UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 4
Science and Ecosystem Support Division
Ecological Support Branch

Standard Operating Procedure (SOP) for the Determination of Particle Size Class Distribution - Wet Sieve

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Approvals

_____ — Sediment Laboratory Administrator — _____ — Date

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- For Documentation of Approval for Use in SESD, EAB

Procedure Section

1.0 Scope and Application

Substrate physical properties are among the most important factors in characterizing habitat suitability for aquatic macroinvertebrates and fish (Howard 1969, USEPA 1973, and Waters 1995). A direct relationship between habitat and biological diversity has been well established (USEPA 1978, Raven et al. 1998, and Pruitt and Howard 2000). The Ecological Assessment Branch (EAB) has developed and used the linkage between substrate physical properties, habitat, and biological diversity in both freshwater and marine ecosystems for over twenty-five years (Hicks et al. 1975, USEPA 1982, and Pruitt et al. 2001). Presently, EAB routinely characterizes substrate physical properties including embeddedness and sediment deposition as an integral part of the rapid bioassessment protocol (RBP, Barbour 1999) in riverine settings.

2.0 Summary of Method

Particle size, which is equivalent to grain size in marine physical science, can be measured in a variety of ways including the diameter, volume, and/or the width of the intermediate surface ("B" dimension) of the particle (Davis 1992 and NRCS 1996). In general, natural sediment particles are three-sided: 1) the "A" dimension is the long axis; 2) the "B" dimension is the intermediate axis; and 3) the "C" dimension is the short axis. In theory, the "B" axis of a three-dimensional particle is the maximum surface width which can pass through a square opening of a specific sieve size. Hence, it is assumed during wet sieving that the sediment particles are oriented properly while the particles are washed in combination with swirling. The particle size scale is based on a modified Wentworth (1922) scale (Table 1.1). The scale is based on a factor of 2 (either multiplier or divisor of 2 from unity). Including the clay fraction, the Wentworth scale segregates particle sizes into 22 classes. Of these, EAB uses eight particle size classes: medium and fine gravel; coarse, medium, fine, and very fine sand; silt; and clay. EAB developed and refined a wet sieve method capable of determining the particle size class distribution (PSD) of wet sediment samples collected from streams, lakes, estuaries, and marine ecosystems. This method entails maintaining the aqueous condition of the sediment sample prior and during sieving. Consequently, the sediment sample is not air dried before sieving.

3.0 Interferences

Not applicable.

4.0 Health and Safety Procedures

Laboratory technicians should use precautions when working with convection ovens and muffle furnaces which operate at extremely high temperatures. The following safety equipment is recommended when removing samples from the convection oven and muffle

furnace: faceshield, gloves, laboratory coats/aprons, and/or long tongs. A dispersing agent (sodium metaphosphate or equivalent) may be used in this procedure. Sodium metaphosphate has no health and safety requirements.

5.0 Special Procedures

Not applicable.

6.0 Analyst Training

Technicians/soil scientists are trained by experienced EPA personnel until proficient.

7.0 Reagents and Standards

Sodium metaphosphate is used as a dispersing agent.

8.0 Apparatus and Materials

8.1 10-liter polyethylene bucket (Nalgene™ or equivalent).
8.2 Six brass or stainless steel sieves which meet current ASTM E11 and ISO 565/3310-1 standards- W.S. Tyler™ certified or equivalent (See Table X.1 for sizes).
8.3 Polyethylene drop-dispensing bottles (Nalgene™ or equivalent).
8.4 Porcelain crucibles (appropriate size based on sample volume) (Coors™ or equivalent).
8.5 Stainless steel utility trays.
8.6 Forced-air convection oven (Fisher™ Isotemp or equivalent).
8.7 Four-place balance (0.1 mg accuracy).
8.8 Desiccator cabins.
8.9 Silica desiccant.
8.10 Gyrotory shaker.
8.11 Centrifuge.
8.12 Graduated cylinder.
8.13 100 cc plastic syringe.
8.14 Dispersing agent (sodium metaphosphate or equivalent).

9.0 Sample Collection and Preservation

This SOP covers sample control and handling once the sample is received by the Sediment Characterization Laboratory (SCL) of EAB. Sediment sample collection and transportation to the laboratory are covered under Sections 7 and 8 (Sediment Sampling and Fluvial Sediment Sampling, respectively). Once received by the SCL, sediment samples are maintained at 0°C in a freezer or at 4°C in a refrigerator at the discretion of the project leader or the anticipated process time.

10.0 Sample Holding Time

No maximum holding time is required for frozen samples.

11.0 Method Calibration

Not applicable

12.0 Sample Analysis and Procedure

12.1 Sample Preparation

Samples are removed from the freezer or refrigerator, and adequate time is allowed for thawing. At the discretion of the project leader, a dispersing agent (e.g., sodium metaphosphate) is added to the sample, and the sample is dispersed for approximately 12 to 24 hours on a gyrotory shaker. Once the sample is adequately dispersed, either the sample is processed by the wet sieve method, laser analyzer, or a combination of the two methods depending on the study objectives. See SOP on *Determination of Particle Size Analysis using the Coulter LS 200* for laser methodology.

12.2 Sieve Method

The following is a general step-wise procedure on the wet sieve procedure. Modifications to the procedure should be requested through the SCL Administrator prior to sample receipt.

12.2.1 Place the sieve with the largest opening size (Table 1.1) in a 10-liter polyethylene bucket, wash the sample, using a drop-dispensing bottle, through the sieve while swirling gently. Use as little deionized water (DI) as possible, and capture the DI and sediment suspension in a 10-liter polyethylene bucket. Once an adequate amount of DI has collected in the bucket, partially submerge the sieve in the DI and swirl gently;

12.2.2 Repeat step 1 with each of five remaining sieves in consecutive order of decreasing opening sizes by decantering the DI and sediment suspension into each sieve. Retain the final DI and sediment suspension for separation of the silt and clay fractions;

12.2.3 Decanter the sediment collected in each of the six sieves into separate pre-weighed porcelain crucibles. Record the clean crucible weights (CCW);

12.2.4 Place the crucibles on stainless steel trays and evaporate the samples at 105°C ±5°C in a convection oven for approximately 24 hours;

12.2.5 Once the samples have dried adequately, remove the samples from the oven and place in a desiccant oven to allow for equilibration to room temperature (20 to 22°C) and moisture absorption; and

12.2.6 Weigh the samples on a four-place balance (0.1 mg accuracy) or at a pre-

specified accuracy required by the project leader. Record the crucible weight plus the dry weight sample (CCW+DWS).

12.3 Silt/Clay Separation (Centrifugation)

Silt and clay fractions are separated using centrifugation. Following centrifugation, it is assumed that the silt fraction is the precipitate and the clay fraction is held in suspension in the supernatant by Browning Movement. Process the DI and silt/clay mixture (hereafter, referred as silt/clay suspension) as follows:

12.3.1 Swirl the silt/clay suspension retained from step 12.2.2 above to a ensure homogenous mixture;
12.3.2 Decanter approximate equal volumes of the silt/clay suspension into appropriate centrifugation bottles;
12.3.3 Centrifuge for approximately five minutes at approximately xx RPMs (i.e., 22 on the speed scale);
12.3.4 Carefully decanter the supernatant (clay fraction) into a appropriately-sized graduated cylinder and record the volume to the nearest five ml;
12.3.5 If the clay suspension exceeds 300 ml, homogenize the sample thoroughly in a bucket and draw-off (by syringe) three representative, 100 ml aliquots and decanter into three pre-weighed porcelain crucibles and place on a stainless steel tray;
12.3.6 Initially, evaporate the clay suspensions at approximately 95°C;
12.3.7 Follow steps 12.2.4 to 12.2.6 above;
12.3.8 Re-suspend the precipitate (silt fraction in the centrifugation bottle) with DI water using a drop-dispensing bottle;
12.3.9 Initially, evaporate the silt suspensions at approximately 95°C; and
12.3.10 Follow steps 12.2.4 to 12.2.4 above.

12.4 Percent Volatile Procedure

Two methods are available to determine percent volatiles: 1) loss by ignition using a muffle furnace; or 2) loss by ignition using a total gravimetric analyzer (Leco™ Model TGA-601 or equivalent). In addition, an elemental analyzer (Leco™ Model CNS-2000) can be used to determine by infrared detection concentrations of total carbon, nitrogen, and sulfur. An appropriate method should be selected based on the project objectives and the required level of detection. See SOPs on *Determination of Volatile Residues using the Leco TGA-601* and *Determination of Total Carbon, Sulfur, and Nitrogen using the Leco CNS-2000* for volatile residues and elemental analysis, respectively. This SOP includes methods specific to loss by ignition using a muffle furnace as follows:

12.4.1 Ignite the eight fractions in a muffle furnace at 500°C for one hour;
12.4.2 Carefully, remove the samples from the oven and place in a desiccant oven to allow for equilibration to room temperature (20 to 22°C) and moisture

absorption; and

12.4.3 Weigh the samples on a four-place balance (0.1 mg accuracy) or at a pre-specified accuracy required by the project leader. Record the crucible weight plus the ashed-weight sample (CCW+AWS).

13.0 Quality Control

At the discretion of the project leader, quality control will be maintained with duplicate samples. In addition, sediment samples of known particle size distribution and weight may be utilized to determine loss of material during sieving.

14.0 Data Analysis and Calculation

The dry weight sample of each fraction is the difference between the clean crucible weight plus the dry weight sample and the clean crucible weight as follows:

$$DWS = (CCW+DWS) - CCW \quad (1)$$

where:

CCW = clean crucible weight
DWS = dry weight sample

The organic ash-free dry weight of each fraction is the difference between the CCW plus the dry weight sample and the CCW plus ashed-weight sample as follows:

$$AFDW = (CCW+DWS) - (CCW+AWS) \quad (2)$$

where:

AFDW = ash-free dry weight
AWS = ashed weight sample

The inorganic ashed weight sample is the difference between the CCW plus AWS and the CCW as follows:

$$AWS = (CCW+AWS) - CCW \quad (3)$$

15.0 Pollution Prevention

See *SESD Safety, Health and Environmental Management Program (SHEM) Procedures and Policy Manual*, Section 5.8.

16.0 Waste Management

Waste management and disposal procedures are described in the *SESD Safety, Health and Environmental Management Program (SHEM) Procedures and Policy Manual*, Section 2.5.

Table 1.1. Modified Wentworth (1922) scale with EAB sieve sizes.

Limiting Particle Diameter			Class Terminology		EAB Sieve Sizes	
Particle Size (mm)	ϕ Units	μm	Size Category	Size Class	SFSC U.S. #	Size (mm)
2048	-11		Very large	Boulders		
1024	-10		Large			
512	-9		Medium			
256	-8		Small			
128	-7		Large	Cobbles		
64	-6		Small			
32	-5		Very coarse	Gravel		
16	-4		Coarse			
8	-3		Medium		8	8
4	-2		Fine		10	4
2	-1		Very fine			
1	0		Very coarse	Sand		
½	+1	500	Coarse		35	0.500
¼	+2	250	Medium		60	0.250
1/8	+3	125	Fine		120	0.125
1/16	+4	62	Very fine sand		230	0.0625
1/32	+5	31	Very coarse	Silt		0.0625 < Silt < 0.002
1/64	+6	16	Coarse			
1/128	+7	8	Medium			
1/256	+8	4	Fine			
1/512	+9	2	Very fine			

		<2		Clay		<0.002

17.0 References

Barbour, M.T., J. Gerritsen, B.D. Snyder, and J.B. Stribling. 1999. *Rapid Bioassessment Protocols for Use in Streams and Wadeable Rivers: Periphyton, Benthic Macroinvertebrates and Fish,* Second Edition. EPA 841-B-99-002. U.S. Environmental Protection Agency, Office of Water, Washington, D.C. USA.

Davis, R.A., Jr. 1992. *Depositional Systems: An Introduction to Sedimentology and Stratigraphy*. Prentice Hall, Englewood Cliffs, New Jersey, USA.

Hicks, D.B., T.R. Cavinder, B.J. Carroll, R.L. Raschke, and P.M. Murphy. 1975. *Finger-fill canal studies*, Florida and North Carolina. U.S. Environmental Protection Agency, Surveillance and Analysis Division, Athens, Georgia, USA.

Howard, H.S. 1969. *Factors affecting the distribution of aquatic insects in Fowler's Mill Creek. Masters Thesis*. The University of Georgia, Athens, Georgia, USA.

Leopold, L.B. M.G. Wolman, and J.P. Miller. 1964. *Fluvial processes in geomorphology*. W.H. Freeman and Company, San Francisco, California. 522 p., USA.

NRCS. 1996. *Soil Survey Laboratory Methods Manual*. Soil Survey Investigations Report No. 42, Version 3.0. U.S. Department of Agriculture, Natural Resources Conservation Service, National Soil Survey Center, Washington, D.C., USA.

Pruitt, B.A. and H.S. Howard. 2000. *South Forth Broad River TMDL project: aquatic ecological survey*. U.S. Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Ecological Assessment Branch, Athens, Georgia, USA.

Pruitt, B.A., D.L. Melgaard, H. Howard, M.C. Flexner, and A.S. Able. 2001. *Chattooga River Watershed Ecological/Sedimentation Project*. In proceeding of 7th Federal Interagency Sedimentation Conference, Reno, Nevada, March 26-30, 2001.

Raven, P.J., N.T.H. Holmes, F.H. Dawson, P.J.A. Fox, M.Everard, I.R. Fozzard, and K.J. Rowen. 1998. *River Habitat Quality: The physical character of rivers and streams in the UK and Isle of Man.* Environment Agency. ISBN1 873760 42 9. Bristol, England.

USEPA. 1973. *Biological field and Laboratory methods for measuring the quality of surface waters and effluents*. Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, USA.

USEPA. 1978. *Water quality and pollution source monitoring: field and laboratory procedures*. U.S. Environmental Protection Agency, National Training and Operational Technology Center, Cincinnati, Ohio, USA. EPA-430/1-78-015.

USEPA. 1982. Hydrographic, water quality and biological studies of freshwater canal systems, South Carolina, Mississippi, and Florida. U.S. Environmental Protection Agency, Environmental Services Division, Athens, Georgia, USA.

Waters, T.F. 1995. Sediment in Streams: Sources, Biological Effects, and Control. *American Fisheries Society Monograph 7.* American Fisheries Society, Maryland, USA.

Wentworth, C.K. 1922. A scale of grade and class terms for clastic sediments. *Journal of Geology*, 30:377-392., USA.

Science and Ecosystem Support Division, Region 4, U.S. Environmental Protection Agency. September, 2000. *Ecological Assessment Branch Laboratory Operations and Quality Assurance Manual.*

Science and Ecosystem Support Division, Region 4, U.S. Environmental Protection Agency. May, 1998. *Safety, Health and Environmental Management Program Procedures and Policy Manual.*