**High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry**
PCCDs/PCDFs (EPA Method 1613B), PCB Congeners (EPA Method 1668C), and Organochlorine Pesticides (EPA Method 1699)
**Stage 2A Review**
**Data Quality Control (QC)**

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|---|---|
| Site: PHSS-Swan Island Basin | SDG #: B7696 |
| Laboratory: SGS Wilmington (NC) | Project #: DT2002.03.03.03.01 |
| HydroGeoLogic, Inc. Validator: Ken Rapuano | Validation Date: 06.08.23 |
| HGL QC Reviewer: Justin Hersh | Peer Review Date: 07.06.2023 |

| Client Sample ID | Laboratory Sample ID | Matrix |
|---|---|---|
| SIB-SE-AAM131-FRONT-030923 | B7696-001 | Pre-filter & sediment |
| SIB-SE-AAQ005-FRONT-030923 | B7696-002 | Pre-filter & sediment |
| SIB-SE-AAM169-FRONT-030923 | B7696-003 | Pre-filter & sediment |
| SIB-SE-AAM107-FRONT-030923 | B7696-004 | Pre-filter & sediment |
| SIB-SE-AAP957-FRONT-030923 | B7696-005 | Pre-filter & sediment |
| SIB-SW-AAM131-BACK-030923 | B7696-006 | PUF Cartridge |
| SIB-SW-AAQ005-BACK-030923 | B7696-007 | PUF Cartridge |
| SIB-SW-AAM169-BACK-030923 | B7696-008 | PUF Cartridge |
| SIB-SW-AAM107-BACK-030923 | B7696-009 | PUF Cartridge |
| SIB-SW-AAP957-BACK-030923 | B7696-010 | PUF Cartridge |
| SIB-SW-EB03-030923 | B7696-011 | Equipment blank |

The following Stage 2A review was performed on the requested analyses. Results for extraction standards (ES) associated with PCDD/PCDFs, PCB congeners and organochlorine pesticides in sample SIB-SW-AAM107-BACK-030923 results were rejected for all the associated analytes. Analytical completeness is 94% for this SDG.

Narrative and Completeness Review – The case narrative and data package were checked for completeness. No completeness issues were noted. The narrative noted that severe matrix interference was experienced in the extraction and analysis of sample SIB-SW-AAM107-BACK-030923, causing low labeled standard %Rs and elevated sensitivity limits. See Labeled Standards section below for qualification.

*Qualification:* None required.

Sample Delivery and Condition – All samples arrived intact at the laboratory in acceptable condition and temperature and were properly preserved.

*Qualification:* None required.

Holding Times – All samples were prepared and analyzed within their required holding times.

*Qualification:* None required.

Method Blanks – The method 1613B Method Blank was contaminated with 1,2,3,4,7,8-HxCDD and 1,2,3,7,8,9-HxCDD; detections below the qualification threshold should be qualified U. The method 1668C Method Blank was contaminated with multiple PCBs; detections below the qualification threshold should be qualified U. The 1699M Method Blank was free from contamination.

***Qualification:* Detections of contaminated compounds detected below the qualification threshold are qualified U; reason code MBH or MBL.**

Trip Blanks – A trip blank was not submitted with the samples in this SDG.

*Qualification:* None required.

Rinsate Blanks – See Ambient Blank below.

*Qualification:* None required.

Ambient Blanks – Sample SIB-SW-EB03-030923 was identified as an equipment blank. This blank consisted of a PUF cartridge handled like a field sample but not used in the HVS system and then packaged and shipped like an environmental sample; it is only associated with the aqueous phase ("BACK") samples in this SDG. In this regard, it resembles an ambient blank instead of an equipment blank. Due to the anomalous results shown by this blank, which included several PCB congeners detected in the blank at higher concentrations than in any of the associated samples, the project Senior Chemist initiated a series of inquiries with the laboratory and the field team as to a possible source for these anomalous results. The field team indicated that although the PUF cartridge for SIB-SW-EB03-030923 was compromised and returned to the laboratory for reconditioning or disposal, this was not indicated on the chain of custody and the laboratory subsequently received, prepared, and analyzed this compromised PUF cartridge. As the medium for sample SIB-SW-EB03-030923 was compromised, the results for this blank are considered to be unusable and cannot be used to qualify any field sample results.

*Qualification:* None required.

Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) – All LCS/LCSD %Rs and RPDs were within QAPP control limits with the exception of high %Rs for PCDF 1,2,3,4,6,7,8-HpCDF and pesticides O,P'-DDD, P,P'-DDD, and O,P'-DDT.

***Qualification:* Detected results for 1,2,3,4,6,7,8-HpCDF, O,P'-DDD, P,P'-DDD, and O,P'-DDT are qualified J; originally detected results qualified U due to ambient blank contamination receive a final qualifier of UJ; reason code LCSH.**

Labeled Standards – Labeled standards reported by the laboratory as ESs were evaluated as being analogous to surrogates with respect to performing a Stage 2A validation. Based on the guidance in the NFG, if ES %Rs are below the lower limit, associated results are qualified J (detects) or R (non-detects); based on the project Senior Chemist judgment, if a %R is low but is ≥80% of the value of the lower limit, associated ND results may be qualified UJ instead of R. If a labeled standard %R is above the upper limit, detected results are qualified J and non-detected results do not require qualification. The following discrepancies were noted in ES recoveries:

PCDD/PCDFs:

- Sample SIB-SW-AAM107-BACK-030923 had ESs reported below the lower limit for all 17 ESs associated with the target PCDD/PCDFs. All results were reported EDLs elevated above the laboratory's EDLs reported for other samples by factors of ~10 up to ~50x. All PCDD/PCDF results reported for sample SIB-SW-AAM107-BACK-030923 are non-detections and are qualified R-LCLX.

PCB Congeners:

- Sample SIB-SE-AAM131-FRONT-030923 had a %R above the upper limit for ES PCB-155; the associated detected results for PCB-136 and PCB-150 in this sample are qualified J.
- Sample SIB-SE-AAM169-FRONT-030923 had a %R below the lower limit for ES PCB-1; the associated detected result for PCB-1 in this sample is qualified J.
- Sample SIB-SW-AAM107-BACK-030923:
    - Results reported for PCB-16, PCB-17, PCB-18, PCB-19, PCB-24, PCB-27, and PCB-32 as "R" due to a non-calculable %R for ES PCB-19 used to quantify these congeners. The results for these analytes were qualified R-LSLX.

- All other results for labeled standards were recovered low and were below 80% of the lower control limit; all non-detected congener results for this sample are qualified R and all detected congener results are qualified J; reason code LSLX.
- Sample SIB-SW-AAP957-BACK-030923 had a %R below the lower limit for ES PCB-1; the associated detected result for PCB-1 in this sample is qualified J.

Organochlorine Pesticides:

- Samples SIB-SE-AAM131-FRONT-030923 and SIB-SW-AAP957-BACK-030923 had low %Rs for ES dieldrin below 80% of the lower control limit. The detected dieldrin results for both samples are qualified J-LSLX.
- Samples SIB-SE-AAQ005-FRONT-030923, SIB-SE-AAM169-FRONT-030923, SIB-SE-AAP957-FRONT-030923, and SIB-SW-AAM169-BACK-030923 had low %Rs for ES dieldrin above 80% of the lower control limit. The detected dieldrin results for these samples are qualified J-LSL.
- Sample SIB-SE-AAM107-FRONT-030923 has a low %R for ES dieldrin above 80% of the lower control limit. The non-detected dieldrin result for this sample is qualified UJ-LSL.
- Sample SIB-SW-AAM107-BACK-030923 had low %Rs for 5 of the 7 ES compounds, with three of those %Rs below 80% of the lower limit. The narrative noted a strong matrix interference for this sample and the MDLs reported for pesticides in this sample are approximately an order of magnitude above those reported for the other samples; in the judgment of the project Senior Chemist, all pesticide results for this sample should be qualified R, with reason code LSLX or VJ.

***Qualification:*** **See itemized list of qualified results above.**

Matrix Spike/Matrix Spike Duplicate (MS/MSD) – MS/MSD was not performed for this SDG.

*Qualification:* None required.

Field Duplicate – A field duplicate was not submitted with the samples in this SDG.

*Qualification:* None required.

Laboratory Duplicate – A laboratory duplicate was not performed in this SDG.

*Qualification:* None required.

Compound Quantitation – Analyte non-detections were reported on the result summary pages either as ND with the associated MDL or as the MDL in parenthesis. These reporting formats is equivalent to reporting non-detected data as MDL U. Analytes detected between the MDL and RL were reported as J-qualified results by the laboratory. These J qualifiers were retained unless superseded by a more severe qualifier.

If a detected analyte has an ion ration outside the characteristic ion ratio window, the result is reported as an estimated maximum potential concentration. All results reported as an EMPC are qualified J-EMPC unless superseded by a higher priority qualifier.

*Qualification:* None required.

Qualification Summary Table (concentrations in pg/sample):

| Sample | Analyte | Lab Value | Lab Qualifier | Validated Qualifier | Interpreted Qualifier | Reason Code |
|---|---|---|---|---|---|---|
| SIB-SE-AAM131-FRONT-030923 | 1,2,3,4,6,7,8-HpCDF | 182 | -- | J | J | LCSH |
| | PCB-136 | 963 | -- | J | J | LSH |
| | PCB-150 | 11.3 | J | J | J | LSH |
| | o,p'-DDD | 4580 | -- | J | J | LCSH |
| | Dieldrin | 3260 | -- | J | J | LSLX |
| | o,p'-DDT | 9030 | -- | J | J | LCSH |
| SIB-SE-AAQ005-FRONT-030923 | 1,2,3,4,6,7,8-HpCDF | 141 | -- | J | J | LCSH |
| | o,p'-DDD | 315 | J | J | J | LCSH |
| | Dieldrin | 1170 | -- | J | J | LSL |
| | o,p'-DDT | 1390 | J | J | J | LCSH |
| SIB-SE-AAM169-FRONT-030923 | 1,2,3,4,6,7,8-HpCDF | 35.9 | -- | J | J | LCSH |
| | 1,2,3,4,7,8-HxCDD | 6.84 | J B EMPC | UJ | UJ | EMPC,MBL |
| | 1,2,3,7,8,9-HxCDD | 12.7 | J B | U | U | MBL |
| | PCB-1 | 151 | -- | J | J | LSL |
| | o,p'-DDD | 483 | J | J | J | LCSH |
| | Dieldrin | 726 | J | J | J | LSL |
| | o,p'-DDT | 538 | J | J | J | LCSH |
| SIB-SE-AAM107-FRONT-030923 | 1,2,3,4,6,7,8-HpCDF | 12.5 | J EMPC | J | J | EMPC,LCSH |
| | 1,2,3,4,7,8-HxCDD | 4.61 | J B EMPC | UJ | UJ | EMPC,MBL |
| | 1,2,3,7,8,9-HxCDD | 4.55 | J B EMPC | UJ | UJ | EMPC,MBL |
| | PCB-85 | 52.3 | C85 | U | U | MBL |
| | PCB-45 | 13.6 | J B EMPC | UJ | UJ | EMPC,MBL |
| | o,p'-DDD | 209 | J | J | J | LCSH |
| | Dieldrin | 206 | U | UJ | UJ | LSL |

| Sample | Analyte | Lab Value | Lab Qualifier | Validated Qualifier | Interpreted Qualifier | Reason Code |
|---|---|---|---|---|---|---|
| SIB-SE-AAP957-FRONT-030923 | 1,2,3,4,6,7,8-HpCDF | 35.6 | -- | J | J | LCSH |
| | 1,2,3,4,7,8-HxCDD | 5.05 | J B EMPC | UJ | UJ | EMPC,MBL |
| | 1,2,3,7,8,9-HxCDD | 7.31 | J B | U | U | MBL |
| | PCB-1 | 7.44 | J B | U | U | MBL |
| | PCB-3 | 13.2 | J B | U | U | MBL |
| | PCB-4 | 35 | B | U | U | MBL |
| | PCB-19 | 14.8 | J B EMPC | UJ | UJ | EMPC,MBL |
| | Dieldrin | 707 | J | J | J | LSL |
| | o,p'-DDT | 723 | J | J | J | LCSH |
| SIB-SW-AAM131-BACK-030923 | 1,2,3,4,7,8-HxCDD | 2.94 | J B EMPC | UJ | UJ | EMPC,MBL |
| | 1,2,3,7,8,9-HxCDD | 2.29 | J B | U | U | MBL |
| | PCB-174 | 14.6 | J B | U | U | MBL |
| | PCB-179 | 9.05 | J B | U | U | MBL |
| | PCB-180 | 22 | J B C180 | U | U | MBL |
| | PCB-187 | 16.3 | J B EMPC | UJ | UJ | EMPC,MBL |
| | PCB-129 | 72.8 | B C129 | U | U | MBL |
| | PCB-132 | 30.8 | B | U | U | MBL |
| | PCB-135 | 42.9 | B C135 | U | U | MBL |
| | PCB-158 | 6.63 | J B EMPC | UJ | UJ | EMPC,MBL |
| | PCB-83 | 9.8 | J B | U | U | MBL |
| | PCB-85 | 27.5 | J B C85 | U | U | MBL |
| | PCB-35 | 8.81 | J B | U | U | MBL |
| SIB-SW-AAQ005-BACK-030923 | 1,2,3,4,6,7,8-HpCDF | 12.9 | J EMPC | J | J | EMPC,LCSH |
| | o,p'-DDT | 381 | J EMPC | J | J | EMPC,LCSH |
| SIB-SW-AAM169-BACK-030923 | 1,2,3,4,6,7,8-HpCDF | 3.72 | J | J | J | LCSH |
| | PCB-189 | 7.5 | J B | U | U | MBL |
| | o,p'-DDD | 1360 | J | J | J | LCSH |
| | Dieldrin | 3250 | -- | J | J | LSL |

| Sample | Analyte | Lab Value | Lab Qualifier | Validated Qualifier | Interpreted Qualifier | Reason Code |
|---|---|---|---|---|---|---|
| SIB-SW-AAM107-BACK-030923 | All ND PCDD/PCDF results | varies | U | R | R | LSLX |
| | All ND PCB congener results | varies | U | R | R | LSLX |
| | o,p'-DDD | 968 | U | R | R | LSL,VJ |
| | Dieldrin | 2860 | U | R | R | LSLX |
| | o,p'-DDE | 1510 | U | R | R | LSLX |
| | o,p'-DDT | 1520 | U | R | R | LSL,VJ |
| | p,p'-DDD | 1090 | U | R | R | VJ |
| | p,p'-DDE | 802 | U | R | R | LSLX |
| | p,p'-DDT | 1550 | U | R | R | VJ |
| SIB-SW-AAP957-BACK-030923 | 1,2,3,4,6,7,8-HpCDF | 7.98 | J EMPC | J | J | EMPC,LCSH |
| | PCB-1 | 3440 | -- | J | J | LSL |
| | PCB-4 | 172000 | E | J | J | ACR |
| | o,p'-DDD | 1550 | J | J | J | LCSH |
| | Dieldrin | 2350 | -- | J | J | LSLX |