Laboratory Manager Approval: Ralph Schulz / 08/24/2021

QA Manager Approval: Jeffrey Moore / 08/24/2021

Spike Witnessing in the GC/MS 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 GC/MS Laboratory in which surrogate spike solutions or QC spike solutions are added to samples before extraction 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. (SOP Reference 13.1)

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. (SOP Reference 13.2)

5 Apparatus and Equipment

5.1 Volumetric pipettes: various sizes
5.2 Analytical accuracy syringe(s)
5.3 Glass beakers: various sizes

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 The SOC 10µL fixed volume auto-pipette (exclusively used for 525.2 sample extractions) must be volume verified before use.
8.1.1 A 10µL (0.01ug) aliquot of water is auto-pipetted onto a pan balance
8.1.2 The weight deviation measured at the balance of the aliquot must be between 0.0097ug and 0.0103ug.

9 Quality Control

9.1 Refer to the Quality Control and Quality Assurance criteria for the applicable extraction or sample preparation SOP.
9.2 All syringes must be rinsed multiple times with solvent before their first use.
9.3 If the same syringe is to be used to spike different mixtures, the syringe must be rinsed multiple times with a glass beaker of solvent before inserting into the next spike mixture.
9.4 If the fixed auto-pipette will be used to spike different spike mixtures, a new glass pipette must be attached and rinsed with solvent before inserting into the next spike mixture.
9.5 The witness must meet the following criteria:
9.5.1 Must be a member of the GC/MS Laboratory staff.
9.5.2 Must be a Scientist 1 or higher, a GC/MS Supervisor or Manager.
9.5.3 Must be certified by a GC/MS Laboratory Supervisor or 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.*)
9.5.4 Certification consists of a GC/MS Laboratory Supervisor or Manager training the individual who is to be a witness. A training certificate is completed and filed in the employee's training folder.

10 Procedure

10.1 Section 10.2 of this SOP applies to all extractions and sample spike preparation steps. Specifics to TO-13A extractions are in section 10.3 of this SOP.
10.1.1 The person who serves as a spike witness must witness all of the spikes performed. Written verification of spike witnessing must be in the form of initials on the extraction form to identify who witnessed each spike addition.

10.1.2 Each extraction or sample preparation form should have a blank field for the extraction where the spike witness involved with the extraction can write their initials and verify spike additions.

10.2 Preparation and Procedures for Spike Witness

10.2.1 Prior to the addition of any spiking solutions (QC, MDL or Surrogates) to samples (QC or collected), the analyst must have collected and have ready the syringe(s) and/or the auto-pipette(s) of appropriate volume(s). Due to previous failed extraction results, the lab does not use variable volume auto-pipettes.

10.2.3 The analyst must have ready (from the extraction sheet) the auto-pipette and syringes. They must be of appropriate size for the volumes to be measured (for example, it would not be appropriate for the analyst to select a 1000µL syringe to measure 50µL of solution). The analyst should indicate to the witness which syringes/pipettes are associated with each spike solution to be used.

10.2.4 The analyst must have ready the spike mixture standard vial numbers recorded on the extraction sheet.

10.2.5 The analyst must indicate to the spike witness when more than one spiking solution is to be used for a sample. Prior to the first addition the analyst must indicate to the spike witness which solution he/she is using. When the analyst changes to a different spiking solution, he/she must indicate to the spike witness the new spike vial and the volume to be added. The spike witness must be aware of these changes in syringes, pipettes, volumes added or solutions used for each sample.

10.2.6 The analyst must arrange the samples in the order listed on the extraction sheet to make the spiking easier for the spike witness to follow. The spike witness must review the extraction sheet and verify all the samples being spiked are listed on the extraction sheet

10.2.7 The spike witness must verify that the spiking solutions have not expired.

10.2.8 The spike witness is responsible to match the spike vial number and extraction sheet vial number, and that the vial concentration is the correct concentration as preprinted or filled in by the analyst 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.

10.2.9 The spike witness must monitor the spiking progress and avoid a spiked sample becoming a “double spiked” sample. If the spike witnessing is properly performed and documented, an accidental double spiked sample can still be used by recording the error, verifying the later with instrument analysis of the sample, creating a corrective action, and then mathematically correcting the results before the sample is reported.

10.3 Preparation and Extraction of TO13A Samples

10.3.1 The TO-13A PUF is 2 foam disks with XAD resin beads sandwiched between them.

10.3.2 All the spiking procedures from section 10.2 apply except for the actual spiking process.

10.3.3 For the spiking process, the spike needle must be inserted into the top foam disk, the needle tip must be inserted deep enough to try to contact the XAD resin layer

10.3.4 Caution must be exercised in how far the needle is pushed into the foam, the spike needle cannot be pushed so deeply into the layers that the needle tip could pass though the top foam, the XAD layer, and the bottom foam.

10.3.5 If there are issues with spiking, the top foam disk may be removed, and the tip of spike needle is placed on top of the XAD beads to perform the spike. After the spike the top foam disk is placed back on top of the XAD beads.

10.3.6 If the quartz paper filter is used (i.e. for the method blank sample) the spike is performed before the filter is placed on top of the PUF.

10.3.7 Before the PUF is sent out into the field for collection, only the 2 Field Surrogate compounds are spiked into the PUF. The PUF shipping logbook contains the spike compound information, standard log number, and spike witness signature.

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. (SOP Reference 13.3)

13 References

13.1 GA EPD Laboratory Quality Assurance Plan, online revision.

13.2 GA EPD Laboratory Safety/Chemical Hygiene Plan & Fire Safety Plan, online revision.

13.3 GA EPD Laboratory SOP – EPD Laboratory Waste Management SOP, SOP 6-015, online revision.

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