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 #: 20693
Laboratory: CFA	Project #: DT2002.03.03.04.01
HydroGeoLogic, Inc. Validator: John Tracy	Validation Date: 07.28.23
HGL Peer Reviewer: Ken Rapuano	Peer Review Date: 8.4.23

Client Sample ID	Laboratory Sample ID	Matrix
SIB-029-OHW-0-1-10252022	20693001	Riverbank Soil
SIB-028-OHW-0-1-10252022	20693002	Riverbank Soil
SIB-027-OHW-0-1-10252022	20693003	Riverbank Soil
SIB-026-OHW-0-1-10252022	20693004	Riverbank Soil
SIB-025-OHW-0-1-10252022	20693005	Riverbank Soil
SIB-024-OHW-0-1-10252022	20693006	Riverbank Soil
SIB-023-OHW-0-1-10252022	20693007	Riverbank Soil
SIB-022-OHW-0-1-10252022	20693009	Riverbank Soil
SIB-125-OHW-0-1-10262022	20693013	Riverbank Soil
SIB-125-MLW-0-1-10262022	20693014	Riverbank Soil
SIB-124-TOB-0-1-10262022	20693015	Riverbank Soil
SIB-124-OHW-0-1-10262022	20693016	Riverbank Soil
SIB-124-MLW-0-1-10262022	20693017	Riverbank Soil
SIB-123-TOB-0-1-10262022	20693018	Riverbank Soil
SIB-123-TOB-0-1-10262022 MS	20693019	Riverbank Soil
SIB-123-TOB-0-1-10262022 MSD	20693020	Riverbank Soil
SIB-123-OHW-0-1-10262022	20693021	Riverbank Soil

The following Stage 2A review was performed on the requested analyses. No results were rejected, and analytical completeness is 100%.

Narrative and Completeness Review – The case narrative and data package were checked for completeness. The laboratory narrative noted that results for sample SIB-125-TOB-0-1-10262022 and the associated MS and MSD (logged in as laboratory sample IDs 20693010, 20693011, and 20693012, respectively) are not provided in this package due to a spiking error resulting in no extraction standards. The results for these samples were provided in a revised data report made available to the data validation team on 9.25.23.

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 associated with preparation batch 52436 was free from contamination with the following exceptions. All detections below qualification limits should be qualified U-MBL. See table below.

Analyte	Result (pg/g)	Qualification Limit (pg/g)
1,2,3,4,6,7,8-HpCDD	0.73	3.65
1,2,3,4,6,7,8,9-OCDD	5.21	26.05
1,2,3,4,7,8-HxCDF	0.174	0.87
1,2,3,4,6,7,8-HpCDF	0.686	3.43
1,2,3,4,6,7,8,9-OCDF	1.01	5.05

The method blank associated with batch 52782 was contaminated with 1.82 pg/g OCDD and 0.262 pg/g, leading to qualification limits of 9.1 pg/g and 1.31 pg/g, respectively.

All detected results in the samples associated with these two method blanks were greater than the qualification limit and no qualification was required.

Qualification: None required.

Rinsate Blanks – Rinse blank EB04-10262022 (results reported in SDG 20699) is associated with all samples in this SDG and was free from contamination with the exception of 1,2,3,4,6,7,8,9-OCDD, which was detected at 3.95 pg/L leading to a qualification limit of 1.975 pg/g. All detections are above the qualification limit and no qualification is required.

Qualification: None required.

Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) – The LCS/LCSD associated with batch 52436 had all %Rs within QAPP control limits; all LCS/LCSD RPDs were within QAPP control limits with the exception of 1,2,3,6,7,8-HxCDD and 2,3,7,8-TCDF. Detections for these analytes in all samples should be qualified J-LCSP. The LCS/LCSD associated with batch 52782 had all %Rs and RPDs in control.

***Qualification:* The 1,2,3,6,7,8-HxCDD results in all samples and 2,3,7,8-TCDF results in samples SIB-125-OHW-0-1-10262022, SIB-124-MLW-0-1-10262022, SIB-123-TOB-0-1-10262022, and SIB-123-OHW-0-1-10262022 are qualified J-LCSP.**

Surrogates – All surrogates (labeled standards) were within control limits.

Qualification: None required.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) – An MS/MSD was performed using sample SIB-123-TOB-0-1-10262022. All %Rs were within QAPP control limits, with the exception of 1,2,3,4,6,7,8-HpCDD and 1,2,3,4,6,7,8,9-OCDD, which recovered outside control limits in the MS and/or MSD, however, the sample concentrations were >4x the spike concentration and the %R results are not applicable. All RPDs were within control limits.

An MS/MSD was performed using sample SIB-125-TOB-0-1-10262022. All %Rs were within QAPP control limits, with the exception of high %Rs for 1,2,3,4,6,7,8-HpCDD, OCDD, and OCDF which recovered above control limits in the MS and/or MSD. The sample concentration for OCDD was >4x the spike concentration and the %R results are not applicable. All RPDs were within control limits. The 1,2,3,4,6,7,8-HpCDD and OCDF results for sample SIB-125-TOB-0-1-10262022 should be qualified J-MSH.

Qualification: **The 1,2,3,4,6,7,8-HpCDD and OCDF results for sample SIB-125-TOB-0-1-10262022 are qualified J, reason code MSH.**

Field Duplicate – Parent sample SIB-125-MLW-0-1-10262022 was analyzed with this SDG, while its duplicate, FD15-10262022, was analyzed with SDG 20694. All precision criteria were met with the exception of 1,2,3,4,6,7,8-HpCDD, which failed RPD criteria and should be qualified J-FDPR.

Parent sample SIB-123-TOB-0-1-10262022 was analyzed with this SDG, while its duplicate, FD18-10262022, was analyzed with SDG 20694. All precision criteria were met.

Parent sample SIB-125-TOB-0-1-10262022 was analyzed with this SDG, while its duplicate, FD16-10262022 was analyzed with SDG 20694. All precision criteria were met with the exception of 1,2,3,4,6,7,8-HpCDF and 1,2,3,4,6,7,8,9-OCDF, which failed RPD criteria and should be qualified J-FDPR.

Qualification: **In sample SIB-125-MLW-0-1-10262022, the 1,2,3,4,6,7,8-HpCDD result is qualified J-FDPR. In sample SIB-125-TOB-0-1-10262022, the 1,2,3,4,6,7,8-HpCDF and 1,2,3,4,6,7,8,9-OCDF results are qualified J-FDPR.**

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 7 affected samples are qualified J-ACR.

Sample SIB-125-OHW-0-1-10262022 was originally reported from a 1x dilution with several analytes having a laboratory qualifier of E. When the laboratory submitted a revised laboratory report and EDD due to the issue with sample SIB-125-TOB-0-1-10262022 (see Narrative and Completeness Review above), the laboratory also revised the reported results for sample SIB-125-OHW-0-1-10262022 to include a 5x diluted analysis but did not include the 1x analysis originally reported. The revised EDD, however, included results for both levels of dilution performed on sample SIB-125-OHW-0-1-10262022. The HGL reviewer qualified the 1,2,3,4,6,7,8-HpCDD and OCDD results reported from the 1x dilution with DNR-EXC and retained the laboratory-applied “No” in the reportable_result field for both analytes. The HGL reviewer also retained the laboratory-applied “No” in the reportable_result field for the total HpCDDs and total HpCDFs reported from the 1x analysis. The 1,2,3,4,6,7,8-HpCDD, OCDD, total HpCDDs and total HpCDFs results from the 5x analysis were retained with no qualification and reportable_result of “Yes”. All other target PCDD/PCDFs in the 5x dilution were qualified DNR-EXC with reportable_result of “No”; all total homologues other than HpCDDs and HpCDFs had reportable_result changed to “No” but were not qualified.

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.

- **7 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.**
- **Three Total HpCDFs results and one 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, these results are reportable.**
- **The 1,2,3,4,6,7,8-HpCDD and OCDD results from the 1x dilution of sample SIB-125-OHW-0-1-10262022 are qualified DNR-EXC.**
- **The 1,2,3,4,6,7,8-HpCDD, OCDD, total HpCDDs, and total HpCDFs results from the 5x dilution of sample SIB-125-OHW-0-1-10262022 are usable and retain the reportable_result of "Yes". All other target PCDD/PCDFs in the 5x dilution were qualified DNR-EXC with reportable_result of "No"; all total homologues other than HpCDDs and HpCDFs had reportable_result changed to "No" but were not qualified.**

Qualification Summary Table (results in pg/g):

Sample	Analyte	Lab Value	Lab Qualifier	Validated Value	Validated Qualifier	Reason Code
SIB-029-OHW-0-1-10252022	1,2,3,4,7,8,9-HEPTACHLORODIBENZOFURAN	523	K	523	J	EMPC
SIB-029-OHW-0-1-10252022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	108	JK	108	J	EMPC
SIB-029-OHW-0-1-10252022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	631	K	631	J	LCSP,EMPC
SIB-028-OHW-0-1-10252022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	142	K	142	J	EMPC
SIB-028-OHW-0-1-10252022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	327	--	327	J	LCSP
SIB-028-OHW-0-1-10252022	OCTACHLORODIBENZO-P-DIOXIN	114000	E	114000	J	ACR
SIB-027-OHW-0-1-10252022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	2.91	JK	2.91	J	EMPC
SIB-027-OHW-0-1-10252022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	3.81	J	3.81	J	LCSP
SIB-027-OHW-0-1-10252022	2,3,4,6,7,8-HEXACHLORODIBENZOFURAN	1.63	JK	1.63	J	EMPC
SIB-026-OHW-0-1-10252022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	3.76	JK	3.76	J	EMPC
SIB-026-OHW-0-1-10252022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	18.4	--	18.4	J	LCSP
SIB-026-OHW-0-1-10252022	1,2,3,7,8,9-HEXACHLORODIBENZOFURAN	2.32	JK	2.32	J	EMPC
SIB-026-OHW-0-1-10252022	1,2,3,7,8,9-HEXACHLORODIBENZO-P-DIOXIN	4.07	JK	4.07	J	EMPC
SIB-026-OHW-0-1-10252022	2,3,4,6,7,8-HEXACHLORODIBENZOFURAN	3.98	JK	3.98	J	EMPC
SIB-026-OHW-0-1-10252022	2,3,4,7,8-PENTACHLORODIBENZOFURAN	1.06	JK	1.06	J	EMPC
SIB-026-OHW-0-1-10252022	OCTACHLORODIBENZO-P-DIOXIN	7170	E	7170	J	ACR
SIB-025-OHW-0-1-10252022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	71	JK	71	J	EMPC
SIB-025-OHW-0-1-10252022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	245	JK	245	J	LCSP,EMPC
SIB-024-OHW-0-1-10252022	1,2,3,4,7,8,9-HEPTACHLORODIBENZOFURAN	79	K	79	J	EMPC
SIB-024-OHW-0-1-10252022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	36.7	JK	36.7	J	EMPC
SIB-024-OHW-0-1-10252022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	20.4	JK	20.4	J	EMPC
SIB-024-OHW-0-1-10252022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	96.3	--	96.3	J	LCSP
SIB-024-OHW-0-1-10252022	2,3,4,6,7,8-HEXACHLORODIBENZOFURAN	15.6	JK	15.6	J	EMPC
SIB-024-OHW-0-1-10252022	OCTACHLORODIBENZO-P-DIOXIN	46500	E	46500	J	ACR
SIB-023-OHW-0-1-10252022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	18.1	JK	18.1	J	EMPC
SIB-023-OHW-0-1-10252022	1,2,3,4,7,8-HEXACHLORODIBENZO-P-DIOXIN	11.3	JK	11.3	J	EMPC
SIB-023-OHW-0-1-10252022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	45.9	J	45.9	J	LCSP
SIB-022-OHW-0-1-10252022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	16.7	--	16.7	J	LCSP
SIB-022-OHW-0-1-10252022	1,2,3,7,8,9-HEXACHLORODIBENZOFURAN	2.46	JK	2.46	J	EMPC

Sample	Analyte	Lab Value	Lab Qualifier	Validated Value	Validated Qualifier	Reason Code
SIB-022-OHW-0-1-10252022	OCTACHLORODIBENZO-P-DIOXIN	8990	E	8990	J	ACR
SIB-125-TOB-0-1-10262022	1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN	63.4	--	63.4	J	FDPR
SIB-125-TOB-0-1-10262022	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN	260	--	260	J	MSH
SIB-125-TOB-0-1-10262022	1,2,3,7,8,9-HEXACHLORODIBENZOFURAN	0.857	JK	0.857	J	EMPC
SIB-125-TOB-0-1-10262022	1,2,3,7,8,9-HEXACHLORODIBENZO-P-DIOXIN	5.2	K	5.2	J	EMPC
SIB-125-TOB-0-1-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	4.17	--	4.17	DNR	EXC
SIB-125-TOB-0-1-10262022	OCTACHLORODIBENZOFURAN	190	--	190	J	MSH,FDPR
SIB-125-OHW-0-1-10262022	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN	2020	E	2020	DNR	EXC
SIB-125-OHW-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	62.8	--	62.8	J	LCSP
SIB-125-OHW-0-1-10262022	1,2,3,7,8,9-HEXACHLORODIBENZOFURAN	14.6	K	14.6	J	EMPC
SIB-125-OHW-0-1-10262022	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN	4.84	K	4.84	J	EMPC
SIB-125-OHW-0-1-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	3.38	K	3.38	J	LCSP,EMPC
SIB-125-OHW-0-1-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	5.21	--	5.21	DNR	EXC
SIB-125-OHW-0-1-10262022	2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN	0.744	K	0.744	J	EMPC
SIB-125-OHW-0-1-10262022	OCTACHLORODIBENZO-P-DIOXIN	13700	E	13700	DNR	EXC
SIB-125-OHW-0-1-10262022 (5x dilution)	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN	2020	--	2020	--	--
	OCTACHLORODIBENZO-P-DIOXIN	13700	--	13700	--	--
	All other results	varies	varies	varies	DNR	EXC
SIB-125-MLW-0-1-10262022	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN	30.3	--	30.3	J	FDPR
SIB-125-MLW-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	1.7	J	1.7	J	LCSP
SIB-124-TOB-0-1-10262022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	17.2	JK	17.2	J	EMPC
SIB-124-TOB-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	8.13	JK	8.13	J	EMPC
SIB-124-TOB-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	93.7	K	93.7	J	LCSP,EMPC
SIB-124-TOB-0-1-10262022	1,2,3,7,8,9-HEXACHLORODIBENZO-P-DIOXIN	47.2	JK	47.2	J	EMPC
SIB-124-TOB-0-1-10262022	OCTACHLORODIBENZO-P-DIOXIN	55300	E	55300	J	ACR
SIB-124-OHW-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	100	--	100	J	LCSP
SIB-124-OHW-0-1-10262022	1,2,3,7,8-PENTACHLORODIBENZOFURAN	10.4	JK	10.4	J	EMPC
SIB-124-MLW-0-1-10262022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	7.33	K	7.33	J	EMPC

Sample	Analyte	Lab Value	Lab Qualifier	Validated Value	Validated Qualifier	Reason Code
SIB-124-MLW-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	3.62	JK	3.62	J	EMPC
SIB-124-MLW-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	7.72	--	7.72	J	LCSP
SIB-124-MLW-0-1-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	0.646	JK	0.646	J	LCSP,EMPC
SIB-124-MLW-0-1-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	1.36	--	1.36	DNR	EXC
SIB-123-TOB-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	2.11	JK	2.11	J	EMPC
SIB-123-TOB-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	11.3	--	11.3	J	LCSP
SIB-123-TOB-0-1-10262022	2,3,4,7,8-PENTACHLORODIBENZOFURAN	2.16	JK	2.16	J	EMPC
SIB-123-TOB-0-1-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	2.07	--	2.07	J	LCSP
SIB-123-TOB-0-1-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	2.58	--	2.58	DNR	EXC
SIB-123-TOB-0-1-10262022	2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN	0.379	JK	0.379	J	EMPC
SIB-123-OHW-0-1-10262022	1,2,3,4,7,8,9-HEPTACHLORODIBENZOFURAN	4.47	JK	4.47	J	EMPC
SIB-123-OHW-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	3.8	JK	3.8	J	EMPC
SIB-123-OHW-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	40.2	--	40.2	J	LCSP
SIB-123-OHW-0-1-10262022	1,2,3,7,8,9-HEXACHLORODIBENZOFURAN	3.4	JK	3.4	J	EMPC
SIB-123-OHW-0-1-10262022	1,2,3,7,8,9-HEXACHLORODIBENZO-P-DIOXIN	26.2	K	26.2	J	EMPC
SIB-123-OHW-0-1-10262022	2,3,4,6,7,8-HEXACHLORODIBENZOFURAN	4.82	JK	4.82	J	EMPC
SIB-123-OHW-0-1-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	3.34	--	3.34	J	LCSP
SIB-123-OHW-0-1-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	3.45	K	3.45	DNR	EXC
SIB-123-OHW-0-1-10262022	OCTACHLORODIBENZO-P-DIOXIN	8620	E	8620	J	ACR