

# Appendix B

## Methods for Laboratory Analysis of Sediments

---

### Methods for Sediment Physical Analyses (Moisture Content, Bulk Density, and Organic Content)

**Moisture Content Method:** Gravimetric - weight loss on drying at 103-105 °C

**Bulk Density Method:** Gravimetric - determination of ratio of sediment mass to known volume

**Reference:** S. E. Allen et al. (1974). *Chemical analysis of ecological materials*. Wiley, New York, 21–22.

#### Equipment:

- a. Analytical balance
- b. Air-circulation oven
- c. Disposable aluminum weighing dishes
- d. Desiccator

#### Moisture Content/Bulk Density Procedure:

- a. Weigh to nearest 0.001 g (1 mg) disposable aluminum weighing dish of known volume, record weight on analysis data form, and label dish

vis-a-vis sample. Prepare three replicates of each sediment sample (when sufficient amounts of sediment are available).

- b.* Homogenize fresh sediment and completely fill weighing dish. Exercise care to exclude all air from sediment by making small additions of sediment while agitating the weighing dish. This can be accomplished by rapping weighing dish on countertop. Note: Be careful not to cause a change in the volume of the dish by denting or bending it.
- c.* Screenshot excess sediment from weighing dish to ensure that sediment equals known volume of dish.
- d.* Weigh sediment and dish to nearest 0.001 g, record weight on analysis data form.
- e.* Place sediment/dish in the air-circulation oven, set temperature to 105 °C, and dry for at least 24 hours. Note: If oven is being used for drying other type samples, e.g., plant tissues, and temperature setting is 80 °C, dry sediment for at least 24 hours at 80 °C, then transfer samples to muffle furnace set at 105 °C and dry for an additional 24 hours.
- f.* Remove from oven and place in desiccator, allow to cool to ambient temperature (constant weight is achieved when successive weighings do not differ more than 1 or 2 mg).
- g.* Weigh and record weight on analysis data form.

### Calculations:

$$\text{Moisture content (\%)} = [\text{sediment weight loss (g)} \times 100] / \text{sediment wet weight (g)} \quad (\text{B1})$$

where

- a.* Sediment wet weight = (weight of fresh sediment + weight of dish) - weight of dish
- b.* Sediment dry weight = (weight of sediment after drying + weight of dish) - weight of dish
- c.* Loss in weight on drying = sediment wet weight - sediment dry weight

$$\text{Bulk density} = \text{sediment dry weight (g)} / \text{volume of aluminum dish} \quad (\text{B2})$$

**Loss-On-Ignition Methods:** Loss of sediment mass on combustion at 550 °C

### References:

- a. Modification of S. E. Allen et al. (1974). *Chemical analysis of ecological materials*. Wiley, New York, 22–23.
- b. Modification of R. H. Plumb, Jr. (1981). “Procedures for handling and chemical analysis of sediment and water samples,” Technical Report EPA/CE 81-1, U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS, 3–59, 60.

### Equipment:

- a. Analytical balance
- b. Muffle furnace for operation at 550 °C
- c. Porcelain crucibles
- d. Soil grinding mill
- e. Desiccator

### Loss-on-Ignition Procedure:

- a. Combust empty porcelain crucibles in muffle furnace at 550 °C for 1 hour.
- b. Place crucibles, after partial cooling, into desiccator until constant weight is achieved.
- c. Weigh to nearest 0.001 g and record.
- d. Grind sediment used in moisture content analysis to a fine powder using either mortar and pestle or soil mill.
- e. Weigh about 1 g of ground sediment to nearest 0.001 g (1 mg) in each porcelain crucible and record total weight (weight of sediment + crucible).
- f. Place crucibles containing sediment (uncovered) into muffle furnace.  
NOTE: DO NOT PREHEAT MUFFLE FURNACE.
- g. Set temperature to 550 °C, power up furnace, and allow temperature to rise slowly while combusting samples for 24 hours.
- h. CAREFULLY remove crucibles from furnace (DO NOT ALLOW ASH TO BE BLOWN FROM CRUCIBLES DURING HANDLING) and place in desiccator after partial cooling.

- i. Allow crucibles to cool to constant weight, weigh, and record.

### Calculations:

$$\text{Loss-on-Ignition (\%)} = (\text{sediment weight loss (g)} \times 100) / \text{sediment dry weight (g)} \quad (\text{B3})$$

where

- a. Sediment weight loss = sediment dry weight (prior to combustion) - sediment dry weight (following combustion)
- b. Sediment dry weight = (ground sediment weight = crucible weight) - crucible weight

## Particle Size Distribution Analysis (Sand, Silt, and Clay)

### Method: Hydrometer

### References:

- a. William H. Patrick, Jr. (1958). "Modification of method of particle size analysis." *Proceedings - Soil Science Society of America*. 22:366-367.
- b. Paul R. Day. (1956). "Report of the Committee on Physical Analyses (1954-1955)." *Proceedings - Soil Science Society of America*. 20:167-169.

### Equipment and Materials:

- a. ASTM D152H standard hydrometer with Bouyoucos scale in grams per liter
- b. Glass sedimentation cylinders with sufficient diameter so that the 1000-mL mark is  $36 \pm 2$  cm from the bottom on the inside
- c. Rubber stoppers to cover sedimentation cylinders
- d. Thermometers, -20 to 50 °C, immersion type
- e. Electrically driven mixer
- f. Timer with cumulative seconds counter

**Reagents:**

- a.* Calgon dispersing agent (10 percent solution): Add about 500 mL distilled water to a 1000-mL beaker and place on stirrer-hot plate. Heat on low until warm, add 100 grams of sodium metaphosphate, and stir to dissolve completely. Remove from heat, allow to cool, then while stirring adjust the pH of the solution to 8.3 with the addition of sodium carbonate solid. Transfer to 1-L volumetric flask and add distilled water to the mark.
- b.* Amyl alcohol (syn. 3-methyl 2-butanol)

**Preparation of Blank Solution/s:**

- a.* Add 50 mL of 10 percent "Calgon" solution to a sedimentation cylinder and make up to 1000-mL mark with distilled water.
- b.* Stopper and invert several times to mix.
- c.* Allow to stand overnight to reach ambient temperature (between 20-30 °C).

**Preparation of Sample Suspension/s:**

- a.* Weigh out 40.0 grams dry weight equivalent of fresh wet sediment. (Dry weight equivalent can be calculated following the determination of sample percent moisture content (Equation B1).) Record on data sheet in "Wt of Sediment Used (g)."
- b.* Place sample in a 250-mL beaker and add 50 mL of 10 percent "Calgon" dispersing solution. Mix gently with glass rod and let stand for at least 10 minutes.
- c.* Transfer to dispersing cup (blender/mixer cup). Sparingly use distilled water from a wash bottle to remove all sediment from beaker.
- d.* Mix for exactly 5 minutes in a Waring Lab Mixer.
- e.* Transfer to labeled sedimentation cylinder using distilled water from a wash bottle to remove all sediment from mixer cup.
- f.* Make up to 1000-mL mark with distilled water, stopper, and allow to stand overnight to reach ambient temperature.
- g.* Put a piece of tape on the counter by each cylinder for recording the times associated with the analysis on the following day.

- h.* Let sit overnight to ensure that the solutions in each cylinder are the same temperature.

### **Calibration of Hydrometer/s (Blank Solution/s):**

- a.* Remove stoppers from the sedimentation cylinder and measure temperature of the blank solution.
- b.* Carefully lower the hydrometer into the blank solution and determine the scale reading  $R_L$  at the upper edge of the meniscus surrounding the stem. All meniscus readings should be made at the upper edge because the bottom of the meniscus will not be evident when examining those samples containing sediment. The position of the meniscus can be determined by viewing it from an angle of 10 to 20 deg above the plane of the liquid.
- c.* Record on data sheet in “Morning Blank Reading” column.

### **Determination of 50 $\mu$ Separation:**

- a.* With a rubber stopper in place on sedimentation cylinder, invert cylinder 10 times to mix thoroughly. After inverting first time, shake cylinder to loosen sediment attached to the bottom.
- b.* After mixing, return cylinder to upright position on laboratory counter and immediately start timer (counting up).
- c.* If surface of suspension is covered with foam, add one (1) drop of amyl alcohol to suspension.
- d.* Carefully lower hydrometer into suspension and read the scale (at the top of the meniscus) at 35 seconds elapsed time.
- e.* Using Table B1, determine the time corresponding to the measured  $R$  value and suspension temperature (the nearest whole degree) and make final reading of hydrometer scale at the time indicated. For example:
  - (1) Suspension temperature = 24 °C
  - (2)  $R$  value at 35 seconds = 27
  - (3) Therefore cumulative time to final reading = 48 seconds
- f.* Record the final  $R$  value for the 50 $\mu$  separation on bench data sheet in “Morning Reading” column.
- g.* Replace stopper on cylinder, write down the actual time of day on the tape next to each cylinder, and do not further disturb. Any disturbance of cylinders will result in having to remix the suspension and start the process over for the 50 $\mu$  and 2 $\mu$  separations.

### Determination of 2 $\mu$ Separation:

- a.* After 7-8 hours elapsed time, remove the stopper from the blank suspension. Measure the temperature. Carefully place the hydrometer in the suspension, and read the scale at the top of the meniscus.
- b.* Using Table B2, determine the time corresponding to the measured *R* value and suspension temperature (the nearest whole degree). For example:
  - (1) Suspension temperature = 24 °C
  - (2) *R* value at about 6 hours = 19
  - (3) Therefore cumulative time to final reading = 9 hours and 20 minutes
- c.* Make final reading of hydrometer scale at the time indicated. Record final *R* value for the 2 $\mu$  separation on bench data sheet in the “Evening Reading” column.

<b>Table B1 Sedimentation Time for 50<math>\mu</math> Separation</b>											
<i>R</i>	Cumulative Time, sec, to Final Reading for Temperature, °C										
	20	21	22	23	24	25	26	27	28	29	30
56	32	31	30	30	29	28	28	27	27	26	26
55	32	32	31	30	30	29	28	28	27	27	27
54	33	32	32	31	30	30	29	29	28	28	27
53	34	33	32	32	31	30	30	29	29	28	28
52	35	34	33	32	32	31	30	30	29	29	28
51	35	34	34	33	32	32	31	30	30	29	29
50	36	35	34	34	33	32	31	31	30	30	29
49	37	36	35	34	33	33	32	31	31	30	30
48	37	37	36	35	34	33	33	32	31	31	30
47	38	37	36	36	35	34	33	33	32	32	31
46	39	38	37	36	35	35	34	33	33	32	32
45	40	39	38	37	36	35	35	34	33	33	32
44	40	39	38	38	37	36	35	35	34	33	33
43	41	40	39	38	37	37	36	35	34	34	33
42	42	41	40	39	38	37	36	36	35	34	34
41	43	42	41	40	39	38	37	36	36	35	34
40	43	42	41	40	39	39	38	37	36	35	35
39	44	43	42	41	40	39	38	38	37	36	35
38	45	44	43	42	41	40	39	38	37	37	36
37	46	44	43	42	41	40	40	39	38	37	37
36	46	45	44	43	42	41	40	39	39	38	37
35	47	46	45	44	43	42	41	40	39	38	38
34	48	47	45	44	43	42	41	41	40	39	38
33	48	47	46	45	44	43	42	41	40	40	39
32	49	48	47	46	45	44	43	42	41	40	39
31	50	49	48	46	45	44	43	42	42	41	40
30	51	49	48	47	46	45	44	43	42	41	40
29	51	50	49	48	47	46	45	44	43	42	41
28	52	51	50	49	47	46	45	44	43	42	42
27	53	52	50	49	48	47	46	45	44	43	42
26	54	52	51	50	49	48	47	46	45	44	43
25	54	53	52	51	49	48	47	46	45	44	43
24	55	54	53	51	50	49	48	47	46	45	44
23	56	55	53	52	51	50	49	47	46	45	44
22	57	55	54	53	51	50	49	48	47	46	45
21	57	56	55	53	52	51	50	49	48	47	46
20	58	57	55	54	53	52	50	49	48	47	46
19	59	58	56	55	54	52	51	50	49	48	47
18	60	58	57	56	54	53	52	51	49	48	47
17	60	59	58	56	55	54	52	51	50	49	48
16	61	60	58	57	56	54	53	52	51	50	48

**Table B2**  
**Sedimentation Time for 2 $\mu$  Separation**

R	Cumulative Time, hr:min, to Final Reading for Temperature, °C												
	20	21	22	23	24	25	26	27	28	29	30	31	32
45	6:50	6:40	6:30	6:20	6:10	6:05	5:55	5:45	5:40	5:30	5:25	5:20	5:15
44	6:55	6:45	6:35	6:25	6:20	6:10	6:00	5:55	5:45	5:40	5:30	5:25	5:20
43	7:05	6:55	6:45	6:35	6:25	6:15	6:10	6:00	5:55	5:45	5:40	5:30	5:25
42	7:15	7:00	6:50	6:40	6:35	6:25	6:15	6:05	6:00	5:50	5:45	5:40	5:30
41	7:20	7:10	7:00	6:50	6:40	6:30	6:20	6:15	6:05	6:00	5:50	5:45	5:40
40	7:30	7:20	7:05	6:55	6:45	6:40	6:30	6:20	6:15	6:05	5:55	5:50	5:45
39	7:35	7:25	7:15	7:05	6:55	6:45	6:35	6:25	6:20	6:10	6:05	5:55	5:50
38	7:45	7:35	7:20	7:10	7:00	6:50	6:45	6:35	6:25	6:20	6:10	6:05	5:55
37	7:50	7:40	7:30	7:20	7:10	7:00	6:50	6:40	6:30	6:25	6:15	6:10	6:00
36	8:00	7:50	7:40	7:25	7:15	7:05	6:55	6:50	6:40	6:30	6:25	6:15	6:10
35	8:10	7:55	7:45	7:35	7:25	7:15	7:05	6:55	6:45	6:35	6:30	6:20	6:15
34	8:15	8:05	7:55	7:40	7:30	7:20	7:10	7:00	6:50	6:45	6:35	6:25	6:20
33	8:25	8:10	8:00	7:50	7:40	7:30	7:20	7:10	7:00	6:50	6:40	6:35	6:25
32	8:30	8:20	8:10	7:55	7:45	7:35	7:25	7:15	7:05	6:55	6:50	6:40	6:30
31	8:40	8:25	8:15	8:05	7:55	7:40	7:30	7:20	7:10	7:05	6:55	6:45	6:40
30	8:45	8:35	8:25	8:10	8:00	7:50	7:40	7:30	7:20	7:10	7:00	6:50	6:45
29	8:55	8:40	8:30	8:20	8:05	7:55	7:45	7:35	7:25	7:15	7:05	7:00	6:50
28	9:05	8:50	8:40	8:25	8:15	8:05	7:50	7:40	7:30	7:20	7:15	7:05	6:55
27	9:10	9:00	8:45	8:35	8:20	8:10	8:00	7:50	7:40	7:30	7:20	7:10	7:00
26	9:20	9:05	8:55	8:40	8:30	8:15	8:05	7:55	7:45	7:35	7:25	7:15	7:10
25	9:25	9:15	9:00	8:50	8:35	8:25	8:15	8:00	7:50	7:40	7:30	7:20	7:15
24	9:35	9:20	9:10	8:55	8:45	8:30	8:20	8:10	8:00	7:50	7:40	7:30	7:20
23	9:40	9:30	9:15	9:00	8:50	8:40	8:25	8:15	8:05	7:55	7:45	7:35	7:25
22	9:50	9:35	9:20	9:10	8:55	8:45	8:35	8:20	8:10	8:00	7:50	7:40	7:30
21	9:55	9:45	9:30	9:15	9:05	8:50	8:40	8:30	8:20	8:05	7:55	7:45	7:35
20	10:05	9:50	9:35	9:25	9:10	9:00	8:45	8:35	8:25	8:15	8:05	7:55	7:45
19	10:10	10:00	9:45	9:30	9:20	9:05	8:55	8:40	8:30	8:20	8:10	8:00	7:50
18	10:20	10:05	9:50	9:40	9:25	9:15	9:00	8:50	8:35	8:25	8:15	8:05	7:55
17	10:30	10:15	10:00	9:45	9:35	9:20	9:05	8:55	8:45	8:30	8:20	8:10	8:00
16	10:35	10:20	10:05	9:55	9:40	9:25	9:15	9:00	8:50	8:40	8:30	8:15	8:05
15	10:45	10:30	10:15	10:00	9:45	9:35	9:20	9:10	8:55	8:45	8:35	8:25	8:15
14	10:50	10:35	10:20	10:10	9:55	9:40	9:30	9:15	9:05	8:50	8:40	8:30	8:20
13	11:00	10:45	10:30	10:15	10:00	9:50	9:35	9:20	9:10	9:00	8:45	8:35	8:25
12	11:05	10:50	10:35	10:20	10:10	9:55	9:40	9:30	9:15	9:05	8:50	8:40	8:30
11	11:15	11:00	10:45	10:30	10:15	10:00	9:50	9:35	9:20	9:10	9:00	8:45	8:35
10	11:20	11:05	10:50	10:35	10:20	10:10	9:55	9:40	9:30	9:15	9:05	8:55	8:40
9	11:30	11:15	11:00	10:45	10:30	10:15	10:00	9:50	9:35	9:25	9:10	9:00	8:50
8	11:35	11:20	11:05	10:50	10:35	10:20	10:10	9:55	9:40	9:30	9:15	9:05	8:55
7	11:45	11:30	11:15	11:00	10:45	10:30	10:15	10:00	9:50	9:35	9:25	9:10	9:00
6	11:50	11:35	11:20	11:05	10:50	10:35	10:20	10:10	9:55	9:40	9:30	9:15	9:05