TOP:027.4

January 18, 2017

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and

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Ludington Biological Station
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and

Fisheries and Oceans Canada
Sea Lamprey Control Centre
1219 Queen Street East
Sault Ste. Marie, Ontario P6A 2E5
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TECHNICAL OPERATING PROCEDURE

PROCEDURE TITLE:

Verification of Percent and Purity of Active Ingredient in Lampricide Vendor Production Batches

APPLICABILITY:

U.S. Geological Survey (USGS), Hammond Bay Biological Station (HBBS), Millersburg, MI; and the Upper Midwest Environmental Sciences Center (UMESC), La Crosse, WI.

PRINCIPLE:

To document the necessary procedures to verify percent and purity of the active ingredient of vendor production batches of all formulations of lampricides 3-trifluormethyl-4-nitrophenol (TFM) and 2',5-dichloro-4'-nitrosalicylanilide (niclosamide) used to control larval sea lampreys in the Great Lakes and Lake Champlain. This includes TFM, TFM Bar, Bayluscide Technical, Bayluscide 70% Wettable Powder, Bayluscide 20% Emulsifiable Concentrate, and 3.2% Bayluscide Granular Sea Lamprey Larvicide. The active ingredient in all lampricides is either TFM or niclosamide.

SAMPLE COLLECTION AND PRESERVATION:

Sampling of TFM batches--follow the procedures listed in HBBS SOP LAB 330 (Appendix Q). Vendors provide samples of Niclosamide products.

EQUIPMENT REQUIRED:

I. Analytical balances

A. Mettler AT 400

B. Sartorius Model R200D or equivalent five place balance.

II. High Performance Liquid Chromatography system (HPLC)

A. Waters Breeze HPLC System (Waters 1525 binary pump, 2487 detector, 717 autosampler, Breeze software)

B. Agilent 1100 Series HPLC with degasser, binary pumping system, autosampler, column heater, and diode array detector or equivalent HPLC system.

C. Agilent 1260 Series HPLC with degasser, binary pumping system, autosampler, column heater, and diode array detector of equivalent HPLC system.

III. Barnant Series 10 Electric Mixer, model # 700-5400 with a stainless steel dual propeller shaft

IV. Sampling rods and pipettes

V. Spatulas - stainless steel, 23 mm

VI. Volumetric flasks - Class A, 25 - 1000 m

VII. HPLC sample vials - amber or clear glass

IIX. Test tubes (20 mL) with screw caps

IX. One 250 mL plastic jar with screw cap

X. One 1.0 L plastic jar with screw cap

POTENTIAL INTERFERENCES:

Avoid chemical contamination.

SAFETY:

I. Standard laboratory PPE (lab coats, gloves, and safety glasses) should be worn when handling chemicals and solvents.

II. Follow MSDS/SDS for chemicals used in the procedures.

DISPOSAL:

Chemical and solvent wastes produced in the described procedures are considered hazardous and will be transported to an approved hazardous waste disposal facility.

REAGENTS:

I. Reagents used:

A. HPLC grade water

B. HPLC grade methanol

C. Sodium acetate/acetic acid buffer (25 mL glacial acetic acid and 10.526 g sodium acetate trihydrate (reagent grade)) in 1L HPLC grade water

D. HPLC grade dimethylformamide (DMF)

E. HPLC grade acetonitrile

PROCEDURES:

I. Assay of lampricide vendor production batches of TFM

A. Prepare three analytical standards of technical TFM of about 700 mg/L, 1100 mg/L, and 1500 mg/L following procedures in HBBS SOP LAB 423 (Appendix Q).

B. TFM Sample Preparation

1. Tare a 100 mL volumetric flask on the analytical balance.

2. With a graduated, disposable plastic pipette, add approximately 0.3 mL of the TFM sample to the tared flask and record the weight to the nearest 0.1 mg.

3. Dilute to 100.0 mL with deionized or HPLC water and mix well by inverting several times.

4. Transfer about 3 mL of sample from the 100 mL volumetric flask to a 4 mL HPLC vial. Do not fill the HPLC vial to the top.

C. HPLC Analysis of the TFM samples

1. Use HPLC procedure (HBBS SOP LAB 322; Appendix Q) to produce a three-point standard curve from the three analytical TFM standards. The three standards will be used for instrument calibration. The peak area for the TFM sample from the HPLC chromatogram is compared to the three-point standard curve to determine the concentration of TFM in the sample.

D. Calculations

1. % TFM = TFM HPLC concentration (mg/L)*0.1 L/sample weight (mg)*100%

2. TFM percent purity = TFM peak area/total peak area *100%

II. Assay of lampricide vendor production batches of TFM Bar, Bayluscide Technical, Bayluscide 70% Wettable Powder (WP), Bayluscide 20% Emulsifiable Concentrate (EC), and Bayluscide 3.2% Granular Sea Lamprey Larvicide

A. Follow procedures in UMESC SOP AEH 409 and applicable procedures in UMESC SOP AEH 004, AEH 007, AEH 011, AEH 214, AEH 407, AEH 901 (Appendix Q).

REFERENCES:

This procedure has been reviewed and approved by the undersigned representatives of the U.S. Fish and Wildlife Service and Fisheries and Oceans Canada.

REVIEWED/APPROVED_____ DATE 2/13/2017
Field Supervisor (U.S.)

REVIEWED/APPROVED_____ DATE 2/8/2017
Division Manager (Canada)