

STANDARD OPERATING PROCEDURE Procedure for Analyzing Hydrogen Peroxide Concentrations in Water

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RECORD OF AMENDMENTS:

No.	Date	Type	No.	Date	Type
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STANDARD OPERATING PROCEDURE

Procedure for Analyzing Hydrogen Peroxide Concentrations in Water

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 water for concentrations of hydrogen peroxide. The method is appropriate for measuring hydrogen peroxide levels of a prospective ballast treatment system (BTS) that involves hydrogen peroxide. Hydrogen peroxide is a reactive compound and its concentration may change rapidly when added to water. Measurement of concentrations must be determined over time.

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).

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

- Spectrophotometer capable of measurement at 351 nm.
- 1 cm cuvettes for spectrophotometer.
- Wash bottle with deionized water.
- Top-loading balance capable of weighing to 0.01g.
- Weighing pans.
- Scoopulas.
- 500 mL volumetric flasks (2).
- 100 mL volumetric flasks (5).
- 100-1000 μL pipettor with disposable tips.
- 10-100 μL pipettor with disposable tips.
- 1000-5000 μL pipettor with disposable tips.
- 30 mL beakers.
- Personal protective equipment (i.e., protective eyewear, laboratory coat, etc.).
- Potassium iodide.
- Sodium hydroxide.
- Ammonium molybdate tetrahydrate.
- Potassium hydrogen phthalate.
- 30 % hydrogen peroxide.

REAGENTS

Reagent A – 33 g potassium iodide, 1.0 g sodium hydroxide, and 0.1 g ammonium molybdate tetrahydrate dissolved and diluted to 500 mL with deionized water. This solution should be kept in the dark to inhibit the oxidation of I^- . If the solution becomes colored, it should be remade.

Reagent B – 10.0 g of potassium hydrogen phthalate (KHP) dissolved in deionized water and diluted to 500 mL.

PROCEDURE

1. Prepare an intermediate hydrogen peroxide (H_2O_2) standard by diluting 60 μL of 30 % H_2O_2 to 100 mL with deionized water in a volumetric flask. The concentration of this standard is 200 mg/L H_2O_2 .
2. Use the 200 mg/L standard to prepare a series of working standards as follows (table 1):

Table 1. Working Standards.

Conc. (mg/L)	mL of 200 mg/L H_2O_2 standard	Final volume with deionized water (mL)
0.0	0.0	100
0.5	0.25	100
1.0	0.50	100
2.0	1.0	100
3.0	1.5	100

3. Collect the required samples from the appropriate location(s) depending on the type of test being conducted. For example, collect a minimum of 3.0 mL of solution per sample, collect a minimum of 10 % of samples in duplicate, and collect an additional aliquot from 10 % of samples to use for determination of spike recoveries.
4. Analyze the standards and samples by pipetting 3.0 mL of “Reagent A”, 3.0 mL of “Reagent B”, and 3.0 mL of standard or sample into a 30 mL beaker. Note: If samples are likely to have a H_2O_2 concentration higher than 3.0 mg/L, they will need to be diluted so that their diluted concentration falls into the range of the calibration standards.
5. Swirl the beaker to mix the contents. Allow the contents to react for a minimum of 5 minutes before reading the absorbance of the solution at 351 nm. If the water to be tested is colored or turbid, use an aliquot of the water that contains no H_2O_2 to prepare a blank for correcting the absorbances of samples that have been treated with H_2O_2 .
6. Prepare a calibration curve by plotting the concentration of the standards versus the blank-corrected absorbance of the standards. Use the slope and y-intercept of the calibration curve to determine the H_2O_2 concentration of the samples based on their blank corrected absorbances.

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. Whenever possible, spike at least 10 % of the samples with a spiking solution containing hydrogen peroxide.
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 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

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.

Eaton, AD, Clesceri, LS, Rice, EW, and AE Greenberg, Eds. (2005). Standard Methods for the Examination of Water and Wastewater, 21st Edition. American Public Health Association, Washington, DC.

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

Great Ships Initiative website: www.greatshipsinitiative.org.

GSI/QAPP/1 - Quality Assurance Project Plan (QAPP) for Great Ships Initiative Bench-Scale and Land-Based Biological Tests (2009).

“H₂O₂ Determination by the I₃⁻ Method and by KMnO₄ Titration”, Analytical Chemistry, Vol. 66, No. 18, September 15, 1994.

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