, in milligrams per liter,
V_s = volume of withdrawn supernatant, in liters, and
$V_t = V_c + V_s$ = volume of sample collected, in liters.

For example, if:

1. Volume of sample collected is 50 L (V_t),
2. Volume of withdrawn supernatant is 30 L (V_s),
3. Volume of concentrated water-sediment mixture is 20 L (V_c),
4. Sediment concentration in the supernatant is 40 mg/L (C_s),
5. Total sediment (all particle sizes) concentration in concentrated mixture is 832 mg/L (C_c), and
6. Particle-size classes with fall diameters finer than 0.0625, 0.0312, 0.0156, 0.0078, and 0.0039 mm have sediment concentrations of 701, 584, 391, 262, and 222 mg/L, respectively, in the concentrated mixture (C_c),

then suspended-sediment concentrations of the specified-size classes in the native water are:

Total (all particle sizes) sediment concentration (ppm) $= \dfrac{832 \text{ mg/L} \times 20 \text{ L}) + (40 \text{ mg/L} \times 30 \text{ L})}{50 \text{ L}} = 357$

Particles finer than 0.0625-mm diameter (ppm) $= \dfrac{(701 \times 20) + (40 \times 30)}{50} = 304$

Particles finer than 0.0312-mm diameter (ppm) $= \dfrac{(584 \times 20) + (40 \times 30)}{50} = 258$

Particles finer than 0.0156-mm diameter (ppm) $= \dfrac{(391 \times 20) + (40 \times 30)}{50} = 180$

Particles finer than 0.0078-mm diameter (ppm) $= \dfrac{(262 \times 20) + (40 \times 30)}{50} = 129$

Particles finer than 0.0039-mm diameter (ppm) $= \dfrac{(222 \times 20) + (40 \times 30)}{50} = 113$

7.2 Calculation of recoverable chemical concentrations associated with particle-size classes: Procedure 7.2.1A outlines how to calculate the concentration of chemicals associated with particle-size classes for samples that were processed in accordance with section 6.1.1A. Procedure 7.2.1B outlines how to calculate the concentration of chemicals associated with particle-size classes for samples that were processed in accordance with sections 6.1.1B through 6.1.5B.

7.2.1A A total recoverable chemical analysis includes the concentration of the suspended chemical (associated with sediments) and the dissolved chemical. Subtraction of the dissolved-chemical concentration from the total recoverable chemical concentration provides an estimate of suspended chemical concentrations associated with a particle-size class. For example, if:

1. Pb concentration of total sample, including all particle sizes of sediments (suspended plus dissolved) is 200 ug/L,
2. Pb concentration associated with particles finer than 0.0625-mm fall diameter, including dissolved, is 170 ug/L, and
3. Dissolved Pb concentration is 10 ug/L,

then the partitioning of Pb associated with sediments is as follows:

Pb associated with total (all particle sizes) suspended sediments = 200-10 = 190 ug/L

Pb associated with particles finer than 0.0625-mm fall diameter = 170-10 = 160 ug/L

7.2.1B Chemical concentrations in native water that required withdrawal of supernatant to concentrate the sediments should be calculated as follows:

$$\text{Chemical concentration } (\mu g/L) = \frac{[(x_c - X_d)\ (V_c)] + [(X_s - X_d)\ (V_s)]}{V_t}$$

where

X_c = the chemical concentration in the concentrated water-sediment mixture that is associated with the total sample (includes all particle-size classes) or with a specific particle-size class, in micrograms per liter,

X_d = concentration of the dissolved chemical, in micrograms per liter,

V_c = volume of the concentrated water-sediment mixture, in liters,

X_s = the chemical concentration in withdrawn supernatant, in micrograms per liter,

V_s = volume of the withdrawn supernatant, in liters, and

$V_t = V_c + V_s$ = total volume of sample, in liters.

For example, if:

1. Total recoverable manganese concentration in the concentrated mixture is 1,000 ug/L (X_c),
2. Particle-size classes with fall diameters finer than 0.0625, 0.0312, 0.0156, 0.0078, and 0.0039 mm have associated manganese concentrations of 900, 700, 500, 400, and 200 ug/L, respectively, in the concentrated mixture (X_c),
3. Dissolved manganese concentration is 10 ug/L, (X_d),
4. Volume of concentrated water-sediment mixture is 20 L, (V_c),
5. Total recoverable manganese concentration in the supernatant is 40 ug/L (X_s),
6. Volume of the withdrawn supernatant is 30 L (V_s), and
7. Total volume of sample collected is 50 L, (V_t),

then

the manganese concentrations (in micrograms per liter) associated with particle-size classes are:

Mn associated with total (all particle sizes) sediments

$$= \frac{[(1000-10)\ \mu g/L\ (20\ L)] + [(40-10)\ \mu g/L\ (30\ L)]}{50\ L} = 414$$

Mn associated with particles finer than 0.0625-mm diameter

$$= \frac{[(900-10)\ 20] + [(40-10)\ 30]}{50} = 374$$

Mn associated with particles finer than 0.0312-mm diameter

$$= \frac{[(700-10)\ 20] + [(40-10)\ 30]}{50} = 294$$

Mn associated with particles finer than 0.0156-mm diameter

$$= \frac{[(500-10)\ 20] + [(40-10)\ 30]}{50} = 214$$

Mn associated with particles finer than 0.0078-mm diameter

$$= \frac{[(400-10)\ 20] + [(40-10)\ 30]}{50} = 174$$

Mn associated with particles finer than 0.0039-mm diameter

$$= \frac{[(200-10)\ 20] + [(40-10)\ 30]}{50} = 94$$

Report

8. Report

8.1 Report inorganic constituent concentrations in accordance with Skougstad and others (1979).

8.2 Report CBOD concentrations in accordance with Quality of Water Branch Technical Memorandum 80.28.

8.3 Report organic carbon concentrations as follows: if less than 1 mg/L, to the nearest 0.1 mg/L; if greater than 1 mg/L, to two significant figures.

Precision

9. Precision

Precision data for samples containing different concentrations of suspended sediment and dissolved solids are presently (1982) unavailable for this method. The reproducibility of the results when using this method is dependent on the following:

1. <u>Sampling techniques</u>: Quality control for suspended-sediment sampling is discussed in a report by Guy and Norman (1970).

2. <u>Sample-splitting techniques, using the cone splitter</u>: The procedure for testing the precision and accuracy of using the USGS cone splitter for splitting sample volumes is outlined in Quality of Water Branch Memorandum 80.17. To check reproducibility, replicate splits should also be analyzed for suspended-sediment concentrations and particle-size distributions. Table 3 shows a comparison of particle-size analyses (processed in accordance with secs. 6.1.1A and 6.3) on two splits of a composite storm-water sample.

3. <u>Sample-processing techniques (separating sediments into particle-size classes)</u>: The precision of a particle-size analyses is dependent on the dissolved-solids concentration, suspended-sediment concentration, and particle-size distribution. Table 4 shows how indeterminate weighing errors vary with selected dissolved-solids concentrations for a hypothetical particle-size distribution. The values shown in table 4 assume that the standard deviation for a single weight determination is ±0.2 mg. When the sediment concentration and particle-size distribution vary, the precision data shown in table 4 will also vary. If the sediment concentrations shown in table 4 were doubled, the relative deviations would be reduced by about 50 percent.

4. Analytical techniques for determining chemical concentrations: Precision data for chemical analyses are discussed in a report by Skougstad and others (1979). Analyzing replicate samples of particle-size classes will provide precision data for sample-processing and analytical techniques.

Table 3.--Comparison of particle-size analyses on two splits of a composite storm-water sample

[Data in milligrams per liter. Triplicate analyses of the dissolved-solids concentrations were 56, 58, and 61 mg/L]

Particle-size distribution	Suspended-sediment concentration	
	Split 1	Split 2
Total (all particle sizes)	455	453
Particles finer than 0.0625-mm diameter	390	424
Particles finer than 0.0156-mm diameter	204	202
Particles finer than 0.0078-mm diameter	120	140
Particles finer than 0.0039-mm diameter	108	111

Table 4.--Estimated precision of a particle-size analysis dependent on selected dissolved-solids concentrations

[Data in milligrams per liter. Standard deviations are based on indeterminate weighing errors]

Particle-size class	Suspended-sediment concentration	Dissolved-solids concentration			
		50	100	500	2,500
		Standard deviation of sediment concentration			
Total (all particle-sizes)	500	±7	±8	±8	±24
Particles finer than 0.0625-mm diameter	450	±12	±14	±16	±26
Particles finer than 0.0312-mm diameter	350	±11	±13	±15	±26
Particles finer than 0.0156-mm diameter	225	±11	±13	±15	±25
Particles finer than 0.0078-mm diameter	125	±10	±12	±14	±24
Particles finer than 0.0039-mm diameter	100	±10	±12	±14	±24

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PRELIMINARY DATA

EXAMPLES OF DATA RELATING CHEMICAL CONCENTRATIONS TO SEDIMENT PARTICLE-SIZE CLASSES

Urban storm-water samples were collected from the Fanno Creek basin in the southwestern part of Portland, Oreg. The basin has an evenly distributed, light-residential land use (McKenzie and Miller, 1976). Tables 5, 6, and 7 show the analyses of three separate composite samples that were processed in accordance with section 6.1.1A. The metal and nutrient samples were processed by one person within 6 hours after collection, and the organic sample was processed by two people within 6 hours after collection.

Table 5.--Metal analyses of composite Fanno Creek sample collected February 18, 1981, between 1415 and 1433 hours

[Data in micrograms per liter except as indicated. Particle-size class analyses of metals include dissolved constituents. Multiple values indicate replicate analyses; n.a., not applicable]

Subsample type or particle-size class	Cd	Cr	Co	Cu	Pb	Mn	Ni	Zn	Suspended sediment (mg/L)
Total recoverable (suspended plus dissolved)	<1 <1 <1	28 29 31	16 17 24	33 36 40	130 160	1,200 1,300 1,300	14 14 16 [1]20	250 260 260	1,210
Particles finer than 0.0625-mm diameter	<1	29	24	40	150	1,100	18 [2]24	270	1,090
Particles finer than 0.0312-mm diameter	<1	30	24	32	130	900	8 [2]20	200	802
Particles finer than 0.0156-mm diameter	<1	27	24	28	100	620	8 [2]16	150	466
Particles finer than 0.0078-mm diameter	<1	20	16	28	72	320	20	100	298
Particles finer than 0.0039-mm diameter	<1	22	16	20	68	290	12 [2]12	110	238
Dissolved	<1	1	<1	5	<1	<1	3	20	n.a.

[1] Rerun value of the sample reported as 16 mg/L in this table.
[2] Rerun value of concentration reported in this table.

Table 6.--Nutrient analyses of composite Fanno Creek sample collected December 2, 1980, between 0750 and 0820 hours

[Data in milligrams per liter. Particle-size class analyses of nutrients include dissolved constituents. Multiple values indicate replicate analyses; n.a., not applicable]

Subsample type or particle-size class	Phosphorus (as P)	Orthophosphate (as P)	Kjeldahl nitrogen (as N)	Nitrite + nitrate (as N)	Ammonia (as N)	Suspended sediment
Total (suspended plus dissolved)	0.69	0.10	1.33	0.52	0.09	453
	.71	.11	1.35	.52	.09	455
	.72	.14	1.56	.52	.09	
Particles finer than 0.0625-mm diameter	.56	.09	1.38	.52	.07	390
	.60	.13	1.84	.52	.07	424
Particles finer than 0.0156-mm diameter	.40	.10	1.08	.51	.06	202
	.56	.12	1.35	.51	.08	204
Particles finer than 0.0078-mm diameter	.34	.10	.74	.52	.06	120
	.44	.10	.99	.52	.07	140
Particles finer than 0.0039-mm diameter	.35	.10	.85	.52	.03	108
	.36	.11	.89	.52	.07	111
Dissolved	.14	.06	.45	.51	.06	n.a.
	.38	.06	.50	.51	.08	n.a.
	.44	.07	.55	.52	.08	n.a.

Table 7.--CBOD ultimate (CBODu) and organic carbon analyses of a Fanno Creek sample collected February 18, 1981, between 1438 and 1528 hours

[Data in milligrams per liter. CBODu analyses include dissolved constituents. Multiple values indicate replicate analyses; n.a., not applicable]

Subsample type or particle-size class	$CBOD_u$	Suspended organic carbon [1] (SOC)	Suspended sediment
All particle sizes (Total CBODu, suspended SOC, and suspended sediment)	9.1 10.4	8.6 8.8 8.8	832
Particles finer than 0.0625-mm diameter	8.8	7.9	701
Particles finer than 0.0312-mm diameter	7.8	6.5 8.2	584
Particles finer than 0.0156-mm diameter	7.1	6.6 7.2	391
Particles finer than 0.0078-mm diameter	5.6	5.4 5.6	262
Particles finer than 0.0039-mm diameter	4.9	3.5 3.6	222
Dissolved	[2] 3.4	[3] 4.9 [3] 4.9 [3] 5.4	n.a. n.a. n.a.

[1] Analyses of sediments excluding dissolved organic carbon.
[2] Estimated value.
[3] Dissolved organic carbon.

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