

## STANDARD OPERATING PROCEDURES LABORATORY

**PROCEDURE TITLE:** Preparation and packaging of TFM Field Standards for Sea Lamprey Control Agents.

**AREA OF APPLICABILITY:** This procedure is applicable to the chemist and chemistry lab technician at the Hammond Bay Biological Station.

**SCOPE:** To describe procedures for preparing and packaging TFM field standards for sea lamprey control agents.

### PRECAUTIONS:

#### A. Safety

1. Standard laboratory safety apparel (lab coats, gloves, and safety glasses) should be worn when handling solvents and chemicals.
2. Review the Safety Data Sheets for chemicals used in this procedure.

### PROCEDURE:

#### A. Reagents used:

1. Saturated sodium borate solution:
  - a. Add ~38g  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$  to 1L deionized water and mix.
  - b. Crystallized sodium borate should remain in the bottom of flask to indicate saturation
  - c. Stability: indefinitely
2. Treatment-grade TFM (~33 % active ingredient as 3-trifluoromethyl-4-nitrophenol)
3. 99% pure analytical grade TFM for making calibration standards
4. HPLC grade methanol
5. HPLC grade water
6. Deionized water for field standard diluent
7. Mobile phase 1: 747 mL HPLC water, 250 mL HPLC methanol, 770 mg ammonium acetate, 3 mL glacial acetic acid
8. Mobile phase 2: 997 mL HPLC methanol, 770 mg ammonium acetate, 3mL glacial acetic acid

## B. Equipment:

1. HPLC with analytical software
2. Waters  $\mu$ Bondapak C-18 reverse phase column (150 x 3.9mm, 10  $\mu$ m)
3. Stand mixer
4. Heat sensitive shrink wrap sized for 250 mL bottles, or tamper evident tape
5. Heat gun for applying shrink wrap
6. Leak proof, polyethylene bottles, 250 mL
7. Latex dispensing line to attach to each container, with some type of flow controlling clamp.

## C. Preparation of standards

1. Fill a large vessel (example: 5-gallon polyethylene carboy with bottom dispensing spigot) with enough deionized water to prepare the desired volume of TFM
2. Add saturated sodium borate buffer in the ratio of 4 mL/L of water
3. Using the following formula, add appropriate amount of technical grade TFM to achieve desired concentration. Add only borate buffer to the 0 mg/L field standard.

$$X \text{ Liters t.g. TFM} = \frac{(\text{desired conc. TFM mg/L})(\text{final volume TFM L})}{\text{conc. stock soln. mg/L}}$$

X=unknown

4. Using the mixer, mix the solution for 5 or more minutes. Ensure complete mixing of any concentrate splashed on inner sides of container by capping and mixing by hand as well. Dispense solution through spigot several times during mixing to avoid leaving unmixed solution in the dispensing line.

## D. Analytical Standard Preparation

1. Prepare TFM stock solution (~1000 mg/L) SOP LAB 423.0
2. Prepare 3 or 4 TFM standards which bracket the expected concentration of the prepared standards (prepared field standards are 4.0, 8.0 and 12.0 mg/L TFM)

E. Verification of concentration procedure

1. Sample each TFM container through the dispensing line.
2. Using HBBS SOP No. LAB 322.7 determine the concentration of the prepared solutions.

F. Adjusting concentration of TFM field standards

1. Depending on results, add deionized water or TFM to achieve desired concentration.
  - a) If the TFM concentration is greater than the desired concentration, add DI water to the TFM field standard.

i. Determine L of DI water to add:

$$X_{\text{final volume}} = \frac{(\text{determined TFM mg/L})(\text{Vol of TFM std})}{\text{Desired TFM mg/L}}$$

$$X_{\text{final volume}} - \text{Vol}_{\text{prepared std}} = \text{Vol of DI water to be added}$$

- b) if the TFM concentration is less than the desired concentration, add TFM to the TFM field standard.

i. Determine mL of TFM to add:

$$\text{Liters conc. stock sol. to be added} = \frac{(\text{desired conc. mg/L} - \text{determined conc. mg/L})(\text{Vol. of TFM std.})}{\text{Conc. stock}}$$

2. If a change is made in the concentration, the standard must be thoroughly mixed again, following procedures in section C4 and again analyzed to determine concentration.
3. Once desired concentration is reached, dispense 10-30 mL of each standard into an appropriate shipping container. One sample from each concentration is sent to Upper Midwest Environmental Science Center, LaCrosse, WI, for confirmation of the concentration.

4. Label the samples as follows: 19UMESC01.04 where the first two digits designate the year, followed by initials UMESC, followed by sample number and a period. Digits following the period describe the concentration of the sample which will be 00, 04, 08, or 12.

G. Packaging procedure

1. After confirmation of concentrations by UMESC, standards are ready to be packaged into 250 mL leak-proof bottles.
2. Dispense each standard and cap in a timely manner to avoid evaporation or contamination.
3. Apply labels formatted as follows: ex: 19LBS031.04 where the first two digits indicate year, followed by initials of receiving control unit, followed by a unique designated serial number and a period. The two digits following the period describe the concentration of the sample in the bottle (00, 04, 08, or 12)
4. Apply the required GHS labels and any necessary safety warnings.
5. Apply shrink wrap using heat gun or apply tamper evident tape.
6. Include documentation of transfer and appropriate safety data sheets with each shipment to a control unit.