Method for Predicting the Efficiency of Proprietary Storm Water Sedimentation Devices (1006)

Wisconsin Department of Commerce
Wisconsin Department of Natural Resources
Conservation Practice Standard

Introduction and Organization

Both regulators and the regulated community must be able to predict how well proprietary sedimentation devices will perform in the field. These predictions will be used in storm water management planning and for evaluating compliance with regulatory and grant programs.

The purpose of this standard is to establish a uniform process for predicting the site-specific efficiency of proprietary sedimentation devices. There are two approaches that may be used in Wisconsin to meet state regulatory and grant requirements:

- One is to use an acceptable model that calculates efficiency based on Stokes' Law settling.
- The other is to use an acceptable model that contains device-specific efficiency data in lieu of Stokes' Law settling.

This technical standard is separated into four divisions. The first division is the core of the technical standard, and includes modeling and reporting requirements for predicting device efficiency using either Stokes' Law settling or device-specific efficiency data. The second division is Appendix A, which establishes criteria for acceptable models. The third division is Appendix B, which establishes laboratory testing criteria for defining device-specific efficiency curves when used in lieu of Stokes' Law settling. The fourth division is Appendix C, the required method for using a coulter counter to quantify small sediment particles under the laboratory testing protocol.

Throughout the text of this standard and its appendices:

- the term "Section" refers to portions of the technical standard proper;
- the term "Part" refers to portions of the appendices;
- criteria are requirements that must be met to comply with the standard; and
- considerations include additional background information and recommendations, which may be followed at the discretion of the user.

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I. Definition

This standard includes modeling, data and reporting requirements for predicting the efficiency of *proprietary flow-through storm water sedimentation devices* (*devices*) in reducing *total suspended solids* mass loads and concentrations. This standard also includes device installation and maintenance requirements necessary to assure devices are installed consistent with modeling assumptions. This standard does not constitute a *general product approval method*.

II. Purpose

This standard is used to predict the reduction in the *average annual* mass load of total suspended solids and to predict the concentration of total suspended solids discharged from a sedimentation device when installed to treat runoff from a specific drainage area of defined characteristics. Application of this standard provides information necessary for regulators and the regulated community to predict the effectiveness of these devices in meeting *regulatory*, grant-based and other storm water management requirements and goals.

III. Applicability

A. This standard applies to devices installed to control total suspended solids, through *sedimentation processes*, from *development*, *new development, re-development* and *infill areas*.

B. These methods and procedures are acceptable as a basis for evaluating whether predicted device performance meets State of Wisconsin regulatory and grant requirements for urban storm water management.

Note: See Consideration VI.A and VI.B. for information about state requirements.

IV. Federal, State and Local Laws

Users of this standard shall be aware of applicable federal, state and local laws, rules, regulations or permit requirements governing the installation, maintenance and required treatment efficiency of proprietary devices. This standard does not contain the text of any federal, state or local laws.

V. Criteria

A. **Modeling Requirements**

1. **Accepted Model Required**. An accepted model shall be used to predict the reduction in the average annual mass load of total suspended solids and to predict the concentration of total suspended solids discharged from a sedimentation device installed to treat runoff from a specific drainage area of defined characteristics.

   a. The Source Loading and Management Model (SLAMM) is accepted for this use when applied in accordance with the modeling procedures specified in Appendix A, Parts 1.0 and 2.0.

   b. The administering authority may approve other models using the approval process set forth in Appendix A, Part 3.0.

2. **Model Process Sub-routines**. The model may predict pollution control efficiency based on either of the following:

   a. **Theoretical Sedimentation Modeling Method**. This method predicts the total suspended solids reduction efficiency of a device based on principles of gravity settling (Stokes' Law and Newton's Law).

   Note: See Consideration VI.C for a discussion of Stokes' and Newton's law settling.

   b. **Laboratory Data-Based Sedimentation Modeling Method**. This method predicts the total suspended solids reduction efficiency of a device based on device-specific efficiency data generated in a laboratory in lieu of generic gravity settling algorithms.

   i. The efficiency data for tested devices shall be generated in accordance with the laboratory testing protocol and reporting requirements presented in Appendix B.

   ii. Laboratory data collected and evaluated in accordance with Appendix B may be scaled for use with untested devices in the same device classification. Scaling shall meet the requirements of Appendix B, Part 3.2.A and the analysis and reporting requirements of Appendix B, Part 6.0.

   Note: In this method, the device pollutant reduction efficiency reflects the sum total of gravimetric and enhanced settling processes provided by the device. Although scour is not modeled as a separate process, scour testing is required to identify the design treatment flow rate and by-pass requirements for modeling and installation.

B. **Requirements for Reporting Performance Predictions**. The following information shall be reported to the *administering state agency* in support of performance predictions for a device installed to control total suspended solids in a drainage area of specified characteristics.

1. Device name, schematic (plan and elevation) diagrams and model number.
2. Device cross-sectional surface area and dimensions used in making the surface area calculation.
3. *Design treatment flow rate* for the device.
4. Sump information, including: depth of clean sump (in feet) as measured from the bottom of the sediment chamber to the outlet invert; and maximum allowable sediment depth (in feet) as measured from the bottom of the sediment chamber to the top of the maximum allowable sediment depth.
5. By-pass information, including: location (internal, external); flow-rated capacity; and justification for selected by-pass capacity.
6. Tributary area size, land use type, acres of the paved and unpaved surfaces, and the connectedness of these areas to the storm drain system.
7. Identity of model input files.
8. Efficiency determinations:
   a. Average annual % reduction of total suspended solids mass load; and
   b. Range and mean of the event-mean total suspended solids discharge concentrations.

C. **Device Installation and Maintenance Requirements**. Proprietary sedimentation devices shall be installed and maintained in a manner consistent with laboratory testing and modeling assumptions used to predict effectiveness. This includes the following requirements:

1. The device shall be installed in accordance with manufacturer recommendations.
2. The installed device shall be equipped with an internal or external bypass to divert flows in excess of the design treatment flow rate.
   a. For the Theoretical Sedimentation Modeling Method, the design treatment flow rate shall not exceed .08 cfs/ft^2, where ft^2 is the cross sectional area of the primary sedimentation chamber.

   Note: See Considerations VI.D. for the derivation of this factor.

   b. For the Laboratory Data-Based Sedimentation Modeling Method, the design treatment flow rate shall be determined through the scour verification testing conducted under Appendix B, Part 4.0.
3. Accumulated pollutants shall be removed from the device as recommended by the manufacturer. This includes periodic removal of sediment to maintain device efficiency and reduce scour. Sediment shall not be allowed to accumulate to a depth greater than the *maximum recommended sediment storage depth*.
4. If the device is modeled using the Theoretical Sedimentation Modeling Method, the device shall be equipped with either a permanent pool having a depth at least three (3) feet above the maximum sediment storage depth to reduce scour, or shall be equipped with internal flow control structures to reduce scour velocities.

   Note: See Consideration VI.E for a discussion of scour.

VI. Considerations

A. Regulations Comm 20, Comm 60, NR 151 and NR 216, Wis. Adm. Code, either contain or make reference to requirements for reducing the average annual mass load of total suspended solids discharged in storm water runoff to waters of the state. Comm 82, Wis. Adm. Code, establishes requirements for the effluent concentrations of total suspended solids discharged from *storm water plumbing systems* to subsurface dispersal or irrigation areas.

B. Comm 82, Wis. Adm. Code, also includes effluent limitations on the discharge of oil & grease, BOD_5 and fecal coliform from storm water plumbing systems to subsurface dispersal or irrigation systems. This standard does not address the effectiveness of these devices for reducing these pollutants.

C. The theoretical sedimentation model approach applies the upflow (surface overflow) equation to a defined particle size distribution. The predicted reductions apply to the influent load estimated for each runoff event. Load reductions are predicted by particle size class. Scour is not typically modeled as a separate process. The model also predicts the event mean total suspended solids discharge concentrations for each runoff event based on the combined effects of device treatment and by-passing.

The method predicts retention efficiency based on the upflow (surface overflow) equation:

v= Q/A, where:

v = critical particle settling velocity

Q = discharge rate from the sedimentation chamber

A = sedimentation chamber surface area

Stokes' law is for laminar flow conditions and is generally applicable to plain settling for particles up to about 100 µm in size. Newton's law is applicable for turbulent settling, generally for particles larger than 5,000 µm in diameter (assuming a specific gravity of about 2.65). Between these sizes, a smooth transition is used to predict settling. Stokes' Law covers the most critical range, where most of the storm water particles are likely present, and the large particles are "easily" captured by the proprietary devices.

D. For devices modeled using the Theoretical Sedimentation Modeling Method, the design treatment flow rate shall not exceed .08 cfs/ft^2, where ft^2 is the cross sectional area of the primary sedimentation chamber. This limitation is intended to reduce scour by requiring that larger flows by-pass the treatment chamber. The factor of .08 is based on the settling rate of a 250 micron particle size with a specific gravity of 2.7 in water at a temperature of 68^o F, and a safety factor of 1.5. The 250 micron particle size was selected as a basis for scour protection for three reasons. First, an average of 73% of the particles removed from three proprietary devices are 250 microns or greater, thus, limiting the expected mass of material subject to scour (see Table B-7 in Appendix B). Second, it is anticipated that some of the remaining 27% of the trapped load, which would be less than 250 microns in size, would be protected from scour by armoring. Third, an evaluation of design parameters for four selected families of proprietary devices indicates that this by-pass requirement is practical, as it can be met by nearly all of these devices using their existing by-pass capacities.

E. The Theoretical Sedimentation Modeling Method assumes no re-suspension (scour) of previously trapped material, which is known to occur and which will decrease efficiency of the device. The requirement for by-pass or internal flow controls is meant to reduce scour so that modeled efficiency is closer to actual operating efficiency. The Theoretical Sedimentation Modeling Method also does not account for any other processes, such as filtration, which can increase pollution control efficiency.

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VIII. Definitions

Administering state agency (V.B.): The state agency or its agents responsible for administering the storm water regulations applicable to the site. Responsible state agencies are the Department of Natural Resources for NR 151 and NR 216, Wis. Adm. Code, and the Department of Commerce for Comm 20, Comm 60 and Comm 82, Wis. Adm. Code.

Average Annual (II): A condition (such as rainfall or mass load) characterized by a calendar year of precipitation, excluding snow, which is considered typical. Typical average rainfall years for five regions in Wisconsin are available from the Department of Natural Resources.

Design treatment flow rate (V.B.3.): The maximum hydraulic discharge capacity (volume/time) of the sedimentation treatment chamber allowable for installations in Wisconsin. It is the capacity at which scour losses are acceptable, as determined by the requirements of this standard.

Note: The design treatment flow rate has a safety factor built in. The safety factor is 1.5 for devices modeled with the Theoretical Sedimentation Modeling Method (See VI.D.). The safety factor is 1.2 for devices that have had a scour verification test under Appendix B, Part 4.0.

Development (III.A.): As defined in NR 151.002, Wis. Adm. Code.

Devices (I): See definition of *Proprietary flow-through storm water sedimentation device.*

Device classification (Appendix B, Part 1.1): A group or "family" of devices that include similar geometry, flow pattern, sedimentation mechanism and high-flow bypass ability. Devices in the same classification are best thought of as a series of devices of different sizes offered under a similar name by the same manufacturer.

General product approval method (I): A method that gives blanket approval for use of a device.

In-fill area (III.A.): As defined in NR 151.002, Wis. Adm. Code.

Maximum recommended sediment storage depth (V.C.3.) This is the maximum depth of sediment accumulation recommended by the manufacturer to maintain acceptable sediment removal efficiency and reduce scour losses.

For devices modeled using the Theoretical Sedimentation Modeling Method, this depth is specified by the device manufacturer.

For devices modeled using the Laboratory Data-Based Sedimentation Method, it is the sediment depth at which the device passes the scour verification test specified in Appendix B, Part 4.0.

New development (III.A.): As defined in NR 151.002, Wis. Adm. Code.

Proprietary flow-through storm water sedimentation device (I): A chamber or set of chambers (which may include internal baffles or other equipment and associated piping) that is provided as a defined product by a commercial vendor, and is warranted by that vendor to provide specific storm water pollutant removal performance under specified conditions. These devices can consist of prefabricated equipment supplied by a manufacturer, structures constructed on-site, or a combination thereof.

Redevelopment (III.A.): As defined in NR 151.002, Wis. Adm. Code.

Regulatory (II): Decisions made in administering state storm water management requirements. This includes sites regulated by the Department of Natural Resources under NR 151 and NR 216, Wis. Adm. Code, and the Department of Commerce under Comm 20, Comm 60 and Comm 82, Wis. Adm. Code.

Sedimentation processes (III.A.): Removal of sediment by a device through entrainment in the settling chamber(s). Includes basic gravity settling as well as settling enhanced through other physical processes such as centrifugation or tube settling. It does not include the effects of filtration.

Storm water plumbing system (VI.A.): Piping, appliances and devices that convey, hold or treat storm water from building runoff. This includes all piping connected to piping conveying runoff from buildings. The portion of the storm plumbing system under the authority of the Wisconsin Uniform Plumbing Code is that portion conveying storm water to the municipal system or discharging to grade.

Suspended sediment concentration (Appendix B, Part 3.1.C.): Operationally defined as the concentration or mass of sediment determined by testing under method ASTM D3977-97 (1989 Standard Methods).

Total suspended solids (I): Operationally defined as the concentration or mass of sediment determined by testing under method EPA 160.2 (EPA 1979).

Appendix A

Criteria for the Theoretical Sedimentation Modeling Method and Laboratory Data-Based Sedimentation Modeling Method

1.0 Introduction

This appendix contains modeling requirements for predicting the site-specific efficiency of proprietary flow through sedimentation devices. The pollution reduction algorithms used in the model may be based either on basic Stokes' Law settling or on device-specific efficiency data generated under the lab protocol set forth in Appendix B.

SLAMM is an accepted model for both the theoretical sedimentation modeling method and the laboratory data-based modeling method. Part 2.0 of this appendix covers requirements for using the Source Loading and Management Model (SLAMM).

An alternative model to SLAMM may be used, but it must be accepted by the administering authority under Part 3.0 of this appendix.

2.0 Modeling Procedures

Note: See Section V.B of this technical standard for reporting requirements.

2.1. **General Modeling Requirements.** The following requirements apply when using models in either the theoretical sedimentation modeling method or the laboratory data-based sedimentation modeling method.

A. The NURP particle size distribution shall be assumed for the influent storm water.

Note: The NURP particle size distribution is shown in the first two columns of Appendix B, Table B-6.

B. The rainfall files shall meet those specified by the Department of Natural Resources.

Note: DNR requirements for rainfall files can be found either in NR 151, Wis. Adm. Code, or on the DNR Website.

C. The device shall be modeled to by-pass flows greater than the design treatment flow rate. The modeled design treatment flow rate of the device shall not exceed the flows allowed under sections V.C.2.a or V.C.2.b of the standard.

D. Efficiency calculations shall include by-pass effects in final calculations of mass load reduction and concentration of total suspended solids discharged in the device's effluent. Water by-passed around the sedimentation chamber shall be modeled as receiving zero treatment.

E. The device surface area shall be the plan-view area of the settling chamber where the bulk of the sedimentation occurs.

F. Credit shall not be given for sedimentation that occurs, or is predicted to occur, in storm water conveyance pipes leading to or exiting the device.

2.2 **Additional SLAMM Modeling Requirements for the Theoretical Modeling Method**

A. SLAMM version 9.0.1, or later, shall be used. The SLAMM model is available from PV & Associates at http://www.winslamm.com.

B. For model versions 9.0.1 through 9.2.0, the catch-basin subroutine shall be used to model the device. For model version 9.2.1 or later, the hydrodynamic device subroutine shall be used.

C. Parameter files appropriate for use in Wisconsin are identified in Table A-1. File selection depends on the version of SLAMM being used. Parameter files shall be selected in accordance with the following Table A-1.

Table A-1. Parameter Files Required When Using SLAMM for the Theoretical Sedimentation Modeling Method or the Laboratory Data-Based Sedimentation Modeling Method

Parameter File	Model v. 9.0.1	Model v. 9.1.0 – 9.1.2________	Model v. 9.2.0
Rainfall (*.ran)	Select files, start & end dates in accordance with s. NR 151.12(1)	Select files, start & end dates in accordance with s. NR 151.12(1)	Select files, start & end dates and winter season range in accordance with s. NR 151.12(1)
Particle Size Distr.	NURP.cpz	NURP.cpz	NURP.cpz
Pollutant File	WI_GEO01.ppd	WI_GEO01.ppd	WI_GEO01.ppd
Delivery File	WI_DLV01.prr	WI_DLV01.prr	WI_DLV01.prr
Particulate Solids Concentration File	WI_AVG01.psc	WI_AVG01.psc	WI_AVG01.psc
Runoff Coefficient File	WI_SL01.rsv	WI_SL01.rsv	WI_SL06 Dec06.rsv
Street Delivery Files	WI_Com Inst Indust May05.std WI_Res and Other Urban May05.std	WI_Com Inst Indust May05.std WI_Res and Other Urban May05.std Freeway.std	WI_Res and Other Urban Dec06.std WI_Com Inst Indust Dec06.std Freeway Dec06.std

2.3 Additional SLAMM Modeling Requirements for Laboratory Data-Based Modeling Method

A. SLAMM version 9.2.1, or later, shall be used.

B. The hydrodynamic device subroutine shall be used.

C. The parameter files shown in Table A-1 for model version 9.2.1, or later, shall be used.

D. Lab tested efficiency input data – The device performance shall be modeled using efficiency data developed from the data collected and analyzed in accordance with Appendix B.

Note: The Department of Natural Resources will take the data reported for the laboratory testing under Appendix B, Part 6.0 and incorporate it into SLAMM as device-defined efficiency data. Manufacturer's reports on performance projections may be reviewed by a technical committee prior to incorporating the device efficiency data into SLAMM. The administering state agency may make revisions to the manufacturer's performance projections based on comments of the technical committee. The administering state agency will give the manufacturer an opportunity to challenge any such changes.

3.0 Approval of Alternative Models

A. The administering authority may approve the use of a model other than WinSLAMM. In making its determination, the administering authority will use the following process.

B. The applicant shall submit a written request to the administering authority that identifies the proposed model and justification as to why the alternative model should be accepted.

C. If acceptable monitoring data has been collected during field test, the justification for acceptance of the alternative model shall be based on a comparison of modeled device efficiency to monitored device efficiency. In the absence of acceptable monitoring data, the device efficiency determined with the alternative model shall be compared with the device efficiency determined using WinSLAMM.

1. To be acceptable, monitoring data shall have been collected and analyzed using the U.S. EPA Environmental Testing Verification Protocol. In performing the comparative analysis, the site characteristics of the monitored site shall be used as inputs in the model.

Note: In 2007, test data sets were available for Stormceptor, Vortechs, and Downstream Defender devices. The Stormceptor, Vortechs,and Downstream Defender were the subject of intensive monitoring efforts designed to verify the performance of each device and verify the load reductions estimated by WinSLAMM. All the monitoring was conducted by the U.S. Geological Survey (USGS) and the results of the monitoring are available in USGS reports. Verification of the Vortechs and Downstream Defender was part of EPA's Environmental Technology Verification (ETV) program.

D. Comparisons shall be made using the sum of the loads (SOL) method, where:

% Load Reduction =

(inlet SOL – outlet SOL)/inlet SOL * 100, where

Note: The SOL is the combined percent load reduction efficiencies for all the modeled events and provides a measure of the overall performance efficiency for the events sampled during the monitoring period.

E. The administering authority shall compare the applicant's modeling results with the monitored results or the WinSLAMM results for the test site and make a determination whether the alternative model is acceptable. For acceptance based on monitored efficiency, the alternative modeling method must be able to produce an estimate of the device efficiency that is within 15 percentage points of the efficiency measured in the field. For approval based on a comparison with WinSLAMM, the alternative model must be able to produce an estimate of device efficiency within 5 percentage points of the efficiency determined using WinSLAMM.

F. The administering authority will send a written response to the applicant with a decision concerning the acceptability of the alternative model. Until a written acceptance is determined, the proposed model is not accepted for documenting compliance with any regulations at site installations.

Appendix B

Wisconsin Laboratory Testing Method for Determining and Reporting The Performance of Proprietary Storm Water Sedimentation Devices

1.0 Introduction

1.1 Purpose and Overview of Testing Method

The purpose of this testing method is to determine the performance of a full-scale device in a lab setting. The data from this testing will be used to prepare pollutant reduction efficiency curves for incorporation into models that will in turn be used to predict the annual efficiency of the device when deployed in a specific location under a specified annual rainfall sequence.

In this appendix, the word "testing" refers to a suite of tests. The suite of tests for each device includes a set of sedimentation tests and a scour verification test. The set of sedimentation tests includes a defined test repeated for each of four specified flow rates.

In the sedimentation tests, total suspended solids (TSS) and suspended solids concentrations (SSC) of the influent and effluent are measured to determine pollution control efficiency. A mass balance of sediment entering and retained in the device provides supplemental data. Performance data is evaluated by particle size class at four flow rates. Performance may also be reported for untested devices within a *device classification* based on scaling relationships determined from the test data. Data may be reported to the Department of Natural Resources for incorporation into the Source Loading and Management Model (SLAMM), or may be incorporated into an alternative model accepted in accordance with Appendix A, Part 3.0.

The scour verification test is run once at a stepped, increasing flow rate to identify by-pass requirements for the device.

1.2 Testing Objectives

Objective 1. To quantify the mass, by particle size class, of sediment particles trapped by a device under different flow rates.

Objective 2. To present and analyze data to show device efficiency as a function of particle size and flow rate, and to show scaling relationships for predicting the efficiency of untested devices in the same device classification.

Objective 3. To verify that at flows up to 1.2 times the design treatment flow rate, significant scour of previously deposited sediment does not occur.

2.0 Laboratory and Data Analyst Qualifications

2.1. Laboratory Qualifications

A. Laboratory testing shall be conducted by an independent laboratory, or shall be overseen by an independent party if conducted at the manufacturer's own laboratory.

B. The laboratory conducting the performance testing must be able to provide the range of flows, sediment characteristics, measurement and recording systems, and trained personnel necessary to generate reliable test results. A general statement of laboratory qualifications shall be submitted with the required report (see Part 6.0.)

C. If the manufacturer is using its own lab and an independent observer, the observer shall meet the following requirements:

i) The observer shall have no financial or personal conflict of interest regarding the test results.

ii) The observer shall have experience in a hydraulics, sampling and sedimentation lab, be familiar with the test and lab methods specified in this standard and have a professional license in an appropriate discipline.

iii) The observer shall approve the experimental set-up and lab testing protocol and observe the test during its full duration.

D. Prior to initiating tests, the manufacturer shall contact the administering state agency to discuss selection of a laboratory to conduct the required testing. If the manufacturer is using its own lab, it shall contact the administering state agency to discuss selection of an independent observer.

i) For the Department of Natural Resources, contact:

Wisconsin Department of Natural Resources
Attn: State Storm Water Coordinator
Bureau of Watershed Management

101 South Webster Street
P.O. Box 7921
Madison, WI 53707-7921
General Bureau Phone: (608) 267-7694

ii) For the Department of Commerce, contact:

Wisconsin Department of Commerce
Attn: Plumbing Product Review
Safety and Buildings Division
P.O. Box 7162
Madison, WI 53707-7162
Phone: (608) 266-6742

2.2 **Data Analysis**

A. The analysis of lab data shall be performed by a qualified individual. A statement of qualification for the selected individual shall be submitted with the report required under Appendix B, Part 6.0.

B. Prior to initiating data analysis the manufacturer shall contact the administering state agency to discuss selection of an individual to perform this task.

3.0 Sediment Removal Performance Testing

3.1 **Test Parameters**

Note: The scour verification test described under Part 4.0 should be performed first because the results are needed to identify the design treatment flow rate (DTFR). The DTFR is needed to identify flow rates for the sedimentation testing.

A. <u>Flow Rates.</u> Each device shall be tested at a minimum of four discrete steady-state flow rates. These are 5%, 20%, 50% and 100% of the design treatment flow rate.

Note: See Appendix B, Considerations Part 7.0.AA for justification of the selected flow rates.

i) The design treatment flow rate shall not exceed 83% of the maximum flow rate for which the device passes the scour test requirements in Appendix B, Part 4.0.

Note: This provides a safety factor of 1.2.

B. <u>Test Sediment Composition.</u>

i) Test sediment shall be comprised of ground silica mixed in accordance with the proportions shown in Table B-1.

Table B-1. Test Sediment Mix

Total mixed weight: 15.35 lbs.	
US Silica Product Gradation	**Weight**
F 65	0.90 lbs
OK 110	1.2 lbs
Sil-Co-Sil 250	0.25 lbs.
Sil-Co-Sil 106	4.0 lbs.
Sil-Co-Sil 52	1.0 lbs
Min-U-Sil 40	2.0 lbs
Min-U-Sil 30	1.0 lbs
Min-U-Sil 15	1.0 lbs
Min-U-Sil 10	4.0 lbs.

Note: See Appendix B, Considerations Part 7.A. for the derivation of this mix.

ii) A particle size distribution analysis of the dry sediment test mix shall be performed prior to running the lab test and the results shall be reported as part of the requirements set forth under Appendix B, Part 6.0.

C. <u>Influent Concentration</u>. The *suspended sediment concentration* in the influent pipe shall be maintained between 150 mg/l and 250 mg/l. The concentration of inorganic sediment in the influent water prior to adding the test sediment shall be as low as practical.

Note: It is recommended that the concentration of inorganic sediment in the influent water be kept below approximately 10 mg/l prior to mixing with the test sediment.

D. <u>Water Temperature</u>. Water temperature shall be maintained between 50°F and 80°F.

3.2 **Procedure and Data Collection**

A. <u>Number of Devices</u>. When the purpose of the testing is to characterize the efficiency of a series of devices in the same device classification through scaling, testing shall be performed on at least two of the device models.

i) The definition of a device classification shall be the responsibility of the manufacturer. It must be based on technically defensible criteria including similarity between models in geometry, flow pattern, sedimentation mechanism and by-pass.

ii) The devices selected to represent the device classification must reasonably represent the range of device models for which the efficiency curves are being defined. The ratio between the primary sedimentation chamber surface areas of the devices tested shall be at least 2.5.

B. Component tests. For each device model, the required test procedure shall be completed for each of the four flow rates identified in Appendix B, Part 3.1.A.

C. Chamber. A "false floor" shall be constructed in the sediment chamber to simulate a device that is partially filled. The false floor shall be placed to simulate a sediment accumulation of 50% of the maximum recommended sediment storage depth for the device. At the start of the test, the chamber shall be clean of sediment.

D. Test length. Each test shall be run for the duration needed to accumulate a mass of trapped sediment adequate to perform the required analyses.

Note: It is recommended that each sediment removal performance test be run until approximately 5 pounds of material has been trapped. See Appendix B, Considerations Part 7.B for an example calculation of estimated test time to trap this mass of material. If tests can be performed on less than 5 pounds of material, that is acceptable.

E. Sediment sampling frequency. For each test, samples shall be collected and analyzed in accordance with Table B-2. Numbers in parentheses are the minimum number of samples that must be collected and reported for each test flow. Influent samples taken during each test flow may be collected on a random schedule or at equal time intervals. An effluent sample shall be collected immediately after each influent sample.

F. Particle size analysis. The particle size distribution for material in the sediment supply hopper and for material trapped in the sediment chamber shall be determined in accordance with the ASTM standards C117, C136 and D422.

The particle size distribution for samples taken from the influent and effluent pipes shall be determined as follows:

i) Particle sizes 63 microns and greater shall be quantified using ASTM standards C117, C136 and D422.

ii) Particle sizes less than 63 microns shall be quantified using a coulter counter method that conforms to the method set forth in Appendix C.

G. Sample Splitting. Each sample of influent and effluent water shall be collected into three separate bottles to be filled one immediately after the other. One sample bottle is for TSS analysis, one is for SSC analysis and one is for particle size analysis. The TSS, SSC and PSD samples shall be collected in the same order for each flow rate.

Table B-2. Sediment Removal Performance Test: Required Sampling for Each Flow Rate

Sampling Location	Particle Size Distribution	Total Sediment Mass	Total Suspended Solids Concentration	Suspended Sediment Concentration
Sediment Supply Hopper	(1)	Total mass weighed at beginning and end of test		
Influent Pipe	(5)		(5)	(5)
Settling Chamber	(composite from 3 sub-samples of collected mass)	Total mass collected		
Effluent Pipe	(5)		(5)	(5)

H. Flow sampling frequency. Flow shall be monitored throughout the test.

I. Temperature sampling frequency. Water temperature shall be monitored periodically during the course of the test.

4.0 Scour Verification Testing

4.1 Purpose

The purpose of the scour verification test is to verify that the device will not lose a significant amount of pre-deposited sediment at a flow rate up to 1.2 times the design treatment flow rate. This verification test will be used to identify the design treatment flow rate to meet modeling and field installation requirements.

4.2 Pre-loading and Flow

A. The sediment chamber shall be pre-loaded to the maximum recommended sediment storage depth. A false floor may be used to create an apparent sediment depth provided that the depth of sediment placed on the false floor averages at least six (6) inches. Sediment shall be well-mixed and distributed as evenly as practical.

B. The material used to pre-load the device shall be mixed according to the formula presented in Table B-3.

Table B-3. Sediment Specifications for the Scour Verification Test

Material	% by Weight
Concrete Sand (ASTM C33)	15
US Silica: Mauricetown Series – NJ 0 Sand	10
US Silica: Mauricetown Series – NJ 4 Sand	20
US Silica: Ottawa Flint Silica Series-Flint #12	15
US Silica: Ottawa Flint Silica Series-Flint #15	10
US Silica: Ottawa Foundary Sand –F60 Grade	15
US Silica: 20/40 OIL FRAC	10
US Silica: HI-50	5

Note: See Appendix B, Considerations Part 7.C. for derivation of this mix.

C. The device shall be filled with clean water to operating depth prior to initiating the scour test. Sediment suspended during the process of filling the chamber shall be given sufficient time to settle prior to initiating scour test flows.

D. The concentration of inorganic sediment in the influent water shall be as low as practical.

Note: It is recommended that the concentration of inorganic sediment in the influent water be kept below approximately 10 mg/l.

4.3 Scour Test Sampling

A. Once the scour test sediment has been added to the sediment chamber and allowed to settle, the scour test shall be run starting at the lowest test flow and progressing to increasingly greater flows. Do not add new test sediment to the device for each new test flow.

Each test flow shall be constant for a period of 30 minutes or the time it takes to replace 5 volumes of water in the primary sedimentation chamber, whichever is greater. In calculating the volume to be displaced by the test flow, the volume of the sedimentation chamber shall not include any volume below the maximum sediment storage depth.

Samples shall be collected at equal time intervals during each flow. A viewing window shall be installed in the sediment chamber to allow direct observation and video documentation of scour test results. If scour begins between chosen flow increments, testing shall be adjusted to include the start of scour.

B. Samples for each flow rate shall be collected and analyzed in accordance with Table B-4. All samples shall be discrete samples unless otherwise noted. Numbers in parentheses are the minimum number of samples that must be collected and reported.

Table B-4. Required Sampling for Each Flow Rate of the Sediment Scour Test

Sampling Location	Suspended Sediment Concentration
Influent pipe	(5)
Effluent Pipe	(5)

C. <u>Flow sampling frequency</u>. Flow shall be monitored periodically throughout the course of the test.

D. <u>Temperature sampling frequency</u>. Water temperature shall be monitored periodically throughout the course of the test.

4.4 Analysis

A. A device passes the scour test if the average suspended sediment concentration in the effluent pipe does not exceed the average suspended sediment concentration of the influent pipe by more than 25 mg/l.

B. The design treatment flow rate for modeling under Appendix A, Part 2.1.C. shall not exceed 83% of the maximum flow rate for which the device is determined to pass the scour verification test.

Note: This provides a safety factor of 1.2.

5.0 Quality Assurance and Control

Laboratory data submitted under this technical standard shall be collected under a quality assurance/quality control plan. The QA/QC plan shall include the following:

A. Project description.

B. Project organization & responsibility.

C. Data quality objectives.

D. Project test methods.

i) Sample collection methods.

ii) Methods to adjust for expected background concentrations of material in inflow test water.

iii) Calibration of the system used to dose sediment during the sediment removal performance testing, including calibration of sediment dosing equipment and flow pump rates to assure that influent concentrations are maintained within test parameters and that the mass of sediment added to the influent pipe can be accurately measured.

iv) Equipment cleaning and blanks.

v) Duplicate samples.

vi) Sample preservation methods.

vii) Chain of custody.

E. Laboratory procedures.

i) Constituents for analysis.

ii) Laboratory performance standards.

iii) Analysis method references.

iv) Frequency and type of lab QA samples.

v) Data reporting requirements.

vi) Data validation procedures.

vii) Corrective actions.

6.0 Reporting Test Results

6.1 **Laboratory Report**—A laboratory report shall be prepared and submitted to the administering state agency. The report shall follow the following format. The administering state agency may allow deviation from this format upon request of the manufacturer or the lab.

Chapter 1.0 Executive Summary

Chapter 2.0 Background

2.1 Name of laboratory, principal investigator and subcontractors.

2.2 Qualifications statements for laboratories and data analysts.

2.3 Lab equipment list, including: name, model and dimensions (depth & height) of the device tested; pumps, compressors, mixers, valves, flow and water quality sampling equipment; storage tanks; standpipe and plunge pool; and filtration equipment.

2.4. Settling chamber diameter (L_1) and depth (L_2) measurements.

2.5 Inlet and outlet pipe dimensions.

2.6 Results of scour verification test.

2.7 Modifications made to the device to enhance transportation or test feasibility and explanation of why these modifications are not expected to affect the lab results.

2.8 Process flow diagram showing test device, piping, water source, pump, storage tanks, filters, sediment injection system, sampling locations and flow meter.

Chapter 3.0 Sedimentation Efficiency Testing and Results

The following shall be reported for each device tested.

3.1 <u>Date, flow rate and elapsed time for the test</u>.

3.2 <u>Tabular results of test parameters required under Table B-2</u> (Appendix B, Part 3.2.E). Where particle size data is required, it shall be reported for each of the following 8 particle size classes (in microns):

1) < 20

2) 20 – 40

3) 40 – 63

4) 63 – 80

5) 80 – 125

6) 125 – 250

7) 250 – 300

8) > 300

a. Test Sediment Introduced. Total mass of test sediment placed in the sediment hopper, total mass remaining in the hopper, and total mass (calculated by difference) of test sediment discharged from the hopper during the test. Component mass by particle size class of test sediment placed in the hopper.

b. Influent and Effluent Sampling Results. For each discrete influent and effluent sample, the total suspended solids concentration, the suspended sediment concentration, the component mass and concentration by particle size class.

Note: For each sample, three separate one-liter bottles will need to be filled and submitted to the lab for a specific analysis (SSC, PSD and TSS). Each analysis will be assigned to one of the three bottles, so the order of the analysis will be the same each time. For example, if the first bottle of the three collected is sent to the lab for SSC analysis, this order should be maintained for all samples.

c. Test Sediment Retained. Total mass of test sediment removed from the settling chamber. Component mass by particle size class of sediment removed from the settling chamber.

3.3 Performance Efficiency: Concentration Data. Tabular data for each test flow showing the calculated percent reduction in mass of test sediment based on inlet and outlet concentrations reported in Chapter 3.2 of the lab report. Calculations shall be by total mass and by particle size class.

a. Percent reduction shall be based on a comparison of inlet and outlet concentrations. Discrete sample results must be combined to perform this analysis.

% Reduction = (inlet – outlet)/inlet * 100

b. The report shall describe how the inlet and outlet concentrations determined from discrete sampling are combined in calculating the percent reduction for each test flow.

c. The tabular analysis shall be presented in the following format:

Flow Rate (cfs)	Total Mass Reduction (%)	% Reduction by Particle Size Class (Microns) Based on Inlet/Outlet Concentrations							
		<20	20-40	40-63	63-80	80-125	125-250	250-300	>300
.10*DTFR [1]									
.20*DTFR									
.50*DTFR									
1.00*DTFR									

[1]DTFR = design treatment flow rate as determined by the scour verification test.

d. The tabular data set above shall also be presented in graphical form. A separate graph for each particle size class shall be presented that shows the percent reduction (y) as a function of flow rate (x) for the particle size class. A formula shall be developed for each graph.

Note: See Appendix B, Considerations Part 7.D. for an example of how these data may be graphically reported.

3.4 Performance Efficiency: Mass Retained. Tabular data for each test flow showing the calculated percent reduction based on mass entering the device and mass retained. Calculations shall be by total mass and by particle size class. Particle size classes shall include those identified under Chapter 3.2 of the lab report.

a. Percent reduction shall be based on a comparison of mass of sediment introduced to the sediment chamber and the mass of sediment retained in the sedimentation chamber, where:

% Reduction = (mass retained/mass in) * 100

b. The tabular analysis shall be presented in the following format:

Flow Rate (cfs)	Total Mass Reduction (%)	% Reduction by Particle Size Class (Microns) Based on Mass Introduced and Mass Retained in the Sediment Chamber							
		<20	20-40	40-63	63-80	80-125	125-250	250-300	>300
.10*DTFR [1]									
.20*DTFR									
.50*DTFR									
1.00*DTFR									

[1]DTFR = design treatment flow rate as determined by the scour verification test.

c. Graphical representation of this data is not required.

Chapter 4.0 Scaling Relationships

4.1 Method Documentation

a. Scaling formula.

b. Theoretical basis and verification.

Note: See Appendix B, Considerations Part 7.E. for one approach to scaling.

4.2 Application of Formula to Specific Devices

a. Device characteristics, including critical dimensions and design treatment flow rate.

b. Tabular and graphic results for device (see 3.3.c and 3.3.d above).

Chapter 5.0 Scour Test and Results

5.1 Test date and elapsed time for test.

5.2 Test flow rate.

5.3 Test material used to pre-load the device.

5.4 Influent and effluent concentration measurements.

5.5 Data interpretation.

5.6 Calculated design treatment flow rate for use in Wisconsin.

Note: The calculated design treatment flow rate will be 0.83 times the flow rate at which the device passes the scour test.

Chapter 6.0 Quality Assurance and Control Test Data

Chapter 7.0 Signatures for Report Submittal

The report shall be signed by the laboratory director or his designee, the person responsible for data analysis and reporting and, if applicable, the independent observer. The signers shall attest that the laboratory testing and data analysis has been conducted in accordance with the requirements of this technical standard.

7.0 Considerations

AA. The majority of the annual runoff volume to a properly sized device can be expected to occur during runoff events having peak flow discharges well below the design treatment flow rate. Sediment testing for each device will generate only 4 data points, one for each test flow rate. The flow rates for which data is collected should be reflective of the flow rates that the device will encounter most often when modeled.

Table B.4.A shows modeling results for a theoretical device having a design treatment flow rate of 0.5 cfs and an impervious tributary area of 0.5 acres. The test file included 109 rainfall events. Of the runoff events that did not by-pass the device, most (81%) generated peak flow rates less than or equal to 25% of the DTFR and few events (8%) generated peak flow rates over 50% of the DTFR. This phenomenon has also been observed at actual field installations. Based on this information, test flow rates equal to 5%, 20%, 50%, and 100% of the design treatment flow rate are required. If a manufacturer desires to get additional definition in the efficiency curve for low flows, it can add additional flows at its discretion.

Table B.4.A. Frequency Distribution of Runoff Event Peak Flows Modeled for a Theoretical Device Installation Having 109 Rainfall Events, a DTFR of 0.5 cfs and a Tributary Area of 0.5 Impervious Acres

Peak Flow Class (% of the Design Treatment Flow Rate, or DTFR)	Runoff Events in the Class (number)	Portion of Peak Runoff Events in Class
0 – 25%	81	81%
25 – 50%	11	11%
50 – 75%	5	5%
75 – 100%	3	3%

Note: This modeling exercise includes 109 rainfall events. Nine (9) events exceeded the DTFR and would have by-passed the device. Statistics are based on 100 events.

A. The ground silica mixture required for sediment testing is a modification of a base mix prepared to meet the NURP particle size distribution. The base mix formula was calculated by Hydro, International using a selection of standard ground silica products and a computer program. A batch of the base mix was prepared by Hydro and sent to Wisconsin DNR for lab testing to validate that it closely matches the NURP particle size distribution. The base mix formula (shown in the table below) was shown by lab testing to be very close to the NURP particle size distribution. The results of the lab testing are shown in the second table.

Table B-5. Base Mix Formula for Sediment Testing

Total mixed weight: 14.3 lbs.	
US Silica Product Gradation	**Weight**
F 65	0.45 lbs
OK 110	0.6 lbs
Sil-Co-Sil 250	0.25 lbs.
Sil-Co-Sil 106	4.0 lbs.
Sil-Co-Sil 52	1.0 lbs
Min-U-Sil 40	2.0 lbs
Min-U-Sil 30	1.0 lbs
Min-U-Sil 15	1.0 lbs
Min-U-Sil 10	4.0 lbs.

Note to Table B-5: Do not use this table to make the test mix.

Note to Table B-6: Do not use this table to make the test mix. Although the base mix accurately matches the NURP particle size distribution, there are not enough sand sized particles to allow an evaluation of how the test device deals with these coarser particles. To correct this problem, the base mix was adjusted by doubling the amount of OK110 (from 0.6 to 1.2 pounds) and F65 (from .045 to 0.90 pounds). Almost all the particles in the OK 110 are between 90 and 125 microns, while the F65 contains particles that are primarily in the range of 106 to 250 microns.

Table B-6. Results of Verification that Compares Base Mix with the NURP Particle Size Distribution

Particle Size, Microns	NURP, % Finer Than	Test Material, % Finer Than
1	2	11
2	14	17
3	23	23
4	29	31
5	35	35
6	41	40
7	46	45
8	51	49
9	53	52
10	56	54
11	58	56
12	60	-
13	62	-
14	63	62
15	65	63
20	71	68
25	75	73
30	78	76
35	80	80
40	82	83
50	84	86
60	87	88
63	-	88
80	89	90
100	91	93
125	-	95
150	94	96
200	95	97
250	-	98
300	97	99
500	99	100

B. The sediment removal performance test under Appendix B, Part 3.0 should probably be run until at least 5 pounds of material has been trapped. Assuming an influent concentration of 250 mg/l suspended sediment concentration, a control efficiency of 10% (using the NURP particle size distribution) and a test flow rate of 0.5 cfs, it should take approximately 120 minutes to run this test once the flow has achieved equilibrium assuming there is no significant scour. The mass of test sediment placed in the supply hopper would have to be at least 50 pounds.

C. The Department of Natural Resources provided Hydro, International with a particle size distribution based on the material measured in the sedimentation chambers of three field installations (Vortechs, Downstream Defender, and StormCeptor). Hydro used a program to develop the specified mix. The average particle size distribution from monitored devices is shown in Table B-7.

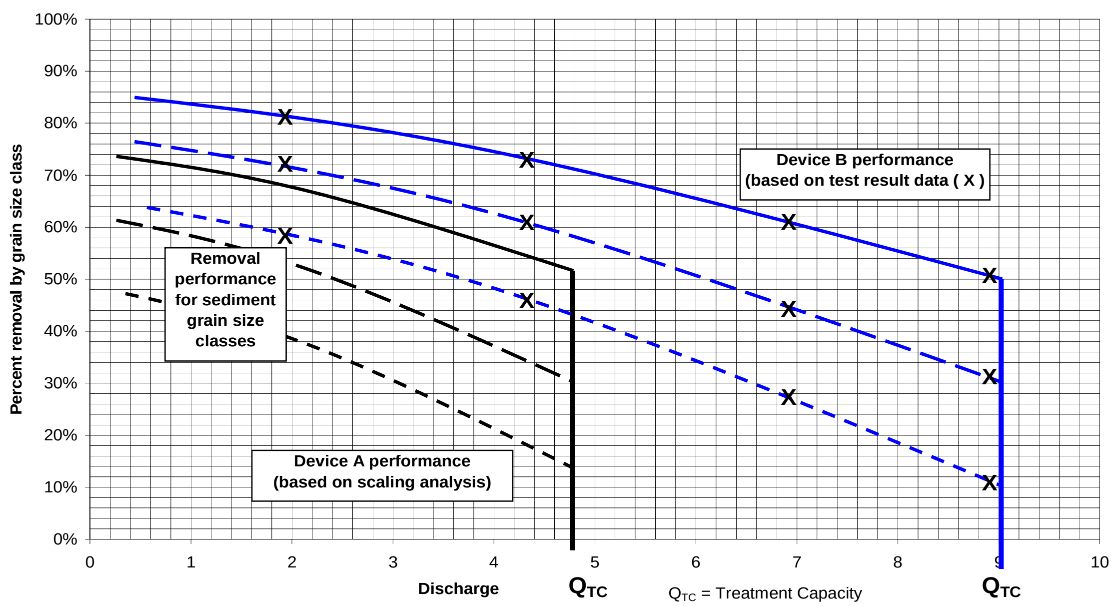
Table B-7. Particle Size Distribution for Sediment Removed from Treatment Chambers of Three Proprietary Devices. (Average of data from three devices: Vortechs, Downstream Defender, StormCeptor)

Particle Size, Microns	Percent Finer Than
8000	97
4000	93
2000	86
1000	75
500	56
250	27
125	12
63	6

D. Suggested graphical presentation of sedimentation test data showing data for multiple devices on the same graph.

Illustration of performance data required for Propriettary Storm Water Sedimentation Devices

Note: Only three grain size clsses shown



E. Manufacturers are encouraged to consider an approach to developing a predictive formula for scaling device performance using the following format:

Percent Reduction = Function (L_1*L_2*Vs)/Q

Where:

L1 = Device characteristic length 1
L2 = Device characteristic length 2
Vs = Particle size settling velocity
Q = discharge through the device

Manufacturers are also encouraged to provide the most accurate predictive methodology for their devices, including approaches other than that listed above.

APPENDIX C

ESS INO METHOD 355.3
Beckman Coulter Multisizer 3, Particle-Size Counter
(Beckman Coulter)

Title: Beckman Coulter Multisizer 3, Particle-Size Counter
ESS INO METHOD 355.3, Revision 0
Effective Date: April 2007
Wisconsin State Laboratory of Hygiene, Environmental Health Division, Inorganic Chemistry Department

1. Scope and Application

1.1 Evaluating the size distribution of particles <32-μm in diameter has become a critical tool in assessing the environmental impact of point/non-point source pollution runoff in urban areas. The potential effects of the smaller-sized particles on receiving waters are not well understood. Consequently the ability to quantify and characterize this size category is extremely important for designing storm water control devices and future decision-making policy.

1.2 There are a wide range of methods available for determining particle size distributions. However, each is based upon different assumptions and principles. Consequently there is not one specific method that is ideal for every application. For example, settling velocities of particles are directly affected by several variables including size, shape, specific gravity, etc. Most standardized methods were established with soils and sediments and ultimately categorize particles <32-μm in diameter into the typical size breaks for sands, silts and clays (15.1, 16.1). Particles carried by storm water runoff may not "fit" into the traditional categories due to their non-terrestrial nature.

1.3 Typically the size distribution of particles in water is established by sieving the sample through a series of sieves (15.3). Each sieve is certified by the size of mesh, and the material trapped on the sieve is quantified, gravimetrically, and expressed as a percentage of the entire sample. Quantifying the mass of material smaller than 32 μm by sieving can be labor intensive, less accurate and at times, impossible due to the small amount of material available for current standard practices (e.g., sieve-pipette method, visual acuity tubes, sediment counters).

1.4 The Wisconsin State Laboratory of Hygiene (WSLH) has developed a method for estimating the distribution of particles that are <32-μm in diameter, by combining data from gravimetric analysis with results obtained from a Beckman Coulter® *Multisizer 3*™ Particle Size Counter (15.2).

1.5 The original Coulter® Principle (*aka* "Electrical Sensing Zone" method) allows for simultaneous counting and sizing of particles in a homogeneous suspension. The sensing zone is established with two electrodes that are separated by a small cylindrical opening (*aperture*). A small amount of electrical current flows through the aperture and between the electrodes. The resistance created by the restricted area separating the electrodes produces current density within the area of the aperture. Particles passing through the aperture displace the volume of the conducting liquid, which creates changes in electrical impedance. The change in the impedance produces a small but proportional flow of current into an amplifier, which further converts the current fluctuation into voltage. The change in magnitude of the current is small (typically 1 mA) but significant enough to generate a voltage large enough to be measured. The Coulter® Principle states that the amplitude of the voltage pulse is directly proportional to the volume of particle that produced it. This principle was developed in the 1940's by Wallace Coulter, who originally developed and patented this technique for blood cell analyses. This technology has evolved over the years to include many industrial applications.

1.6 The Coulter® Principle is applied to particle-size analysis by adding aliquots of sieved sample to an electrolytic solution (i.e., conducting liquid) to facilitate suspension of the particles.

1.7 Urban runoff conditions from specific locations can be monitored both spatially and temporally with WSLH methodology.

1.8 With the appropriate aperture, the Coulter® *Multisizer 3*™ Particle Size Counter can provide particle sizing and counting capabilities within an overall size range of 0.4 to 1200 μm.

2. Summary of Method

2.1 Each sample is processed through a series of standard sieves to trap all particles $\geq$ 32 μm (15.2). Approximately 250 to 1000 mL of well mixed sample (<32-μm in diameter) is recovered after sieving for analysis by the Coulter® *Multisizer 3*™ Particle Size Counter and microfiltration (gravimetric component).

2.2 A metered portion of sample suspension (sample + electrolyte) is drawn through a 50-μm aperture (sensing zone) at a steady rate. The 50-μm aperture provides sizing and counting resolution to 1 to 60% of aperture size (i.e., 2 - 30 μm).

2.3 Data from the instrument is integrated with software to produce a "percent less than" result based upon size breaks assigned by the analyst.

2.4 The percent distribution results from the Coulter® *Multisizer 3*™ Particle Size Counter are applied to gravimetric results from 0.4-μm filtration data and mathematically converted to concentration (mg/L).

2.5 Finally, the estimated concentration data in the size fractions less than 32 μm are compared to the total concentration of particles in the sample. A percent distribution is developed within the Laboratory Information Management System (LIMS) for subsequent report generation.

2.6 Coulter® *Multisizer 3*™ Particle Size Counter data combined with the sieve results provides the WSLH with the ability to mathematically estimate the complete particle size distribution in a sample from $\geq$ 500 to below 0.4 μm.

2.7 Coulter® *Multisizer 3*™ Particle Size Counter offers a high degree of flexibility in size ranges obtained by simply changing the size of the instrument's aperture.

3. Safety and Waste Management

3.1 General safety practices for all laboratory operations are outlined in the Chemical Hygiene Plan for the Environmental Health Division (15.4).

3.2 All laboratory waste, excess reagents and samples must be disposed of in a manner that is consistent with applicable rules and regulations.

3.3 Waste disposal guidelines are described in the University of Wisconsin Laboratory Safety Guide (15.5).

4. Sampling Handling and Preservation

4.1 Samples to be processed and analyzed for particle sizing are typically collected in 1-gallon, polyethylene containers.

4.2 Prior to analysis commencing, WSLH personnel will weigh the sample container on a high-capacity analytical balance to establish the original mass/volume of sample received at WSLH (15.6).

4.3 After sieving, WSLH personnel will recover approximately 250 to1000 mL of the <32μm fraction in a WSLH quart bottle. The bottle will be assigned the same WSLH sample Identification number (ID) and reserved for analysis with the Coulter® *Multisizer 3*™ Particle Size Counter and microfiltration at 0.4μm.

4.4 Samples are stored at 4C.

4.5 Samples collected for particle size determinations are not preserved.

4.6 Although a specific holding time for particle size samples has not yet been established, every effort should be made to process the sample within 30 days of collection for best results.

5. Interferences

5.1 Samples containing a large amount of particles may clog apertures.

5.2 Each aperture allows the measurement of particles within 2 to 60% of the nominal diameter of the aperture. For example, a 100-μm aperture allows sizing of particles between 2 and 60 μm, not inclusive.

5.3 Particles in samples may aggregate or clump during storage and can cause clogging of the aperture. For best results, samples should be at room temperature and mixed thoroughly prior to analyzing.

5.4 Aliquots of sample should be combined with a diluent to facilitate dispersion and minimize clogging of the aperture.

6. Reagents and Standards

6.1 ASTM Type-1 Water (*MQ*).

6.2 Conductance/electrolyte solution: ISOTON® II diluent (Beckman Coulter®).

6.3 Particle Characterization/Sizing Standards: Certified sizing standards (e.g., polystyrene latex beads or polymer microspheres in an aqueous medium) are available from Beckman Coulter, Duke Scientific, etc. and should be used for performing or validating the instrument calibration and for use as a Quality Control

Standard. The standards should be NIST traceable. Calibration or verification is only needed at one size for each aperture, preferably between 5 and 20% of the aperture diameter (15.2).

6.4 Aperture Instrument Concentration Control (Beckman Coulter®): Control standard used to verify instrument count accuracy performance (units = **#Total Particles/mL**); acceptable results are typically within ±10% of the assay value.

7. Apparatus

7.1 Beckman Coulter® *Multisizer 3*™ Particle Size Counter (M3).

7.2 Electronic pipette.

7.3 Beakers of assorted sizes.

7.4 Cuvettes, 20 mL, e.g., Accuvette™ II container (Beckman Coulter®).

8. Quality Control

8.1 **Corrective Action** documentation for QC failures within analytical runs will include: a) identifying the QC failure and cause, if known; b) specific corrective actions that were performed; c) the next action that will be taken.

8.1.1 Attached to each analytical run will be lists of specific analytical items to be checked in the event of a QC failure. The lists will be tailored to the specific method and instrumentation as an aid in documenting corrective action. If the analytical failure cannot be identified, the analyst will note: "*Analytical Checks ok; Unknown cause*" on the benchsheet.

8.2 **An instrument logbook** is maintained for each instrument. Maintenance, performance problems, date calibrated, analyst, and other pertinent information are documented in the logbook.

8.3 **A Quality Control Standard (QCS)** is analyzed with each run. The analytical result must be within ± 10% of the true value to continue the analysis. If the recommended limits are exceeded, corrective action includes reanalyzing the QCS or the analyst may recalibrate if necessary. Choose a QCS with certified particle size that is within the analytical range of the aperture (15.2).

8.4 **A Laboratory Reagent Blank (LRB)**, aka "Check Blank (CB). For purposes of this method, a LRB/CB is not applicable for particle size determinations in environmental sample. However, if samples of a biological nature are analyzed, the dispersion agent may be utilized as the LRB/CB (aka "Control Blank").

8.5 **Laboratory Fortified Blank (LFB): not applicable for this method**.

8.6 **Matrix Duplicates:** Prepare **a minimum of 10%** of the samples, per matrix, as duplicates**. Matrix Spikes are not applicable for this method**. Refer to the QL dataset in LIMS for a detailed listing of all QC limits used for various sample matrices. If the duplicate (precision QA) is not met, the matrix group should be reanalyzed unless clogging of the aperture is a problem. If limits are exceeded a second time, a smaller volume of sample from this matrix group may be added to the diluent (6.2) and reanalyzed. If limits are exceeded a second time, qualify the matrix group (15.8) as a comment or memo. Because M3 data is used for LIMS calculations, data cannot be qualified as "* *result*."

8.7 **An Instrument Performance Check (IPC)** is not applicable for this method. The instrument performance is based upon a Calibration Verification Check (9.1 – 9.6), which is analyzed at the beginning of each batch. The M3 software will notify the analyst if the instrument is not within calibration based upon the size of aperture installed at the time of calibration. Choose a calibration standard or verification standard as recommended by the manufacturer (15.2). A new calibration check should be performed whenever a new or different aperture is installed.

8.8 **Initial Demonstration of Capability (IDOC):** Initial DOC and annual continued proficiency checks are performed according to ESS INO QA 115 (15.9). The QCS (6.3) may be used for this procedure.

8.9 **Limit of Detection (LOD, 15.10): not applicable for this method** and is defined by the size limit of the aperture installed at the time of use.

9. Method Calibration

9.1 Allow the instrument to warm up a minimum of 15 minutes prior to operation.

9.2 Calibrate every new aperture following the M3 Operator's manual (15.2). Once a particular aperture has been calibrated, a verification standard should be analyzed prior to each analytical batch. Calibration of an aperture should be performed whenever a verification procedure fails, or whenever a new aperture is installed.

9.3 Prepare a calibration/verification standard by adding approximately 30 drops of standard solution and diluting to the 20-mL mark on the M3 cuvette. Mix thoroughly.

9.4 Open the door to the sample compartment on the M3 and lower the sample platform.

9.5 Secure the cuvette containing the calibration or verification standard into the platform. Raise the platform until the electrode and aperture are submerged in the standard solution.

9.6 Close the door.

9.7 Check the concentration level of the suspension by selecting ***Preview*** from the left-hand status panel. The concentration index bar will be displayed and notify the analyst if the concentration is ok; the manufacturer recommends a concentration of 10% for best results. If the concentration is high, calibration may be incorrect; if too low, the time required for calibration will be too long.

9.8 Exit the ***Preview*** mode by selecting <cancel>.

9.9 Activate the ***Calibrate*** mode via the M3 software.

9.9.1 If calibrating for the first time, choose the appropriate size calibrator and click on the calibration icon. The Calibrator Size box will open; enter the modal value of the calibrator—this is the certified value provided by the manufacturer. Beckman Coulter recommends repeating the calibration ten times and record the ***Kd*** each time. Calculate the mean ***Kd*** for the aperture and enter this value into the "Aperture Tube list" along with the serial number of each aperture. The "Aperture Tube list" can be accessed via the *<Change Aperture Tube Wizard…>*.

9.9.2 Once the calibration standard has been analyzed, the instrument is ready for analyzing samples and need not be calibrated again unless the daily verification standard is exceeded. Future verifications of **this** calibration should always be within ± 4% of the mean value obtained in 9.9.1 (15.2).

9.10 If the aperture has already been calibrated, the analyst needs only to ***Verify the calibration***.

9.10.1 Prepare the verification standard (9.3 – 9.8).

9.10.2 Activate the ***Verify*** mode via the M3 software.

9.10.3 Enter the modal value of the verification standard in the Calibrator Size box (9.9.1). Note: If the same aperture is being used for each batch, the Calibrator Size box will retain the certified modal value of the previous verification standard.

9.10.4 Press *<Start>* from the Calibrator Size box to activate the *Verification* process.

9.10.5 The software will automatically notify the analyst if the verification has been successful. The software will prompt the analyst of the change between the old ***Kd*** and the new ***Kd***. Record the new ***Kd*** in the instrument logbook to maintain a record for each specific aperture.

9.11 Always verify aperture calibration prior to analyzing samples.

9.12 Recalibrate any time the verification process fails or if a new aperture is installed.

10. Procedure

10.1 Select the appropriate analytical settings for the M3 from the Main Menu. Alternatively, *Load* the desired Standard Operating Procedure (SOP) by selecting **Settings** from the Main Menu bar.

10.1.1 An M3 SOP consists of pre-selected analytical settings that have been saved as a "Standard Operating Method (SOM)." See the Beckman Coulter Operator's Manual for detailed directions for creating and/or changing an SOM (15.2).

10.1.2 Although the size settings can be altered at any time, it is helpful to configure the SOM for the desired size breaks in the *Cumulative % <* format for Volume, Number and Surface Area.

10.1.2.1 Check the ***Cumulative, %<*** data table at the end of each run report. If only "<100%>" shows for each size break on the table, extra digits after the decimal point are needed. In the chart window, select <Analyze>, <Convert Pulses to Size Settings>; select <2% to 60%> to expand the *x* axis on the chart window to the maximum resolution of the aperture. Turn off the “Multisizer II” edit box, then select <ok>.

10.1.2.2 Check the data table again to view the cumulative, %< size breaks on the data table. You should now have values less than 100% for each size break. These percentages are recorded on a *Worklist* (*WL*) and used by LIMS to estimate the overall percent distribution of particles below 32 μm.

10.2 Pipette an aliquot of sample into the cuvette.

10.2.1 The volume of sample may range from one to 15 mL, at the analyst’s discretion. Samples containing noticeably large amounts of particles should be diluted approximately 1:20 with diluent prior to analysis to minimize clogging of aperture.

10.3 Dilute the volume of sample to the 20-mL mark on the cuvette with diluent (6.2).

10.4 Mix the cuvette by inversion.

10.5 Modify the sample and batch information as appropriate under the Sample Information section of the Status Panel.

10.5.1 *Group ID*: Enter the WSLH batch ID.

10.5.2 *Sample ID*: Enter the WSLH sample ID.

10.5.3 *Control Sample*: Check this box whenever a QC sample is being analyzed.

10.5 4 NOTE: If the concentration of particles in the sample (i.e., counts) is a desired result, the following data fields must be completed:

10.5.4.1 Sample volume or mass (weight or volume of sample used for the analysis; the volume or mass combined with electrolyte).

10.5.4.2 Electrolyte volume (volume of electrolyte used).

10.5.4.3 Analytical volume (volume of sample suspension being analyzed, where: *suspension = sample + electrolyte*).

10.6 Open the door to the sample compartment on the M3 and lower the sample platform.

10.7 Secure the cuvette into the platform. Raise the platform carefully until the electrode and aperture are submerged into the sample solution. Note: When using the 20-mL cuvette for sample analysis, the glass stirrer should always be adjusted with the stirrer knob such that the paddles are moved to the right of the cuvette; i.e., the stirrer does not fit in the cuvette.

10.8 Check the concentration level of the suspension by selecting ***Preview*** from the left-hand status panel. The concentration index bar will be displayed and notify the analyst if the concentration is ok. Although the manufacturer recommends a concentration of 10% for best results, previous work at WLSH demonstrates that samples prepared at 3 to 5% concentration level perform best (i.e., higher concentration levels tend to clog the aperture).

11. Calculations

11.1 The raw instrument data for each size break is entered on a *Worklist, WL* (15.15). These results are estimates of the percent size distribution in water samples that have been sieved down to 32 μm.

11.2 Once the raw data has been entered into LIMS, the data is processed automatically and mathematically converted to yield both concentration (i.e., mg/L) and percent distribution (i.e., “% <”) for the entire sample, based upon the total mass received.

12. Data Management

12.1 The *WL* (15.15) and the *QAWRKSHT* (15.14), where all quality control is calculated for pass/fail criteria, will be reviewed for quality control prior to accepting results (see section 8) by an experienced chemist who did not run the original analysis (15.13). The reviewer must initial and date the cover sheet as an indication of the run’s acceptable results.

12.2 Final QC-reviewed results will be submitted for manual data entry into LIMS (15.14).

12.3 Whenever possible, data will be electronically exported to LIMS.

13. Definitions

13.1 Definitions of terms in this SOP may be found in the reference method (15.2). General definitions of other terms that may be used in this method are found in Section 19 of the WSLH Quality Assurance Manual (15.8).

14. Method Performance

14.1 Where applicable, the laboratory's initial accuracy and precision data (LOD's and DOC's) were generated in compliance with the reference method and the Inorganic Chemistry Department's standard operation procedures: ESS INO QA 115 (15.9) and ESS INO QA 116 (15.10). Supporting data will be retained according to the applicable Records Disposition Authority (RDA). Data generated within the last two years will be kept on file within the Inorganic Chemistry Department. Data older than two years may be archived in the basement.

15. References

15.1 American Society of Testing and Materials (ASTM), 2002. *Standard Test Method for Particle-Size Analysis of Soils*, D 422-63.

15.2 Beckman Coulter® *Multisizer 3*™ Particle Size Counter, Operator's Manual. Beckman Coulter, Inc. Fullerton, CA 92835.

15.3 Wisconsin State Laboratory of Hygiene, ESS INO 355.1. *Particle-Size Determinations by Sieving and Microfiltration.*

15.4 Wisconsin State Laboratory of Hygiene, EHD GENOP 026. *Chemical Hygiene Plan for the Environmental Health Division, State Laboratory of Hygiene, Agriculture Drive*.

15.5 University of Wisconsin-Madison, Chemical Radiation Protection Office, Safety Department (262-8769). 2004. *Laboratory Safety Guide*. http://www.fpm.wisc.edu/safety.

15.6 Wisconsin State Laboratory of Hygiene, ESS GENOP 202. *Calibration, Maintenance, and Accuracy Verification Procedure for Balances*.

15.7 Wisconsin Department of Natural Resources Lab Certification Program, July 1997 PUBL-TS-007-97.

15.8 Wisconsin State Laboratory of Hygiene. *Quality Assurance Procedures and Policies*.

15.9 Wisconsin State Laboratory of Hygiene. ESS INO QA 115, Initial DOC and Annual Continued Proficiency Check Procedures.

15.10 Wisconsin State Laboratory of Hygiene. ESS INO QA 116, *LOD Procedures*.

15.11 Wisconsin State Laboratory of Hygiene. ESS INO GENOP 200, *Pipette Performance Checks*.

15.12 Wisconsin State Laboratory of Hygiene. ESS INO QA 101, Bottle Check Procedure.

15.13 Wisconsin State Laboratory of Hygiene. ESS INO QA 107, *Q.C. Audits of Analytical Runs for ESS Wet Chemistry Area*.

15.14 Wisconsin State Laboratory of Hygiene. ESS INO QA 114, *LIMS Quality Assurance Worksheet Procedures*.

15.15 Wisconsin State Laboratory of Hygiene. ESS INO GENOP 108. *Procedure for Creating a WL Worklist*.

16. Tables, Figures, Diagrams, Charts, Checklists, Appendices, Definitions

16.1 **Table 1.** Recommended scale of particle size breaks for sediment analysis (15.1).

Description	Size (μm)
Sands:	
Very coarse	1000-2000
Coarse	500-1000
Medium	250-500
Fine	125-250
Very fine	62-125
Silts:	
Coarse	31-62
Medium	16-31
Fine	8-16
Very fine	4-8
Clay:	
Coarse	2-4
Medium	1-2
Fine	0.5-1
Very fine	0.24-0.5

Written by: Lorrine D. Edwards Date: 06/2007

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Unit: ESS Inorganic Chemistry Unit

Reviewed by: ______________________ Date: ______________

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Accepted by: ______________________ Date: ______________

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