

STANDARD OPERATING PROCEDURE Procedure for Analyzing Total Suspended Solids (TSS)

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RECORD OF AMENDMENTS:

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STANDARD OPERATING PROCEDURE

Procedure for Analyzing Total Suspended Solids

BACKGROUND

The [Great Ships Initiative](#) (GSI) is a collaborative effort to end the problem of ship-mediated invasive species in the Great Lakes-St. Lawrence Seaway System through independent research and demonstration of environmental technology, financial incentives and consistent basin-wide harbor monitoring. To that end, GSI has established research capabilities at three scales—bench, land-based, and shipboard. Each scale is dedicated to addressing specific evaluation objectives, with protocols as consistent with IMO and federal requirements as practicable. Developers of ballast treatment systems apply for GSI research services [online](#), and awards are offered based on an objective review process. GSI incubation/testing will allow meritorious ballast treatment systems to progress as rapidly as possible to an approval-ready and market-ready condition.

GSI bench-scale tests take place year-round at the University of Wisconsin-Superior's Lake Superior Research Institute (LSRI) in Superior, Wisconsin. The LSRI is amply equipped with staff expertise and resources to conduct the tests, and has a long history of successfully undertaking similar tests.

The overarching goals of GSI bench-scale testing are to explore dose-effectiveness, chemical degradation, residual toxicity, and sensitivity to challenge conditions of a proposed ballast treatment method about which little is known. To that end, the tests are “range-finding” missions, to determine the optimal treatment dose/intensity that would maximize effectiveness and minimize residual toxicity. Findings help treatment developers better design an effective system and/or to move to the next stage of treatment evaluation. The tests are also a form of trouble-shooting to encounter possible problems with the proposed treatment in advance of more extensive and larger scale tests.

INTRODUCTION

This GSI Standard Operating Procedure (SOP) describes the method used to analyze total suspended solids (TSS) in water samples. TSS are organic (e.g., algae) and inorganic (e.g., soil particles) material suspended in the water column. As TSS increase, the turbidity of the water and the absorption of light are also increased. Analyses of TSS are determined on a well-mixed sample that is filtered through a pre-weighed standard glass fiber filter. The retained residue on the filter is dried to a constant weight at 103-105 °C. The increase in the weight of the filter is due to the TSS present in the filtered sample.

DEFINITIONS

Brackish Water (BW): Synthetic water created from laboratory water (LW) with the addition of commercially prepared salts, such as Instant Ocean, to obtain a salinity of 16 parts per thousand (as measured by a refractometer).

High Organic Content Laboratory Water (HOC-LW): Synthetic water created from laboratory water (LW) that is used as a surrogate in place of Duluth-Superior Harbor water.

Laboratory Water (LW): City of Superior, Wisconsin municipal water that has been dechlorinated by passage through an activated carbon filter. Note: Based on data from previous testing, background levels of chlorine from below the limit of detection (i.e., $> 10 \mu\text{g/L}$) are expected in dechlorinated laboratory water, depending on where the dechlorinated water is taken from.

Prospective Ballast Treatment System (BTS): A system containing an active substance and/or component that mechanically, physically, chemically, or biologically serves to remove, render harmless, or avoid the uptake or discharge of potentially invasive organisms within ballast water (IMO, 2005).

Salt Water (SW): Synthetic water created from laboratory water (LW) with the addition of commercially prepared salts, such as Instant Ocean, to obtain a salinity of 32 parts per thousand (as measured by a refractometer).

EQUIPMENT LIST

- Glass-fiber Filter Disk, 47 mm (i.e., Whatman 934AH, Gelman Type A/E).
- Membrane Filter Funnel.
- Deionized Water.
- 1-L Filtration Flask.
- Forceps.
- Graduated Cylinders, 50 and 1000 mL.
- Aluminum Weighing Pan, 42 mL.
- Drying Oven, for Operation at 103-105 °C.
- Vacuum Pump.
- Vacuum Tubing.
- Analytical Balance.
- Desiccator with Indicating Desiccant.

PROCEDURE

1. Conduct procedure in a vented work area, taking appropriate health and safety measures.
2. Glass-fiber filter preparation procedure (to be conducted before sample filtration):
 - a. Place the filter into the membrane filter funnel with the wrinkled side facing up.
 - b. Insert the rubber stopper of the filter holder into the neck of the filtration flask and connect the vacuum pump to the side arm on the filtration flask using vacuum tubing.

- c. Apply the vacuum, rinsing the filter with three 20-mL aliquots of deionized water. Continue the suction to remove all traces of water and discard the washings.
 - d. After all traces of water have been removed, continue to draw air through the filter for approximately 1 minute. This will help to ensure that the filter does not stick to the aluminum weighing pan after it is dried in the oven.
 - e. Carefully remove the washed filter from the filter holder and transfer it to a labeled aluminum weighing pan.
 - f. Place the pan containing the filter into a drying oven at 103-105 °C and dry for a minimum of 1 hour.
 - g. After drying, remove the weighing pan containing the filter from the oven and place in a desiccator to cool.
 - h. Weigh the pan containing the filter on an analytical balance to 0.1 mg. Record the weight.
3. Filter a volume of sample that will yield a mass of between 2.5-200 mg of dried residue. For samples with low suspended solids concentrations, a volume of 1000 mL should be filtered. If filtration requires more than ten minutes, reduce the sample volume being filtered. Record the volume of filtered sample in a log book or data sheet.
4. Sample filtration procedure:
- a. Assemble the filtering apparatus containing a previously prepared glass-fiber filter (step 2).
 - b. Wet the filter with a small amount of deionized water to seat it properly in the filter holder.
 - c. If filtering a sample volume of 100 mL or less, thoroughly mix the sample, and transfer the homogenized sample from the sample bottle into a beaker. Stir the sample with a magnetic stirrer and pipette the volume of sample to be filtered from a point both mid-depth and midway between wall and vortex of the stirred sample.
 - d. If filtering a sample volume of greater than 100 mL, shake the sample bottle vigorously, and pour the volume of sample to be filtered into a 1000-mL graduated cylinder. Filter the sample transferred to the filter holder by applying a vacuum to the filtration flask. Rinse the graduated cylinder with several aliquots of deionized water to ensure that all solids have been transferred to the filter apparatus.
 - e. Rinse the walls of the filter holder with three aliquots of deionized water to transfer any particles adhering to the walls onto the filter. Continue the vacuum for an additional 3 minutes after the filtration is complete.
 - f. Carefully transfer the filter containing the residue back into its aluminum pan.
 - g. Dry for a minimum of 1 hour at 103-105 °C, cool in a desiccator, and weigh the pan and filter to 0.1 mg. Record the weight.
 - h. Repeat the drying, cooling in the desiccator, and weighing until the weight change is less than 4 % of the previous weight or 0.5 mg, whichever is less.

- i. Duplicate determinations should be conducted on at least 10 % of the samples and duplicates should agree within 5 % of their average TSS concentration.
5. Calculate the total suspended solids in each sample:

$$\text{Total Suspended Solids (mg/L)} = \frac{(A - B) * 100}{\text{Sample volume (mL)}}$$

Where:

A = weight of aluminum pan, filter, and dried residue (mg)

B = weight of aluminum pan and filter (mg)

QUALITY ASSURANCE/QUALITY CONTROL

1. Conduct all quality assurance/quality control procedures according to the GSI/QAPP/1 - Quality Assurance Project Plan (QAPP) for Great Ships Initiative Bench-Scale and Land-Based Biological Tests (2009). Analyze data to ensure that all applicable data quality criteria are met.
2. Collect and analyze in duplicate at least 10 % of the samples to document sampling and analytical variability. Duplicates should agree within 5 % of their average.
3. Follow all procedures outlined in this SOP. Any deviations known ahead of time must be approved by the GSI Lead Investigator for Bench-Scale Studies. Any deviations made during the experiment must be recorded and also approved by the GSI Lead Investigator for Bench-Scale Studies as soon as practicable.
4. Record data on data collection forms or in specific laboratory notebooks. All instrument data output (e.g., chromatograms, absorbance scans, and/or measurements) and data forms must be stored in a project-specific three-ring binder. Ensure hard copies of instrument data output and data collection forms are scanned and stored electronically.

DATA STORAGE AND ARCHIVING

1. Store and archive data according to GSI/QAPP/1 - Quality Assurance Project Plan (QAPP) for Great Ships Initiative Bench-Scale and Land-Based Biological Tests (2009).
2. Archive all hard- and electronic-copies of data and records generated for a period of five years.

REFERENCES AND RELATED DOCUMENTS

American Public Health Association (2005). Total suspended solids dried at 103-105 °C. Part 2540 D. In *Standard Methods for the Examination of Water and Wastewater*, 21st edition. Washington, DC, pp 2-58 to 2-59.

Cangelosi AA (2006). RDTE Facility for the Great Ships Initiative (GSI) (OAR-SG-2006-20000364). Project Proposal to the National Oceanic and Atmospheric Administration/U.S. Fish and Wildlife Service. Northeast-Midwest Institute, Washington DC.

Great Ships Initiative Standard Operating Protocols: <http://www.nemw.org/GSI/protocols.htm>.

Great Ships Initiative website: www.greatshipsinitiative.org.

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International Maritime Organization (IMO) (2005). Guidelines for Approval of Ballast Water Management Systems (G8) Adopted by Resolution MEPC.125 (53). London, England.