High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry
PCCDs/PCDFs (EPA Method 1613B)
Stage 2A Review
Data Quality Control (QC)

Site: PHSS-Swan Island Basin	SDG #: 20543
Laboratory: CFA	Project #: DT2002.03.03.04.01
HydroGeoLogic, Inc. Validator: John Tracy	Validation Date: 07.19.23
HGL Peer Reviewer: Ken Rapuano	Peer Review Date: 8.3.23

Client Sample ID	Laboratory Sample ID	Matrix
SIB-111-TOB-0-1-10052022	20543001	Riverbank Soil
SIB-110-MLW-0-1-10052022	20543002	Riverbank Soil
SIB-110-TOB-0-1-10052022	20543003	Riverbank Soil
SIB-109-OHW-0-1-10052022	20543004	Riverbank Soil
SIB-109-TOB-0-1-10052022	20543005	Riverbank Soil
SIB-108-TOB-0-1-10052022	20543006	Riverbank Soil
SIB-107-OHW-0-1-10052022	20543007	Riverbank Soil
SIB-107-TOB-0-1-10052022	20543008	Riverbank Soil
SIB-106-OHW-0-1-10052022	20543009	Riverbank Soil
SIB-106-TOB-0-1-10052022	20543010	Riverbank Soil
SIB-105-OHW-0-1-10052022	20543011	Riverbank Soil
SIB-105-TOB-0-1-10052022	20543012	Riverbank Soil

The following Stage 2A review was performed on the requested analyses. One result was rejected, and analytical completeness is 99.5%.

Narrative and Completeness Review – The case narrative and data package were checked for completeness. No completeness issues were noted.

Qualification: None required.

Sample Delivery and Condition – With the exception of several labeling discrepancies, which were resolved, all samples arrived intact at the laboratory in acceptable condition and temperature and were properly preserved. No other discrepancies were noted.

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 the following analytes:

- 0.48 pg/g of 1,2,3,4,6,7,8-HpCDD, leading to a qualification limit of 2.4 pg/g (24 pg/g for 10x dilutions)
- 2.31 pg/g of 1,2,3,4,6,7,8,9-OCDD, leading to a qualification limit of 11.55 pg/g (115.5 pg/g for 10x dilutions)
- 0.118 pg/g of 1,2,3,4,7,8-HxCDF, leading to a qualification limit of 0.59 pg/g (5.9 pg/g for 10x dilutions)
- 0.076 pg/g of 1,2,3,6,7,8-HxCDF, leading to a qualification limit of 0.38 pg/g (3.8 pg/g for 10x dilutions)

- 0.328 pg/g of 1,2,3,4,6,7,8-HpCDF, leading to a qualification limit of 1.64 pg/g (16.4 pg/g for 10x dilutions)
- 0.43 pg/g of 1,2,3,4,6,7,8,9-OCDF leading to a qualification limit 2.15 pg/g (21.5 pg/g for 10x dilutions)

All detections below qualification limits should be qualified U-MBL.

> ***Qualification:*** **The 1,2,3,4,7,8-HxCDF results in samples SIB-110-MLW-0-1-10052022, SIB-110-TOB-0-1-10052022, and SIB-109-OHW-0-1-10052022 are qualified U-MBL. The 1,2,3,6,7,8-HxCDF results in samples SIB-110-MLW-0-1-10052022, SIB-110-TOB-0-1-10052022, and SIB-109-OHW-0-1-10052022 are qualified U-MBL. The 1,2,3,4,6,7,8-HpCDF result in sample SIB-110-MLW-0-1-10052022 is qualified U-MBL. The 1,2,3,4,6,7,8,9-OCDF result in sample SIB-110-MLW-0-1-10052022 is qualified U-MBL.**

Rinsate Blanks – Rinse blank EB01-10052022 (results reported in SDG 20574) is associated with all samples in this SDG and was free from contamination.

> *Qualification:* None required.

Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) – All LCS/LCSD %Rs and RSDs were within QAPP control limits.

> *Qualification:* None required.

Surrogates – All surrogates (labeled standards) were within control limits with the exception of sample SIB-109-OHW-0-1-10052022. Labeled standards 13C-OCDD, 13C-1,2,3,7,8,9-HxCDF, 13C-1,2,3,4,6,7,8-HpCDF, and 13C-1,2,3,4,7,8,9-HpCDF recovered below control limits. Associated analytes should be qualified J/R-LSL.

> ***Qualification:*** **In sample SIB-109-OHW-0-1-10052022, the 1,2,3,4,6,7,8,9-OCDD, 1,2,3,4,6,7,8-HpCDF, and 1,2,3,4,7,8,9-HpCDF results are qualified J-LSL and the 1,2,3,7,8,9-HxCDF result is qualified R-LSL.**

Matrix Spike/Matrix Spike Duplicate (MS/MSD) – An MS/MSD was performed using sample SIB-111-TOB-0-1-10052022. All %Rs were within control limits with a few exceptions. 1,2,3,4,6,7,8-HpCDD and 1,2,3,4,6,7,8,9-OCDD recovered below control limits in the MS and MSD, however, the sample concentration was >4x the spike concentration and the %R results are not applicable. Additionally, 1,2,3,6,7,8-HxCDD, 1,2,3,4,6,7,8-HpCDF, and 1,2,3,4,6,7,8,9-OCDF recovered below control limits in the MS and/or MSD and these results in the parent sample should be qualified J-MSL. All RPDs were within QAPP control limits.

> ***Qualification:*** **In sample SIB-111-TOB-0-1-10052022, the results for 1,2,3,6,7,8-HxCDD, 1,2,3,4,6,7,8-HpCDF, and 1,2,3,4,6,7,8,9-OCDF are qualified J-MSL.**

Field Duplicate – No field duplicates were submitted with this SDG.

> *Qualification:* None required.

Compound Quantitation – Analyte non-detections were reported as the EDL and qualified U. Analytes detected between the EDL and PQL 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 (a “K” flag is present in the laboratory qualifier) are qualified J-EMPC unless superseded by a higher priority qualifier.

2,3,7,8-TCDF is not fully resolved on the DB-5MS column; when detected above the PQL on the DB-5MS column, the result was confirmed on a DB-225 column using the instrument ID listed in the case narrative. In cases where two results are reported for 2,3,7,8-TCDF for a sample, the result from the DB-225 column is selected for use and the result from the DB-5MS is qualified DNR-EXC.

In cases where a target analyte is detected at a concentration above the calibrated range, the laboratory's practice is to report the result with a laboratory qualifier of E without running a dilution so long as the detector is not saturated. The OCDD results for 3 affected samples are qualified J-ACR.

Qualification: **The following results are qualified:**

- **All results reported with a laboratory qualifier of K are qualified J with reason code EMPC.**
- **All 2,3,7,8-TCDF results reported from the DB-5MS column that are paired with a 2,3,7,8-TCDF result from the DB-225 column are qualified DNR, reason code EXC; ; note that the reportable_result field for these results are populated with "Yes" by the laboratory and are changed to "No" for the affected results.**
- **3 OCDD results reported with a laboratory qualifier of E are qualified J with reason code ACR; note that the reportable_result field for these results are populated with "No" by the laboratory and are changed to "Yes" for the affected results.**
- **Two Heptachloro-p-dioxin results reported with a laboratory qualifier of E have the reportable_result field populated with "No" by the laboratory and are changed to "Yes". Although total congener results are not validated, this results are reportable.**

Qualification Summary Table (results in pg/g):

Sample	Analyte	Lab Value	Lab Qualifier	Validated Value	Validated Qualifier	Reason Code
SIB-111-TOB-0-1-10052022	1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN	102	--	102	J	MSL
SIB-111-TOB-0-1-10052022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	90.9	--	90.9	J	MSL
SIB-111-TOB-0-1-10052022	2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN	0.466	JK	0.466	J	EMPC
SIB-111-TOB-0-1-10052022	OCTACHLORODIBENZOFURAN	58.4	--	58.4	J	MSL
SIB-111-TOB-0-1-10052022	OCTACHLORODIBENZO-P-DIOXIN	11300	E	11300	J	ACR
SIB-110-MLW-0-1-10052022	1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN	1.41	BJ	1.41	U	MBL
SIB-110-MLW-0-1-10052022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	0.205	BJ	0.205	U	MBL
SIB-110-MLW-0-1-10052022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	0.217	BJ	0.217	U	MBL
SIB-110-MLW-0-1-10052022	1,2,3,7,8,9-HEXACHLORODIBENZO-P-DIOXIN	0.332	JK	0.332	J	EMPC
SIB-110-MLW-0-1-10052022	OCTACHLORODIBENZOFURAN	1.35	BJ	1.35	U	MBL
SIB-110-TOB-0-1-10052022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	0.534	BJ	0.534	U	MBL
SIB-110-TOB-0-1-10052022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	0.292	BJ	0.292	U	MBL
SIB-110-TOB-0-1-10052022	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN	0.288	JK	0.288	J	EMPC
SIB-109-OHW-0-1-10052022	1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN	42.9	J	42.9	J	LSL
SIB-109-OHW-0-1-10052022	1,2,3,4,7,8,9-HEPTACHLORODIBENZOFURAN	4.3	JK	4.3	J	LSL,EMPC
SIB-109-OHW-0-1-10052022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	3.52	J	3.52	U	MBL
SIB-109-OHW-0-1-10052022	1,2,3,4,7,8-HEXACHLORODIBENZO-P-DIOXIN	7.09	JK	7.09	J	EMPC
SIB-109-OHW-0-1-10052022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	1.98	J	1.98	U	MBL
SIB-109-OHW-0-1-10052022	1,2,3,7,8,9-HEXACHLORODIBENZOFURAN	2.6	QU	--	R	LSL
SIB-109-OHW-0-1-10052022	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN	5.22	JK	5.22	J	EMPC
SIB-109-OHW-0-1-10052022	2,3,7,8-TETRACHLORODIBENZOFURAN	2.48	JK	2.48	J	EMPC
SIB-109-OHW-0-1-10052022	OCTACHLORODIBENZO-P-DIOXIN	3650	--	3650	J	LSL
SIB-109-TOB-0-1-10052022	2,3,7,8-TETRACHLORODIBENZOFURAN	2.19	--	2.19	DNR	EXC
SIB-109-TOB-0-1-10052022	OCTACHLORODIBENZO-P-DIOXIN	6170	E	6170	J	ACR
SIB-108-TOB-0-1-10052022	2,3,7,8-TETRACHLORODIBENZOFURAN	0.417	JK	0.417	J	EMPC
SIB-107-OHW-0-1-10052022	2,3,7,8-TETRACHLORODIBENZOFURAN	2.25	--	2.25	DNR	EXC
SIB-107-TOB-0-1-10052022	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN	2.57	K	2.57	J	EMPC
SIB-107-TOB-0-1-10052022	2,3,7,8-TETRACHLORODIBENZOFURAN	1.8	--	1.8	DNR	EXC
SIB-107-TOB-0-1-10052022	2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN	0.543	K	0.543	J	EMPC

Sample	Analyte	Lab Value	Lab Qualifier	Validated Value	Validated Qualifier	Reason Code
SIB-107-TOB-0-1-10052022	OCTACHLORODIBENZO-P-DIOXIN	4080	E	4080	J	ACR
SIB-106-OHW-0-1-10052022	2,3,4,6,7,8-HEXACHLORODIBENZOFURAN	1.21	JK	1.21	J	EMPC
SIB-106-OHW-0-1-10052022	2,3,4,7,8-PENTACHLORODIBENZOFURAN	0.918	JK	0.918	J	EMPC
SIB-106-OHW-0-1-10052022	2,3,7,8-TETRACHLORODIBENZOFURAN	0.75	JK	0.75	J	EMPC
SIB-106-TOB-0-1-10052022	2,3,7,8-TETRACHLORODIBENZOFURAN	0.746	JK	0.746	J	EMPC
SIB-106-TOB-0-1-10052022	2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN	0.488	JK	0.488	J	EMPC
SIB-105-OHW-0-1-10052022	2,3,7,8-TETRACHLORODIBENZOFURAN	0.329	JK	0.329	J	EMPC
SIB-105-TOB-0-1-10052022	2,3,7,8-TETRACHLORODIBENZOFURAN	1.19	--	1.19	DNR	EXC
SIB-105-TOB-0-1-10052022	2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN	0.781	K	0.781	J	EMPC