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 #: 20694
Laboratory: CFA	Project #: DT2002.03.03.04.01
HydroGeoLogic, Inc. Validator: John Tracy	Validation Date: 08.15.23
HGL Peer Reviewer: Ken Rapuano	Peer Review Date: 7.28.23

Client Sample ID	Laboratory Sample ID	Matrix
SIB-122-TOB-0-1-10262022	20694001	Riverbank Soil
SIB-122-TOB-0-1-10262022 MS	20694002	Riverbank Soil
SIB-122-TOB-0-1-10262022 MSD	20694003	Riverbank Soil
SIB-122-OHW-0-1-10262022	20694004	Riverbank Soil
SIB-122-MLW-0-1-10262022	20694005	Riverbank Soil
SIB-122-MLW-0-1-10262022 MS	20694006	Riverbank Soil
SIB-122-MLW-0-1-10262022 MSD	20694007	Riverbank Soil
SIB-121-TOB-0-1-10262022	20694008	Riverbank Soil
SIB-121-TOB-0-1-10262022 MS	20694009	Riverbank Soil
SIB-121-TOB-0-1-10262022 MSD	20694010	Riverbank Soil
SIB-121-OHW-0-1-10262022	20694011	Riverbank Soil
SIB-121-OHW-0-1-10262022 MS	20694012	Riverbank Soil
SIB-121-OHW-0-1-10262022 MSD	20694013	Riverbank Soil
SIB-121-MLW-0-1-10262022	20694014	Riverbank Soil
FD15-10262022	20694015	Riverbank Soil
FD16-10262022	20694016	Riverbank Soil
FD17-10262022	20694017	Riverbank Soil
FD18-10262022	20694018	Riverbank Soil
FD19-10262022	20694019	Riverbank Soil
FD20-10262022	20694020	Riverbank Soil
SIB-022-MLW-0-1-10252022	20694021	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. 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 bank was contaminated with several target analytes including the following:

- 1,2,3,4,6,7,8-HpCDD at 0.312 pg/g, leading to a qualification limit of 1.56 pg/g
- 1,2,3,4,6,7,8,9-OCDD at 1.15 pg/g, leading to a qualification limit of 5.75 pg/g
- 1,2,3,7,8-PeCDF at 0.162 pg/g, leading to a qualification limit of 0.81 pg/g
- 1,2,3,4,7,8-HxCDF at 0.222 pg/g, leading to a qualification limit of 1.11 pg/g
- 1,2,3,6,7,8-HxCDF at 0.146 pg/g, leading to a qualification limit of 0.73 pg/g
- 2,3,4,6,7,8-HxCDF at 0.112 pg/g, leading to a qualification limit of 0.56 pg/g
- 1,2,3,4,6,7,8-HpCDF at 0.438 pg/g, leading to a qualification limit of 2.19 pg/g
- 1,2,3,4,6,7,8,9-OCDF at 0.482 pg/g, leading to a qualification limit of 2.41 pg/g

Detections in associated samples less than or equal to the qualification limits should be qualified U-MBL.

Qualification: **The following results were qualified U-MBL; the detect_flag for the affected results is changed from Y to N.**

- **The 1,2,3,7,8-PeCDF result in sample FD15-10262022**
- **The 1,2,3,4,7,8-HxCDF result in sample FD19-10262022**
- **The 1,2,3,6,7,8-HxCDF results in samples SIB-122-TOB-0-1-10262022, SIB-122-MLW-0-1-10262022, and FD19-10262022**

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) – LCS/LCSD %Rs and RPDs were within QAPP control limits.

Qualification: None required.

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-122-TOB-0-1-10262022. The MSD had high %R and RPD for 1,2,3,4,6,7,8,9-OCDD . All other %Rs and RPDs were within control limits. The 1,2,3,4,6,7,8,9-OCDD result in sample SIB-122-TOB-0-1-10262022 should be qualified J-MSH,MSP.

An MS/MSD was performed using sample SIB-122-MLW-0-1-10262022. 1,2,3,4,6,7,8-HpCDD recovered below control limits in the MS and above control limits in the MSD. 1,2,3,4,6,7,8,9-OCDD had an extremely low recovery in the MS and recovered above control limits in the MSD, however the sample concentration was >4x the spike concentration and the %R results are not applicable. The RPDs for 1,2,3,4,6,7,8-HpCDD and 1,2,3,4,6,7,8,9-OCDD were above control limits. All other %Rs and RPDs are within control limits. The 1,2,3,4,6,7,8-HpCDD result in samples SIB-122-MLW-0-1-10262022 and FD17-10262022 (field duplicate of the parent sample) should be qualified J-MSL,MSH,MSP. The 1,2,3,4,6,7,8,9-OCDD result in samples SIB-122-MLW-0-1-10262022 and FD17-10262022 should be qualified J-MSP.

An MS/MSD was performed using sample SIB-121-TOB-0-1-10262022. 1,2,3,4,6,7,8,9-OCDD recovered below control limits in the MSD, however the sample concentration was >4x the spike concentration and the %R results are not applicable. All other %Rs and RPDs were within control limits.

An MS/MSD was performed using sample SIB-121-OHW-0-1-10262022. 1,2,3,4,6,7,8-HpCDD and 1,2,3,4,6,7,8,9-OCDD recovered outside control limits in the MS and/or MSD, however the sample concentration was >4x the spike concentration and the %R results are not applicable. All other %Rs and RPDs were within control limits.

An MS/MSD was performed using sample SIB-125-TOB-0-1-10262022, with results reported in SDG 20693. The MS/MSD results caused the 1,2,3,4,6,7,8-HpCDD and OCDF results in the parent sample to be qualified J-MSH. Sample SIB-125-TOB-0-1-10262022 is also the parent sample of field duplicate sample FD16-10262022, which was analyzed in this SDG. The 1,2,3,4,6,7,8-HpCDD and OCDF results for sample FD16-10262022 should be qualified J-MSH.

> ***Qualification:*** **For sample SIB-122-TOB-0-1-10262022, the 1,2,3,4,6,7,8,9-OCDD result is qualified J-MSH,MSP. For samples SIB-122-MLW-0-1-10262022 and FD17-10262022, the 1,2,3,4,6,7,8-HpCDD result is qualified J-MSL,MSH,MSP and the 1,2,3,4,6,7,8,9-OCDD result is qualified J-MSP. The 1,2,3,4,6,7,8-HpCDD and OCDF results for sample FD16-10262022 are qualified J-MSH.**

Field Duplicate – Field duplicate FD15-10262022 was analyzed with this SDG, while its parent sample, SIB-125-MLW-0-1-10262022, was analyzed with SDG 20693. 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.

Field duplicate FD16-10262022 was analyzed with this SDG, while its parent sample, SIB-125-TOB-0-1-10262022 was analyzed with SDG 20693. 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.

Sample FD17-10262022 is a field duplicate of sample SIB-122-MLW-0-1-10262022. All precision criteria were met.

Field duplicate FD18-10262022 was analyzed with this SDG, while its parent sample, SIB-123-TOB-0-1-10262022, was analyzed with SDG 20693. All precision criteria were met.

Sample FD19-10262022 is a field duplicate of sample SIB-121-MLW-0-1-10262022. All precision criteria were met.

Sample FD20-10262022 is a field duplicate of sample SIB-121-TOB-0-1-10262022. All precision criteria were met.

> ***Qualification*****: For sample FD15-10262022, the 1,2,3,4,6,7,8-HpCDD result is qualified J, reason code FDPR. For sample FD16-10262022, the 1,2,3,4,6,7,8-HpCDF and 1,2,3,4,6,7,8,9-OCDF results are qualified J, reason code 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 4 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.**
- **Four 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.**
- **One total HpCDD result and one total HpCDF result reported with a laboratory qualifier of E had the reportable_result field populated with "No"; although these results are not validated, the reportable_result field is changed to "Yes", as these results are usable.**

Qualification Summary Table (results in pg/g):

Sample	Analyte	Lab Value	Lab Qualifier	Validated Value	Validated Qualifier	Reason Code
SIB-122-TOB-0-1-10262022	1,2,3,4,7,8,9-HEPTACHLORODIBENZOFURAN	0.5	JK	0.5	J	EMPC
SIB-122-TOB-0-1-10262022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	1.93	BJK	1.93	J	EMPC
SIB-122-TOB-0-1-10262022	1,2,3,4,7,8-HEXACHLORODIBENZO-P-DIOXIN	0.838	JK	0.838	J	EMPC
SIB-122-TOB-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	0.486	BJ	0.486	U	MBL
SIB-122-TOB-0-1-10262022	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN	1.29	JK	1.29	J	EMPC
SIB-122-TOB-0-1-10262022	OCTACHLORODIBENZO-P-DIOXIN	375	--	375	J	MSH,MSP
SIB-122-OHW-0-1-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	3.14	--	3.14	DNR	EXC
SIB-122-OHW-0-1-10262022	OCTACHLORODIBENZO-P-DIOXIN	4920	E	4920	J	ACR
SIB-122-MLW-0-1-10262022	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN	134	--	134	J	MSL,MSH,MSP
SIB-122-MLW-0-1-10262022	1,2,3,4,7,8-HEXACHLORODIBENZO-P-DIOXIN	1.67	JK	1.67	J	EMPC
SIB-122-MLW-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	0.655	BJ	0.655	U	MBL
SIB-122-MLW-0-1-10262022	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN	0.875	JK	0.875	J	EMPC
SIB-122-MLW-0-1-10262022	OCTACHLORODIBENZO-P-DIOXIN	840	--	840	J	MSP
SIB-121-TOB-0-1-10262022	1,2,3,4,7,8,9-HEPTACHLORODIBENZOFURAN	0.905	JK	0.905	J	EMPC
SIB-121-TOB-0-1-10262022	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN	2.03	JK	2.03	J	EMPC
SIB-121-TOB-0-1-10262022	2,3,4,7,8-PENTACHLORODIBENZOFURAN	1.09	JK	1.09	J	EMPC
SIB-121-TOB-0-1-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	4.06	--	4.06	DNR	EXC
SIB-121-TOB-0-1-10262022	2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN	0.304	JK	0.304	J	EMPC
SIB-121-OHW-0-1-10262022	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN	2.87	K	2.87	J	EMPC
SIB-121-OHW-0-1-10262022	2,3,4,6,7,8-HEXACHLORODIBENZOFURAN	3.78	JK	3.78	J	EMPC
SIB-121-OHW-0-1-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	4.68	--	4.68	DNR	EXC
SIB-121-OHW-0-1-10262022	2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN	0.668	K	0.668	J	EMPC
SIB-121-OHW-0-1-10262022	OCTACHLORODIBENZO-P-DIOXIN	4440	E	4440	J	ACR
SIB-121-MLW-0-1-10262022	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN	1.2	JK	1.2	J	EMPC
FD15-10262022	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN	57.1	--	57.1	J	FDPR
FD15-10262022	1,2,3,7,8,9-HEXACHLORODIBENZO-P-DIOXIN	1.22	JK	1.22	J	EMPC
FD15-10262022	1,2,3,7,8-PENTACHLORODIBENZOFURAN	0.45	BJK	0.45	UJ	MBL,EMPC
FD15-10262022	2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN	0.178	JK	0.178	J	EMPC

Sample	Analyte	Lab Value	Lab Qualifier	Validated Value	Validated Qualifier	Reason Code
FD16-10262022	1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN	31.1	--	31.1	J	FDPR
FD16-10262022	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN	168	--	168	J	MSH
FD16-10262022	1,2,3,4,7,8,9-HEPTACHLORODIBENZOFURAN	2.01	JK	2.01	J	EMPC
FD16-10262022	1,2,3,7,8-PENTACHLORODIBENZOFURAN	6.05	K	6.05	J	EMPC
FD16-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	3.41	--	3.41	DNR	EXC
FD16-10262022	OCTACHLORODIBENZOFURAN	91.5	--	91.5	J	FDPR, MSH
FD17-10262022	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN	184	--	184	J	MSL,MSH,MSP
FD17-10262022	1,2,3,4,7,8-HEXACHLORODIBENZO-P-DIOXIN	1.64	JK	1.64	J	EMPC
FD17-10262022	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN	1.2	JK	1.2	J	EMPC
FD17-10262022	2,3,4,7,8-PENTACHLORODIBENZOFURAN	1.02	JK	1.02	J	EMPC
FD17-10262022	OCTACHLORODIBENZO-P-DIOXIN	1130	--	1130	J	MSP
FD18-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	2.18	K	2.18	DNR	EXC
FD18-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	1.97	K	1.97	J	EMPC
FD19-10262022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	0.559	BJ	0.559	U	MBL
FD19-10262022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	0.429	BJK	0.429	UJ	MBL,EMPC
FD20-10262022	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN	4.97	JK	4.97	J	EMPC
FD20-10262022	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN	1.27	JK	1.27	J	EMPC
FD20-10262022	2,3,4,6,7,8-HEXACHLORODIBENZOFURAN	1.13	JK	1.13	J	EMPC
FD20-10262022	2,3,4,7,8-PENTACHLORODIBENZOFURAN	0.992	JK	0.992	J	EMPC
FD20-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	4.2	--	4.2	DNR	EXC
FD20-10262022	2,3,7,8-TETRACHLORODIBENZOFURAN	3.29	K	3.29	J	EMPC
SIB-022-MLW-0-1-10252022	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN	22.2	JK	22.2	J	EMPC
SIB-022-MLW-0-1-10252022	OCTACHLORODIBENZOFURAN	42500	E	42500	J	ACR
SIB-022-MLW-0-1-10252022	OCTACHLORODIBENZO-P-DIOXIN	102000	E	102000	J	ACR