

APPENDIX 9 – Laboratory Methods Stormwater Discharge Monitoring

Stormwater discharge monitoring is intended to characterize stormwater runoff quantity and quality at a limited number of locations in a manner that allows analysis of loadings and changes in conditions over time and generalization across the Permittees' jurisdiction.

QAPP Preparation

Permittees shall prepare a Quality Assurance Project Plan (QAPP) in accordance with *Quality Assurance Project Plan Guidance, Special Condition S8.D, Phase I Municipal Stormwater Permit*, December 2010 (Ecology Publication no. 10-10-075 <http://www.ecy.wa.gov/pubs/1010075.pdf>). The QAPP shall be developed by qualified staff or contractors with experience in applying Ecology's or EPA's QAPP Guidelines. The QAPP shall describe each stormwater discharge monitoring site and associated drainage basin in detail. The QAPP shall also describe why and how each site was selected.

Stormwater discharge monitoring QAPPs shall be submitted to Ecology in accordance with the deadlines in S8.

Permittees are responsible for maintaining an up-to-date approved QAPP for stormwater discharge monitoring. Significant changes should be reviewed by Ecology and reflected in a revised QAPP. Significant changes can include, but are not limited to:

- Land disturbing activities over 10 acres in size within the sampled drainage area.
- Relocating a monitoring station.
- Introducing new sampling equipment.
- Unanticipated back water conditions, base flow or tidal influences.
- Changes in laboratories, analytical methods or reporting limits.

Site Selection

Stormwater monitoring sites shall have the tributary conveyance system and drainage area mapped, and be suitable for permanent installation and operation of flow-weighted composite sampling equipment. Additional site selection guidance, and information about how to estimate a rainfall to runoff relationship is available in *Standard Operating Procedure for Automatic Sampling for Stormwater Monitoring, ECY002* (<http://www.ecy.wa.gov/programs/wq/stormwater/municipal/SOPAutomatedSampling.pdf>).

Permittees may identify a sampling site upstream in the conveyance system (*i.e.*, upgradient of the outfall) in order to achieve the desired land use, to accommodate the installation of sampling equipment, and/or to avoid or minimize back water or tidal interference.

The QAPP must describe how each site was selected, the size of the drainage basin, the percentage of area in the drainage basin representing the following land uses: high density residential, low density residential, commercial, industrial, agriculture, and transportation right-of-way.

1 Sites must be evaluated for a rainfall to runoff relationship in order to ensure that the site will
2 receive enough runoff for sufficient sample volume. This rainfall to runoff relationship will also
3 assist in programming the automatic sampling equipment. In order to establish the rainfall to
4 runoff relationship, one year of continuous flow recording (including base flow and all storm
5 events) is necessary.

6 **Monitoring Frequency**

7 Permittees shall sample each stormwater discharge monitoring site according to the frequency
8 described below. Documented good faith efforts with good professional practice by the
9 Permittee which do not result in collecting a successful sample for the full number of required
10 storms may be considered as contributing toward compliance with this requirement.

11
12 The Permittee shall sample and analyze eleven (11) qualifying storm events per water year.
13 Qualifying storm event sampling must be distributed throughout the year, approximately
14 reflecting the distribution of rainfall between the wet and dry seasons (with a goal of 60-80% of
15 the samples collected during the wet season and a goal of 20-40% of the samples collected in the
16 dry season).

17
18 Additionally, the Permittee shall analyze up to a maximum of three (3) samples that are collected
19 as a result of attempts to sample the eleven (11) required storm events and do not meet the
20 rainfall volume storm event criterion but do meet the other storm event and sample criteria. The
21 maximum number of sampled storm events to be analyzed is fourteen (14) per year.

22 **Qualifying Storm Event Criteria**

23 The wet season is from October 1 through April 30. A qualifying wet season storm event is
24 defined as follows:

- 25 • Rainfall volume: 0.20” minimum, no fixed maximum
- 26 • Rainfall duration: No fixed minimum or maximum
- 27 • Antecedent dry period: Less than or equal to 0.02” rain in the previous 24 hours
- 28 • Inter-event dry period: 6 hours

29 The dry season is from May 1 through September 30. A qualifying dry season storm event is
30 defined as follows:

- 31 • Rainfall volume: 0.20” minimum, no fixed maximum
- 32 • Rainfall duration: No fixed minimum or maximum
- 33 • Antecedent dry period: less than or equal to 0.02” rain in the previous 72 hours
- 34 • Inter-event dry period: 6 hours

35 **Types of Sampling**

36 Storm events shall be sampled using flow-weighted composite sampling techniques. Automatic
37 samplers shall be programmed to begin sampling as early in the runoff event as practical and to
38 continue sampling past the longest estimated time of concentration for the tributary area. Refer to
39 Standard Operating Procedure for Automatic Sampling for Stormwater Monitoring, ECY002
40 (<http://www.ecy.wa.gov/programs/wq/stormwater/municipal/SOPAutomatedSampling.pdf>) for
41 guidance on how to conduct flow weighted composite sampling.
42

1 For storm events lasting less than 24 hours, samples shall be collected for at least seventy-five
2 percent (75%) of the storm event hydrograph. For storm events lasting longer than 24 hours,
3 samples shall be collected for at least seventy-five percent 75% of the hydrograph of the first 24
4 hours of the storm.

5
6 Each composite sample must consist of at least 10 aliquots. Composite samples with 7 to 9
7 aliquots are acceptable if they meet the other sampling criteria and help achieve a representative
8 balance of wet season/dry season events and storm sizes.

9
10 Continuous flow recording of all storm events (not just sampled storm events) is necessary for at
11 least one year to establish a baseline rainfall/runoff relationship. Ongoing continuous flow
12 monitoring is necessary to properly operate the flow weighted composite sampling. Precipitation
13 data shall be collected from the nearest rain gauge reporting at least hourly rainfall amounts.
14 Grab samples are necessary for some parameters (see below) and shall be collected early in the
15 storm event. Refer to *Standard Operating Procedure for Grab Sampling for Stormwater*
16 *Monitoring, ECY001*
17 (<http://www.ecy.wa.gov/programs/wq/stormwater/municipal/SOPGrabSampling.pdf>).

18
19 Sediment samples shall be collected once per water year at each stormwater discharge
20 monitoring site, or in the vicinity of each stormwater monitoring site. Use of in-line sediment
21 traps or similar collection system is preferred; refer to *Standard Operating Procedure for*
22 *Collection of Stormwater Sediments using In-Line Sediment Traps, ECY003*
23 ([http://www.ecy.wa.gov/programs/wq/stormwater/municipal/SOPSedimentTrapStormwaterSam](http://www.ecy.wa.gov/programs/wq/stormwater/municipal/SOPSedimentTrapStormwaterSampleCollection.pdf)
24 [pleCollection.pdf](http://www.ecy.wa.gov/programs/wq/stormwater/municipal/SOPSedimentTrapStormwaterSampleCollection.pdf)). Sampling of receiving water sediment deposits is an alternative where
25 approved by Ecology.

26 **Parameters**

27 *Flow-weighted composite samples* shall be analyzed for the following parameters utilizing an
28 accredited laboratory and the methods and reporting limits as provided in this appendix.

- 29 • Conventional Parameters: TSS, turbidity, Conductivity, Chloride, Biochemical oxygen
30 demand (BOD₅), Hardness, and Methylene Blue Activating Substances (MBAS).
- 31 • Nutrients: Total phosphorus, Orthophosphate, Total Kjeldahl Nitrogen, and Nitrate plus
32 nitrite.
- 33 • Metals: total and dissolved copper, zinc, cadmium, and lead; mercury shall also be
34 sampled in commercial and industrial land use areas.
- 35 • Organics:
 - 36 ○ PAHs including: Acenaphthene, Acenaphthylene, Anthracene,
 - 37 Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(ghi)perylene,
 - 38 Benzo(k)fluoranthene, Chrysene, Dibenzo(a,h), Fluoranthene, Fluorene,
 - 39 Indeno(1,2,3-cd)pyrene, Naphthalene, Phenanthrene, and Pyrene.
 - 40 ○ Bis(2-Ethylhexyl)phthalate.

41 If the volume of stormwater sample collected from a qualifying storm is insufficient to allow
42 analysis for all parameters listed above, the sample shall be analyzed for as many parameters as
43 possible in the following priority order: 1. Metals and hardness; 2. TSS; 3. Organics; 4.
44 Nutrients; 5. Conductivity; and 6. BOD₅. If insufficient sample exists to run the next highest

1 priority pollutant, that analysis should be bypassed and analyses run on lower priority pollutants
2 in accordance with the remaining priority order to the extent possible.

3
4 Grab samples shall be analyzed for the following parameters utilizing an accredited laboratory
5 and the methods and reporting limits provided in this appendix.

- 6 • Total Petroleum Hydrocarbons (TPH) using NWTPH-Gx and NWTPH-Dx and BTEX
7 (benzene, toluene, ethyl-benzene, and xylenes). The lube oil fraction, not the diesel
8 fraction, is targeted for NWTPH-Dx.

9
10 Sediment samples shall be analyzed for the following parameters utilizing an accredited
11 laboratory and the methods and reporting limits provided in this appendix. If the volume of
12 sediment sample is insufficient to analyze for all of the parameters listed below, the sample shall
13 be analyzed for as many parameters as possible in the following priority order:

- 14 • Grain size (visual, qualitative determination only), total organic carbon, copper, zinc,
15 lead, cadmium, PAHs, percent solids.

16 A minimum of one sediment sample per year shall be collected. Parameters that are below
17 detection limits after two years of data may be dropped from the analysis.

18 **Recordkeeping and Reporting**

19 For each stormwater monitoring site, calculate the following:

- 20 • Event Mean Concentrations (EMCs)
- 21 • Total annual pollutant load by parameter
- 22 • Seasonal pollutant loads by parameter for the wet and dry seasons

23
24 The annual pollutant load calculations must be based on a water year and include wet and dry
25 season loads and total annual load (wet plus dry season load). The loadings shall be expressed as
26 total pounds and as pounds per acre, and must take into account potential pollutant load from
27 base flow. Loadings shall be calculated following *Standard Operating Procedure for*
28 *Calculating Pollutant Loads for Stormwater Discharges, ECY004*
29 ([http://www.ecy.wa.gov/programs/wq/stormwater/municipal/SOPPOLLUTANTLOADINGCALCULATIONS.](http://www.ecy.wa.gov/programs/wq/stormwater/municipal/SOPPOLLUTANTLOADINGCALCULATIONS.pdf)
30 pdf). Pollutant loading information is required for water quality parameters only.

31
32 Annual Monitoring Reports shall be submitted with each Annual Report beginning with the first
33 Annual Report following the first full water year of monitoring. Annual Monitoring Reports shall
34 provide all monitoring data collected during the preceding water year (October 1 – September
35 30). Annual Monitoring Reports shall consist of a narrative report and a submittal to Ecology's
36 Environmental Information Management (EIM) database. Guidance for EIM data submittals is
37 provided in *Stormwater Monitoring Report Guidance, Phase I Municipal Stormwater Permit,*
38 *Reporting Requirements for Special Condition S8, November 2010 (Ecology Publication No. 10-*
39 *10-028).* For the Annual Monitoring Report to be considered on time, the EIM data submission
40 process must be initiated before March 1 of each relevant year, and completed by April 30 of
41 each relevant year.

42
43 Annual Monitoring Reports shall include:

- 1 • A brief summary of each monitored drainage basin (full details of the monitoring
2 drainage basin should be in the QAPP), including any changes within the contributing
3 drainage area or changes to the monitoring station that could affect hydrology and/or
4 pollutant loading.
- 5 • A description of each flow-weighted composite and grab sampled storm event,
6 including:
 - 7 ○ General summary about storm event criteria, including:
 - 8 • Precipitation data including antecedent dry period and rainfall
9 distribution throughout the event.
 - 10 • Flow and hydrograph data including sampled and total runoff time
11 periods and volumes.
 - 12 • Total number of qualifying and non-qualifying storm events captured
13 and analyzed at each monitoring location (specify which criteria were
14 not met for each sampled non-qualifying event).
 - 15 • Whether or not 3 storm events were captured which did not reach the
16 0.20" rainfall depth criterion (how many and date of storm events).
 - 17 • Distribution of storms collected between wet and dry seasons (permit
18 goals include 60-80% of storms during the wet season and 20-40% of
19 storms during the dry season).
 - 20 • Logistical problems associated with any storm event criterion.
 - 21 ○ A hietograph and a hydrograph for each sampled storm event. Include
22 properly labeled graphs that display the following:
 - 23 • Date of the storm event,
 - 24 • Time of day versus precipitation information,
 - 25 • Time versus flow rate, and,
 - 26 • Time versus aliquot collection
 - 27 • Display the total duration of the storm event, not just the duration
28 when samples were collected (remember your pollutant load
29 calculation must include flow for the entire storm event, not just
30 the water quality sampled portion)
 - 31 ○ A summary of (or in the graph) the total runoff volume in gallons/other
32 appropriate unit of measure.
 - 33 ○ A rainfall/runoff relationship table used to estimate the un-sampled storm
34 events (where water quality samples were not collected). This is used for
35 future estimations of annual and seasonal loads.
 - 36 ○ Whether or not any chemicals were removed from the list of analysis due to
37 two years of non-detect data.
 - 38 ○ A brief summary with storm event dates where insufficient volumes were
39 collected. Include the parameters analyzed.
- 40 • A description of the sediment sampling event, including:
 - 41 ○ Whether or not any chemicals were removed from the list of analysis due to
42 two years of non-detect data.

1 o A summary of sediment sampling (including dates) where insufficient
2 volumes were collected. Include the parameters analyzed.

3 • Event Mean Concentrations (EMCs)

4 • The wet and dry season pollutant loads and annual pollutant load based on water year
5 for each site expressed in total pounds, and pounds/acre. Include the following:

6 o For storm events where water quality samples were collected, the load for
7 each parameter for each sampled storm event, include date of storm events.

8 o An estimated seasonal pollutant load for each parameter at each site. This is
9 calculated using all storm events (where water quality samples were collected
10 and were not collected).

11 o A total annual pollutant load (wet season load + dry season load) for each
12 parameter (include estimated events).

13 o The rainfall/runoff relationship including your pollutant load estimates for un-
14 sampled events.

15 o Note that if any data is unavailable to effectively estimate your rainfall to
16 runoff relationship due to an incomplete water year, submit this information in
17 the next year's stormwater monitoring report.

18 • Quality Assurance/Quality Control information for each *sampled storm event* at each
19 site and *sediments* sampled at each site, including:

20 o A narrative summary of your field and laboratory verification, validation results
21 and quality control checks performed.

22 o A narrative analysis of your field and laboratory quality control sample results
23 and how they compare with your data quality objectives/indicators in your QAPP.

24 o Corrective actions reported/taken.

25
26 • An explanation and discussion of results from each *sampled storm event* at each
27 monitoring site and *sediments* collected at each site, including:

28 o A narrative analysis of the event mean concentrations for each parameter.

29 o Any conclusions based on trend data that may result from this study or from
30 previously collected data from these sites.

31 o A description of Stormwater Management Program activities currently taking
32 place or planned within the monitoring station's drainage area that may have
33 affected or may potentially affect future monitoring results.

34
35 If the Permittee monitors any pollutant more frequently at the stormwater discharge monitoring
36 sites, then the results of this monitoring shall be included in the annual monitoring report
37 reflecting the water year in which the monitoring occurred.

38 After 3 water years of data, the Annual Monitoring Report shall include an evaluation of the data
39 as it applies to the SWMP, and shall identify any stormwater management activities that can be
40 adjusted to respond to this data.

Laboratory Methods

The Permittee’s stormwater discharge monitoring program shall use the following analytical methods unless alternative methods are approved by Ecology. ~~in the Permittees’ QAPP the following analytical methods shall be used by Permittees when analyzing stormwater as required by section S8—Monitoring of this permit.~~ Any alternative method proposed by the Permittee must have a similar reporting limit, or must be justified as adequate for the likely range of concentrations. Permittees are not guaranteed approval of their alternative methods or reporting limits.

In cases where smaller volumes of water are expected to be collected, or to save analytical costs, Permittees may propose that some of the analyses be optimized for specific parameters or groups. The Permittee must, in consultation with a qualified chemist, define the exact volumes and optimization steps and include them in the QAPP.

Table 9-1 Analytical Procedures in Stormwater

Analyte (or Surrogate)	Method in Water	Reporting Limit Target ^a
Conventional Parameters		
Total suspended solids	SM 2540B ^b or SM 2540D	1.0 mg/L
Turbidity	EPA Method 180.1 or SM2130B	± 0.2 NTU
Conductivity	SM 2510 or EPA Method 120.1	± 1 umhos/cm
Chloride	EPA Method 300.0, EPA Method 325.2, or SM4110B	0.2 mg/L
BOD ₅	SM5210B	2.0 mg/L
Particle Size Distribution	Coulter Counter, Laser diffraction, or comparable method - <i>see attached method</i>	NA
Grain Size	Ecology method sieve and pipette (ASTM 1997), PSEP 1986/2003, or comparable method	NA
pH	EPA Method 150.2 or SM 4500H ⁺	0.2 units
Hardness as CaCO ₃	EPA Method 200.7, SM2340B(ICP), SM2340C (titration) or SM 3120B	1.0 mg/L
Methylene Blue Activated Substances (MBAS)	SM 2340B (ICP) or 2340C (Titration)CHEMetrics Colorimetric or SM5540C	0.025 mg/L
Bacteria		
Fecal Coliform	SM 9221E	2 min., 2E6 max.
Nutrients		
Total phosphorus	EPA Method 365.3, EPA Method 365.4, SM 4500-P E or SM4500-P F	0.01 mg P/L
Orthophosphate	EPA Method 365.3, EPA Method 365.1, SM 4500-P E or SM4500-P F	0.01 mg P/L
Total kjeldahl nitrogen	EPA Method 351.2, EPA Method 351.1, SM 4500 Norg-B, SM 4500 Norg-C, SM 4500 NH3-D, SM 4500 NH3-G, SM 4500 NH3-E or	0.5 mg/L

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	SM4500 NH3-F	
Nitrate-Nitrite	EPA Method 353.2 or SM 4500 -NO3 ⁻ E	0.01 mg/L
Metals		
Total recoverable zinc	EPA Method 200.8 (ICP/MS), EPA Method 200.7 (ICP/MS) or SM 3125 (ICP/MS)	5.0 µg/l
Dissolved zinc	EPA Method 200.8 (ICP/MS), or SM 3125 (ICP/MS)	1.0 µg/l
Analyte (or Surrogate)	Method in Water	Reporting Limit Target^a
Total recoverable lead	EPA Method 200.8 (ICP/MS), or SM 3125 (ICP/MS)	0.1 µg/l
Dissolved lead	EPA Method 200.8 (ICP/MS), or SM 3125 (ICP/MS)	0.1 µg/l
Total recoverable copper	EPA Method 200.8 (ICP/MS), or SM 3125 (ICP/MS)	0.1 µg/l
Dissolved copper	EPA Method 200.8 (ICP/MS), or SM 3125 (ICP/MS)	0.1 µg/l
Total recoverable cadmium	EPA Method 200.8 (ICP/MS), or SM 3125 (ICP/MS)	0.2 µg/l
Dissolved cadmium	EPA Method 200.8 (ICP/MS), or SM 3125 (ICP/MS)	0.1 µg/l
Total Mercury	EPA Method 7470 (CVAA), EPA Method 245.7, or EPA Method 1631E	0.1 µg/l
Dissolved Mercury	EPA Method 7470 (CVAA), EPA Method 245.7, or EPA 1631E	0.1 µg/l
Organics		
PAH Compounds	EPA Method 8310 or 8270D SIM	0.1 µg/l
Bis(2-ethylhexyl)Phthalates	EPA Method 8270D	0.250 µg/l
Herbicides (2,4-D, MCPP, Triclopyr, Dichlobenil, Pentachlorophenol)	EPA Method 8270D SIM or 8151	0.01—1.0 µg/l
Pesticides, Nitrogen (Prometon)	EPA Method 8270D SIM	0.01—1.0 µg/l
Pesticides, Organophosphates (Diazinon)	EPA Method 8270D SIM or 8141	0.01—1.0 µg/l
Petroleum Hydrocarbons		
NWTPH-Dx	Ecology, 1997, (Publication No. 97-602) or EPA SW-846 method 8015B; lube oil fraction	0.1 25-0.50 mg/L
NWTPH-Gx	Ecology, 1997, (Publication No. 97-602)	0.1 25 mg/L
BTEX	EPA Method 602	
Toxicity		
Environment Canada Trout Embryo Viability	E-test in Env. Canada EPS-1/RM/28. See also Ecology publication no. WQ-R-95-80.	NA

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- a. All results below reporting limits should also be reported and identified as such. These results may be used in the statistical evaluations.
- b. To ensure accurate results, Ecology recommends modifying these methods to analyze (filter) the entire field sample. Research results indicate that errors may be introduced by decanting a subsample, although using a

funnel splitter may help. The analyst may also consider analyzing several premixed subsamples from the same sample container to determine if significant variability occurred due to stratification. Reports shall indicate whether the entire field sample or a subsample was used.

NA – Not applicable
SM – Standard Methods

Table 9-2 Analytical Procedures in Sediments

Analyte (or Surrogate)	Method in Sediment	Reporting Limit Target ^a
Conventional Parameters		
Total Percent Solids	or SM 2540B	NA
Total Organic Carbon	Puget Sound Estuary Protocols (PSEP 1997), SM 5310B, SM 5310C, SM 5310D or EPA Method 9060	0.1%
Grain-size	Ecology Method Sieve and Pipette (ASTM 1997), ASTM F312-97, ASTM D422 or PSEP 1986/2003	NA
Total Phosphorus	EPA Method 365.3, EPA Method 365.4, SM 4500 P E or SM 4500 P F	0.01 mg/kg
Total Volatile Solids	EPA Method 160.4 or SM 2540E	0.1%
Metals		
Total Recoverable Zinc	EPA Method 200.8 (ICP/MS), EPA Method 6010, EPA Method 6020 or SM 3125 (ICP/MS), or EPA Method 200.7 (ICP)	5.0 mg/kg
Total Recoverable Lead	EPA Method 200.8 (ICP/MS), EPA Method 6010, EPA Method 6020 or SM 3125 (ICP/MS)	0.1 mg/kg
Total Recoverable Copper	EPA Method 200.8 (ICP/MS), EPA Method 6010, EPA Method 6020 or SM 3125 (ICP/MS)	0.1 mg/kg
Total Recoverable Cadmium	EPA Method 200.8 (ICP/MS), EPA Method 6010, EPA Method 6020 or SM 3125 (ICP/MS)	0.1 mg/kg
Total Recoverable Mercury	EPA Method 245.5 or EPA Method 7471B	0.1 mg/kg
Organics		
PAH Compounds	EPA Method 8270D ^b	70 µg/Kg dry
Phthalates	EPA Method 8270D^b	70 µg/Kg dry
Phenolics	EPA Method 8270D^b or PSEP 1997	70 µg/Kg dry
PCB's	EPA Method 8082	80 µg/Kg dry
Pentachlorophenol	EPA Method 8270D SIM or EPA Method 8151	1.0 µg/kg
Diazinon	EPA Method 8270D SIM or EPA Method 8141	50 µg/kg
Chlorpyrifos and Malathion	EPA Method 8270D SIM or EPA Method 8141	25 µg/kg
Petroleum Hydrocarbons		
NWTPH-Dx	Ecology, 1997 (Publication No. 97-602) or EPA SW-846 method 8015B	25.0-100.0 mg/Kg
BTEX	EPA Method 8320	

- 1
2 a. All results below reporting limits shall also be reported and identified as such. These results may be used in
3 the statistical evaluations.
4 b. Sample preparation procedures followed: 3550, 3640, 3660G, and 3620
5 NA – Not applicable
6 SM – Standard Methods
7

8 **~~WET SIEVING AND MASS MEASUREMENT~~**
9 **~~FOR LASER DIFFRACTION ANALYSIS~~**

10
11
12 **~~WET SIEVING~~**

13
14 **~~Sample Collection/Handling~~**

15 ~~Samples should be collected in HDPE or Teflon containers and held at 4 degrees C during the~~
16 ~~collection process. If organic compounds are being collected, the sample containers should be~~
17 ~~glass or Teflon.~~

18
19 **~~Preservation/Holding Time~~**

20 ~~Samples should be stored at 4° C and must be analyzed within 7 days (EPA, 1998). Samples~~
21 ~~may not be frozen or dried prior to analysis, as either process may change the particle size~~
22 ~~distribution.~~

23
24 **~~Sonication~~**

25 ~~Do not sonicate samples prior to analysis to preserve particle integrity and representativeness.~~
26 ~~Laboratories using laser diffraction will have to be notified not to sonicate these samples at any~~
27 ~~time during the analysis. It is recommended that this request also be written on the chain-of-~~
28 ~~custody form that the analytical laboratory receives in order to assure that sonication is omitted.~~
29

30
31 **~~LABORATORY PROCEDURES~~**

32
33 **~~Equipment~~**

- 34
35 ~~— 2 Liters of stormwater sample water (total sample required for analysis (ASTM D 3977))~~
36 ~~— Drying oven (90 degrees C ±2 degrees)~~
37 ~~— Analytical balance (0.01 mg accuracy)~~
38 ~~— Desiccator (large enough diameter to accommodate sieve)~~
39 ~~— Standard sieves – larger than 2" diameter may be desirable~~
40 ~~— 500 um (Tyler 32, US Standard 35)~~
41 ~~— 250 um (Tyler 60, US Standard 60)~~
42 ~~— Beakers – plastic (HDPE)~~
43 ~~— Funnel (HDPE – Large enough diameter to accommodate sieve)~~
44 ~~— Wash bottle~~
45 ~~— Pre-measured reagent grade water~~
46

1 **Sample Processing**

- 2
- 3 ● Dry 250 um and 500 um mesh sieves in a drying oven to a constant weight at $90 \pm 2^\circ \text{C}$.
 - 4 ● Cool the sieves to room temperature in a desiccator.
 - 5 ● Weigh each sieve to the nearest 0.01 mg.
 - 6 ● Record the initial weight of each dry sieve.
 - 7 ● Measure the volume of sample water and record.
 - 8 ● Pour the sample through a nested sieve stack (the 500 um sieve should be on the top and
 - 9 the sieve stack should be stabilized in a funnel and the funnel should be resting
 - 10 above/inside a collection beaker).
 - 11 ● Use some of the pre-measured reagent grade water in wash bottle to thoroughly rinse all
 - 12 soil particles from sample container so that all soil particles are rinsed through the sieve.
 - 13 ● Thoroughly rinse the soil particles in the sieve using a pre-measured volume of reagent-
 - 14 grade water.
 - 15 ● The particles that pass through the sieve stack will be analyzed by laser diffraction
 - 16 Particle Size Distribution (PSD) analysis using the manufacturers recommended protocols
 - 17 (with the exception of no sonication).
 - 18 ● Particles retained on the sieve ($>250 \text{ um}$) will not be analyzed with the laser diffraction
 - 19 PSD.
 - 20 ● Dry each sieve (500 um and 250 um) with the material it retained in a drying oven to a
 - 21 constant weight at $90 \pm 2^\circ \text{C}$. The drying temperature should be less than 100°C to
 - 22 prevent boiling and potential loss of sample (PSEP, 1986).
 - 23 ● Cool the samples to room temperature in a desiccator.
 - 24 ● Weigh the cooled sample with each sieve to the nearest 0.01 mg.
 - 25 ● Subtract initial dry weight of each sieve from final dry weight of the sample and sieve
 - 26 together.
 - 27 ● Record weight of particles/debris separately for each size fraction ($\geq 500 \text{ um}$ and $499-$
 - 28 250 um).
 - 29 ● Document the dominant types of particles/debris found in this each size fraction.
- 30

31

32 **Laser Diffraction (PSD)**

33

34 PSD results are reported in ml/L for each particle size range. Particle size gradations should

35 match the Wentworth grade scale (Wentworth, 1922).

36

37 **Mass Measurement**

38

39 **Equipment**

- 40
- 41 == Glass filter—0.45 um (pore size) glass fiber filter disk (Standard Method D 3977) (larger
 - 42 diameter sized filter is preferable)
 - 43 == Drying oven (90 degrees C \pm 2 degrees)
 - 44 == Analytical balance (0.01 mg accuracy)

- ~~== Wash bottle~~
- ~~== Reagent grade water~~

Procedure

- ~~• Dry glass filter in drying oven at $90 \pm 2^\circ \text{C}$ to a constant weight.~~
- ~~• Cool the glass filter to room temperature in a desiccator.~~
- ~~• Weigh the 0.45 um glass filter to the nearest 0.01mg.~~
- ~~• Record the initial weight of the glass filter.~~
- ~~• Slowly pour the laser diffraction sample water (after analysis) through the previously weighed 0.45 um glass filter and discard the water.~~
- ~~• Use reagent grade water in wash bottle to rinse particles adhering to the analysis container onto glass filter~~
- ~~• Dry glass filter with particles in a drying oven at $90 \pm 2^\circ \text{C}$ to a constant weight.~~
- ~~• Cool the glass filter and dried particles to room temperature in a desiccator.~~
- ~~• Weigh the glass filter and particles to the nearest 0.01mg.~~
- ~~• Subtract the initial glass filter weight from the final glass filter and particle sample weight.~~
- ~~• Record the final sample weight for particles $< 250 \text{ um}$ in size.~~

Quality Assurance

~~Dried samples should be cooled in a desiccator and held there until they are weighed. If a desiccator is not used, the particles will accumulate ambient moisture and the sample weight will be overestimated. A color indicating desiccant is recommended so that spent desiccant can be detected easily. Also, the seal on the desiccator should be checked periodically, and, if necessary, the ground glass rims should be greased or the "O" rings should be replaced.~~

~~Handle sieves with clean gloves to avoid adding oils or other products that could increase the weight. The weighing room should not have fluctuating temperatures or changing humidity. Any conditions that could affect results such as doors opening and closing should be minimized as much as possible.~~

~~After the initial weight of the sieve is measured, the sieve should be kept covered and dust free. Duplicate samples should be analyzed on 10% of the samples for both wet sieving and mass measurements.~~

Reporting

~~Visual observations should be made on all wet sieved fractions and recorded. For example if the very coarse sand fraction (2,000-1,000 um) is composed primarily of beauty bark, or cigarette butts, or other organic debris this should be noted. An option might also be for a professional geologist to record the geological composition of the sediment as well.~~

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