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### **Spike Witnessing in the Organics Laboratory**

Access to this SOP shall be available within the laboratory for reference purposes; the official copy of this SOP resides on the official Georgia EPD website at <https://epd.georgia.gov/about-us/epd-laboratory-operations>. Printed copies of this SOP will contain a watermark indicating the copy is an uncontrolled copy.

#### **1 Scope and Application**

- 1.1 Spike Witnessing is conducted to assure that valid spiking solutions are added in correct amounts to the appropriate samples and to verify that the spiking procedure is performed correctly. As one analyst prepares samples for analysis, a second analyst observes, verifies and records that spiking solutions are added as appropriate for each sample. This procedure applies to all sample preparations performed in the Organics Laboratory in which surrogate spike solutions or QC spike solutions are added to samples unless otherwise noted.

#### **2 Definitions**

- 2.1 Refer to Section 3 and Section 4 of the Georgia EPD Laboratory Quality Assurance Manual for quality control definitions.
- 2.2 Analyst: In this document, the term “analyst” refers to the individual performing the spike additions.
- 2.3 Witness: In this document, the term “witness” refers to the individual witnessing and verifying the spike additions.

#### **3 Interferences**

- 3.1 Pipettes must be scrupulously cleaned and free of interfering contaminants. Syringes should be rinsed with appropriate solvents prior to use and thoroughly rinsed with one or more appropriate solvents after use.
- 3.2 Contaminants can be introduced by improper handling of pipettes and syringes such as laying on contaminated surfaces or allowing the delivery device to come in contact with a sample.

#### **4 Safety**

- 4.1 Refer to the Laboratory Chemical Hygiene Plan, online revision.

#### **5 Apparatus and Equipment**

- 5.1 Volumetric pipettes: various sizes
- 5.2 Aspirator pipette bulb(s)
- 5.3 Analytical accuracy syringe(s)
- 5.4 Glass beakers: various sizes
- 5.5 Single channel (variable volume) pipettor:
  - 5.5.1 1 – pipettor capable of a measurement from 0.100 ml to 1.00 ml
  - 5.5.2 1 – pipettor capable of a measurement from 1.00 ml to 10.00 ml

## **6 Reagents and Standards**

- 6.1 Appropriate Surrogate, MDL and/or QC spiking solutions

## **7 Sample Collection**

- 7.1 Not Applicable

## **8 Calibration**

- 8.1 Not Applicable

## **9 Quality Control**

- 9.1 Refer to the Quality Control and Quality Assurance criteria for the applicable extraction or sample preparation SOP.
- 9.2 The witness must meet the following criteria:
  - 9.2.1 Must be a member of the Organics Laboratory staff.
  - 9.2.2 Must be a Scientist 1 or higher, an Organics Laboratory Supervisor or Manager.
  - 9.2.3 Must be certified by an Organics Laboratory Supervisor, the Organics Manager. The QA Manager and Laboratory Director may also certify spike witnessing. (*Exception: members of the GCMS Laboratory should spike witness the spiking of samples extracted for BNA analysis whenever possible.*)
    - 9.2.3.1 Certification consists of an Organics Laboratory Supervisor or Manager training the individual who is to be a witness.
    - 9.2.3.2 Certification is completed when a training certificate is completed and filed in the employee's training folder.

## **10 Procedure**

- 10.1 Section 10.1.2 of this SOP applies to extractions and sample preparations performed by the Organics Laboratory except for Trihalomethanes (THMs, SOP 1-027, online revision) and Haloacetic Acids (HAAs, SOP 1-008, online revision). For THMs and HAAs, refer to section 10.1.3 of this SOP.
  - 10.1.1 It is possible that the person who begins an extraction as a witness may not be the person who witnesses all of the spikes performed. Written verification of spike witnessing must be in the form of initials, not merely check marks or Xs, so as to identify who witnessed each spike addition.

- 10.1.1.1 Each extraction or sample preparation form should have a field for each sample where witnesses involved with the extraction can write their initials and verifying spike additions.
- 10.1.2 Extractions and Preparations **other** than THMs and HAAs
- 10.1.2.1 Prior to the addition of any spiking solutions (QC, MDL or Surrogates) to samples (QC or collected), the witness verifies the following:
- 10.1.2.1.1 Syringes and/or pipettes of appropriate volumes and accuracy have been collected and readied. The analyst should indicate to the witness which syringes/pipettes are associated with each spike solution to be used.
- 10.1.2.1.2 Pipettes are of the volumes indicated on the extraction sheet. The witness also verifies that the pipettes are of the appropriate type (i.e. measured as “t.d.” (“To Deliver”) and Class A accuracy).
- 10.1.2.1.3 Syringes and variable volume pipettes are of appropriate sizes for the volumes to be measured (for example, it would not be appropriate for the analyst to select a 1000 $\mu$ L syringe to measure 50 $\mu$ L of solution). It is highly recommended that the use of variable volume pipettes and syringes be avoided, if possible.
- 10.1.2.1.4 Standard Numbers indicated on the extraction sheet must match the Standard Numbers of the solutions used.
- 10.1.2.1.5 Concentrations of the solutions to be used must match any concentrations that may be indicated on the extraction sheet(s). However, the witness is not responsible for that concentration being the appropriate concentration to use unless the information is preprinted on the extraction sheet. The analyst is responsible for selecting the correct concentration spiking solutions and volumes for the work he or she is performing.
- 10.1.2.1.6 Ascertaining whether or not QC spikes contain surrogates so as to prevent “double spiking” of surrogates in samples that also receive QC Spikes. If spike witnessing is otherwise properly performed and documented, double spiking will be recorded and verified and can be taken into account when reporting samples.
- 10.1.2.1.7 Spiking solutions have not expired.
- 10.1.2.2 Upon this verification, the witness writes his or her initials near the volume numbers and Standard Numbers indicated on the extraction sheet.
- 10.1.2.3 When more than one spiking solution is to be used, prior to the first addition, the analyst must indicate to the witness which solution is in current use. When the analyst changes to a different spiking solution, he or she must indicate to the witness this solution and the volume to be added prior to the additions. Throughout the spike witnessing process, the witness must be aware of changes in syringes, pipettes, volumes added or solutions used.

10.1.2.4 Immediately prior to adding a spiking solution to a sample, the analyst will call out the identity of the sample being spiked. The witness will verify the number assigned to the sample on the extraction vessel is actually what the analyst stated. The witness will locate the appropriate field on the extraction form and upon the analyst actually adding the spike to the sample will write his or her initials in that field to verify the addition. This “real time” written verification must be completed before another sample is spiked.

10.1.3 Extractions and Preparations of THMs and HAAs

10.1.3.1 The nature of these extractions makes it unreasonable to have a witness to each and every spiking step. However, these extractions are performed in a way that allows for the analyst to develop a “system” that can aid in the prevention of erroneous spiking. However, QC and MDL Spikes (but not Surrogate Spikes) must be witnessed.

10.1.3.1.1 The analyst must demonstrate to an Organics Laboratory Supervisor or Manager that they can use a technique that is an appropriate substitution for spike witnessing of Surrogate additions. The analyst must be consistent in the application of the system in order to continually assure that the Surrogate Spikes are performed correctly.

10.1.3.1.2 A recommended technique for accomplishing this is a scheme of vial movements (i.e. move a vial to a specific position, add the Surrogate Spike, move the vial to another position, repeat with the next sample). The vials and Styrofoam pieces used to hold them work well for such a scheme.

10.1.3.1.3 QC and MDL Spike additions for THMs and HAAs must be witnessed according to Sec. 10.1.2 above.

**11 Calculations**

11.1 Not Applicable

**12 Waste Management**

12.1 See GA EPD Laboratory SOP – EPD Laboratory Waste Management Standard Operating Procedures, online revision

**13 References**

13.1 Not Applicable

**14 Reporting Limits (RLs), Precision and Accuracy Criteria and Quality Control Approach**

14.1 Not Applicable

**15 Associated LabWorks Test Codes**

15.1 Not Applicable